Publisher version: https://doi.org/10.1016/j.fuproc.2012.05.031; Licence: CC BY-NC-ND

Accepted version:

OPTIMISATION OF PELLETISATION CONDITIONS FOR POPLAR ENERGY CROP

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Abstract

Solid biomass usually undergoes a stage of pretreatment with the aim of being prepared for its energy utilisation. Within pretreatment stage, pelletisation process can be considered. Nowadays the main raw materials used for pellets production are wood wastes from sawmills and wood processing industries. However, searching for other raw materials is necessary in order to guarantee the supply. In this work, poplar energy crop has been studied as raw material. The results showed that poplar is a tough material with regard to its pelletisation and maize starch and/or lignosulphonate (a by-product in cellulose production) addition increases the process stability and decreases the specific energy demanded. It has been observed that with maize starch addition, the specific energy demanded during the pelletisation process is lower than with lignosulphonate addition, but, on the other hand, when lignosulphonate is used, physical quality of pellets is higher than with maize starch utilisation. Finally, the optimum conditions found to pelletise poplar are: milling particle size below 4 mm, specific pelletisation surface 5.6 cm²/kW, die compression 26 mm and 4 wt.% (d.b.) of dry additive (3% maize starch plus 1% lignosulphonate).

Keywords

Additive, lignosulphonate, maize starch, pelletisation, poplar

1. Introduction

Production and utilisation of biofuels entail social and environmental benefits compared to fossil fuels. Solid biomass is obtained in origin with some characteristics which decrease its quality for its direct energy utilisation. Consequently, a previous stage of pretreatment must be carried out and in that sense, pelletisation process can be considered. During pelletisation, temperature increases as a consequence of a pressure raise and friction forces. An effect of this temperature increase is the auto-agglomeration of the biomass raw material due to the fluidification of the lignin which is an essential component of most of biomass materials.

Knowledge of the fundamental agglomeration properties of biomass particles is essential to optimise the densification process. It is also important to understand the compaction mechanism in order to design energy-efficient compaction equipment and to quantify the effects of some process variables on the pellets physical quality [1]. Moreover, additives can be added in order to improve the compression process and the physical quality of the obtained pellets. In this sense, maize starch and lignosulphonate have demonstrated to be successful when they are used in fodder and pellets industries [2-6].

Lignosulphonate is obtained as a by-product in cellulose production from wood by sulphite acid process. Binding effect of lignosulphonate is due to lignin-lignin and lignin-water interactions because it is a compound with hydrophobic and hydrophilic groups [7, 8]. Different studies have shown that, in general, addition of lignosulphonate during pelletisation process improves pellets physical quality and decreases energy demand [2, 3, 9].

On the other hand, it has been proved that maize starch can also act as a binder. It has been demonstrated that binding action is improved when starch is gelatinized and mechanical shearing during the densification process improves starch gelatinization [3]. In the presence of heat and water, starch granules are able to swell by adsorbing considerable amounts of water, and then, they lose their semi-crystalline structure, amylose is lixiviated and, finally, viscosity of the suspension increases [10-14]. Other works show that starch addition decreases the abrasion that raw material produces on the pelletisation equipment, because starch has a lubricating effect [15, 16].

Most of the raw materials processed by pellet production plants in Europe are sawdust and shavings coming from sawmills and wood processing industries. Nevertheless, taking into account the increasing demand of pellets for energy and non-energy purposes, there is a growing interest in the utilisation of alternative raw materials, such as bark, logging residues, agricultural residues and energy crops [17-19]. An example is the poplar, which shows advantages as short rotation energy crop, such as the wide knowledge of its species, its high yields (15-25 dry t/ha·year in Spain with 3-5 years crops) [20, 21] and its low need of fertilizers. However, it must be considered its high demand of water, fact which determines the places where this crop can be grown [22].

The objective of this work is to evaluate the pelletisation process of poplar as well as the characteristics of the pellets obtained, in order to define the optimum conditions to pelletise this biomass raw material.

2. Materials and methods

2.1 Raw materials

The poplar (*Populus* sp.) utilised in this work was obtained in nurseries from the Confederación Hidrográfica del Duero and it was received as chips.

The starch used was native maize starch.

The Ca-Mg-lignosulphonate was kindly supplied by Borregaard Ligno-Tech Ibérica.

2.2 Comminution equipment

Comminution of poplar chips was carried out using two machines:

A primary crusher which was a double bearing horizontal axis knife mill with a drive of 35 kW power and 640 r.p.m. rotation speed. The exchangeable insert screen had an opening in the upper part to feed the material into the chamber perpendicularly to the rotor axis. The rotor carried nine knives and two counter-knives that reached a peripheral velocity of 20 m s⁻¹. The inner diameter of the grinding chamber was 600 mm.

A secondary grinder which was a single bearing horizontal axis hammer mill with a drive of 11 kW power and 3000 r.p.m. rotation speed. The grinding chamber was surrounded by a closed exchangeable perforated screen with an inner diameter of 485 mm. The feeding of the biomass was from

the front side of the chamber, parallel to the rotor axis. The number of hammers was 24 and the peripheral velocity was 71 m s⁻¹.

2.3 Pellet plant

The facility used in the experiments was a pilot plant (Figure 1) which included a blending system, a pellet mill and cooling and bagging equipment. The pellet press was a flat die type Amandus Kahl 33-500: die diameter 500 mm, hole diameter 6 mm and drive power 30 kW.

