**ORIGINAL PAPER**



# **Energy Recovery from Olive Pomace by Hydrothermal Carbonization on Hypothetical Industrial Scale: a LCA Perspective**

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# **Abstract**

This work aimed to analyze and compare the environmental impacts of hydrothermal carbonization (HTC) process performed on olive pomace, aimed at energy recovery. Twelve alternative pathways, considering variable process conditions in terms of temperature (260, 280 and 305 °C), heat recovery layouts and the subsequent combustion of the generated solid product, were analyzed. Results, related only to the processing phases (i.e. without including avoided efects), showed, as expected, that the fnal environmental performance depends on the HTC process temperature. Moreover, it was noted that the resulting impacts are from 1.4 to 2.0 lower for the layouts with heat recovery, for all the analyzed impact categories, with exception of Freshwater Toxicity. It was also reported that by substituting the fuel in marginal processes of heat production, environmental load reduction potential, regarding all impact categories, can be achieved. The highest benefts were achieved by substituting coal with HTC pellet in the heat production, followed by wood pellets and natural gas. Concerning the contribution analysis, the combustion processes of HTC and wood pellet represent the major contributions to the Climate Change and Acidifcation impacts, the combustion processes of HTC and wood pellet represent the major contributions, around the 95% and the 75% of the total, respectively, for the cases with the highest HTC temperature and no recovery scheme. For the Eutrophication potential, biomass production contributes signifcantly to the fnal efect. Regarding the Freshwater Ecotoxicity, the major part of the impact is coming from the HTC process and the relative liquid phase emissions assumed to be composed by phenols and furfurals released from the process. However, this part of the system needs to be further analyzed in detail.

#### **Graphic Abstract**



**Keywords** Biomass upgrading · Energy balance · Life cycle assessment · Olive mill waste · Waste valorization

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#### **Statement of Novelty**

The novelty of this work lays in the effort made to preliminary design the HTC process, applied to olive pomace, on industrial scale. The aim is to estimate, rather accurately, the energy consumptions and the fuel production, in order to carry out the energy balance and, more in general, to defne the inputs/outputs for the evaluation of the process from the environmental point of view, in the Life Cycle Assessment (LCA) perspective. Diferent alternative pathways are compared by LCA with the aim of providing useful information for the efective choices of design and realization of the process on industrial scale.

# **Introduction**

Olive oil is one of the strategic agriculture products in Mediterranean countries. The European Union (EU) is the leading producer of the olive oil. According to data reported by FAOSTAT [\[1](#page-14-0)] around 10.8 Mha of olive crop were cultivated in 2017 worldwide. Total olive fruit production in the EU accounted for 12.9 Mt, 61.3% of total worldwide yield. The major producer being Spain with 6.54 Mt, followed by Greece with 2.72 Mt and Italy with 2.57 Mt. As far as oil production is considered, in 2017, 2.2 Mt/year of oil was produced in the EU, accounted for around 70% of total worldwide production [\[2](#page-14-1)]. Italy with the annual olive oil production of 430 kt/year in 2017 represented the second largest producer in the EU after Spain [[3\]](#page-14-2). Italian olive production covers approximately 1.7 Mha, 80% of which are located in the south of Italy, mainly in Puglia, Calabria and Sicily [\[4](#page-14-3)].

During olive oil production several by-products are generated. They can be categorized as low/medium-moisture (olive tree biomass residues, olive stones, and pomaces) and high-moisture residues (wastewaters) [\[5](#page-14-4)]. As reported by many authors  $[6-9]$  $[6-9]$  $[6-9]$  $[6-9]$  amount of residues coming from olive oil production can vary signifcantly depending on the process of production. As an example, the 3-phase process uses of large amounts of water to facilitate the extraction and separation of oil, while in the 2-phase or traditional process, only small amount of washing water is added. As a consequence, the 2-phase process produces more solid residues with higher moisture and oil contents, but relatively small volumes of wastewater. It is reported that for every 1000 kg of olive input, traditional, 2-phase and 3-phase production processes generate around 500, 735–800, 550–580 kg of solid wastes and 0.65, 0.2,  $1.0-1.6$  m<sup>3</sup> of wastewater respectively.

During the peak period of the olives processing, the olive oil mills, due to the severe operating conditions, have no way to operate modern and efficient disposal and recycling and thus they often discharge liquid and solid waste on the soil.

Olive pomace (OP) difers in composition depending on the production process (two or three-phase). Olive pomace represents the main residue after both types of separation systems and it is constituted by crushed olive stones, process water and all material coming from the olive fruits except the olive oil [[5\]](#page-14-4). The indicative chemical composition of the olive pomace generated from diferent methods is shown in Table [1](#page-1-0), based on data reported in the literature.

OP can be processed to extract the oil and obtain the so-called "pomace oil" and a fnal solid residue is generated called "extracted dry pomace residue," which can be integrated for energy production, as it contains a signifcant contribution of carbon, or other applications. Solid olive mill wastes can be converted into useful forms of energy through diferent energy utilization pathways [[6](#page-14-5)]. The conversion to energy can be performed through two main technological pathways, namely thermochemical (pyrolysis, gasifcation and co-combustion) and biochemical/biological (anaerobic digestion and fermentation). However, before being efectively used as a combustion fuel, it has to be pre-treated employing drying and oil extracting treatments [\[10](#page-14-7)]. As an alternative, OP can be upgraded into enhanced fuel, called hydrochar, by using hydrothermal carbonization process (HTC). HTC is combined dehydration and decarboxylation of raw biomass to raise its carbon content with the aim of achieving products with a higher calorifc value. The HTC process consists in heating the end-products of the oil industry (e.g., olive pomace and mill wastewater) in a reactor where high temperatures (typically in the range 150–300 °C) and pressure are kept for several hours. The pressure in the reactor has to be high enough to avoid water vaporization and therefore drastically reducing the energy requested by the process, especially if compared with drying. With this method, the solid and organic fractions of the end-products are transformed in a lignite-like, easy to handle fuel with

