

Mapping of the best solutions for physical pre-treatment of the biomass

Assessment of technical solutions for pre-treatment of raw materials

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Summary

The aim of these activities is pointing out the available solutions for mixing, sizing, and drying pomace and olive pruning and selecting the most appropriate option for each type of raw material.

The main expected outcome is defining the best technical solutions for pre-treatment of raw materials. In this line, a lab protocol for producing pellets to be tested in the lab gasifier will be defined.

Technical solutions for pre-treating raw material were found by taking into account the state of art technologies and CEN EN Biomass standards. The task has been conducted in coordination with WP3: the pellets once produced were sent to Fraunhofer to feed the lab-scale gasifier.

ISAFoM produced an overall amount of 100 kg of pellets, divided into nine batches with different composition: 0, 25, 50, 75 and 100 % olive pruning over olive oil waste.

In the second year a production of a further amount of 100 Kg, divided into two batches, was performed

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1. Introduction

The main expected outcome of the work associated with this deliverable is defining the best technical solutions for pre-treatment of raw materials.

In this context, a lab protocol for producing pellets to be tested in the lab gasifier was defined. The present work investigates the properties of blends of pelletised residues from olive pomace and olive pruning analysed with the aim to find the best blend composition to be used to produce fuel by means of the FFW process involving gasification, and subsequent Fischer-Tropsch and methanation processes.

In the Mediterranean areas of the Southwest of Europe, agro-industrial business plays a paramount role in the local economy, and therefore during the agricultural activities a significant amount of residues are generated; above all, olive pomace stands out as one of the most abundant. The management of these residues involves a problem for these industries due to their potential as pollutants in some cases and to the costs associated to the treatments needed for their proper removal.

An interesting opportunity to recycle such waste is to reuse it as biofuel through densification.

Indeed, pelletised biofuels, due to their increased density, are characterised by homogeneous size, which facilitates automatic or semi-automatic treatments.

Furthermore, their use also reduces the costs associated with handling and transportation.

The olive pomace, once subjected to a drying process, can be properly used as a source to produce fuel such as syngas and biodiesel. It is worth noting that its oleaginous characteristics prevent the densification during the pelletizing process. Moreover, their high concentrations of certain elements exceed the specifications given by the corresponding standards. Therefore, it is necessary to blend the olive oil by-products with other biomass residues to make products suitable for gasification. [1].

1.1 Background

Technical solutions for pre-treating raw material have been found taking into account the

state of art technologies and CEN EN Biomass standards.

The most appropriate mixture of the different residues has been assessed taking into account availability, efficiency of the process and the requirements of the subsequent treatment (Gasification).

2. Lab scale protocol description

The goal of this task is to describe a lab scale protocol to be scaled up by means of a pilot test during subsequent activities.

2.1 Standard references for pellets production

In order to determine the applicability of biological raw materials for the production of pellets, usable raw materials were defined in compliance with the European standards. According to the standard EN 14961, the material is classified as biofuel in origin and source. With reference to *Table 1 Classification for origin and sources of solid biofuels*+EN 1496-1¹, the material used in this work belongs to Group 3. Fruit biomass (cf. group 3.2.1.4, 3.2.2.4, 3.2.3), which refers to fruit biomass material that is pure or left over after industrial handling and treatment.[3]

In this respect, the raw materials used to produce pellets must comply with prEN 14961-6 which is the standard defining the allowed constituents of pellets.[4]

Further parameters for the evaluation of biological materials as raw material for pellets are predetermined by the pelletisation technique and also by the gasification technique with parameters influencing the gasification behaviour. Thus, our analysis is based upon the feedback given by WP3 leaders.

2.2 Pellets production

2.2.1 Unit operations layout

Pellets are the product of a mechanical process based on mechanical and thermal phenomena able to activate chemical and physical transformation of raw material,