(Figure 1. Layout of the pilot plant used in the experiments located at CEDER-CIEMAT)

2.4 Experimental design

Two degrees of comminution were tested: a first size defined by a screen opening of 10 mm in the knife mill and a second size defined by a further grinding operation with 4 mm screen opening in the hammer mill. Batches of approximately 500 kg of poplar chips were used in each experiment.

After comminution, the milled batches were pelletised. During the tests, which lasted 1 hour, the raw material was put in the pellet press mixed with the suitable quantity of water in order to get a right operation of the machine (i.e. stable power demand and low vibration) and low moisture content pellets (moisture content < 10%). With the aim of setting the quantity of water to add, it was taken into account that the facility utilised in this work to pelletise was a pilot plant (drive power 30 kW), and that, consequently, moisture content at the inlet of the pellet press can be modified as process goes on due to mainly the feeding flow and the warming of the pellet press. Thus, in order to define this moisture content, previous tests were carried out and a range of moisture content was established to get a right pelletisation process and pellets with appropriate moisture content ($\leq 10\%$).

Five factors were taken into account during the pelletisation tests:

1. Particle size distribution of the raw material, studying the pelletisation of two different degrees of comminution: 10 mm (knife mill) and 4 mm (hammer mill).

2. Specific pelletisation surface. Three different values were used: 9.6, 8.0 y 5.6 cm²/kW. Specific pelletisation surface is calculated as follows:

$$\mathbf{S}_{\mathrm{p}} = (\mathbf{N} \mathbf{x} \mathbf{S}_{\mathrm{o}}) / \mathbf{P}$$

where:

S_p: specific pelletisation surface.

N: number of die holes which are covered by the rollers.

S₀: surface of one hole. It is calculated with: S₀ = $(\pi \times D^2) / 4$, where D is the diameter of the straight part of the hole.

P: drive power of the pellet press.

3. Die compression (pressway). It is defined as the way between the beginning of the inlet cone and the end of the straight part of the die hole (1 + L in Figure 2). Initially, 20 and 24 mm die compression were tested with 9.6 cm²/kW of specific pelletisation suface. However, this configuration resulted in a severe warming and vibration of the pellet press during the tests. Consequently, a lower die compression (17 mm) was tested. On the other hand, specific pelletisation surfaces of 8.0 and 5.6 cm²/kW required a higher pressway (26 mm).

(Figure 2. Schematic representation of a die hole)

4. Additive addition. Poplar presented difficulties during its pelletisation and it was necessary to add maize starch and lignosulphonate. In order to optimise the kind and the dosage of additive, three different dosages of maize starch and lignosulphonate were added (2.5, 5.0 and 7.0 wt.% (d.b.) of dry additive) during the pelletisation tests. Maize starch (powder) was put in the mixer previous to the pellet press with a mixing time of 3 minutes and lignosulphonate (liquid with 45% of moisture content) was added in the pellet press with a pump. As a result of these tests, it was noticed that blends of maize starch and lignosulphonate in order to pelletise poplar, and four blends where tested, restricting the quantity of lignosulphonate in order to keep sulphur content in the final fuel below 0.10 wt.%., stated value for heating oil by a Spanish act [23].

5. Physical characterization of pellets, which was compared to the values stated in EN 14961-1:2010 [24], which are depicted in Table 1.

(Table 1. Adaptation from table for pellets characterization from EN 14961-1:2010 [24])

Process variables recorded in milling and pelletisation tests were the specific mass flow (in kg of dry matter/h kW of drive power) and the specific energy (in kWh/t of dry matter). The specific mass flow is calculated as the mass of milled or pelletised material (in kg of dry matter) divided by the time utilised to mill or to pelletise it (in hours) and by the power of the mills (knife mill: 35 kW; hammer mill: 11 kW) or the pellet press (30 kW). The specific energy is calculated as the electric energy demanded by the different equipments to mill or to pelletise the material, divided by the mass of material which has been milled or the mass of pellets which has been obtained (in tonnes of dry matter).

2.5 Analytical procedures

The sampling procedure of the milled biomass and the pellets was based on the technical specification UNE-CEN/TS 14778-1 EX [25]. Therefore, and taking into account that the sampling was made from moving material (at the outlet of the mill and at the outlet of the bagging bin of the pellet pilot plant), the number of increments was calculated as:

 $n=3+0.026\ x\ M_{lot}$ for milled material

 $n=5+0.040 \ x \ M_{lot} \ for \ pellets$

where

n is the minimum permitted number of increments, round off to the nearest whole number;

M_{lot} is the mass of the lot or sub-lot in tonnes.

Thus, the number of increments for milled material was three and the number of increments for pellets was five. All the increments (milled material and pellets separately) were placed into one container to form a combined sample which was analysed in the laboratory.

The utilised analytical procedures were based on extensive investigations, norms and scientific literature. The methods utilised for milled material and pellets characterisation at CEDER-CIEMAT are resumed as follows:

Moisture content: the determination was based on heating the sample at 105 °C until a constant weight was achieved. It was derived from the norms ASTM D 2016-65 "Moisture content of wood" and ASTM 871-872 "Moisture analysis of particle fuels".