<span id="page-1-0"></span>**Table 1** Characteristics of the olive pomace derived from diferent oil production processes [[8\]](#page-14-8)

| Parameters              | Press process    | 2-phase process   | 3-phase process   |
|-------------------------|------------------|-------------------|-------------------|
| Moisture $(\%)$         | $27.2 \pm 1.048$ | $56.80 \pm 2.188$ | $50.23 \pm 1.935$ |
| Fats and oils (%)       | $8.72 \pm 3.254$ | $4.65 \pm 1.736$  | $3.89 \pm 1.449$  |
| Proteins (%)            | $4.77 \pm 0.024$ | $2.87 \pm 0.014$  | $3.43 \pm 0.017$  |
| Total sugars $(\%)$     | $1.38 \pm 0.016$ | $0.83 \pm 0.010$  | $0.99 \pm 0.012$  |
| Cellulose $(\%)$        | $24.1 \pm 0.283$ | $14.54 \pm 0.170$ | $17.37 \pm 0.203$ |
| Hemicellulose<br>$(\%)$ | $11.0 \pm 0.608$ | $6.63 \pm 0.366$  | $7.92 \pm 0.438$  |
| Lignin $(\%)$           | $14.1 \pm 0.291$ | $8.54 \pm 0.175$  | $10.21 \pm 0.209$ |
| Total carbon $(\%)$     | $42.9 + 3.424$   | $25.37 + 2.025$   | $29.03 + 2.317$   |
|                         |                  |                   |                   |

well-defned properties, whereas the water contained in the initial charge is sterilized and it is characterized by a near to zero residual chemical oxygen demand (COD).

Existing literature studies on HTC of olive mill wastes focus on the laboratory scale experimental research. As an example, Volpe and Fiori [[11\]](#page-14-9) performed the HTC of two types of agro-industrial wastes: olive tree trimmings and olive pulp under diferent process conditions regarding temperature, and biomass to water ratio. The heating value of olive pomace, reported by [\[11\]](#page-14-9) was equal to 21.7 MJ/ kg. The HHV values of produced hydrochar were enhanced with the increase of process temperature and increasing of biomass to water ratio. In another study, Missaoui et al. [[12\]](#page-15-0) performed hydrothermal carbonization of dried olive pomace, obtained from 3-phase centrifugal extraction process, under varying temperature conditions (180–250 °C), varying residence time (0–120 min) and varying biomass to water ratios (1:2–1:6). Similarly, to the work presented by Volpe and Fiori [\[9](#page-14-6)], they evaluated the solid mass yields and quality of the hydrochars depending on the process conditions obtaining the same conclusions. These studies are aimed to prove and optimize the olive wastes HTC process at the laboratory scale.

As no real industrial scale HTC system is in operation processing OP (presently only an industrial HTC plant is operating in Valencia, with a design capacity of 14,000 t/y processing garden waste and organic fraction of municipal solid waste) [\[13](#page-15-1)], there exists a considerable gap in the evaluation of HTC systems. Results in literature are based on many simplifcations and scale-up of experimental results. The results synthesis is of great importance in the development of the olive pomace HTC process from the laboratory to the pilot and industrial scale. There are no commercial HTC plants in operation and the best available data for the HTC reaction is currently based on laboratory-scale batch experiments. Available works confirm high energy efficiency and scalability of the HTC process [[14,](#page-15-2) [15](#page-15-3)]. Stemann et al. [\[14\]](#page-15-2) proposed a process fowchart of HTC upscaling the 200 ml HTC vessel into a hypothetical plant converting annually 40,000 tons (5 714 kg/h) of empty palm fruit bunches into hydrochar on industrial scale. Lucian and Fiori [\[15](#page-15-3)] attempted to design and model an industrial scale HTC of two raw moist biomass: of—specifcation compost and grape marc, basing on the experimental data for equivalent laboratory process. They considered HTC process capable to convert 20,000 tons per year (2500 kg/h) of organic waste, finding that plant efficiency was strongly dependent on initial feedstock moisture content.

To consider environmental aspects, Life Cycle Assessment (LCA) can be applied to compare HTC systems with other alternative treatments.

Numerous studies suggest the HTC as an environmentally advantageous process for transformation of diferent waste

and biomass flows, including sewage sludge  $[16]$  $[16]$  $[16]$ , wet biomass [\[17](#page-15-5)], food wastes [\[18](#page-15-6)], or algae [[19\]](#page-15-7) into added-value product. These studies also indicate the potential environmental benefts of HTC, when compared to more traditional energy valorization pathways. Regarding olive mill waste energy valorization pathways, LCA has been applied in the cases of a pyrolysis system [\[20](#page-15-8)], energy recovery plant [\[21](#page-15-9)], torrefaction [[22](#page-15-10)]. Studies on HTC of olive mill waste are limited.

Study by Benavente et al. [[23\]](#page-15-11) used LCA to compare environmental impacts of two-phase olive mill waste HTC with conventional energy valorization processes, such as aerobic composting, anaerobic digestion, and incineration. In contrast to other studies on HTC, their results indicated that generally agricultural valorization of olive mill wastes is more favorable pathway, regarding the global warming potential, and, among the energy valorization concepts, the HTC is not as environmentally advantageous as drying and incineration with energy recovery. However, it should be noticed that in their study simplifed HTC process layout, without any heat recovery scheme, was considered. The HTC process layout of their study was composed of the main HTC reactor, rotary dryer for drying the obtained hydrochars and a combustor combusting a combination of hydrochars and auxiliary materials. When considering the use of HTC to treat OP on industrial scale it should be determined whether the integration of HTC with and without heat recovery is more advantageous when compared to a direct utilization of OP.

