VOL. 9, NO. 1, JANUARY 2014 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

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EVALUATION OF EQUILIBRIUM MOISTURE CONTENT IN LIGNO-CELLULOSIC RESIDUES OF OLIVE CULTURE

Antonio Messineo[1], Giuseppina Ciulla[2], Simona Messineo[3], Maurizio Volpe [1] and Roberto Volpe [1]
[1]Facoltà di Ingegneria e Architettura, Università Enna "Kore", Cittadella Universitaria, Enna, Italy
[2]Dipartimento dell'Energia, Università di Palermo, Viale delle Scienze, Palermo, Italy
[3]Siciliacque S.p.A., Via Gioacchino di Marzo, Palermo, Italy
E-Mail: antonio.messineo@unikore.it

ABSTRACT

The use of ligno-cellulosic residuals from agriculture as means for renewable energy production is well known; nonetheless significant problems still exist on development of bioenergy value chains. Moisture content and hygroscopicity are among these problems. Higher moisture content of the biomass means lower calorific value and higher perishability, hence storage difficulties. For this reason it is important to evaluate how the moisture content of the material varies while it is stored and how the calorific value of the feedstock is affected by it. The purpose of this study is to determine the variation of moisture content and its influence on lower calorific value (LCV) of four types of solid residual biomass: oven dried chipped olive tree trimmings, milled olive pomace; oven dried olive tree trimmings and fresh olive tree trimmings. Moisture content of the samples is calculated by means of thermogravimetric analysis (TGA) according to standard CEN/TS 14774-2:2004, while the LCVs are calculated by means of a calorimeter, following standard UNI EN 14918:2010. All the biomass samples were left to reach equilibrium moisture content (EMC) in a temperature and humidity controlled environment. Two different phases were analyzed with respect to moisture uptake rates: 1) fast moisture uptake rate phase (first four hours of exposure) and 2) slow moisture uptake rate, (the days following the first four hours of exposure). Samples experiencing fast moisture uptake rate, during the first four hours of exposure, were kept in a monitored ambient at T=22 ± 1°C and RH=59 ± 2%, while samples exposed to slow moisture uptake rate were kept in a climatic chamber at three different set of temperature and humidity controlled environment simulating the climatic conditions in different periods of the year in Enna province: 10°C - 80% RH (winter), 15°C - 70% RH (spring/autumn) and 20°C - 55% RH, (summer). The results obtained show that the olive pruning chips (0.425 mm to 1.00 mm and 1.00 mm to 2.00 mm particle size ranges) stored in a heap in a controlled climatic chamber require approximately 20 days to reach EMC. Depending on the particle size range EMCs reach the values of 6.2 and 7.5% by weight in the "summer" condition, 14.3% and 16.9% in "spring/autumn" condition, 24.1% and 28.2% in "winter" condition. Moisture is absorbed gradually over time and results show that in the first four hours, the dry sample exposed to ambient condition (T=22 ± 1 °C and RH=59 ± 2%) reaches a moisture content between 0.75 and 0.96% of its weight; the LCV at equilibrium is evaluated between 18, 576 J•odg-1 and 18, 793 J•odg-1, the higher value related to the bigger particle size range examined. Pomace heap under the same experimental set up and time period, reaches an equilibrium moisture content of 8.5% and 9.7% (summer conditions), 19.2% and 22.0% (spring/autumn condition) and between 30.9% and 34.1% (winter condition). Olive pomace accumulates moisture relatively faster than olive trimmings as dry sample recovers about 1.61% to 1.97% of moisture in the first four hours of exposure. The LCV at equilibrium is determined between 20, 145 J•odg-1 and 20, 436 J•odg-1. Pruning dried samples reach an EMC equal to 6.1% in approximately 25 days, with a LCV equal to18, 921 J•odg-1 ± 1.9%. Heaps of fresh prunings reach an EMC of 8.2% in approximately 20 days; LCV of the feedstock is 19, 356 J•odg-1± 1.6%.

Keywords: olive residues, moisture content, storage, stabilization.