Particle size distribution: this test consists of the separation of the sample into defined size fractions that are expressed in weight percent. This procedure was adapted from the norm ASTM E 828-81 "Designating the size of refuse derived fuel-3 from its sieve analysis".

Bulk density: this procedure was adapted from the norm ASTM E 873-82. "Bulk density of densified particulate biomass fuels".

Particle density: it was calculated by means of the ratio between pellet weight and pellet volume, which was determined using the geometrical measures with the help of a caliber.

Mechanical durability: this analysis evaluates the resistance of the densified biomass to repeated beating (transport and handling). The determination was made according to the Austrian norm ÖNORM M7134. A machine called "lignotester", which functions by blowing air at 70 mbar into a pellet chamber, was utilised for determining the durability in pellets. The durability is defined as the final weight of pellets that remains in the pellet chamber divided by the initial weight and multiplied by 100, to express it in percentage.

Sulphur analysis was carried out by ion chromatography after combustion of the sample in a calorimeter and lixiviation of the ashes with ultrapure water. This procedure derives from the technical specification prCEN/TS 15289 "Solid biofuels – Determination of total content for sulphur and chlorine".

3. Results and discussion

3.1 Comminution tests

Poplar milling behaviour is characterised by two process variables (Section 2.4), specific mass flow and specific energy, whose values are shown in Table 2. As can be seen, the accumulated energy requirement to obtain the raw material milled with the finest degree of comminution (4 mm) is 124 kWh/t (97 kWh/dry t + 27 kWh/dry t).

(Table 2. Specific mass flow and specific energy in milling for poplar)

As was explained in section 2.4, the values of the specific mass flow for the screen pore size of 10 mm are related to the knife mill throughput and the values for 4 mm are referred to the hammer mill throughput.

The particles obtained after comminution have mainly fusiform shape, as can be seen in Figure 3, where three photographs of poplar chips and milled materials are shown. Particle size distribution of the milled poplar as well as pine sawdust, which has been considered as reference material, are represented in Figure 4. As can be seen, 10 mm milled poplar shows larger sizes than pine sawdust and 4 mm milled poplar shows smaller sizes than pine sawdust. However, it must be considered that the method used for determining the particle size distributions is by horizontal sieving and with this method, the fusiform particles can pass trough the sieve by their narrowest dimension but not by the longest one.

(Figure 3.Original raw material and milled products)

(Figure 4. Particle size distribution for milled poplar)

A comparison of the bulk density of the milled poplar and pine sawdust appears in Table 3. As can be noted, the finer the milling, the higher the bulk density is. On the other hand, 4 mm milled poplar and pine sawdust have a similar bulk density.

(Table 3. Bulk density of raw material)

3.2 Pelletisation tests

Initially, during pelletisation tests, biomass feeding flow to pellet press was regulated to be as high as possible in order to get the maximum production, whenever pellet press power did not exceed the upper limit value and obtained pellets had an acceptable physical quality. Furthermore, poplar was put in the pellet press mixed with the suitable quantity of water in order to soften the material and to get a stable work of the pellet press, but without diminishing the pellets quality.

As can be seen in Figure 5, firstly, two milling sizes (10 and 4 mm) and two die compressions (20 and 24 mm), which had been successful for pine sawdust pelletisation in a previous work [26], were

utilised with the specific pelletisation surface of 9.6 cm²/kW. It was observed that 10 mm milled poplar could hardly go through the tested dies and, therefore, this milling size was rejected for the rest of the tests. Moreover, the two die compressions assayed (20 and 24 mm) proved to be too high for the specific pelletisation surface of 9.6 cm²/kW, because during the tests, important vibration occurred in the pellet press and a high increase in its temperature was observed. Consequently, die compression was decreased to 17 mm.

Once completed the tests with 9.6 cm²/kW specific pelletisation surface, 4 mm milled poplar was pelletised with 17 mm die compression and specific pelletisation surfaces of 8.0 and 5.6 cm²/kW. For these specific surfaces, it was noticed that the die compression utilised was low, because the raw material went through the die holes without being properly pelletised. As a result, it was decided to increase the die compression up to 26 mm.

The values of moisture content at the inlet of the pellet press, specific mass flow and specific energy for the described tests appear in Table 4. As can be observed, a high quantity of water is necessary to be added (moisture content higher than 25% at the inlet), the energy demanded is very high and the specific mass flow is very low, especially for specific pelletisation surfaces of 9.6 and 8.0 cm²/kW. Furthermore, it must be considered that, during the tests, the pellet press worked in an unstable way with power peaks, vibration and powder production (raw material which goes through the die without being pelletised).

(Figure 5. Poplar pelletisation scheme)

(Table 4. Specific mass flow and specific energy in poplar pelletisation process)

Although the first test could not be carried out with the same die compression, it can be noticed that as specific pelletisation surface is decreased, specific mass flow increases and specific energy decreases.

Physical characteristics of pellets are shown in Table 5. It can be observed that poplar pellets have low moisture content (M10 classification according to EN 14961-1:2010 [24]) and high densities

and mechanical durability, especially for 5.6 cm²/kW specific pelletisation surface (BD700 and DU97.5 classification according to EN 14961-1:2010 [24]).