The present study contributes to existing literature by extending knowledge on environmental impact of HTC process applied to olive pomace. In our study, while taking into account the processes layout of HTC on industrial scale with and without heat recovery scheme, 12 diferent HTC valorization concepts of olive pomace are analyzed in detail, based on empirical data. The aim is to identify the most promising concept and layout of HTC from a LCA perspective. Firstly, laboratory scale experiments were performed to determine mass yields and elemental analyses. The results were used to upscale and model the environmental performance of hypothetical industrial HTC plant. The specifc objectives of this work are to: (i) evaluate the environmental impact associated with the diferent OP HTC process conditions, mainly temperature (260, 280 and 305 °C), and the subsequent combustion of the generated solid product; (ii) evaluate environmental impacts of OP HTC considering diferent scenarios of energy recovery, including heat recovery and self-sufficiency by integrating boilers fired by produced HTC hydrochar; (iii) understand how key parameters associated with each treatment approach (e.g. process temperature, heat recovery, self-sufficiency of the process) influence system environmental impacts; (iv) provide fnal recommendations for process selection from an environmental perspective.

# **Materials and Methods**

In this work, the LCA approach is applied using general methodological framework and standards for LCA defned by ISO 14040 and ISO 14044 series [[24](#page-15-12), [25](#page-15-13)] and by the International Reference Life Cycle Data (ILCD) System [[26\]](#page-15-14). The methodology is composed of four phases: Definition of the goal and scope of the system, Life Cycle Inventory, Life Cycle Impact Assessment, and Life Cycle Interpretation (results section). Life Cycle Inventory phase is based on demanding experimental and modelling works.

# **Goal and Scope Defnition**

This work aims to analyze and evaluate, from the environmental point of view, the treatment of OP by HTC, comparing possible alternatives of supply the required thermal energy to the HTC process, with the aim of making the proposed system the most possible self-sufficient from an energy point of view. In particular the following possibilities of energy supply are investigated: combustion of the solid product from the HTC process or combustion of other biomass available in the olive mill nearby. The study is conducted with reference to hypothetical industrial scale layout. The analysis includes variable process conditions in terms of temperature (260, 280 and 305 °C).

Additionally, two possible process layouts were considered: with and without internal heat recovery.

The analysis includes system exchanges with energy systems as well as waste and wastewater systems. Consequences on the background systems are included in the analysis by implementing the system expansion, by including the efects caused by substitution of products in marginal processes by the process products and co-products in the analyzed solutions [[27\]](#page-15-15).

The mass and energy balances for all the scenarios are modelled assuming the same quantity and quality of input OP. The function of the HTC plant is to treat OP, producing as fnal output the upgraded fuel. The Functional Unit (FU) and reference fow for all the alternative are defned as treatment and upgrade of 1 kg of OP in the HTC plant. All technological steps required to convert raw OP into pelletized hydrochar are accounted for.

Foreground and background systems are included in the system boundary. Foreground system refers to operation of the HTC plant fulflling the FU. Background system considers all the processes of production of entering flows and dealing with the exiting fows. Contributions of the construction phase are excluded from this study.

#### **HTC Plant Process Layout**

Concept of HTC industrial plant layout is developed and modelled considering setup used in the experiment and



Emission to air, solid wastes

<span id="page-3-0"></span>**Fig. 1** HTC industrial process layout

design schemes suggested in the literature [[14](#page-15-2), [15\]](#page-15-3). Figure [1](#page-3-0) shows a schematic diagram of the HTC process under consideration.

Firstly, OP is transported from olive mill to HTC plant and then stored (ST). Feedstock is therefore processed in grinder (G) and mixed (M) with water in order to obtain homogenous mixture stream (MIX) with desired biomass to water ratio. Mixture is transported by pump (P) to the top of HTC reactor (HTC\_R). The designed treatment capacity of HTC plant is assumed to be 2500 kg/h. The energy requirements for the HTC\_R were estimated for three different temperature levels: 260, 280 and 305 °C, with residence time of 3 h. Burner (B1) heats up reactor diathermic oil jacket, transferring the heat required to raise the temperature of the mixture inside the reactor, from initial temperature to the set operating one.

Gaseous products of HTC (stream HTC\_G) are released from the reactor by valve (V) and are estimated according to laboratory scale results. Wet HTC slurry (stream HTC\_S1) containing liquid and solid products of HTC is extracted from the bottom of the reactor and then cooled down and depressurized. Liquid–solid separation is performed in two steps. First step includes mechanical dewatering by decanting (DEC), fltration and centrifugation (F). Wet hydrochar stream (HTC\_S2) enters the convective air dryer (D), fed by air heated up by the second burner (B2), pushed by air blower (BL). Part of the remained liquid stream (HTC\_L) is recirculated to the mixer, in order to prepare mixture stream (MIX), while the remaining part should be sent for wastewater treatment. Actually, the wastewater treatment is not included within the system boundary and wastewaters with the simplifed composition of phenols and furfurals are assumed to be discharged into the environment. This assumption is mainly linked to lack of detailed information about the composition and, consequently, on the required appropriate treatment. This limitation of the study will be highlighted in the results. Additional water makeup is considered, when recirculated water stream in not sufficient to obtain the desired dry OP to water ratio. The dry hydrochar stream, exiting the decanter (stream HTC\_S3), is directed to the pelletizer.

The basic described layout can be improved, by integrating the heat recovery from some streams. In this case, the entering slurry (MIX) is previously preheated in the recovery heat exchanger HX1. The sensible heat transferred to the slurry is provided by using steam recovered during cooling and depressurizing of HTC exiting slurry (HTC\_S1) in the fash tank (FT). Secondly, vapor leaving HX1 enters HX2 in order to preheat fresh air before it enters the convective air dryer. In this case, burner B1 and B2 provide the remaining heat required to reach, respectively, operational HTC temperature and air temperature. Burners B1 and B2 are fred by biomass pellets (wood pellet or hydrochar pellet).

