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CISTUS LADANIFER SOLAR DRYING, MILLING AND PELLETIZING – SAWDUST PARTICLE DISTRIBUTION AND ITS INFLUENCE ON PELLETS QUALITY

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Abstract. Cistus ladanifer *also known as rockrose, referred on literature by its use in the perfumery and cosmetic industry was used in this work for pellet production aiming to study the energy requirements to obtain high quality* Cistus ladanifer *pellets. This study verifies the influence of particle size distribution and burnt particles on further pellet quality.*
Initially, solar drying process was studied, in a solar kiln previously instrumented with appropriate sensors, data loggers and software to record real time solar radiation, temperature and air relative humidity data. Also, a device was used to monitor sample's water content loss. Eleven days drying cycles were carried out and variables related to the drying process calculated. As the drying process occurred a decrease on the drying efficiency and an increase on the energy needed was registered.
Milling and pelletizing machines were also previously instrumented to monitor manufacturing conditions. In general, pellets produced presented dimension values acceptable as established by EN 14961-2. Concerning durability and water resistance, the best results obtained (representing high quality pellets for commercialization) were respectively 98.7 and 28.9%, respectively, after adding Cistus ladanifer sawdust with mean particle diameters of 716 µm and a moisture content (in wet basis) of 13.7%. Pellets produced with forest fire burned raw material presented a durability of 74.6% and negligible water resistance (they practically disintegrate as they have acquired significant hygroscopic characteristics). An energy balance was made. Finally, higher incidence of finer particles with wider distributions increases pellet's quality.

Keywords: Cistus ladanifer, *solar drying, pelletization, particle size distribution, pellet quality.*

1. INTRODUCTION

The global Energy demand currently is fulfilled essentially by fossil fuels, which leads to the scarce of this raw material and to an increase on air pollution (produced by the transformation procedures to obtain energy, mainly fuel combustion). Facing a potential energy crisis, biomass appears as a carbon neutral and renewable source capable of being used for energy purposes in a sustainable manner, bringing socio-economic and environmental benefits (*Poddar et al.*, 2014).

Even being an attractive raw material, biomass seasoning, difficult in handling, high costs of transportation (that is the second higher cost after capital cost in a power plant), and low bulk density forces to find alternative process of biomass transformation (Kummar *et al.*, 2003). These disadvantages can be reduced by densifying the raw material and using abundant species on the local flora. Iberian Peninsula soils are particularly rich in *Cistus ladanifer*, a species also known as rockrose which grows in lands after being burnt by fire. Rockrose has been used for industrial applications, such as perfumery or cosmetics. In addition, labdanum (a sticky resin that characterizes the plant with a distinct odor) has been used as a sedative, hemostatic and anti-infective agent (Robles et al., 2003; Hernández-Rodríguez et al., 2013).

A wide variety of studies were performed on densification processes namely the pellet production area. For instance Tabil and Sokhansanj (1996a and 1996b) worked on understanding the compression characteristics of alfalfa pellets, or Adapa *et al.* (2002) that studied pelleting fractionated alfalfa products. Because until obtain a good quality pellet, during the production steps there are complex interactions between particles that are affected by some variables. There are variables related with the raw material and others related with the production process (Relova *et al.*, 2009). Regarding the raw material, depending on wood species there are expected some composition variation, which is one of the major variables affecting the pellet quality. Plants are composed by low molecular weight (substances including organic matter and inorganic matter) and macromolecular components (cellulose, hemi-cellulose, and lignin). Thereby this affects bonds between particles (Mohan *et al.*, 2006). Still related to the raw material, moisture content, particle mean diameter and particle distribution also pays an important role at the palletization process. In particular, the particle size distribution seems to play an important role on the pellet production and further pellet quality, even though being described on the literature that the sawdust must be with 3-8 mm particle sizes (Mani et al., 2004), there must be present finer particles to fill the gap left by the oversized ones, allowing a uniform bond between particles (Mani et al., 2003). In what concerns to the production process itself, pressure applied and temperatures achieved along the pellet production influence plastic lignin deformation that leads to final pellet quality variations (Vam Dam *et al.*, 2004).