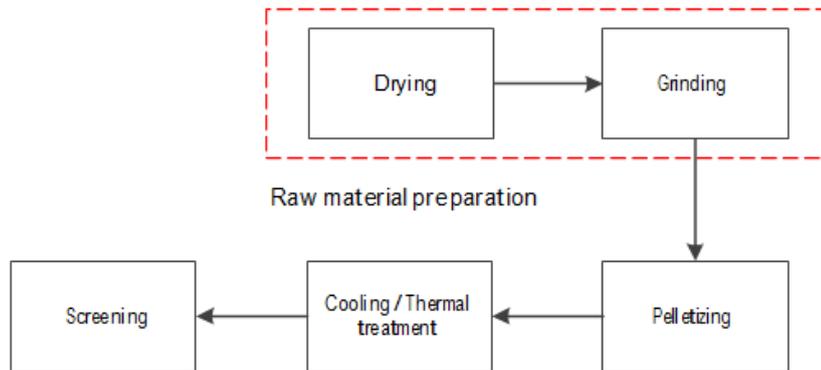
¹ See CENEN 14961-1 table 1

more relevant in the case of blended materials. Generally, unit operations in pellets manufacturing process are: drying, grinding, pelletising, thermal treatments and screening so fines can be recycled and pelletised.

The sequence of the most suitable unit process operation individuated during this task is illustrated in Figure 1.

The timeline of the sequence in Figure 1 can be rearranged due to chemical and physical characteristic of the raw material, and for desired final characteristics of the products. Starting from preliminary production tests, the unit operations were optimised in order to produce a good quality pellet in terms of both durability and energy quality. Once the raw material is dried and suitably reduced in size, it is extruded in the pelletizing unit. During extrusion, due to high friction forces, forming pellets are thermalized up to a temperature able to set natural bonding agents conferring the suitable hardness to the product. [2]

Figure 1 Unit operations layout of the pelletising process



2.2.2 Raw material preparation

The product of the last clearing of the olive tree consists of pruning residues. For the purpose of this work, representative samples from an olive orchard, typical of Central Italy have been considered; those residues have not been barked. This circumstances have to be taken into account for the proper interpretation of the results, expecting higher ash content than in the case of a fined-down residue.

Likewise, the olive pomace has been collected from local manufacturers from Central Italy regions. The olive pomace is a product deriving from two and three phase methods.

In order to have the best selection of blends to be used in lab production tests, experiments are performed in almost any field of enquiry and are used to study the performance of processes and systems. Some of the process variables are controllable although they may be controllable for the purpose of a test, while other variables are not.

The objectives of the experiment include: determining which variables are the most related to the response, determining where to set the controllable variables so that the response is usually similar to the desired optimal value, so that the variability in the response is small, so that the effects of uncontrollable variables are minimized.

-Determination of batches composition

Olive mill husk (OMH) from two- and three- phase decanter, collected from two local mills, and olive tree pruning (OP) have been included among raw materials for preparing pellets to be processed according to the guidelines of the FFW project. Exhausted OMH was excluded for ecological and market reasons; in fact, the chemical extraction process requires previous drying of the husk, which is costly, especially for two-phase husk; in addition, extraction plants release into the air solvents (hexane) that are pollutants. At the moment and at least in Italy, the production of olive husk oil is decreasing, due also to the very low quality oil production, if compared to the more valuable extra virgin olive oil, derived from mechanical extraction.

Due to the high moisture content, both ingredients were dried before grinding.

Drying of OMH was performed in wintertime using a greenhouse provided by a heating system and by putting the raw materials in thin layers over heated beds, reducing waiting time associated with natural drying. To speed up the drying process OMH was periodically turned by hand (at least once per day). OP was previously mechanically chipped before drying which was performed in the same environment.

When both raw materials reached moisture content below 20%, they were ground using a laboratory miller (RetschZM200), equipped with sieves as larger as possible, for reducing energy consumption, but ensuring correct pelletizing; with this regards, according to the feed-back obtained, a 4 mm sieve was the best choice.

With the aim to produce pellets characterized by different chemical composition to be tested in the following FFW procedure (chemical pre-treatment), after grinding, the resulting powders were mixed in different proportion by weight. In our case, it sounds reasonable to analyse the response of the system by varying the composition of the blend. In order to obtain a likelihood characterization, it is necessary to analyse the behaviour of the composition over a wide spectrum. In this respect the following sets of compositions are proposed.

Table 1 shows the different blends of pruning and olive oil waste batches that have been prepared within this task, and the labels which have been adopted:

Table1 – Batches label and compositions

PR	100% olive pruning
3PH	100% three phase method olive waste
2PH	100% two phase method olive waste

Table 2 – Categorisation of samples

PR/2PH	0% / 100%	25% / 75%	50% / 50%	75%/25%	100% / 0%
PR/3PH	0% / 100%	25% / 75%	50% / 50%	75%/25%	

One of the main matters to be considered is the pomace stability related to the high moisture content. Hence, the lowering of moisture content (MC) to less than ca. 20wt% is required in order to achieve a product stability limiting microorganisms activity. The most convenient method, from technical and economic point of view, to obtain stable pomace is to perform natural drying. In our case, pomace (2PH and 3PH) was arranged in a greenhouse, in a 3 cm bed, and it was periodically moved in order to facilitate the drying process.