As far as heat supply to the process is considered, two scenarios are taken into account. First scenario assumes external biomass fuel supply and as consequence, all the generated hydrochar is used to replace the fuel in the conventional heat production system. In this case, biomass fuel is assumed to be wood pellets (WP—purple stream in Fig. [1](#page-3-0)). In the second scenario, heat is generated using pellets derived from internally produced hydrochar, so only the excess hydrochar pellets are directed to substitute marginal heat production outside the system boundaries (dotted green line in Fig. [1](#page-3-0)).

Generally, as hydrochar is a coal-like material, it is likely that generated hydrochar will be transported and ultimately combusted in coal-fred power plants, ultimately substituting coal-derived heat and avoiding the relative impacts (reference marginal production system, depicted in the "avoided heat production" box in Fig. [1](#page-3-0)).

Alternatively, the avoided heat production was also evaluated considering the substitution of other heat generation systems: heat natural gas boilers as well as wood pellet stove [[28,](#page-15-16) [29\]](#page-15-17). Product inventories are compared and the diferences of impacts are calculated at the level of a functional unit. The diference may be either negative or positive. Positive diferences are commonly referred to as avoided emissions.

#### **Life Cycle Inventory Analysis**

The inventory analysis includes the required energy and materials (inputs) flows as well as products, co-products, emissions and wastes (outputs) exchanged with the environment by the system boundary. Inventory analysis was developed using both primary and secondary data. Primary data for the main processes in the foreground system, i.e. HTC yields as well as products and substrates compositions, were upscaled on the basis of the HTC experimental trials performed by the authors on olive pomace [[30](#page-15-18)]. The upscaled HTC process layout was depicted and energy consumption was estimated for each device, as detailed in the following. The secondary data related to processes of the background system, namely transport, electricity and heat generation, raw material production as well as waste treatments, are retrieved from the European Reference Life Cycle Database (ELCD) and the Ecoinvent database version 3.2 with the cut-off system model. For consistency, average EU mixes regarding electricity, materials and other resources were considered. The OP as an input element is treated as a waste. Thus it is assumed as a zero burden input of the system [[31,](#page-15-19) [32](#page-15-20)].

#### **Laboratory Scale Primary Data**

Primary data were obtained from laboratory scale discontinuous process, performed in a batch reactor. The batch reactor with a volume of 5.0 l, designed for maximum pressure of 100 bars and maximum temperature of 310 °C made from AISI Type 304 Stainless Steel, was designed and constructed to investigate the HTC process. During HTC experiments OP was kept in subcritical water at 260, 280 and 305 °C and autogenous saturated vapor pressures. Dry OP to water ratio was maintained at 1:6.

Feedstock and hydrochar compositions were evaluated by means of total solid (TS), and ultimate analysis providing carbon (C), hydrogen (H) and nitrogen (N) content, as weight percentage on dry basis. The ash content was assumed to be 3% in the raw material [[30](#page-15-18)]. Elemental analysis of both raw and hydrothermally carbonized OP was performed according to the procedure set in the standard UNI EN 15104:2011. Higher heating value (HHV) of both feedstock and product was determined, according to the procedure from ASTM D 5865-13. Table [2](#page-5-0) reports the elemental analysis and the HHV on a dry basis of OP and produced hydrochars, after 180 min at diferent temperatures.

Gaseous phase composition was measured using gaschromatographic analysis [\[33](#page-15-21), [34\]](#page-15-22). Liquid phase composition was not measured in detail. Total organic carbon (TOC) analysis was performed on the liquid residue allowing the determination of the amount of carbon dissolved within this phase after each HTC batch experiment. Due to the lack of detailed liquid analysis, it was assumed that liquid phase is composed of phenol  $(C_6H_6O)$  and 5-(hydroxymethyl)furfural (5-HMF,  $C_6H_6O_3$ , addressed as furfurals in the following) as a representative organic species coming from cellulose, hemicellulose and lignin degradation and water [[15,](#page-15-3) [35](#page-15-23)]. Therefore, starting from the measured TOC of the liquid phase, the moles of C have been divided proportionally between phenol (50%) and furfurals (50%) [\[36](#page-15-24)].

<span id="page-5-0"></span>**Table 2** Characteristics of initial OP and hydrochar produced at different temperatures, with the residence time of 180 min [\[30\]](#page-15-18)

| Test case                 | <b>OP</b> | 1     | 2     | 3     |
|---------------------------|-----------|-------|-------|-------|
| Temperature $(^{\circ}C)$ |           | 260   | 280   | 305   |
| Pressure (bar)            |           | 54    | 86    | 98    |
| Residence time (min)      |           | 180   | 180   | 180   |
| Dry OP: water             |           | 1:6   | 1:6   | 1:6   |
| Dry OP(g)                 |           | 500   | 500   | 500   |
| C (mass fraction)         | 52.47     | 71.4  | 77.8  | 77.20 |
| N (mass fraction)         | 1.53      | 1.71  | 1.62  | 1.81  |
| H (mass fraction)         | 6.25      | 5.87  | 7.84  | 6.42  |
| O (mass fraction)         | 36.75     | 18.02 | 9.74  | 11.57 |
| $HHV$ (MJ/ $kg$ )         | 21.83     | 27.82 | 29.13 | 30.27 |