INTRODUCTION

While energy has become a central topic on the European and Italian political agenda, bioenergies have attracted the attention of researchers, operators and policy makers [1-3]. In particular, in Italy, energy production from biomass is significantly subsidized through incentive schemes designed to economically sustain investments. In last decades, bioenergies have attracted the attention of researchers, operators and policy makers, becoming a central topic on the European and Italian political agenda [4-9]. In particular, in Italy, energy production from biomass is significantly subsidized to economically sustain investments [10]. Several studies have demonstrated how residuals from agriculture and in particular residuals from olive cultivation may constitute viable feedstock for sustainable energy production in Euro-Mediterranean rural environments [11-14]; however, the actual practice is affected by numerous difficulties related to supply chain. In particular, the low energy density of the feedstock, the relatively high moisture content, and the hygroscopicity of the raw material pose serious difficulties on transport and storage of the feedstock and this in turn affects the energy available per kg of feedstock. The agro-food sector is particularly important especially for some agricultural areas of Sicily, where its fostering could significantly contribute to the local agricultural development [15] and the development of alternative resources (such as power generation from agro-waste), is in line with current European agricultural policies and regional energy master-plan [16-19]. Boasting some 158, 000 ha of cultivated land and € 188 mln of revenue, the olive agro-industrial sector is of major economic importance throughout Sicily [15].

Sicilian annual olive oil production totals about 47, 000 t from about 315, 000 t of olives. As a result, more than 268, 000 t of olive milling effluents (OME) and 379, 000 fresh tons of tree prunings and trimmings are produced as a by-products, which could potentially be converted into a low cost solid biofuel. Each of the 700 olive oil mills in Sicily produces on average 382 t of OME and accounts for 540 fresh tons of tree trimmings [20, 21].

Nonetheless the disposal of OME has always been a problem for Sicilian olive oil sector. Traditionally, producers have disposed of by-products by selling OME to the husk-oil industry.

However, nowadays, modern olive oil extraction techniques render husk-oil extraction less and less economic. In addition to this, in recent years, the husk-oil market is significantly shrinking and selling OME has gradually become more difficult. Hence, while in the past years the disposal of OME was a revenue source; nowadays it has become a significant problem for almost all olive oil mills. Olive oil producers are obliged to dispose of OME by spreading it on considerable areas of fields, thereby incurring in significant costs. While is not proven that OME can be used as a fertilizer or as a soil conditioner, the legislation which regulates OME disposal is becoming more and more stringent. High volumes of OME need to be disposed of yearly whilst suitable land is becoming ever less available. Similar conclusions may be drawn on the use of olive tree trimmings. A quota of it is still used in local markets as local low-cost fuel for bread-makers ovens; however the vast majority of it is even more frequently burnt in the open field, a practice which is dangerous for the environment and illegal.

Energy conversion of biomass offers today a valid alternative for disposal of olive cultivation by-products, which could be used as a low cost biofuel for renewable energy production, allowing farmers to access to an additional source of revenue. However, the energy conversion of olive farm by-products is somewhat problematic due to its high moisture content and its high seasonality. OME are mainly composed of a perishable sludge showing in excess of 50% moisture content and their production is concentrated in only two or three months each year. Olive tree prunings and trimmings also show some 45% of moisture content and equally hindering seasonality [21]. In this scenario, a process aiming at sustainable energy conversion of olive cultivation by-products could be a significant potential solution towards overall sustainability of olive cultivations. Energy conversion of by-products could transform a potential problem into a virtuous renewable energy production process.

However, as mentioned, the practical utilization of this feedstock poses significant logistical problems. For the purpose of this study, the supply chain of residual biomass may be divided in the following five steps:

- collection;
- transport;
- size reduction;
- drying - stabilizing;
- storing.

To facilitate the transportation and conservation of feedstock, lingo-cellulosic biomass is normally transformed depending on its original form. Tree trimmings may be chipped or shredded then stored in piles, while OME may be stabilized by means of drying then milled and stored in piles, or densified and finally stored in piles [15].