In previous studies, an influence of average particle size and its distribution in pellets durability was detected, (Almeida et *al.*, 2014). In this work rockrose was used for pellet production aiming to study the energy requirements to obtain high quality *Cistus ladanifer* pellets. This study verifies the influence of particle size distribution and burnt particles on further pellet quality.

2. EXPERIMENTAL SETUP

As it was aimed to monitor the energy required for every step that leads to final pellet production, all equipment were previously instrumented. At the milling and pelletizing process, the energy consumption data was obtained by means of a Hioki Wattmeter Analyzer. This equipment acquires tensions, currents and power factor values of the three electrical phases and calculates the respective energy consumption (Ghorbani et al., 2008).

Throughout this paper, moisture content, is referred to the wet basis and is represented by M_{wb}.

2.1 Solar drying and water content monitoring

Samples of Cistus ladanifer were collected in a local forest and dried in a solar kiln. Eleven day drying cycles were carried out. Aiming to assess the energy needed for the drying, T type thermocouples were used to obtain temperature data and Honeywell HIH 4000 sensors to read internal air relative humidity. For the conversion from analogical to digital signal, the sensors were connected to a Pico Technology TC-08 USB and ADC-20, respectively. Pico Technology Picolog Recorder was the software used for both dataloggers to record data. Solar radiation data were obtained using Apogee SP 110 pyranometers (one of them placed outside the solar drier and the other inside), connected to a NI USB-6008 DAQ using Labview 8.6 software, both from National Instruments.

For monitoring the moisture content of the raw material, each day a sample of Cistus ladanifer was collected from the solar kiln and dried in a Venticelli lab oven at 105 ºC until losing all water (Samuelsson *et al.*, 2006).

A strain sensor was placed inside the solar dryer to monitor the raw material mass, connected to a HBM MGC dalalogger and using the Spider 8-30 software to collect data. Software was programmed to collect data every minute, and one hour averages were then calculated. The collected samples were previously weighed individually in a lab scale and their diameters measured using a caliper, then they were placed into a net basket (43 x 28 cm) suspended in the strain sensor.

2.2 Biomass ground and particle characterization

An Agico Wood crusher was used to grind the raw material. Inside the hammer mill, sieves with diameters of 4 and 6 mm were sequentially used. Also random samples were reintroduced into the machine to obtain finer particles, with wider distribution. Sawdust was characterized in terms of moisture content (Samuelsson *et al.,* 2006) and average mean particle sizes were calculated according to ISO 3310-2 1999-01. Three sawdust samples were sieved in a Retsch AS 200 device. Particle mean diameters were calculated using Eq. (1).

$$\overline{d_p} = \frac{1}{\sum\left(\frac{x}{d_p}\right)} \tag{1}$$

where $\overline{d_p}$ is the value of the average particle diameter (µm), d_p represents the sieve mean diameter at a specified interval (µm) and x the retained mass fraction (g) in each sieve.

2.3 Pellets production and caracterization

In a typical commercial pellets production process, 100 to 135 ºC is the ideal range of temperature to work with, when adding biomass with moisture contents between 8 and 15% (Lehtikangas, 2001). Other authors (Vam Dam *et al.*,

2004) refer wider ranges (80 to 200 ºC) to allow the softness and plastic deformation of lignin for further stronger particle bonding. So having in mind the importance of monitoring temperatures, J and K type thermocouples were used to measure temperatures outside and inside the pelletizer machine. A TC-08 USB board was used to connect thermocouples and recording software was used to acquire data, both from Pico Technology.

After a cooling period, pellets produced were characterized by means of a set of procedures according to ÖNORM M 7135. For that purpose, a random sample of twenty pellets were selected and, for each of them, dimensions were measured (diameter and length) using a caliper with a precision of 0.1 mm. Each pellet was weighed in a precision lab scale (0.0001 g resolution). With data obtained previously, pellets density was calculated, applying a stereometric measurement method (Rabier *et al.*, 2006).

