Energy efficiency of hydrothermal carbonization of olive pomace: scaling up from a laboratory to industrial scale

E. Carnevale[1], A. De Risi[2], L. Lombardi[3], B. Mendecka[1], F. Micali[2],

[1]Department of Industrial Engineering, University of Florence, Florence, 50139, Italy
[2]Department of Innovation Engineering, University of Salento, Lecce, 73100, Italy
[3]Niccolò Cusano University, Rome, 00166, Italy
Keywords: olive residues, hydrochar, energy consumption.
Presenting author email: lidia.lombardi@unicusano.it

Olive oil is one of the strategic agriculture products in Mediterranean countries. The European Union is the leading producer of the olive oil. In 2017, 2.2 Mt/year of oil was produced in the EU, accounted for around 70 % of total worldwide production (The European Commission 2018). Italy with the annual olive oil production of 430 kt/year in 2017 represented the second largest producer in the EU after Spain (Ornella 2017).

Olive oil production yields two main by-products: a solid one (olive pomace) and a liquid one (mill wastewater). 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 discharge liquid and solid waste on the soil. It is reported that for every 1000 kg of olive oil production, about 7500 kg of wastewater and 1260 kg solid wastes are generated (Vlyssides *et al.* 2004).

The solid residue derived from the olive oil production process may represent an interesting energy source as it contains a significant contribution of carbon. The composition of the olive pomace depends on the process applied. The indicative chemical composition of the olive pomace obtained from different methods is available in the literature (Vlyssides *et al.* 2004), highlighting that olive pomace also contains oils and high moisture levels. Thus before being effectively used as a combustion fuel, it has to be pre-treated employing drying and oil extracting treatments (Intini *et al.* 2011).

As an alternative, olive pomace 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 calorific 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 well-defined 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).

Few studies on HTC of olive pomace can be found in the literature. As an example, Volpe and Fiori (Volpe & Fiori 2017) have performed the HTC of two types of agro-industrial wastes: olive tree trimmings (OT) and olive pulp (OP) under different process conditions regarding temperature, and biomass to water ratio. The heating value of olive pomace, reported by (Volpe & Fiori 2017) 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. (Missaoui *et al.* 2017) performed hydrothermal carbonization of dried olive pomace obtained from 3-phase centrifugal extraction process under the varying temperature conditions (180 – 250^{o}C), varying residence time (0-120 min) and varying biomass to water ratios (1:2-1:6).
Similarly to work presented by Volpe and Fiori, 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 in the laboratory scale. However, 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. No literature studies have been dedicated to this area.

In this study, we applied the experimental and fundamental approach to scale – up the HTC process of olive pomace. The experimental part was dedicated to the laboratory scale discontinuous process performed in a batch reactor. During HTC experiments olive pomace was heated in subcritical water at 260, 280 and 305 ^{o}C and autogenic pressures. Olive pomace to water ratio was kept equal to 1: 6. On the basis of the experimental results, we created a general kinetic model assuming that all the occurring reactions are the first order and the Arrhenius equation describes the temperature dependences of reaction rate constants. Obtained kinetic parameters were applied in the solid, liquid and gas mass conversion model. The example mass conversion for the selected process temperature is presented in figure 1.

The second step was aimed to the thermodynamic performance of the laboratory scale HTC reactor. Simple lumped capacitance method was applied to simulate the transient behavior of the reactor during non –

isothermal operation and to estimate the heating energy consumption. The heating energy required is composed of the energy to raise the reaction mixture to a certain temperature and to keep it for the duration of the reaction.