(Table 5. Physical characteristics of poplar pellets)

The results obtained in pelletisation tests with poplar biomass showed that the conditions which led to the highest pellets production, the lowest energy demand and the best pellets physical properties were: 4 mm milling size, 26 mm die compression and 5.6 cm²/kW specific pelletisation surface. However, it must be considered that the energy demanded in such conditions is still high and, moreover, instability in the pellet press was observed during the test, with vibrations and blockages. Accordingly, the use of additives was taken into account and two additives were assayed: maize starch and lignosulphonate, following the scheme shown in Figure 6.

(Figure 6. Poplar + additives pelletisation scheme)

Firstly, maize starch and lignosulphonate were added separately in three dosages (2.5, 5.0 and 7.0 wt.% (d.b.) of dry additive). In order to see the effect on the yield of the process, the pellet press was fed with constant flow to obtain approximately the same pellets production (8 dry kg/h kW) and to avoid the influence of this variable on the energy demanded. Moisture content at the inlet of the pellet press was checked in order to avoid pellets with moisture content higher than 10%, especially when lignosulphonate was added, because this additive is liquid (moisture content: 45%). In Table 6, these values of moisture content are shown and it can be seen that pelletisation with maize starch requires moisture content between 12.5 and 13.0% whereas pelletisation with lignosulphonate needs values between 9.0 and 10.5%.

Table 6 shows the specific energy demanded. It can be observed that, comparing Tables 4 and 6, with the addition of additives, the specific energy decreases; however, an increase in the additive dosage (specially from 5.0 to 7.0 wt.%) does not have an important influence on the specific energy. Furthermore, with regard to pellet press work, it must be emphasized that the addition of maize starch or lignosulphonate results in a general improvement of the press stability when comparing with no-additive addition.

Comparing the specific energy demand, it can be seen that the addition of maize starch results in a lower energy demand than the addition of lignosulphonate. This fact could be due to the lubricating effect of the starch [15, 16].

(Table 6. Poplar pelletisation tests with addition of maize starch and lignosulphonate separately)

Physical characteristics of pellets obtained are shown in Table 7. As can be seen, in general, the quality of pellets obtained with lignosulphonate is better than the quality of pellets obtained with maize starch, for the reason that pellets obtained with lignosulphonate show higher values of bulk and particle densities (according to EN 14961-1:2010 [24], BD600 and BD650 classification for pellets with lignosulphonate versus BD550 for pellets with starch) and mechanical durability (regarding EN 14961-1:2010 [24], DU97.5 classification for pellets with lignosulphonate versus DU95.0 and DU96.5 for pellets with starch). Moreover, with maize starch addition, cracks over the pellet surface appear although 7 wt.% maize starch dosage is utilised. Nevertheless, with lignosulphonate addition, the pellets have a uniform and shiny surface, without cracks.

(Table 7. Physical characteristics of poplar pellets obtained with addition of maize starch and lignosulphonate separately)

Taking into account the obtained results, the option of introducing maize starch together with lignosulphonate was considered interesting, since it had been observed that with maize starch addition, specific energy demand was especially reduced, and with lignosulphonate addition, the physical properties of the pellets were particularly improved. Furthermore, Richardson et al. [27] had observed that starch gelatinization is modified when lignosulphonate is added. In order to define the dosage of additives and with the aim of restricting sulphur emissions in combustion, it was considered the sulphur limit which has been stated for heating oil by a Spanish act [23] (0.10 wt.% (d.b.)). However, it must be thought that this limit value could be higher for biomass, because an important part of the sulphur contained in the biomass is as sulphates which, together with the sulphates formed during the combustion process, could remain retained in the ashes [28-30].

Sulphur content of the pure poplar biomass was analysed, and a value of 0.034% (wt.) (dry basis) was obtained. With the object of confirming if this value would be reasonable, the work of Ciria et al. [31], where samples of nine poplar clones with or without fertilisation were analysed, was consulted. In that work, sulphur content in short rotation poplar (without leaves) varied between 0.030 wt.% (d.b.) without fertilisation and 0.039 wt.% (d.b.) with fertilisation. Consequently, poplar with a sulphur content of 0.034 wt.% (d. b.) was considered as representative.

On the other hand, the used lignosulphonate was analysed and a sulphur content of 6.3 wt.% (d.b.) was determined. Then, appropriate calculations were carried out in order to obtain the additives dosages so that sulphur content in pellets was not higher than 0.10% (wt.) (dry basis). The obtained results are shown in Table 8.

(Table 8. Additives dosages in pellets for sulphur concentration of 0.10% (wt.) (d.b.))

With the purpose of selecting the optimum additives dosage when they are added simultaneously, the pellet press operated in the same conditions than in previous tests (i.e. constant feed flow: 8 dry kg/h kW). Pelletisation tests, moisture content at the inlet and specific energy demanded are shown in Table 9. Moreover, specific energy values versus additives dosages are represented in Figure 7. It can be noticed that, when maize starch and lignosulphonate are added simultaneously, specific energy has intermediate values between those obtained for maize starch addition and lignosulphonate addition.