Plant managers need to decide whether to store the original feedstock fresh or dried, and to what particle size should the chipping process be taken prior to further processing. Knowing the EMC for each type of feedstock is of crucial importance to take this operational decision.

The EMC of each of the feedstock types is also one of the important characteristics to be taken into consideration for the densification process. Recently, Canadian researchers have developed a model for estimating the moisture changes of woody biomass during storage when exposed to weather variability [22].

This study aims at investigating some of the issues connected to the storage of the feedstock in the forms mentioned, when the biomass is stored in a temperature and humidity controlled environment to simulate an indoor storage. In particular, this research evaluates how the moisture content of piles of chipped olive tree trimmings, milled OME and fresh tree trimmings varies with time.

The LCVs of the feedstocks after different storages are also evaluated. Further comparisons are made with piles of tree trimmings, when stored fresh or previously dried.

MATERIALS AND METHODS

The research carried out in this study deals with the evaluation of equilibrium moisture level of biomass at three different constant climatic conditions, simulating condition over the climatic condition over the year in Enna province. Evaluation of LCVs at equilibrium of the following different biomass samples was also performed:

- oven dried piles of chipped olive tree trimmings, 0.425 - 1.000 mm particle size range;
- oven dried piles of chipped olive tree trimmings, 1.000 - 2.000 mm particle size range;
- oven dried piles of milled olive pomace, 0.425 - 1.000 mm particle size range;
- oven dried piles of milled olive pomace, 1.000 - 2.000 mm particle size range;
- oven dried stacks of olive tree trimmings;
- stacks of fresh olive tree trimmings.

Each sample of biomass was exposed at three different sets of temperature and relative humidity, according to the average climatic conditions registered over the year in Enna's region:

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- 10°C - 80% RH (winter);
- 15°C - 70% RH (spring/autumn);
- 20°C - 55% RH, (summer).

The evaluation of moisture content was conducted by means of thermogravimetric analysis using a LECO TGA-701 thermogravimetric analyzer, (CEN/TS 14774-2:2004). The LCV was calculated by means of a LECO AC500 calorimeter, (UNI EN 14918:2010). Biomass moisture uptake to equilibrium was performed by means of a Binder KMF 115 constant climate chamber. Pretreated biomass samples were left to equilibrate inside the climatic chamber until EMC was reached. A DL-UT2 temperature and humidity data logger was used for monitoring the biomass moisture uptake during the first four hours exposure at ambient conditions (T=22 ± 1°C and RH=59 ± 2%). The biomass samples have been chipped using an electrical knife mill. Typical chip size was between 1 and 13 mm. The chipped biomass was then finely ground by means of a Restch PM100 planetary ball mill equipped with a 500 ml corindon jar and 25 corindon spheres of 20 mm in diameter. After comminution, biomass fractions used for the experiments were sieved using 2.000 mm, 1.000 mm and 0.425 mm sieves, thus obtaining two particle size ranges for each biomass sample (0.425-1.000 mm and 1.000-2.000 mm). A Retsch vibratory sieve shaker model AS 200 basic equipped with standard 200 mm diameter and 50 mm height ISO-3310/1 sieves was used for this purpose. Approximately 20 kg of feedstock have been collected from olive trees of ‘Moresca’ variety grown in the Enna Province, Sicily. This variety was selected as it is the most common in that province. Plants are approximately 50 years old and show significant vigour. Trimmings have been collected manually and include branches 1 and 2 years old. The samples were taken randomly, all leaves have been kept and branches showed diameter between 2 and 15 mm, no olives were included in the collection. The collected feedstock has been put fresh into a dark and airtight bag so to avoid significant moisture loss.

The collected feedstock was used to prepare samples for the following categories:

- oven dried piles of chipped olive tree trimmings, 0.425 - 1.000 mm particle size range;
- oven dried piles of chipped olive tree trimmings, 1.000 - 2.000 mm particle size range;
- stacks of fresh olive tree trimmings;
- oven dried stacks of olive tree trimmings.