At a MC lower than 20wt%, pomace was grinded using a hammer mill (RETSCH SM 200) equipped with a screen with holes of 4 mm in size. Lab milling procedures produced some problems in the reduction of material size, mainly due to the high value of moisture content. These problems suggested performing a further drying phase to consistently

reduce MC and produce a procesable pomace. Drying was operated by static oven, lowering water content respectively to ca. 7 and 11% in the case of 2PH and 3PH, as illustrated in Table 3.

Table3 Æ Moisture content of samples %(w.b.)

Material	Initial content	Stability	Before pelletisation
PR	47.40%	-	1.57%
3PH	57.20%	20%	6.97%
2PH	72.86%	20%	11.21%

Moreover, the size distribution of three representative 50 g samples was determined and illustrated in Table 4. This distribution was obtained by manual sieving on a stack of sieves arranged from the largest to the smallest opening. The sieving procedure terminated when no further notable passing took place. For the samples from the 4 mm hammer mill screen size the sieve sizes 3150, 2800, 2000, 1400, 1000, 500 and 250µmused.

Table 4 Æ Size dimension distribution

Size dimension [mm]	PR	2PH	3PH
>3.15	0.00%	0.00%	0.00%
2.80÷3.15	0.12%	0.05%	0.08%
2.00÷2.80	1.45%	1.98%	1.78%
1.40÷2.00	10.16%	8.27%	7.58%
1.00÷1.40	17.56%	24.95%	22.56%
0.50÷1.00	46.54%	47.94%	50.17%
0.25÷0.50	12.16%	9.97%	12.34%
<0.25	7.02%	6.84%	5.50%

Concerning pruning residues, in order to obtain the same low water content, the material has been oven dried starting from branch wood chips and leaves; the final moisture content is shown in Table 3, while the size distribution of PR is shown in Table 4.

2.2.3 Lab-scale production

The overall goal of this activity is to provide conditions to produce pellets from biomass feedstock or bind formulations whose densification characteristics are relatively unknown. One of the most relevant issues to produce strong and durable pellets depends on the physical forces that bond the particles together.

The shape and the size of a densified fuel usually determine the correct choice of feeding technologies as they influence the conveying and combustion/gasification behaviour of the fuel.

The strength and durability of the densified products mainly depend on the nature of the material and on the physical forces that bond the particles together [5].

To perform our test, a lab scale pelletiser designed and produced by a local manufacturer was used.

In our pellet production equipment, the feed material is pressed through open-ended cylindrical holes die made in the periphery of a series of rings. Two small rotating rolls push the feed material into the die holes from inside of the ring towards the outside of the ring. The skin friction between the feed particles and the wall of the die resists the free flow of feed and thus the particles are compressed against each other inside the die to form pellets.

In pressure agglomeration, high forces are applied to a mass of particulate materials within a confined volume to increase the density.

In the case of pellets, their diameter is determined by selecting a die with the correct diameter of the die holes, considering that the bigger the particles are, the more robust feeding appliances have to be used and the required time for the gasification process increases.

Based on the outcomes given by our test, since the pellet production is a very complex process, simple rules can be given:

- “ The pellet diameter is determined by the diameter of the holes.
- “ The pellet density is linked to length of holes and to physical and chemical characteristics of raw materials, as:
 - Mean size of particles;
 - Water content influencing transport phenomena during densification;
 - Lubricant content influencing friction and transport phenomena as well;
 - Chemical composition, ie.: ash, lignin content, water soluble compounds, etc..

With regards to the pellets dimension and shape, the length of pellets was not controlled by pelletising machine setting. Produced pellets have a diameter of 6 mm and a maximum length of 35 mm according prEN 14961-6 that states that pellet length cannot be more than 50 mm. The length is particularly important if pneumatic feeding systems will be used since one single overlong pellet can cause blockings in the feeding system. This can lead to a standstill of the whole system.