(Table 9. Poplar pelletisation tests with addition of maize starch and lignosulphonate simultaneously)

(Figure 7. Poplar pelletisation tests with addition of maize starch and lignosulphonate separately and simultaneously)

With regard to the physical properties of the pellets obtained with the addition of maize starch and lignosulphonate simultaneously (Table 10), it can be observed that the moisture content is low for all the pellets and the mechanical durability for the pellets obtained with 2% of additive (maize starch: 0.95 wt.% and lignosulphonate: 1.05 wt.%) is low (93.2%). Nevertheless, when maize starch addition

increases, mechanical durability rises, although 97.5% value is not reached, which is the value stated by EN 14961-1:2010 [24] for the highest pellets quality. On the other hand, it can be seen that bulk densities are similar for all the additives dosages assayed, whereas, in general, particle density increases when additive dosage is increased. This difference could be explained by the fact that pellets with low additives dosages are broken more easily (as can be seen in their mechanical durability values), and this situation could cause an increase in bulk density, because spaces among pellets could be taken up by lower size pellets, which would be a result of the original pellets breakage.

(*Table 10. Physical characteristics of poplar pellets obtained with addition of maize starch and lignosulphonate simultaneously*)

Comparing these results with those obtained for the addition of additives separately (Table 7), it is observed that, in general, when additives are used simultaneously, bulk and particle densities are improved with regard to pellets obtained with maize starch. Nevertheless, mechanical durability for pellets made with blends of maize starch and lignosulphonate is similar to mechanical durability for pellets made with maize starch alone, but lower than durability for pellets made with only lignosulphonate. Therefore, in order to get a mechanical durability value higher than 97.1%, the lignosulphonate dosage would have to be increased.

Taking into account the quality classification of pellets according to EN 14961-1:2010 [24], it can be noticed that bulk density and mechanical durability are low, with values belonging to BD600, DU95.0-, DU95.0 and DU96.5 categories.

4. Conclusions

Poplar is a tough material and additives are needed to pelletise it in order to carry out a mechanically viable operation. Therefore, the following conclusions can be highlighted:

Maize starch or lignosulphonate addition (in dosages of 2.5, 5.0 and 7.0 wt.% (d.b.) of dry additive) increased the process stability and decreased the specific energy demand, but an increase of additive over 5.0 wt.% did not entail a better process.

- Considering the same additive dosage, with maize starch addition, the specific energy demanded during the pelletisation process was lower than with lignosulphonate addition. The reduction of the energy demanded was between 42 and 43% for maize starch addition and between 7 and 23% for lignosulphonate addition, depending on the dosage utilised.
- Concerning the physical quality of pellets and when the same additive dosage was used, the pellets properties in general were better with lignosulphonate addition than with maize starch addition. Regarding the classification established by EN 14961-1:2010 [24], pellets obtained with maize starch addition belonged to BD550 category according to bulk density and to DU95.0 or DU96.5 categories with regard to mechanical durability; whereas pellets obtained with lignosulphonate addition belonged to BD600 or BD650 categories according to bulk density and to DU97.5 category regarding mechanical durability. In addition, pellets obtained with lignosulphonate showed a shinier surface without cracks.

Regarding the tests carried out and the facilities used, the pelletisation conditions selected as optimal are:

- Milling size: 4 mm.
- Specific pelletisation surface: 5.6 cm2/kW.
- Die compression: 26 mm.
- The most favourable dosage of additives, taking into account a sulphur content limit of 0.10 wt.%, is 4 wt.% (d.b.)of dry additive (3 wt.% of maize starch plus 1 wt.% of lignosulphonate). With this dosage, pellet press worked with stability and the specific energy demanded was low (99 kWh/ dry t); moreover, pellets obtained had an acceptable physical quality (moisture content: 8.0%; bulk density: 610 kg/m³; particle density: 1100 kg/m³; mechanical durability: 97.1%).

Acknowledgements

We gratefully acknowledge the Borregaard Ligno-Tech Ibérica Company for providing the lignosulphonate utilised in this work.

References

 S. Mani., L. G. Tabil., S. Sokhansanj, Effects of compressive force, particle size and moisture content on mechanical properties of biomass pellets from grasses, Biomass and Bioenergy 30 (2006) 648-654.
 S. Van Loo, J. Koppejan, Biomass fuel supply and pre-treatment, in: The Handbook of Biomass Combustion and Co-firing, Earthscan, London, 2008, pp. 54-111.

[3] N. Kaliyan, R. Vance Morey, Factors affecting strength and durability of densified biomass products.Biomass and Bioenergy 33 (2009) 337-359.

[4] E. Smidt, K. Meissl, M. Schmutzer, B. Hinterstoisser, Co-composting of lignin to build up humic substances – Strategies in waste management to improve compost quality, Industrial Crops and Products 27 (2008) 196-201.

[5] M. Olsson, J. Kjällstrand, G. Petersson, Specific chimney emissions and biofuel characteristics of softwood pellets for residential heating in Sweden, Biomass and Bioenergy 24 (2003) 51-57.

[6] F. García, F. Martín, J.J. Rodríguez, Posibilidades de aprovechamiento de la lignina en la industria química, Ingeniería Química 187 (1984) 249-254.

[7] D. Stewart, Lignin as a base material for materials applications: Chemistry, application and economics, Industrial Crops and Products 27 (2008) 202-207.

[8] Q. Shen, T. Zhang, M. Zhu, A comparison of the surface properties of lignin and sulfonated lignins by FTIR spectroscopy and wicking technique, Colloids and surfaces A: Physicochem. Eng. Aspects 320 (2008) 57-60.

[9] M. Thomas, T. van Vliet, A.F.B. van der Poel, Physical quality of pelleted animal feed. 3.Contribution of feedstuff components, Animal Feed Science Technology 70 (1998) 59-78.