Similarly, approximately 20 kg olive pomace obtained by cold squeezing of the same olive variety ‘Moresca’ was collected from an olive mill located in the Enna province. The pomace was collected fresh from a two stage olive mill and placed in dark air-tight barrels in order to avoid premature drying or oxidation. The collected feedstock was then used to prepare the following samples:

- oven dried piles of milled olive pomace, 0.425 - 1.000 mm particle size range;
- oven dried piles of milled olive pomace, 1.000 - 2.000 mm particle size range.

Randomly collected samples of 2 kg of the initially collected feedstock (both trimmings and pomace) were oven dried by means of a fan assisted, electronically controlled Memmert Universal Oven model UF55. The drying process has been performed following the CEN-TS standard so to avoid any local chemical modification of the material [23].

The oven dried 2 kg sample of trimmings was fully chipped by means of an electrical knife mill, to a chip size between 1 and 13 mm. Similarly the 2 kg dried sample of pomace was milled to particle size between 1 and 7 mm. All the obtained material was then finely milled by means of the planetary ball mill. The milled feedstock was then sieved by means of a Retsch vibratory sieve shaker model AS 200 basic, to obtain the following samples categories:

- milled olive tree trimmings, 0.425 - 1.000 mm particle size;
- milled olive tree trimmings, 1.000 - 2.000 mm particle size;
- milled olive pomace, 0.425 - 1.000 mm particle size;
- milled olive pomace, 1.000 - 2.000 mm particle size.

After chipping and milling, the samples were placed in the oven at 105°C for approximately 24 hours, until all the residual moisture was removed. Once fully dried, feedstock was put in the constant climatic chamber and divided into two piles per each of the categories listed above. One pile was used for subsequent sampling and the other as means for control. Biomass moisture content at equilibrium has been dealt with data in literature [22-24]. The EMC is defined as the moisture of the biomass as the feedstock achieves equilibrium with the surrounding environment at a given pressure, temperature and relative moisture, and is calculated as:

$EMC= (w_e- w_d)/w_e$

where
w_e = weight of the biomass at equilibrium;
w_d = weight of the oven dried biomass.

Moisture content of ligno-cellulosic biomass is a dynamic property; it varies with time and in a non-linear way [23]. The further the biomass is from EMC, the faster the increase in moisture content of the feedstock. The rate of increase of moisture of the feedstock is therefore expected to decrease with time. For this reason the variation of moisture content of sample is evaluated in the two separate phases as follows:

- Evaluation of variation of moisture content first 4 hours;

- Evaluation of variation of moisture content until EMC.

Evaluation of variation of moisture content first 4 hours

This analysis was conducted only for the dried samples. A quantity of approximately 5 grams per each of the dried biomass sample's category was collected randomly from a pile and placed in an oven at 105 °C temperature until it was completely dried. Once dried, a number of 3 small samples weighting approximately 0.5 grams where collected from it and placed on an analytical scale 0.0001 precision, to evaluate the increase in weight. During the experiments the external temperature and humidity were registered by means of a DL-UT2 temperature and relative humidity data logger.

The increase in weight was noted at 2 minutes time intervals for the first 30 minutes, then every 10 minutes and then every 20 minutes as the rate of increase in moisture decreased.

Evaluation of variation of moisture content until EMC

This analysis was conducted for all the sample's categories. Moisture content of the biomass was evaluated throughout time for a period of 25 days, which proved sufficient for all the categories of feedstock to reach equilibrium. Samples were collected from the internal and external part of the piles and randomly from the stacks, and were left to reach equilibrium in the constant climatic chamber. The conic piles typically had a diameter of 30 cm and a 20 cm height. Three samples have been collected per each part of the pile (or stack) at regular intervals of 24 h for the first 5 days then at regular intervals of five days and the moisture uptake was calculated as the average of the three values, obtained by thermogravimetric analysis. The variation in moisture content and the time needed to reach equilibrium of both the internal and external part was also calculated. Internal samples within piles have been collected using a long thin spoon from a portion approximately 2-3 cm far from the bottom so to avoid any effects related to contact at the base of the pile. Due to the small amount of samples collected, we can assume that sampling did not appreciably influence the piles moisture uptake and thus the experimental data. Results from the control piles and stacks confirmed this assumption. The samples collected from the piles were typically 2.00 g to 2.50 g for milled olive tree trimmings and olive pomace, 4.00 g to 9.50 g for fresh tree trimmings. Higher quantities of samples were needed to ensure good homogeneity of not milled biomass.