Kinetic models, as well as mass-energy balances, were created using a lumped model for the HTC process of biomass, as proposed by Knežević et al. [\[37](#page-15-25)]. The overall mass balance for the batch reactor can be written as follows (1):

$$
M_{input} = M_{output} \tag{1}
$$

where  $M_{input} = M_{Rdb} + M_{L in} + M_{air}$  and  $M_{output} = M_{HCdb} +$  $M_{L \text{ HTC}} + M_{gas}$  where  $M_{HCdb}$  represents the mass (on a dry basis) of the solid (hydrochar) after thermal treatment,  $M_{\text{Rdb}}$ represents the mass (on a dry basis) of the raw sample before thermal treatment,  $M_{air}$  is the mass of the air in the reactor,  $M_{gas} = M_{CO_2} + M_{CO} + M_{CH_4} + M_{H_2}$  is the total mass of the gas produced,  $M_{L HTC}$  and  $M_{L in}$  are the total mass of the liquid residue recovered after HTC treatment and the total mass of water charged into the reactor before each experiment, respectively. Amount of separated phenols and furfurals in liquid phase was estimated considering the carbon balance. The total amount of water, at the end of the HTC batch process, results from water added to the feedstock and water produced during the HTC reaction. Water produced during the HTC was determined as the diference between total amount of liquid and amount of phenols and furfurals produced. Solid hydrochar mass yield (SY), liquid mass yield (LY) and gas mass yield (GY) can be then calculated according to Eqs.  $(2)$  $(2)$ – $(4)$  $(4)$ :

<span id="page-5-1"></span>
$$
SY = \frac{M_{HCdb}}{M_{Rdb}}\tag{2}
$$

$$
GY = \frac{M_{gas}}{M_{Rdb}}\tag{3}
$$

<span id="page-5-2"></span>**Table 3** Mass yields, gas compositions and TOC [\[30\]](#page-15-18)

| Test case                        | 1     | $\mathfrak{2}$ | 3     |
|----------------------------------|-------|----------------|-------|
| SY                               | 0.422 | 0.455          | 0.321 |
| LY                               | 0.507 | 0.470          | 0.588 |
| $TOC$ (mg/L)                     | 3.91  | 2.23           | 5.77  |
| GY                               | 0.071 | 0.075          | 0.091 |
| $CO2$ (volumetric frac-<br>tion) | 58.10 | 37.60          | 69.90 |
| $H2$ (volumetric frac-<br>tion)  | 0.38  | 0.15           | 0.89  |
| CO (volumetric frac-<br>tion)    | 3.50  | 3.10           | 1.30  |
| $CH4$ (volumetric frac-<br>tion) | 4.30  | 6.30           | 7.20  |
| $O2$ (volumetric frac-<br>tion)  | 33.72 | 52.85          | 20.71 |

$$
LY = \frac{M_{LHTC} - M_{Lin}}{M_{Rdb}}\tag{4}
$$

Table [3](#page-5-2) reports the mass yields, gas compositions and TOC values obtained during HTC batch experiments, after 180 min at diferent temperatures.

On the basis of the experimental results, a general kinetic model, assuming all the occurring reactions to be the frst order and described by the Arrhenius equation, was created. Obtained kinetic parameters were further applied in the solid, liquid and gas mass conversion model. The example mass conversion for the considered process temperatures is presented in Fig. [2.](#page-6-1)

### **Upscaled HTC Process Data**

For determining energy requirements of HTC process, a model connecting input and output variables (Table [4\)](#page-6-2) is required. Previously developed laboratory scale energy and mass balances are implemented to the hypothetical industrial scale. The fundamental approach is used to simulate the industrial HTC process by changing the input parameters.

The scale-up of substrates and products of the process is performed linearly using kinetic model. The energy consumption instead depends on many factors like the size of reactor or construction, insulation materials used as well as reaction parameters (time, temperature). Investigating <span id="page-6-0"></span>shows that consumed heating energy does not behave linearly, meaning that the energy consumption per unit of mass produced decreases with growing scale [\[38](#page-15-26)]. Additionally, the following assumptions regarding energy consumptions are used in the simulation:

- Grinder power consumption is estimated assuming that particle has mean particle size of 10 mm and shall be reduced to a mean size of 0.8 mm in order to allow slurry be easily pumped [\[15\]](#page-15-3).
- Slurry pump power consumption is calculated assuming centrifugal pump efficiency of  $45\%$  [[14](#page-15-2)].
- Model estimating energy consumption during stirring proposed by [\[39](#page-15-27)] and used in the scale-up the procedure of chemical bath reactor by [\[38](#page-15-26)] is applied.
- Temperature of slurry exiting HX1 in the heat recovery scenario is set equal 155 °C.
- Thermal balance of reactor accounts for sensible heat for increasing the temperature of the slurry from the initial value (25  $\degree$ C without heat recovery, 155  $\degree$ C with heat recovery) to the target temperature, supplied by the oil circulating in the reactor jackets (temperature diference between oil and HTC process temperature is 10 K).
- The reactor energy losses are considered in the duration of the residence time.
- The heat of reaction is not considered in the analysis.



<span id="page-6-1"></span>**Fig. 2** Predicted mass conversion (total mass),  $T = 260$ , 280 and 305 °C [\[30\]](#page-15-18)

<span id="page-6-2"></span>

- Biomass-pellets burning furnaces (B1 and B2) provide energy by combustion of wood pellets or hydrochar pellet; softwood (HHV =  $20.08$  MJ/kg) is assumed as reference biomass fuel, boiler excess air fraction is 40%, the exhaust gas temperature is  $130\text{ °C}$  and burner efficiency is 80% [[40\]](#page-15-28).
- Flash tank (FT) is adiabatic and operates on thermodynamic equilibrium.
- Specifc power consumption of fltration and centrifugation is assumed to be equal to 10 kWh/ton of hydrochar [[38\]](#page-15-26).
- Moisture content of dewatered slurry stream exiting decanter (HTC\_S2) is assumed to be equal to 40%.
- Moisture content of dried slurry stream exiting air dryer (HTC\_S3) is assumed to be equal to 10%.
- Temperature of air entering air dryer was set equal to 200 °C.
- Specifc power consumption of pelletizer is assumed equal to 55 kWh/ton of hydrochar [\[15](#page-15-3)].
- The energy consumption of any wastewater treatment is not taken into account.