[10] P.A.M. Steeneken, Rheological properties of aqueous suspensions of swollen starch granules, Carbohydrate Polymers 11 (1989) 23-42.

[11] J.L. Doublier, G. Llamas, M. Le Meur, A rheological investigation of cereal starch pastes and gels.Effect of pasting procedures, Carbohydrate Polymers 7 (1987) 251-275.

[12] L. Copeland, J. Blazek, H. Salman, M.C. Tang, Form and functionality of starch, Food Hydrocolloids 23 (2009) 1527-1534.

[13] A.J.F. Carvalho, Starch: major sources, properties and applications as thermoplastic materials.Monomers, Polymers and Composites from Renewable Sources (2008) 321-342.

[14] P. C. Knight, Structuring agglomerated products for improved performance, Powder Technology 119 (2001) 14-25.

[15] G.A. Holt, T.L. Blodgett, F.S. Nakayama, Physical and combustion characteristics of pellet fuel from cotton gin by-products produced by select processing treatments, Industrial Crops and Products 24 (2006) 204-213.

[16] J.F. Wood, The functional properties of feed raw materials and their effect on the production and quality of feed pellets, Animal Feed Science and Technology 18 (1987) 1-17.

[17] U. Malisius, H. Jauschnegg, H. Schmidl, B. Nilsson, S. Rapp, A. Strehler, H. Hartmann, R. Huber, J.
Whitfield, D. Kessler, A. Geiβlofer, B. Hahn, Wood pellets in Europe. Thermie B DIS/2043/98-AT,
Industrial Network on Wood Pellets (2000).

[18] M. Olsson, J. Kjällstrand, Emissions from burning of softwood pellets, Biomass and Bioenergy 27(2004) 607-611.

[19] C. Rhén, M. Öhman, R. Gref, I. Wästerlund, Effect of raw material composition in woody biomass pellets on combustion characteristics, Biomass and Bioenergy 31 (2007) 66-72.

[20] M.P. Ciria, Efecto del turno de corta y de la densidad de plantación sobre la productividad de diversos clones de chopo en condiciones de corta rotación, Tesis Doctoral, Universidad Politécnica de Madrid, 2000.

[21] M.P. Ciria, E. González, J.E. Carrasco, The effect of fertilization and planting density on biomass productivity of poplar harvested after three-years rotation, Proceedings of the 12th European Conference on Biomass for Energy, Industry and Climate Protection, 2002, pp. 283-286.

[22] C. M. Gasol, X. Gabarrell, A. Anton, M. Rigola, J. Carrasco, P. Ciria, J. Rieradevall, LCA of poplar bioenergy system compared with *Brassica carinata* energy crop and natural gas in regional scenario, Biomass and Bioenergy 33 (2009) 119-129.

[23] España. Real Decreto 61/2006, de 31 de enero, por el que se determinan las especificaciones de gasolinas, gasóleos, fuelóleos y gases licuados del petróleo y se regula el uso de determinados biocarburantes, *Boletín Oficial del Estado*, 17 de febrero de 2006, núm. 41, pp.6342-6357.

[24] AENOR. EN 14961-1:2010 Biocombustibles sólidos. *Especificaciones y clases de combustibles*.*Parte 1: Requisitos generales*, Madrid, AENOR, 2010.

[25] AENOR. UNE-CEN/TS 14778-1 EX. Biocombustibles sólidos. *Muestreo. Parte 1: Métodos de muestreo*, Madrid, AENOR, 2007

[26] I. Mediavilla, L.S. Esteban, M.J. Fernández, P. Pérez, J.E. Carrasco, Pelletization characteristics of different Spanish biomass feedstock, Proceedings of the 15th European Biomass Conference, 2007, pp. 71-77.

[27] G. Richardson, Y. Sun, M. Laungton, A. Hermansson, Effects of Ca- and Na-lignosulphonate on starch gelatinization and network formation, Carbohydrate Polymers 57 (2004) 369-377.

[28] M. J. Fernández Llorente, J. M. Murillo Laplaza, R. Escalada Cuadrado, J. E. Carrasco García, Ash behaviour of lignocellulosic biomass in bubbling fluidised bed combustion, Fuel 85 (2006) 1157-1165.

[29] M. S. Reddy, C. Venkataraman, Inventory of aerosol and sulphur dioxide emissions from India. PartII – Biomass combustion, Atmospheric Environment 36 (2002) 699-712.

[30] R. W. Bryers, Fireside slagging, fouling and high-temperature corrosion of heat-transfer surface due to impurities in steam-raising fuels, Progress in Energy and Combustion Science 22 (1996) 29-120.

[31] M.P. Ciria, J. Pérez, M.J. Fernández, J.E. Carrasco, Nutrients extraction in a high density poplar plantation, 16th European Biomass Conference, 2008, pp. 589-592.

FIGURES

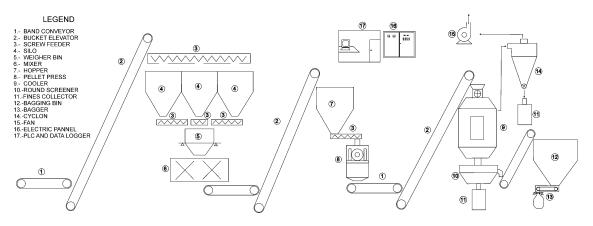


Figure 1. Layout of the pilot plant used in the experiments located at CEDER-CIEMAT

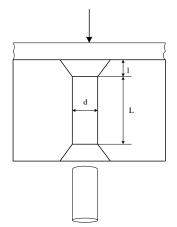


Figure 2. Schematic representation of a die hole.