RESULTS AND DISCUSSIONS

Figure-1 shows the evaluation of oven dried chipped wood moisture uptake for the first 4 hours of exposure, while the environmental conditions were: 22 ± 1 °C and RH=59 ± 2%.

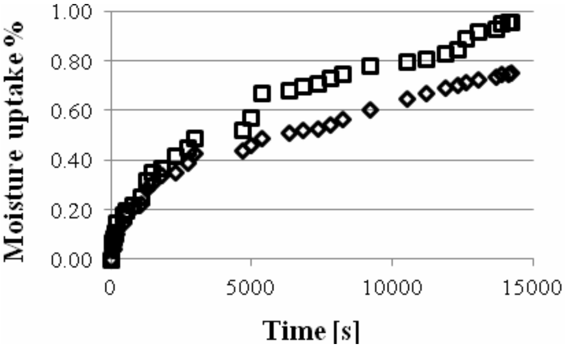

Figure-1. Moisture uptake of chipped wood: 0.425 - 1.000 mm (□) and 1.000 - 2.000 mm (◊) of particle size ranges during first 4 hours of exposure to the environmental conditions (RH = 59 ± 2% and T = 22 ± 1 °C).

The curves show that samples with the particle range size of 0.425 -1.000 mm gradually reach a moisture content of 0.96% in 4 hours time (□), while the samples with the particle range size of 1.000 - 2.000 mm reach a moisture content of 0.75% (◊) in the same interval of time.

Figure-2 shows the moisture uptake of oven dried olive pomace (0.425 - 1.000 mm and 1.000 - 2.000 mm particle size ranges) when exposed for 4 hours to a controlled environment (T=22 ± 1 °C and RH=59 ± 2%).

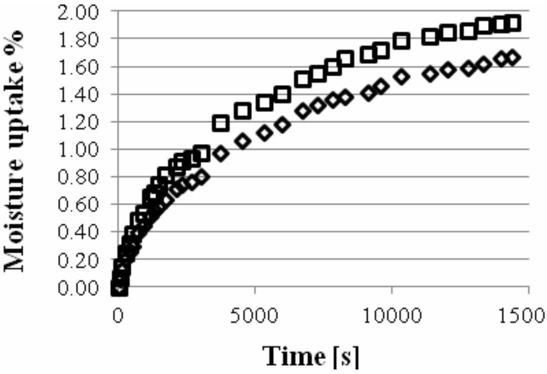

Figure-2. Moisture uptake of olive pomace: 0.425 - 1.000 mm (□) and 1.000 - 2.000 mm (◊) of particle size ranges during first 4 hours of exposure to the environmental conditions (RH = 59 ± 2% and T = 22 ± 1 °C).

The oven dried olive pomace of 1.000 - 2.000 mm particle size range reaches, in four hours, a moisture content of 1.67% while oven dried olive pomace of 0.425 - 1.000 mm particle size range reaches a moisture content of 1.92% in the same time interval.

Table-1 shows the equilibrium moisture contents of the two particle sizes of milled olive tree trimmings and olive pomace when the samples are left in a climatic chamber at different temperatures and relative humidity. All the samples reach equilibrium between 20 and 25 days.

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Results show that after approximately 20 days the moisture uptake of the chipped wood with the particle size range of 0.425 - 1.000 mm stabilises at values of 28.21%, 16.87 and 7.54% by weight at 10°C - RH=80%, 15°C - RH=70% and 20°C - RH= 55% respectively. The higher size fractions of chipped wood (size range of 1.000 - 2.000 mm) show the following moisture uptakes respectively: 24.15%, 14.34% and 5.99%. Values show a difference between the internal part and the external part of the pile for about 10 days, then the two moisture values increase together to the approximately the same final moisture uptake.