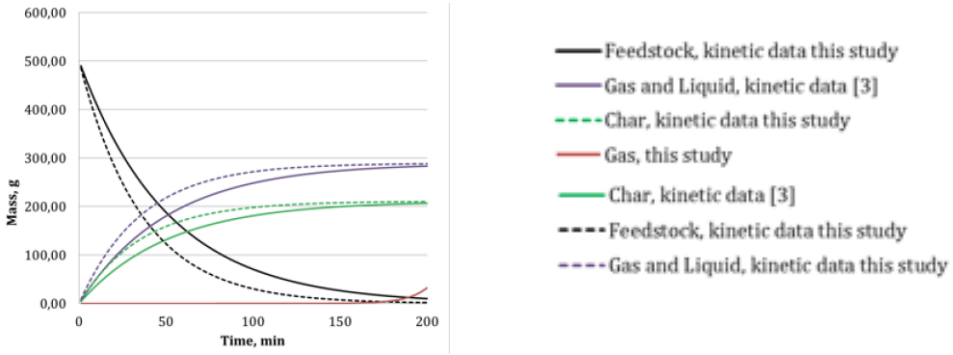

Figure 1 Predicted mass conversion (total mass), T=260°C

The thermal model was validated using the temperature and energy consumption profiles obtained during the experiments. Moreover, during a reaction, a certain amount of stirring energy is consumed. A model estimating energy consumption during stirring proposed by (Zlokarnik 2008) and used in the scale-up the procedure of chemical bath reactor by (Piccinno *et al.* 2016) is applied.

In order to implement the HTC process to a pilot or industrial plant, a model connecting input and output variables (Table 1) is required. Previously developed laboratory scale energy and mass balances were implemented to the hypothetical industrial scale. The fundamental approach was used to simulate the industrial HTC process by changing the input parameters.

Table 1: Input/output to the model

Chemical reactions	Input	Output
Parameters	Raw material mass	Mass and Energy yield (using reaction rate and Arrhenius equation and prediction model) Energy consumption (Lumped capacitance model) Stirring energy
	Temperature	
	Time	
	Reactor geometry	
	Construction materials	

The scale-up of substrates and products of the process is performed linearly because those have to be used in stoichiometric quantities according to the lab procedures. 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 the change of the energy values within the various scales shows that consumed heating energy do not behave linearly, meaning that the energy consumption per kg produced decreases with growing scale.

The proposed scale-up methodology was developed using the phenomenological based numerical model in the complex HTC system. Non – isothermal process HTC in the batch reactor was scaled up from 5 l to …..{???}. The proposed approach can be useful in the design of the industrial batch reactor.

References

Intini, F., Kühtz, S., & Rospi, G. (2011). Energy Recovery of the Solid Waste of the Olive Oil Industries–LCA Analysis and Carbon Footprint Assessment. *J. Sustain. Energy Environ*, **2**(January), 157–166.

Missaoui, A., Bostyn, S., Belandria, V., Cagnon, B., Sarh, B., & Gökalp, I. (2017). Hydrothermal carbonization of dried olive pomace: Energy potential and process performances. *Journal of Analytical and Applied Pyrolysis*, **128**(September), 281–290.

Ornella, B. (2017). Italian Olive Oil Overview 2017, 1–4.

Piccinno, F., Hischier, R., Seeger, S., & Som, C. (2016). From laboratory to industrial scale: a scale-up framework for chemical processes in life cycle assessment studies. *Journal of Cleaner Production*, **135**, 1085–1097.

The European Commission. (2018). *Market situation in the oil and table olives sectors*.

Vlyssides, A. ., Loizides, M., & Karlis, P. . (2004). Integrated strategic approach for reusing olive oil extraction by-products. *Journal of Cleaner Production*, **12**(6), 603–611.

Volpe, M., & Fiori, L. (2017). From olive waste to solid biofuel through hydrothermal carbonisation: The role of temperature and solid load on secondary char formation and hydrochar energy properties. *Journal of Analytical and Applied Pyrolysis*, **124**, 63–72.

Zlokarnik, M. (2008). Scale-Up in Chemical Engineering. In *Ullmann's Encyclopedia of Industrial Chemistry*, Weinheim, Germany: Wiley-VCH Verlag GmbH & Co. KGaA. doi:10.1002/14356007.b01_03.pub2