It is assumed the transportation distance between the olive mill and the HTC plant is equal to 50 km. The same transportation distance was assumed for biomass fuel delivery. The overall transportation inventory is accounted by using the appropriate dataset of Ecoinvent database (Transport, freight, lorry > 32 metric ton, EURO6 {GLO}| market for | Conseq, S).

Because of a lack of data associated with emissions from hydrochar combustion, hydrochar combustion is assumed to

<span id="page-7-0"></span>**Table 5** Inventory of 1 MJ of heat produced by combustion of wood and bio-waste pellets

|                             |       | Wood [29] | Hydrochar [17]    |
|-----------------------------|-------|-----------|-------------------|
| Input from technosphere     | kg    | 0.062     | $0.041 - 0.045$   |
| Pellet                      |       |           |                   |
| <b>HHV</b>                  | MJ/kg | 20.08     | 27.82–30.27       |
| Transportation of fuel      | km.   | 50        | $\Omega$          |
| Output to technosphere      |       |           |                   |
| Heat, pellet combustion     | MJ    | 1         | 1                 |
| Emission to air             |       |           |                   |
| Carbon dioxide, biogenic    | g/MJ  | 128.67    | 86.89–94.54       |
| Carbon monoxide, biogenic   | g/MJ  | 0.200     | $0.001 - 0.002$   |
| Nitrogen oxides             | g/MJ  | 0.093     | $0.162 - 0.176$   |
| Sulfur dioxide              | g/MJ  | 0.003     | $0.0021 - 0.0023$ |
| <b>NMVOC</b>                | g/MJ  | 0.009     | 0.001             |
| Particulates, $< 2.5 \mu m$ | g/MJ  | 0.060     | $0.0018 - 0.0020$ |
| Particulates, $< 10 \mu$ m  | g/MJ  | 0.060     | $0.009 - 0.010$   |
| Solid wastes                |       |           |                   |
| Ash                         | g/MJ  | 0.320     | $1.97 - 2.15$     |

be bio-waste pellet combustion when hydrochar is assumed to feed the process boilers [\[17](#page-15-5)]. Inventory of the combustion of wood and bio-waste pellets are presented in the Table [5.](#page-7-0) Inventory for electricity and fuel entering the HTC processes were assumed to be average EU mixes values.

Furthermore, it is assumed that liquid and gaseous products streams are emitted to atmosphere without any treatment. Carbon dioxide emissions released during HTC process are treated as biogenic emissions. Further, it is assumed that HTC process water is recirculated. Approximately 96% of process water (around 14,500 kg/h) can be recovered and reused after dewatering as feedwater stream. The remaining part, lost mainly during evaporation, has to be made up to compensate for losses. Wastewater composition and emission to air during HTC process are modelled using experimentally measured mass yield and composition of gas and liquid phase, as well as using the previously described assumption about the TOC distribution in the liquid phase composition. Table [6](#page-7-1) shows amounts and organic and mineral loadings of the wastewater and air emissions as resulting from the mass balances simulation.

# **Life Cycle Impact Assessment**

The proposed solutions will be compared according to their energy balance, as well as by Life Cycle Impact Assessment (LCIA) results. The impact assessment phase is carried out adopting the following impact assessment indicators: Climate Change, Acidifcation and Freshwater Eutrophication, representing set of indicators commonly used and recommended in energy system LCAs [[41,](#page-15-29) [42](#page-15-30)]. These indicators were calculated according to ILCD 2011 Midpoint methodology, released by the Joint Research Centre of the European Commission in 2012 [[43](#page-15-31)]. Additionally, Freshwater Ecotoxicity is estimated, using the characterization factors (CFs) from UseTox model, developed by UNEP–Society for Environmental Toxicology and Chemistry (SETAC) Life Cycle Initiative [\[44,](#page-15-32) [45](#page-16-0)]. The CFs for Freshwater Ecotoxicity

<span id="page-7-1"></span>



are expressed in Comparative Toxic Units (CTU) per kg of emission that estimate the Potentially Afected Fraction of species (PAF) integrated over time and volume per unit mass of a chemical emitted (PAF  $m^3$  day kg<sup>-1</sup>). Specifically, PAF  $\times$  m<sup>3</sup>  $\times$  day per kg emitted = CTUe per kg emitted.

The results are presented comparing the values of the calculated indicators, showing the impacts of the recovery processes and the potential avoided impacts due to products and co-products substitution separately. Moreover, the contribution analysis focusing on the diferent phases of the HTC processing and highlighting the most impactful ones was performed.

# **Results and Discussion**

#### **Energy Balance**

Energy balance of the HTC plant is given in Table [7.](#page-8-0) Major part of the thermal energy ranging from 91.6 to 96.9% of the total thermal energy demand is used by Burner B1. As expected, energy demand of B1 is increased with increasing of HTC temperature. Regarding B2, the highest thermal requirements are observed for cases with the highest solid mass yield (test case 2). The higher amount of hydrochar produced implies a greater amount of water has to be evaporated in the dryer, increasing B2 energy demand. It can be further observed that burners fuel demand can be satisfed by produced hydrochar pellets for both scenarios of heat recovery.

In the case of electric energy, the highest consumption (between 43.7 and 47.7%) is attributed to pelletizer. Similarly to B1, the power duty depends on the produced hydrochar amount, which varies with HTC temperature. Second major consumer is air blower requiring from 19.2 to 21.0% of total electricity demand. Also in this case, the variations of electricity consumptions follow a trend depending on hydrochar yield, in turn function of the HTC process temperature. The pump electricity consumption is in the range from 8.8 to 11.4% of total power demand. Electricity demand of the flter and decanter is in the range 8.6 to 9.4% of total power demand. Remained contribution of mixer, grinder, and stirrer is ranging from 13.3 to 17.1% of total electricity consumption. The specifc thermal energy consumptions are presented in Fig. [3](#page-9-0).

