

Impact of the Medium as a moisture source in the Hydrothermal carbonization of raw olive pruning

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In recent years, concerns have emerged within the scientific community regarding the growing increase in olive oil production waste, which has been linked to severe environmental problems including the contamination of groundwater by several pollutants. To address these issues, one of the sustainable solutions lies in adopting biomass as both a source of renewable energy and an adsorbent for water contaminants through thermochemical processes, mainly hydrothermal carbonization (HTC) which stands out as a promising alternative for converting wet biomass (A. A. Azzaz et al., 2020).

The present work aims to use Olive Mill Wastewater (OMWW) as a moisture medium in a calorimetric bomb to process Raw Olive Pomace (ROP) as to produce hydrochars and to compare the yields with those obtained when using distilled water. This approach aims to increase the minerals present in OMWW and their role in a possible enhancement of the physico-chemical properties of the derived hydrochars. Although, the ultimate purpose of this approach is to enhance the physicochemical properties of the hydrochar, this paper focuses only on the impact of the moisture source on yields at different temperatures.

Olive mill wastewater (OMW) is obtained from an olive oil mill employing a continuous three-phase centrifugation system in a semi-industrial facility located in the Morneg region of Tunis, Tunisia. Samples were collected during the olive harvesting season in November 2023. This liquid effluent (OMWW) is sourced directly from the mills during the olive oil extraction process and analyzed for pH, total solids, chemical oxygen demand (COD), and phenolic content. Raw olive pomace (ROP) is air-dried for four days and ground to achieve a particle size of less than 1 mm. It is then mixed with water at a 1:9 ratio. For impregnation, which aims to improve the carbonization process, ROP is combined with OMWW in the same 1:9 ratio, allowing the mixture to absorb the liquid for a specified duration. This method, initially tested by Azzaz et al. [7], utilizes the abundant organic matter and minerals in OMWW to potentially enhance the physicochemical properties of the resulting hydrochars. However, this study focuses specifically on hydrochar yields when OMWW is employed as the liquid medium for ROP carbonization. The hydrothermal carbonization (HTC) process is performed in stainless steel batch reactors designed to withstand high pressure and temperature. The sealed reactors are heated to temperatures between 180 °C and 250 °C, at pressures of 1–5 MPa, with a heating rate of 10 °C/min. These conditions promote the decomposition of organic matter and the formation of hydrochar. Residence times can range from 1 to 12 hours, depending on the desired hydrochar properties. In this work, a 24-hour residence time was selected to achieve higher carbon content and more complete carbonization. Following the process, the resulting products are vacuum-filtered using 0.45 µm Whatman® filter paper and separated into solid hydrochar, a liquid fraction rich in organic compounds, and gases. The hydrochar is dried overnight at 105 °C, weighed, and stored in glass vials. The liquid fractions are filtered twice using 0.2 µm polypropylene syringe filters and stored in 10 mL glass vials at 4 °C.

The results show that an increase in carbonization temperature from 180°C to 220°C leads to a significant decrease in hydrochar yield for the two media (distilled water and olive mill wastewater), which is attributed to the increase in volatile matter content. This decrease is due to the alteration of the cellulose matrix, leading to the formation of a thermodynamically less stable carbon structure (A. Azzaz et al., 2020). The highest carbonization yield, of about 73.75% was observed for the OMWW as a moisture source at 180°C. At all experiments, the OMWWs were the media that yield most hydrochars. These results confirm that this approach takes advantage of the huge amount of organic matter to increase the yields, which would contribute in reducing the olive mill waste volumes.

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