First pelletisation tests performed on PR, 2PH, and 3PH showed that extrusion procedures cannot be accomplished using low moisture material- see Table 3- as by using such

grinded materials binding phenomena are not activated leading to lump product. The reasons can be explained by the influence of water on the thermal conductivity that promotes binding mechanisms, between components of raw materials, and on chemical reaction, that allows activation of natural bonding agent. As a result of this consideration, different pelletisation tests have been performed conditioning grinded raw materials by adding water at three different levels: 5, 10,15wt%.

Pelletisation has been achieved at total moisture content higher than ca.17 wt.%. Together with the moisture content, another key parameter that was investigated to perform a good product is the die temperature. An optimal die temperature in the range of 80-90°C has been identified by the experiment. It is easy to reach this level of temperature with Pruning, while in the case of pomace it was necessary to set up the pelletising machine prior to other unit operation, with high friction material such as i.e.: olive pruning or poplar wood saw dust. The increase in temperature can be related, as Bradfield and Levi [6] stated, to an efficient friction of wood polymers on the die holes. This effect in the case of pomace shows a less efficient friction behaviour due to the presence of fat and moisture that alter the cohesive forces between the material and the die, even if the presence of free water among particles amplify cohesive forces between particles [5]. Once that the material came out from the pelletiser it was gradually air cooled, in order to solidify and strengthen the pellets promoting the action of natural bonding agents. Pellets obtained from 2PH and 3PH resulted almost friable and too wet. MC level was higher than the required one (10%). An additional drying process must be considered in order to reduce the MC and strengthen pomace pellets obtaining sharp strengthen of pellet products.

Figure 2 shows the water content level for 2PH, 3PH, PR for main steps of pelletisation process. To date, we obtained the control of the moisture content in the 2PH and 3PH pomace blend with different composition. Without a doubt, our goal was to achieve a product within the given moisture constraint, which requires a less expensive quantity of energy to dry it. Even if the presence of such high water content causes such a rapid decrease of the die temperature, our effort was to stabilise this parameter within the Above-cited range. Since the die is not thermal controlled to reach the above mentioned activating temperature the rotation speed was regulated upon the material employed.

2.2.3.1 Amounts produced and production plan

ISAFoM produced and supplied to FhG an overall amount of 100 kg pellets, divided in nine

batches with different composition: 0, 25, 50, 75 and 100 % olive pruning over olive oil waste.

After the feedback received from FhG an extra amount of 100 Kg of pellets, divided into two batches, has been produced and sent to WP3 leaders.

It is foreseen that other amounts will be produced during the scaling-up of the pellets production. These amounts will be sent to Luxembourg to be used in the DEMO phase (WP5)

2.3 Chemical-physical fuel properties

2.3.1 Moisture content of raw materials and pellets

The moisture content of pellets is relevant for the net calorific value, the efficiency of gasification process and the combustion temperature. Net calorific value, efficiency and combustion temperature decrease with rising moisture content.

In Figure 2 the moisture content during the whole process is illustrated.



Figure 2 – Moisture content of both pellets and raw materials

2.3.2 Gross calorific value, net calorific value and energy density of pellets

The gross calorific value is a measured value of the specific energy of combustion for a mass unit of a fuel burned in oxygen in a bomb calorimeter under specified conditions. It is an intrinsic property of the raw material and cannot therefore be influenced. The gross calorific value can be determined by using a bomb calorimeter [7]. The net calorific value depends mainly on the gross calorific value, the moisture content and the content of hydrogen in the fuel. Other parameters such as nitrogen, oxygen or ash content have a minor influence. The NCV can be calculated from the GCV.