When considering thermal energy consumption, it can be observed the values ranging between 2.45 and 2.74 kWh/ kg<sub>feedstock</sub> for HTC process without heat recovery scheme, and 1.15 to 1.55 kWh/k $g_{\text{feedback}}$  for HTC process with heat recovery scheme. Therefore, in the HTC process with heat recovery the energy consumption can be reduced from 43 to 53% depending on the process temperature. Electricity values are between 0.04 and 0.05 kWh/k $g_{\text{feedback}}$ . Obtained values are comparable with those reported in literature.

Lucian and Fiori [[15\]](#page-15-3) reported values in the range 1.65–1.85 kWh/k $g_{\text{feedback}}$  for off-specification compost and 0.30–0.38 kWh/k $g_{\text{feedback}}$  for grape marc with HTC process at operating temperatures between 180 and 250 °C with heat recovery. Stemann et al. [[14](#page-15-2)] obtained values of 0.09 kWh/  $kg_{\text{feedback}}$  of electricity and 0.39 kWh/k $g_{\text{feedback}}$  of boiler fuel

#### <span id="page-8-0"></span>**Table 7** Energy balance



<span id="page-9-0"></span>**Fig. 3** Specifc energy consumption (kWh/kgOP) at different HTC temperatures (260, 280, 305 °C) and residence time of 3 h



Electric energy Thermal energy, with heat recovery Thermal energy, without heat recovery

for HTC of empty palm fruit bunches performed at 220 °C. Benavente et al. [\[23](#page-15-11)] reported the electricity consumption of HTC process of olive mill wastes at temperatures between 200 and 250 varying from 0.02 and 0.03 kWh/kgfeedstock and relative thermal energy consumption between 1.47 and 2.10 kWh/kg<sub>feedstock</sub>. The values we obtained for the layouts without internal energy recovery—which can be directly compared with those from Benavente et al. [\[23\]](#page-15-11)—are generally higher, because of assumed higher HTC temperatures; however, in the layouts with internal energy recovery, our values are lower, thanks to the integrated energy recovery schemes.

#### **LCA Impact Assessment Results**

A comparison among the analyzed systems is provided in Figs. [4,](#page-9-1) [5](#page-10-0), [6](#page-10-1) and [7](#page-11-0), reporting the values of Climate Change (units: kgCO<sub>2eq</sub>/kgOP), Acidification (units: mol H +  $_{eq}$ / kgOP), Freshwater Eutrophication (units:  $kgP_{eq}/kgOP$ ) and Freshwater Ecotoxicity (units: (CTU/kgOP).

In Figs. [4](#page-9-1), [5](#page-10-0), [6](#page-10-1) and [7,](#page-11-0) for each case the specifc HTC process impacts and comparative reference system impact with respect to diferent marginal productions are reported. Positive values represent the environmental load while negative



<span id="page-9-1"></span>**Fig. 4** Climate Change impact (kgCO<sub>2eq</sub>/kgOP) at different HTC temperatures (260, 280, 305 °C) and residence time of 3 h: *HR* heat recovery, *WP* wood pellet feeding, *HCP* HTC hydrochar pellet feeding



<span id="page-10-0"></span>**Fig. 5** Acidifcation impact (mol H+eq/kgOP) at diferent HTC temperatures (260, 280, 305 °C) and residence time of 3 h: *HR* heat recovery, *WP* wood pellet feeding, *HCP* HTC hydrochar pellet feeding



<span id="page-10-1"></span>**Fig. 6** Eutrophication impact (kgPeq/kgOP) at diferent HTC temperatures (260, 280, 305 °C) and residence time of 3 h: *HR* heat recovery, *WP* wood pellet feeding, *HCP* HTC hydrochar pellet feeding

values are the benefcial efects due to substitution of the process product in the marginal production process.

Considering only the efective consumptions and emissions from the olive pomace HTC valorization processes, i.e. excluding the comparison with reference systems, impact effects range from 0.468 to 1.024 kgCO<sub>2eq</sub>/kgOP, 0.0007 to 0.0015 mol  $H_{\text{eq}}$ /kgOP, 0.036 to 36.290 g P<sub>eq</sub>/ kgOP and 165.8 to 254.5 CTU/kgOP, in terms of Climate Change, Acidification, Eutrophication and Freshwater



<span id="page-11-0"></span>**Fig. 7** Freshwater ecotoxicity impact (CTU/kgOP) at diferent HTC temperatures (260, 280, 305 °C) and residence time of 3 h: *HR* heat recovery, *WP* wood pellet feeding, *HCP* HTC hydrochar pellet feeding

Toxicity, respectively. These results are in line with those from Benavente et al. [[23\]](#page-15-11).

As expected, the resulting impacts are higher for the scenarios without heat recovery for all the impacts analyzed with exception of Freshwater Toxicity. In fact, for Climate Change, Acidifcation end Eutrophication, it is observed that the impact values for the HTC fed by wood pellet with heat recovery (HTC260/HR/WP, HTC280/ HR/WP, HTC305/HR/WP) is between 1.6 and 2.0 times lower than those obtained when no heat recovery is implemented; similarly HTC hydrochar feeding process with heat recovery (HTC260/HR/HCP, HTC280/HR/HCP, HTC305/HR/HCP) lead to values of impact that are between 1.4 and 2.0 times lower than the corresponding cases with no heat recovery.

Conversely, for the Freshwater Ecotoxicity, no signifcant diferences are obtained between the cases involving different feeding and heat recovery scenarios. For this impact category, the major contribution  $(> 99\%)$  is associated with the liquid phase emission during HTC reaction, that depends only on the liquid yield, thus on the process temperature, as it will be detailed later (Fig. [11](#page-13-0)).