To evaluate pellets quality, water resistance (Kaliyan *et al.*, 2009) and durability tests (Temmerman *et al.*, 2006) were performed.

3. RESULTS AND DISCUSSION

3.1 Solar drying data

A summer eleven days drying cycle was carried out inside the solar kiln previously instrumented. 24.2 kg of fresh rockrose collected on the local forest, was placed on the solar kiln. Fresh trunks presented 12.5 to 41mm diameter, and a 38% M_{wb}. Leafs, even not being too abundant, presented 41% M_{wb}. At the end of the process, trunks and leafs presented 17 and 12% M_{wb}, respectively.

One random trunk was chosen and two sensors (one air relative humidity sensor and one thermocouple) were placed in it surface. Data obtained from trunk surface sensors are shown in Fig.1, where the increase on temperature is accompanied by a decrease on air relative humidity.

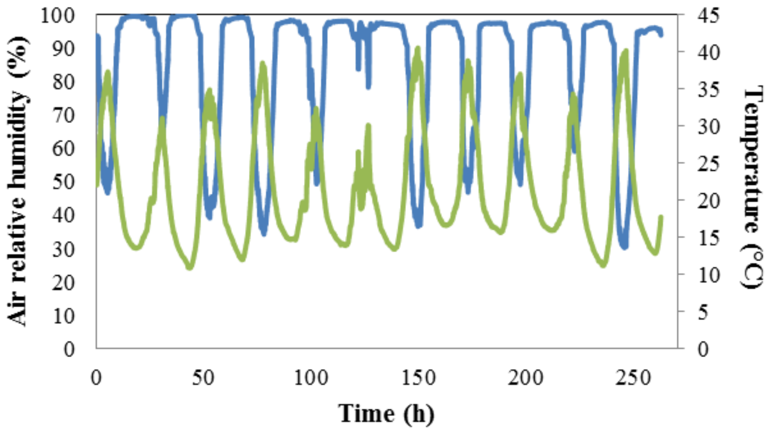

Figure 1. Air relative humidity (blue line) and temperature (red line) data vs solar drying time.

A *Cistus ladanifer* sample with 1 kg was placed in a net basket suspended in a load cell, monitoring the real time sample mass. This 1kg sample, works as a control sample, to verify the amount of water lost during the day, and recurring to Eq. (2) it allows also to calculate de thermal efficiency, η.

$$\eta = \frac{m_v h_{lg}}{I_{av} A_i t} \times 100 \qquad (2)$$

where m_v represents water mass evaporated during the period of the drying process, h_{lg} is the latent heat of vaporization, I_{av} is the average irradiation obtained during the period of the solar drying, A_i is the area exposed to solar radiation and t is the period of time during which drying process took place (Singh *et al.*, 2006).

Integrating irradiation values, assuming a drying period of 800 minutes, one obtained daily thermal efficiency values, present in Tab.1. Daily drying rate data is as well represented in Tab.1.

Table 1. Daily thermal efficiency and drying rate calculated.

Drying days	1	2	3	4	5	6	7	8	9	10	11
η (%)	9.8	11	9.6	9.2	6.4	3.5	4.8	3.8	2.8	3.8	1.3
DR(kg/h)E-4	60	21	29	30	15	6.7	16	11	9.2	8.3	4.6

Analyzing thermal efficiency data, it was verified higher values until the fourth day of the drying process, accompanied by higher drying rates. Later than, the drying process became slower requiring for water movement from the inside cells to the surface, higher amount of energy, leading to a less efficient drying process.

Energy assessment was performed using Eq. (3).

$$S = \frac{I_{av} A_i t}{m} \tag{3}$$

where I_{av} is the average irradiation obtained daily inside the kiln, A_i is the area used to dry raw material, exposed to solar radiation, t is the time during which drying process took place, m represents mass of wet raw material (Singh *et al.*, 2006). A calculated amount of 19,434 kJ/kg was needed to dry the raw material.