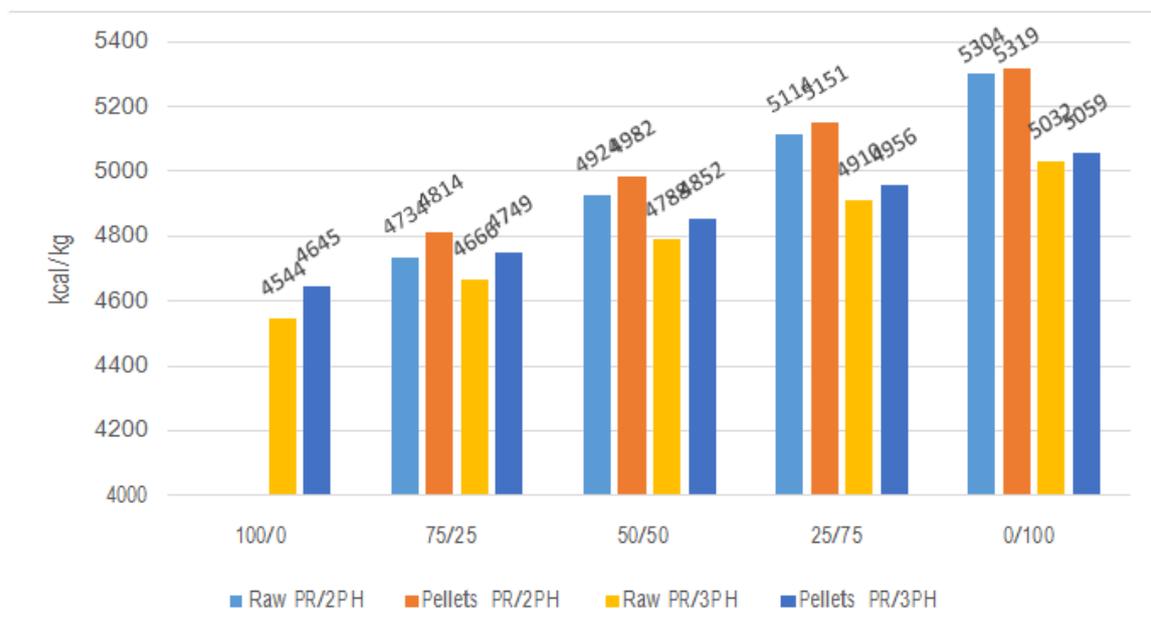


Figure 3– Net calorific value of both pellets and raw materials

2.3.3 Ash content

The ash and moisture content was determined according to the standard ASTM - D5142[8]. These instrumental test methods cover the determination of moisture, volatile matter, and ash, and the calculation of fixed carbon in the analysis of coal and coke samples prepared

in accordance with Method D 2013 and Practice D 346. Results are showed below.

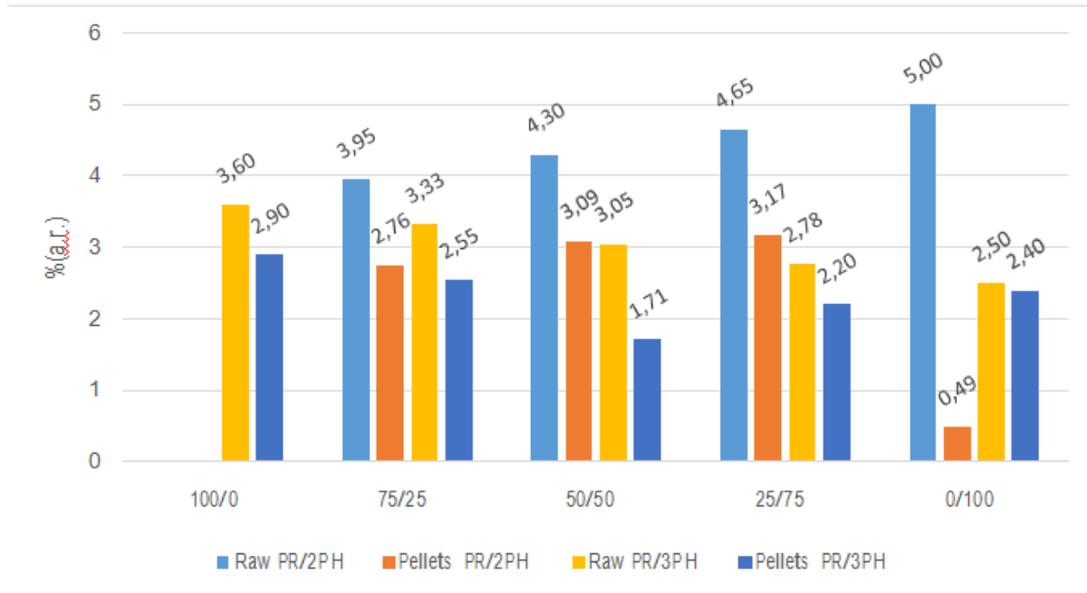


Figure 4 – Ash content of both pellets and raw materials

The main ash forming elements in fruit waste material are calcium, magnesium, sodium and potassium. It is worth noting that the influence of such elements on the ash melting behaviour is directly related to the reliability of the plant.