Poplar chips

10 mm milled poplar

4 mm milled poplar

Figure 3.Original raw material and milled products

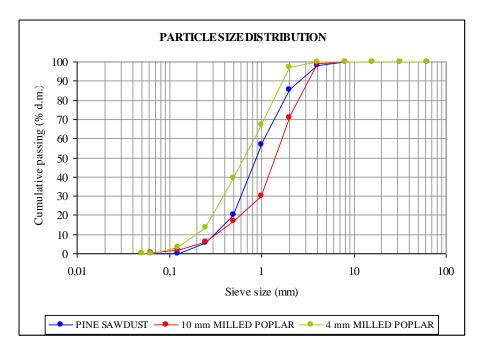


Figure 4. Particle size distribution for milled poplar

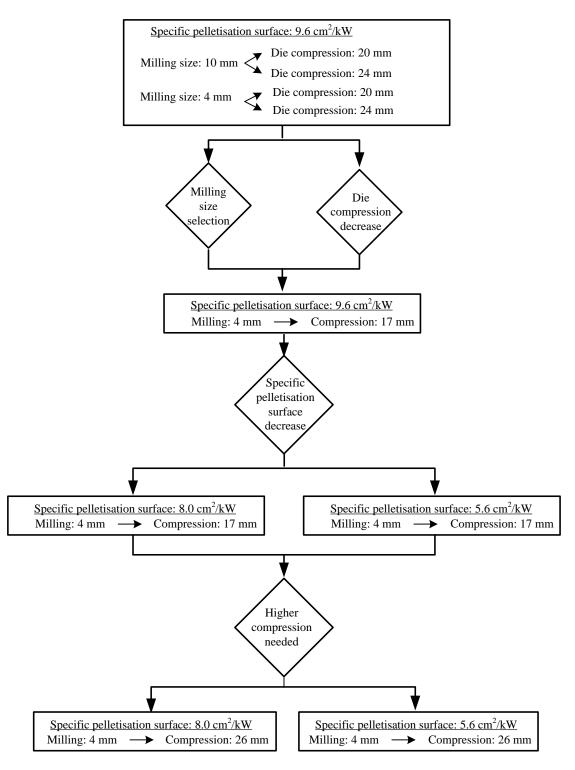


Figure 5. Poplar pelletisation scheme

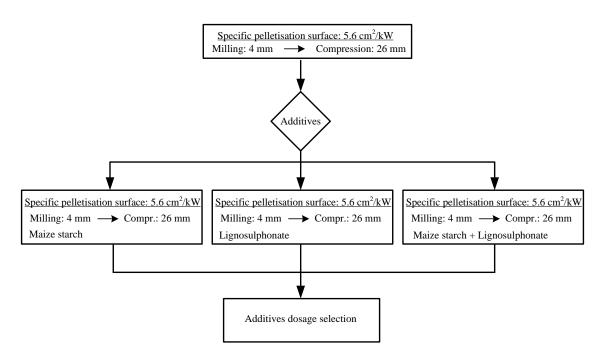
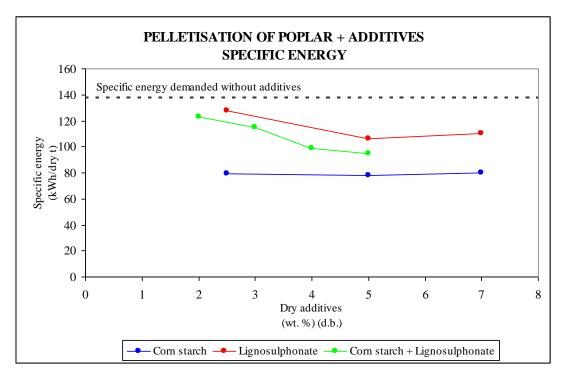


Figure 6. Poplar + additives pelletisation scheme



wt. %: weight %; d.b.: dry basis

Figure 7. Poplar pelletisation tests with addition of maize starch and lignosulphonate separately and simultaneously

TABLES

	PELLETS				
Diameter (D	0) and length (L) (mm)				
D06	6 mm \pm 1.0 mm, and 3.15 \leq L \leq 40 mm				
D08	8 mm \pm 1.0 mm, and 3.15 \leq L \leq 40 mm				
D10	10 mm \pm 1.0 mm, and 3.15 \leq L \leq 40 mm				
D12	12 mm \pm 1.0 mm, and 3.15 \leq L \leq 50 mm				
D25	25 mm \pm 1.0 mm, and 10 \leq L \leq 50 mm				
Moisture cor	ntent: M (wt. %)				
M10	≤ 10%				
M15	$\leq 15\%$				
Mechanical of	durability: DU (wt. %)				
DU97.5	≥97.5%				
DU96.5	≥ 96.5%				
DU95.0	≥95.0%				
DU95.0-	< 95.0%				
Bulk density	: BD (kg/m ³)				
BD550	\geq 550 kg/m ³				
BD600	$\geq 600 \text{ kg/m}^3$				
BD650	$\geq 650 \text{ kg/m}^3$				
BD700	$\geq 700 \text{ kg/m}^3$				
BD700+	$> 700 \text{ kg/m}^3$				
4.0/					