The results calculated for the control pile show that manual collection of samples did not significantly influence the moisture uptake of the pile. Moreover the moisture uptake of the control pile shows no significant difference with the moisture uptake of the sampling pile.

Table-1. Moisture uptake of olive tree trimmings and olive pomace: 0.425-1.000 mm and 1.000-2.000 mm of particle size ranges during after 20-25 days of exposure to three different sets of climatic conditions.

Biomass species	Equilibrium moisture content (%)		
	T = 10°C RH = 80%	T = 15°C RH = 70%	T = 20°C RH = 55%
Olive tree trimmings 0.425-1.000 mm	28.21	16.87	7.54
Olive tree trimmings 1.000-2.000 mm	24.15	14.34	5.99
Olive pomace 0.425-1.000 mm	34.06	21.96	9.57
Olive Pomace 1.000-2.000 mm	30.88	19.25	8.22

The LCV at equilibrium was 18,576 J•odg-1± 1.9% for the sample of 0.425 - 1.000 mm particle size range and 18,793 J•odg-1 ± 1.6% for the sample of 1.000 - 2.000 mm particle size range. On the other hand when the oven dried olive pomace is exposed to the conditioned environments, the 0.425 - 1.000 mm particle size range sample of olive pomace reaches averagely an equilibrium moisture of 34.06% at 10C - RH=80%, 24.96% at 15°C - RH= 70% and 9.57%, while the 1.000-2.000 particle size range samples reach an EMC of 30.88%, 19.25 and 8.22% respectively. In this case low differences in moisture uptakes between internal and external sides of the piles were registered.

The differences were between 0.4-0.7% of the equilibrium moisture uptakes, the internal samples showing lower values. The moisture uptakes values reported in Table-1 are the average ones. Oven dried olive pomace reaches an EMC higher than that of chipped wood. This is probably due to the higher porosity of the feedstock. The LCV at equilibrium is 20,145 J•odg-1 ± 1.5% for the sample of 0.425 - 1.000 mm particle size range and 20,436 J•odg-1 ± 1.6% for the sample of 1.000 - 2.000 mm particle size range.

Figure-3 shows that oven dried olive tree trimmings take approximately 25 days to reach their EMCs of 22.7%, 13.0 and 6.2% at 10°C - RH=80%, 15°C - RH=70% and 20°C - RH= 55% respectively. The EMCs values are close to those obtained for the larger size particles (1.000 - 2.000 mm) of milled biomass samples.

Figure-4 shows that moisture content of fresh olive trims decreases from a value of 47.79% at collection to a value of 27.6% at T=10° - RH=80%, 17.2% at T=15° - RH= 70% and 8.21% at T=20°C - RH=55% Data show that the biomass reach values close to the EMC in 25-30, days the longer time needed for the lower relative humidity condition. In 10 days the biomass lost between 10 and 20% of its moisture content.

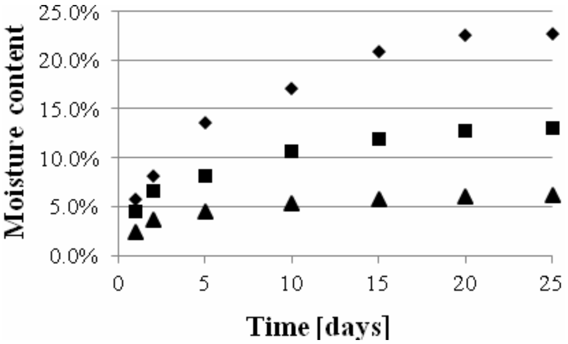

Figure-3. Moisture uptake with time of dried olive tree trimmings: ♦ T=10°C - RH80%, ■ T=15°C - RH= 75%, ▲ T=20°C - RH= 55%.

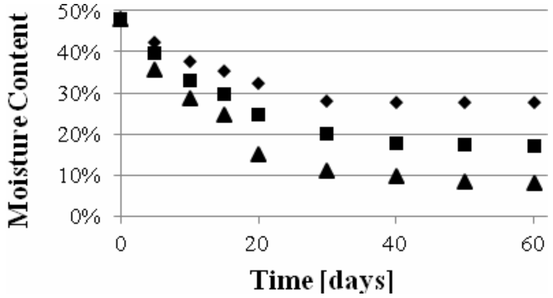

Figure-4. Moisture content variation with time of fresh olive tree: ♦ T=10°C - RH80%, ■ T=15°C - RH= 75%, ▲ T=20°C - RH= 55%.