Moreover, it is notable that especially for pruning residues, phosphorus is relevant since herbaceous materials are rich in this element. Phosphorus is semi-volatile and may also cause ash melting problems by the formation of phosphates. [9]

2.3.4 Bulk density of pellets

Bulk density is an indicator of pellets compaction. It is calculated as the dry weight of pellets divided by its volume. This volume includes the volume of pellets and the volume of pores among pellets. Bulk density is expressed in g/cm³. Since it can change depending on how the material is handled, it is indicated as loose bulk density and tapped one.

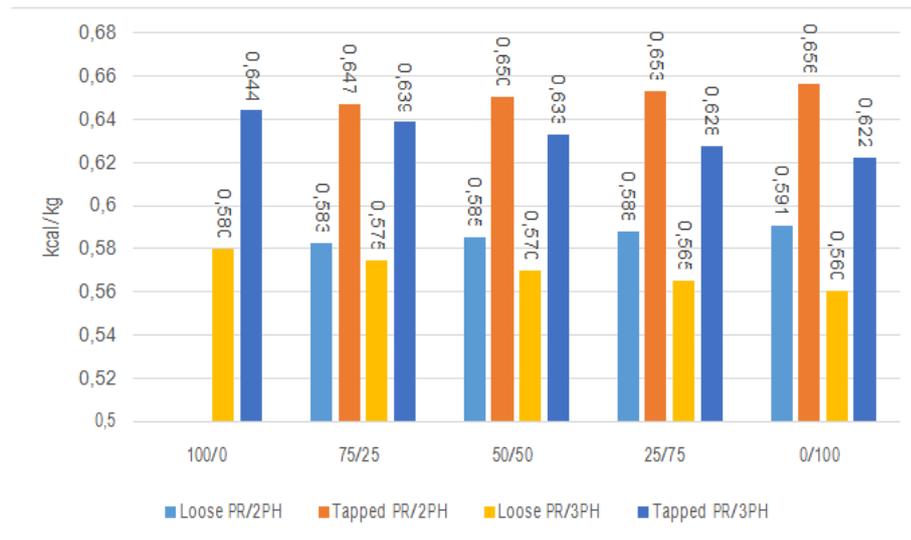


Figure 5 – Bulk density of pellets

2.3.5 Lignin content

The lignin content is determined by the hydrolysis of wood carbohydrates obtaining an insoluble matter.

Since oils, resins, fats, and starch would remain insoluble with the lignin, these are first removed by proper solvents. The 72 % sulphuric acid method for lignin contains two and sometimes three preliminary extractive treatments, namely:

- I. with alcohol, to remove the catechol tannins;
- II. with alcohol-benzene solution, to remove the resins, oils, fats and waxes
- III. with hot water, to remove the water-soluble materials

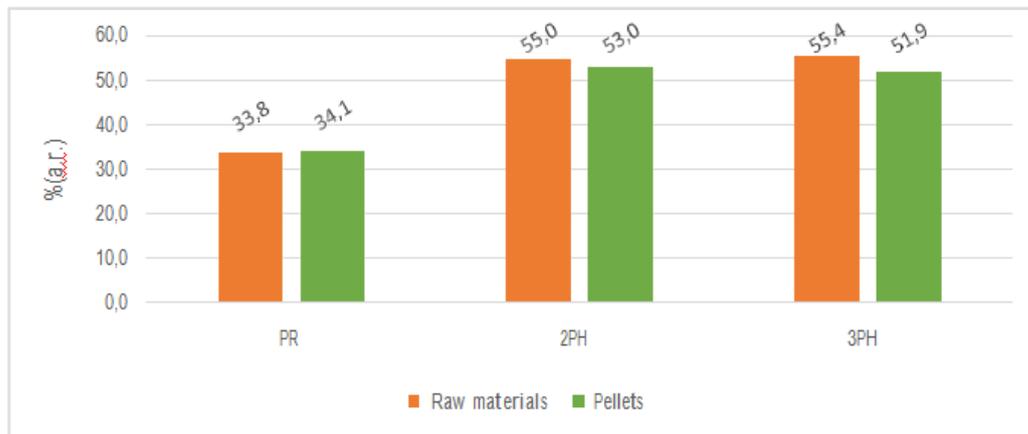


Figure 6 – Lignin content

2.3.6 Sulphur content

The sulphur content test methods cover two alternative procedures for the determination of total sulphur in prepared analysis samples of solid forms of refuse-derived fuel (RDF).

Usually, RDF products are not homogeneous, for this reason it is quite important to consider representative laboratory samples from the RDF to be characterized.