Table 1. Adaptation from table for pellets characterization from EN 14961-1:2010 [24]

wt %: weight %

Table 2. Specific mass flow and specific energy in milling for poplar

Inlet material	Screen pore size (mm)	Specific mass flow (dry kg/h kW)	Specific energy (kWh/dry t)
Poplar chips	10	19	97
Product crushed (10 mm)	4	23	27

Table 3. Bulk density of raw materials

Raw material	Bulk density (dry kg/m ³)
Milled poplar (10 mm)	170
Milled poplar (4 mm)	200
Pine sawdust	210

Table 4. Specific mass flow and specific energy in poplar pelletisation process

Specific pelletisation surface (cm ² /kW)	Milling size (mm)	Die compression (mm)		Specific mass flow (dry kg/h kW)	Specific energy (kWh/dry t)
9.6	4	17	28.0	1.6	408
8.0	4	26	26.2	2.6	313
5.6	4	26	26.6	4.4	138

wt. %: weight %; w.b.: wet basis

Specific pelletisation surface (cm ² /kW)	Milling size (mm)	Die compression (mm)	Moisture content (wt. %) (w.b.)	Bulk density (kg/m ³) (w.m.)	Particle density (kg/m ³) (w.m.)	Mechanical durability (wt. %) (w.m.)
9.6	4	17	9.7	670	1240	97.4
8.0	4	26	8.8	730	1260	97.1
5.6	4	26	8.3	730	1260	97.6

Table 5. Physical	characteristics	of poplar pellets
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wt %: weight %; w.b.: wet basis; w.m.: wet matter

Table 6. Poplar pelletisation tests with addition of maize starch and lignosulphonate separately

Specific pelletisa	ation surface: 5.6 cm	² /kW						
Die compression	Die compression: 26 mm							
Milling size: 4 n	nm							
Dry additives		Moisture content at the	Specific energy					
(wt. %) (d.b.)		inlet (wt.%) (w.b.)	(kWh/ dry t)					
Maize starch	Lignosulphonate							
2.5	0	13.0	79					
5.0	0	12.8	78					
7.0	0	12.5	80					
0	2.5	10.5	128					
0	5.0	9.0	106					
0	7.0	10.0	110					

wt. %: weight %; d.b.: dry basis; w.b.: wet basis

Table 7. Physical characteristics of poplar pellets obtained with addition of maize starch and

Specific pelle	tisation surface: 5.6	cm ² /kW			
Die compress	ion: 26 mm				
Milling size: 4	4 mm				
Dry additives (wt. %) (d.b.)		Physical charac	teristics		
Maize starch	Lignosulphonate	Moisture content (wt. %) (w.b.)	Bulk density (kg/m ³) (w.m.)	Particle density (kg/m ³) (w.m.)	Mechanical durability (wt. %) (w.m.)
2.5	0	9.9	570	970	95.9
5.0	0	9.8	560	960	97.3
7.0	0	9.0	590	1000	96.4
0	2.5	9.9	610	960	98.0
0	5.0	8.5	650	1080	98.8
0	7.0	9.5	620	1060	98.4

lignosulphonate separately

wt %: weight %; d.b.: dry basis ; w.b.: wet basis; w.m.: wet matter

Dry additives	Lignosulphonate	Maize starch
(wt. %) (d.b.)	(wt. %) (d.b.)	(wt. %) (d.b.)
2	1.05	0.95
3	1.06	1.94
4	1.06	2.94
5	1.07	3.93

Table 8. Additives dosages in pellets for sulphur concentration of 0.10% (wt.) (d.b.)

wt %: weight %; d.b.: dry basis

Table 9. Poplar	pelletisation tests w	vith addition of	f maize starch and	d lignosulphon	ate simultaneously

Specific pelletis	ation surface: 5.6 cm	$^{2}/kW$	
Die compression	n: 26 mm		
Milling size: 4 n	nm		
Dry additives		Moisture content at the	Specific energy
(wt. %) (d.b.)		inlet (wt.%) (w.b.)	(kWh/ dry t)
Maize starch	Lignosulphonate		
0.95	1.05	7.2	123
1.94	1.06	8.8	118
2.94	1.06	8.5	99
3.93	1.07	7.9	95

wt. %: % weight; d.b.: dry basis; w.b.: wet basis

Table 10. Physical characteristics of poplar pellets obtained with addition of maize starch and

lignosulphonate simultaneously

Specific pelletisation surface: 5.6 cm ² /kW								
Die compression: 26 mm								
Milling size: 4 mm								
Dry additives (wt. %) (d.b.)		Physical charact	teristics					
Maize starch	Lignosulphonate	Moisture content (% wt.) (w.b.)	Bulk density (kg/m ³) (w.m.)	Particle density (kg/m ³) (w.m.)	Mechanical durability (% wt.) (w.m.)			
0.95	1.05	6.3	630	1070	93.2			
1.94	1.06	8.4	600	1030	95.6			
2.94	1.06	8.0	610	1100	97.1			
3.93	1.07	7.2	610	1130	97.1			

wt. %: weight %; d.b.: dry basis; w.b.: wet basis; w.m.: wet matter