3.2 Biomass ground and particle characterization

At the milling process, internal sieves with 4 and 6 mm diameter were sequentially used not being taken into account the energy consumption variations at this process. An average of 200 kJ/kg was necessary to grind the rockrose.

Figure 2 shows the sieving results related to the sawdust particle distribution obtained using a 4mm screen.

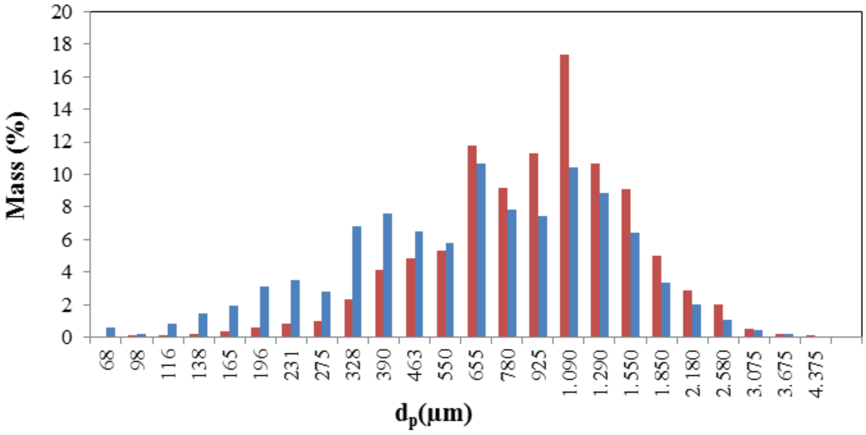

Figure 2. Sieving results – mass *vs* mean diameter (red – 4mm grid; blue – 4 mm grid reintroduced).

To study the influence of a wider particle distribution on the pellets quality, the raw material after being ground in the hammer mill was reintroduced and the sample produced was characterized. As seen in Fig. 2, this reintroduction of the raw material into the hammer mill lead to the production of a higher amount of smaller particles and to a particle mean diameter reduction from 640 to 412 µm.

The same procedure was performed using the 6 mm sieve, and the sieving results can be observed in Fig. 3.

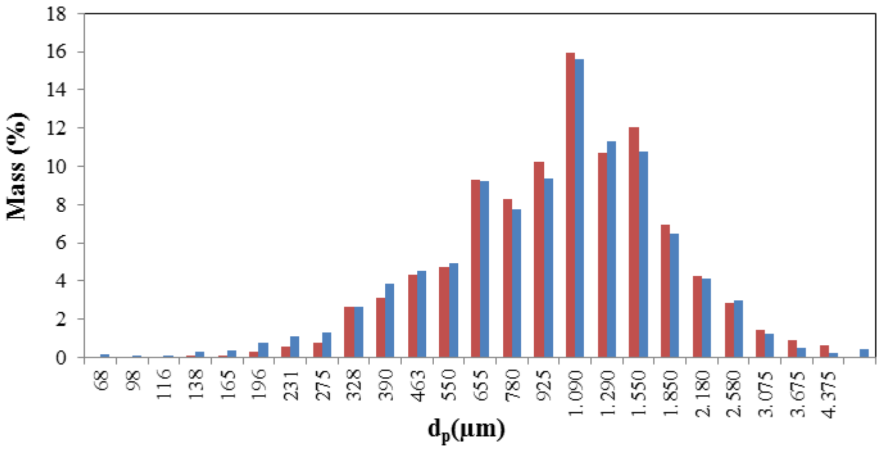

Figure 3. Sieving results – mass *vs* mean diameter (red – 6mm grid; blue – 6 mm grid reintroduced).