To preserve the samples' representative characteristics the procedure must be carefully performed, and, they must be air-dried and particle size reduced to pass a 0.5-mm screen as described in ASTM Practice E 829.

Sulphur is determined in the washings from the oxygen-bomb calorimeter following calorimetric determination in accordance with Test Method E 711. The type of bomb, amount of water in the bomb, oxygen pressure, and amount of sample taken shall be the same as specified under the calorimetric determination. The bomb shall stand in the calorimeter water for not less than 5 min after firing.

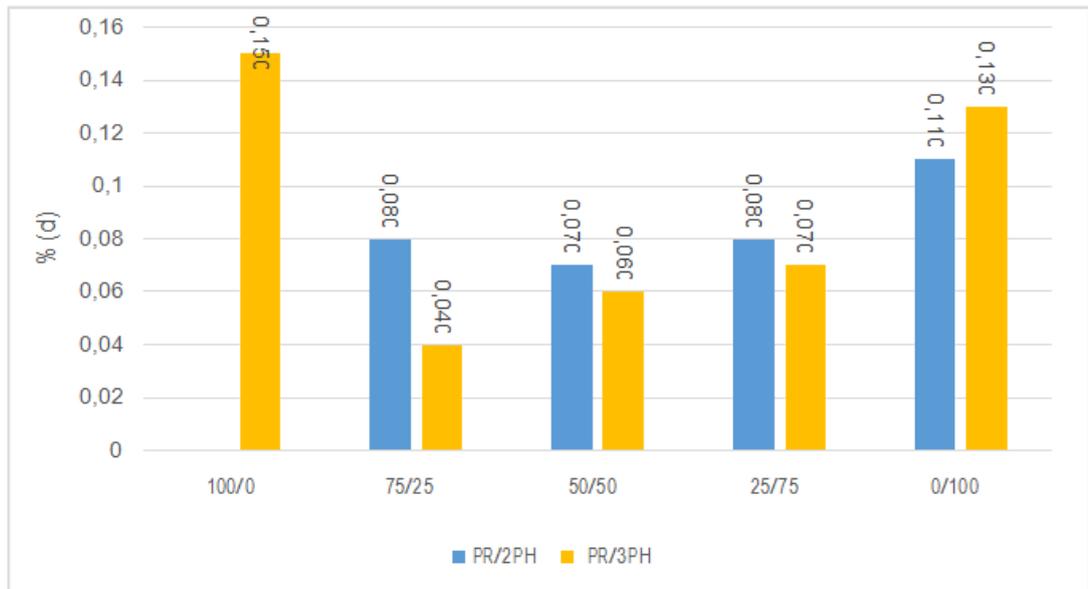


Figure 7 – Sulphur content of pellets

2.4 Technology scouting

The following figure shows the process to be scaled-up.

Once the process has been identified, a preliminary technology scouting was carried out. Since the raw materials to be used for the process are very different, two different drying processes are proposed.

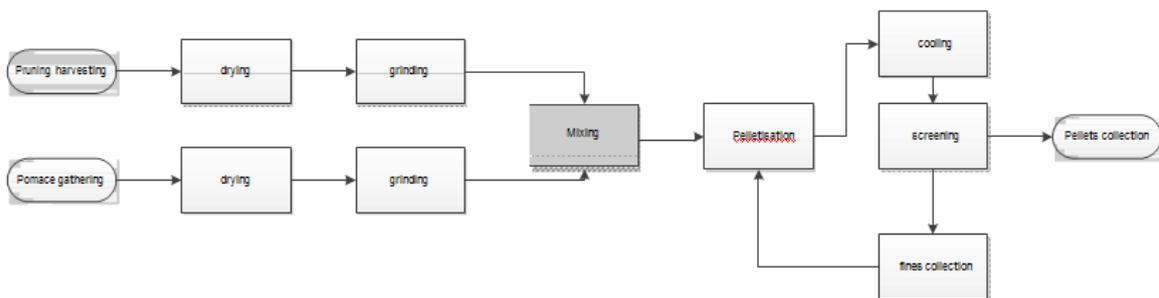


Figure 8 process to be scaled up

The scale up design of the process will be carried out within subsequent activities.

2.4.1 Physical pretreatment of pruning residues

The aim of our project, accordingly to the rapid development of bioenergy is to provide potential outlet for the olive industry waste. To date, management of pruning residue generally represents a disposal problem; our purpose is to highlight an opportunity for additional revenue. Therefore, our interest will be in developing cost-effective solution for the collection, processing and delivery of pruning residue.