Further, by comparing the impact of HTC hydrochar heat production with reference systems, for all the studied cases, negative values are obtained. This means that substitution of the products from marginal processes with HTC product leads to environmental load savings for all of the analyzed impacts. In general, as expected, it can be noted that the highest benefts can be achieved by substituting coal with HTC pellet in the heat production. A less efective solution is represented by substituting wood pellets and natural gas. This is particular evident for Acidifcation and Eutrophication potentials, where the avoided impact in terms of heat produced from coal is particularly high, while those obtained from natural gas and wood pellets are comparable with the HTC impact. On the contrary, for the Freshwater toxicity case, it is observed that avoided efects are found to be negligible with respect to the HTC process impact.

#### **Contribution Analysis**

In this section, the contribution analysis is presented. Figures [8](#page-12-0), [9,](#page-12-1) [10,](#page-13-1) and [11](#page-13-0) display the share of the processes included within the system boundaries.

Regarding Climate Change and Acidifcation impacts, the combustion processes of HTC and wood pellet represent the major contributions, by accounting up to roughly the 95% and the 75% of the total, respectively, for the cases with the highest heat consumption that is for the cases with the highest HTC temperature and no recovery scheme.

By analyzing Fig. [8](#page-12-0) it can be noted that the wood pellet production contributes with negative values decreasing Climate Change impact value by about 20%, because  $CO<sub>2</sub>$  sink during biomass growth is considered. This can be explained by the fact that during the biomass production, signifcant amount of  $CO<sub>2</sub>$  is consumed as a resource exceeding the process emissions. For the remaining impact categories, in particular for the eutrophication potential, biomass production



<span id="page-12-0"></span>**Fig. 8** Contribution analysis of climate change impact (kgCO<sub>2eq</sub>/kgOP) at different HTC temperatures (260, 280, 305 °C) and residence time of 3 h: *HR* heat recovery, *WP* wood pellet feeding, *HCP* HTC hydrochar feeding



<span id="page-12-1"></span>**Fig. 9** Contribution analysis of acidification impact (mol  $H_{\text{eq}}$ /kgOP) at different HTC temperatures (260, 280, 305 °C) and residence time of 3 h: *HR* heat recovery, *WP* wood pellet feeding, *HCP* HTC hydrochar feeding

contributes signifcantly to the fnal efect. This is mainly due to phosphate water emission during the process.

Regarding the Freshwater Ecotoxicity, the major part of the impact is coming from the HTC process and the relative liquid phase emissions. The major responsible for such high



<span id="page-13-1"></span>**Fig. 10** Contribution analysis of Eutrophication impact (kgP<sub>eq</sub>/kgOP) at different HTC temperatures (260, 280, 305 °C) and residence time of 3 h: *HR* heat recovery, *WP* wood pellet feeding, *HCP* HTC hydrochar feeding



<span id="page-13-0"></span>**Fig. 11** Contribution analysis of Freshwater Ecotoxicity impact (CTU/kgOP) at diferent HTC temperatures (260, 280, 305 °C) and residence time of 3 h: *HR* heat recovery, *WP* wood pellet feeding, *HCP* HTC hydrochar feeding

share are phenols and furfurals released from the process, that are characterized by high impact factors (phenols: 933 CTU/kg and furfurals: 387 CTU/kg). We remind that one

strong simplifed assumption of the inventory is related to the characteristics of the liquid phase—that was not analyzed in detail—and for which we assumed the composition

being only phenols and furfurals. More details for the liquid composition are required for future deepening of the assessment. Additionally, we assumed that wastewater is directly discharged into the environment. It is reported that by removing liquid phase contaminants before discharging the water into an external water body, the environmental impact of HTC will be significantly reduced  $[18, 23]$  $[18, 23]$  $[18, 23]$ . This is also an issue that will require further analysis and the inclusion of a devoted wastewater treatment, properly designed in consideration of the real composition of the liquid phase (presently not available), in order to contain the impact on Freshwater Ecotoxicity.

# **Conclusions**

In this paper LCA is applied to evaluate and compare several scenarios of HTC process for olive pomace treatment with energy recovery. Diferent process conditions by means of temperature (260, 280 and 305 °C), subsequent combustion of the generated solid product, as well as diferent scenarios of energy recovery, including heat recovery and self-sufficiency by integrating boilers fred by produced HTC hydrochar, were considered.

Results from this study indicate that the environmental performance of HTC is mainly dependent on its energy consumption. Depicting a complete layout of the process, integrating as much as possible heat recovery from hot exiting streams, to pre-heat cold entering streams, is pivotal to fgure out more realistic estimation of energy consumption and evaluate the process, also in comparison with other alternatives. Process temperature and energy consumption were identifed as the parameters afecting signifcantly fnal environmental impacts.

By implementation of energy recovery scheme in the HTC process it was possible to save up to 53% of energy consumed and as a consequence to obtain values of Climate Change, Acidifcation end Eutrophication Potentials impacts from 1.4 to 2.0 times lower with respect to the process without heat recovery. For Freshwater Ecotoxicity impact no substantial diferences were observed between the cases involving diferent feeding and heat recovery scenarios. Such an impact is depending on the liquid phase emission during HTC reaction, that depends only on the liquid yield, thus on the process temperature.

However, some limitations apply to the results for freshwater ecotoxicity, due to the assumptions about the simplifed composition of HTC wastewaters and exclusion of wastewater treatment before discharge to environment. Further work should deepen the liquid composition analysis and include an appropriate process to treat HTC wastewaters.

By considering only the efective consumptions and emissions from the olive pomace HTC valorization, it was noted that wood pellet feeding was a process more environmentally convenient than HTC pellet feeding. Further, substitution of the products from marginal processes with HTC product led to environmental load savings for all of the impacts analyzed with the highest benefts achieved by substituting coal with HTC pellet in the heat production.

The results of this study may help decision makers for fnding the best options for HTC energy supply in the future.

It is recommended that future works focus on the thermal consumption model development, process fowchart optimization as well as the integration of HTC with other renewable/conventional processes in order to make the whole process more energy efficient.

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