Using the 6 mm sieve internally in the grinding machine, 716 μm particle mean diameter sawdust was produced. After reintroducing samples into the mill, a 652 μm particle mean diameter sawdust was obtained. Even verifying a reduction on the particle mean diameter by reintroducing the sawdust in the mill, the particle distribution seems very similar, as shown in Fig. 3.

3.3 Pellets production and caracterization

The pellet production process occurred reaching 100 °C average temperature, adding sawdust with 120 kg/h feed rate. The sawdust produced with the 4 mm screen presented 12.1% M_{wb} and after reintroducing in the grinding mill presented 11.5% M_{wb}. In what concerns to the raw material produced using the 6 mm sieve, revealed 13.7% M_{wb} and further reintroduction into the mill decreased the M_{wb} to 13.4%.

Table 2 contains data related to the pellet characterization, where "R" represents the raw material reintroduced on the equipment.

Table 2. Pellets characterization.

Sieve hole diameter (mm)	4	4R	6	6R
Diameter (mm)	6.08	6.07	6.04	6.05
Length (mm)	35.57	26.59	27.16	26.8
Mass (g)	1.13	0.846	0.83	0.85
Particle density (m^3/kg)	1096	1099	1063	1106
Fine content (%)	4.45	3.89	1.45	2.17
Moisture (%, w.b.)	8.31	8.05	8.515	8.183
Durability (%)	92.91	97.56	98.73	98.4
Water resistance (%)	28.14	17.87	28.90	25.40
Energy (kJ/kg)	349	335	363	358

Concerning the use of the 4 mm sieve, as seen in Tab.2, the reintroduction of the raw material into the mill improved durability from 92.91 to 97.56% accomplishing the European standards compliance for high quality pellets for commercialization.

Additionally, an improvement occurred in water resistance index from 28.14 to 17.87%. Comparing the physical characteristics, all pellets were similar.

Using the 6 mm sieve, high quality pellets were produced presenting 98.73% durability. Reintroducing sawdust into the milling machine lead to the production of still high quality pellets with 98.4% durability, but an improvement on the quality was not verified.

3.4 Pellets produced from rockrose burnt by fire

After being burn by fire, a sample of raw material was collected from the forest, dried and pelletized. It was possible to pelletize this material after suffer thermal alterations. The sawdust was characterized and presented 680.3 μm as particle mean diameter and 9.87% M_{wb}.

Table 3 represents data from pellet characterization.

Table 3. Rockrose burnt by fire pellets characterization.

Sieve hole diameter (mm)	6
Diameter (mm)	6.02
Length (mm)	23.6
Mass (g)	0.76
Particle density (m^3/kg)	1063
Fine content (%)	26.3
Moisture (%, w.b.)	6.08
Durability (%)	74.6
Water resistance (%)	-
Energy (kJ/kg)	222

Pellets produced with forest fire burned raw material presented a durability of 74.6% and negligible water resistance (they practically disintegrate as they have acquired significant hygroscopic characteristics).

The energy consumed to pelletize the normal raw material ranged between 335 and 363 kJ/kg; 222 kJ/kg for the burned material.

4. CONCLUSIONS

The drying process took eleven days and from all the processes that lead to pellet production, drying needs higher amount of energy to be performed.

Hammer milled sawdust using internal 4 mm sieve revealed lower durability indexes that were increased by reintroducing sawdust into the grinding mill. That procedure made a reduction on the particle mean diameter from 640 to 412 µm, being observed the appearance of a wider distribution, conducting to an increase on pellets durability from 92.91 to 97.56% and a reduction of water resistance from 28.14 to 17.87%.

The best results obtained (representing high quality pellets for commercialization) were respectively 98.7 and 28.9%,, after adding Cistus ladanifer sawdust with mean particle diameters of 716 µm and a moisture content (in wet basis) of 13.7%.

Rockrose is able to be pelletized, even after being burnt by fire.

The energy needed to produce standard sufficiently good quality *Cistus* pellets (pelleting, milling and drying) was approximately 580 kJ/kg.

5. ACKNOWLEDGEMENTS

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