Having a look to the market, it is possible to select machines generally derived from conventional mulchers, equipped with a storage bin or with a blower, the latter designed to direct the flow of comminuted residue to an accompanying trailer [10]. Despite such implements being relatively cheap, generally, they cannot achieve industrial performance, and their productivity is commonly in the range of one tonne per hour [11]. Such a low productivity level may compromise the economic sustainability of the operation, unless the work is conducted with surplus resources obtained at marginal cost.

An alternative solution that can be considered is to harvest the residues once they are air-dried up to their moisture stability.



Figure 9 - Tractor-mounted machine while harvesting olive pruning residues. The high-tipping bin is not installed here and biomass is blown onto a towed trailer (Source:[11])

Once the material has been dried it is necessary to reduce its size by means of industrial granulators, these machines offer a good reliability and low energy consumption. Some tests conducted on a semi-industrial machine demonstrated very interesting results in terms of mechanical properties of pellets.



Figure 10 - Granulator–Size reduction solution used to perform preliminary tests

2.4.2 Physical pretreatment of pomace residues

There are some general guidelines for selecting a drying system, but it should be recognised that the rules are far from rigid and exceptions not uncommon.

In order to obtain a stable pomace residue, for the lab scale application it was proposed that a natural drying process be performed. Despite the air-drying being more convenient from an economical point of view, it is not compatible with the timing related to the other operations involved in the process. Thus, due to the high water content of pomace residues industrial application is performed.

As a rule of thumb, the dryer must operate reliably, safely, and economically.

Drum drying is a method used for drying out liquids from a wet matrix. The raw material is loaded into a heated drum. Once dried, pomace solids are ejected by airflow.

A drum dryer is a mechanical system, which is widely used for various sectors in industry. It has a cylindrical shape.



Figure 11 E Example of rotary dryer to be used in the drying process.

Generally, designing of a single drum is simple, but problematic in practice, even if they are widely used in industry.

The performance of the drum depends on their application, higher capacity; improved product quality and lower power consumption are obtained with a suitable location, adjustment and maintenance.

As well as for the pruning residues the dried pomace has to be reduced by mean of another industrial granulator to obtain the right size reduction.

2.4.3 Blend preparation

When the raw materials to be blended are reduced in size, it is necessary to mix them accordingly to the selected receipt. It is advisable to use bin tumbler, since they are designed for blending and homogenising powders and granulates. The machine rotates containers and is able to generate an efficient mixing effect thanks to the container being tilted by 15° as compared to the rotation axis.

This kind of machines is characterized by:

- High productivity: the containers are hermetically closed so that different bins containing different products can be mixed in sequence on the same machine without the need for cleaning or decontaminating the tumbler.
- In case of "difficult" product blending, the rotation axis of the Trigon System can be inclined also referring to a vertical plane to create a double inclination rotating system.

Moreover, it is possibility to arrange the production layout to save space in the processing area installing the machine "through the wall".

3. Conclusions

The activities show a good progress. In particular, the laboratory scale protocol has been developed and used to produce the first 100 kg of test material.

In concrete, the following activities have been accomplished:

- Literature review of olive farming waste
- Literature review of olive oil waste
- Pellet mill setup
- Technology scouting
- Nine different batches of pellets produced (100 Kg in total)
- Pellets analysis performed
- Shipment of 100 kg of pellets to Fraunhofer
- Lab-protocol definition
- KPI (Key Performance Indicator) Individuation
- Optimisation of the pellet production process
- Production and shipment of further amounts of 100 Kg of pellets to Fraunhofer

The pellets analysis performed included: moisture content of raw materials, net calorific values, and ash content, and further analysis required by WP3, including sulphur content and lignin content.

Concerning the KPIs, it was confirmed that:

- Particle size is one of the key factor involved in the process, since the blend should be as homogenous as possible;
- The temperature of the die affects the activation of most of the bonding mechanisms involved in chemical and physical transformations. An optimal die temperature in the range of 80-90°C has been identified by the experiment;
- The moisture content has to be controlled in order to promote the transport phenomena responsible of the binding process. Pelletisation has been achieved at total moisture content higher than ca.17 wt.%;

Since some experiments evidenced that the raw material behaves differently according to the scale of the process, some pilot scale essays will be carried out in order to validate the lab protocol.

4. References

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