CONCLUSIONS

Results obtained may be summarized as follows:

- Oven dried chipped olive tree trims of 0.425-1.000 mm particle size ranges reach equilibrium moisture contents between 7.5%, 16.9% and 28.2% depending on the climatic condition set for the experiment (T=20°C-RH=55%, T=15°C RH=70% and T=10°C RH=80%); on the other hand olive tree trims of 1.000 - 2.000 mm particle size reach equilibrium moisture values of 6.0%, 14.3% and 24.1%. No appreciable difference in moisture contents at equilibrium is detectable between the internal and the external parts of the piles. Experimental data confirm that the smaller particle size range samples absorb moisture faster than the bigger particle size range, with a moisture uptake in four hours of 0.96 and 0.75% by dried weight respectively. The difference in the rate of moisture uptake and the moisture at equilibrium is probably due to the higher superficial area of the smaller particles. Chipped olive tree trims particle size range 0.425-1.000 mm show a LCV at equilibrium of 18,576 J•odg-1 ± 1.9% while 1.000-2.000 mm particle size feedstock shows a LCV at equilibrium of 18,793 J•odg-1 ± 1.6%.

- Oven dried milled olive pomace, exposed to the above mentioned constant climatic conditions; require between 20 and 25 days to reach their EMC. Samples of particle size range 0.425-1.000 mm reach moisture contents at equilibrium of 9.6%, 22.0% and 34.1% while samples of particle size range 1.000-2.000 mm reach values of 8.2%, 19.2% and 30.88%.
- When exposed to monitored environment conditions (T=22 ± 1 °C and RH=59 ± 2%), the same samples show an initial rate of moisture uptake significantly higher than that of the chipped wood, in fact, samples reach a moisture content of 1.92% (0.425-1.000 mm particle size range) and 1.61% (1.000-2.000 mm particle size range) during first four hours of exposure. LCV at equilibrium was respectively 20,145 J•odg-1± 1.5%; and of 20,436 J•odg-1 ± 1.6%.
- Oven dried olive tree trimmings need approximately 25 days to reach the following EMCs values: 6.2% (T=20°C RH=55%), 13.0% (T=15°C RH=70%) and 22.7% (T=10°C RH=80%); the LCV of the feedstock is 18,921 J•odg-1± 1.9%.
- Piles of fresh olive trims reach the equilibrium values in approximately 40 days: 8.2% (T=20°C RH=55%), 17.2% (T=15°C RH=70%) and 27.6% (T=10°C RH=80%); the LCV of the feedstock is 19,356 J•odg-1± 1.6%.

As it can be seen by the above summarized results, the oven dried olive pomace absorbs some between 2% and 5% more moisture at equilibrium, compared to chipped wood trims. Similarly, the moisture uptake during the first 4 hours of exposure is considerably higher. This seems due to the physical properties of the feedstock.

Environmental conditions and particle size range strongly influence the moisture at equilibrium and the uptake rates. The smaller the feedstock particles, the higher the moisture at equilibrium and the moisture uptake rate. A difference of 25% of relative humidity lead to an increase between 20 and 25% in the moisture content at equilibrium, the smaller the particle size the higher the difference.

Oven dried tree trimmings absorb approximately the same moisture absorbed by the chipped trimmings. However it takes a longer period to reach equilibrium. This apparent benefit is counterbalanced by the higher storage difficulty posed by the tree trimmings when compared to chipped trimmings.

Fresh tree trimmings reach EMC slightly higher than that reached by oven dried trims. Albeit obtained with lab scale heaps, the results of this study refer to sample's particle sizes which are in all similar to those used at industrial scale. The data obtained give useful insight to plant managers with regards to their decision in storage and processing of the feedstock.

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