Valorization of olive mill wastewaters: Novel and sustainable adsorption techniques using activated spent coffee grounds

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During olive-oil production, substantial quantities of by-products laden with phenolic components and degradation-resistant organic matter are generated, with phytotoxic properties, posing a considerable environmental hazard. Olive mill wastewaters (OMW) constitute the primary liquid effluents produced by olive oil production industry. According to several investigations, polyphenols derived from olive fruits and their by-products have significant health benefits (Tsagaraki *et al* 2007). Natural bioactive components from food industry wastes are increasingly acknowledged as promising options for the food, cosmetic and pharmaceutical industries. Given the abundant availability of OMW, particularly in Mediterranean countries like Greece, and its high phenolic content, OMW may turn into a natural source of valuable antioxidants in the coming years (El-Abbassi *et al* 2012).

The objective of the present study is the recovery of these value-added compounds through an innovative, sustainable, and cost-effective technique, such as adsorption. Recently, due to the high cost and need for regeneration of commonly used adsorbent, researchers have shifted their attention toward utilizing biosorbents originated from food industry wastes. Spent coffee grounds (SCG) constitute the primary solid residue produced during brewing of coffee powder (Zuorro and Lavecchia, 2012). SCG stands as a plentiful and inexpensive solid waste after coffee beverage production. According to the International Coffee Organization in 2021, global consumption of coffee exceeded 9.9×10^6 kg (ICO, 2022), while over 6 million tons of SCG are generated annually, with a considerable amount being disposed of in landfills.

Primarily, in the present study, OMW was collected from a three-phase mill in Lesvos island and filtered. SCG underwent a drying process at 45 °C for 24 h, reducing moisture content to around 3% w/w before being employed for phenolic compound extraction. After extraction, solid residue of the filtration was dried. Batch adsorption experiments were conducted in order to investigate equilibrium time and the effects of initial sorbate phenolic concentration, sorbent mass concentration, temperature and solution’s pH on total phenols uptake with a view to identify the optimum adsorption conditions. The highest observed adsorption efficiency was 45.44%, achieved in 20 min, at 30 °C and pH of 8.0, with an initial phenolic concentration of 162.5 mg/L and a sorbent ratio of 0.02 g/mL. While this value may appear relatively modest, in comparison to other methods for OMW purification, it can be deemed satisfactory, notable due to the absence of thermal or chemical pretreatment of biosorbent.

Taking into consideration that adsorption commonly encompasses various mechanisms, this study also aimed to delve deeper into the behavior and mechanism behind utilizing SCG in order to enhance the recovery of OMW polyphenols. With this purpose, the adsorption behavior of the primary OMW phenolics was examined separately to thoroughly understand their sorptive mechanisms. Consequently, adsorption experiments were carried out under the optimum conditions, focusing on the behavior of certain pure phenolics, i.e. p-coumaric acid, caffeic acid, gallic acid, tyrosol, hydroxytyrosol, and oleuropein, which constitute the main phenolic species in OMW.

In the present study, the effect of physical and chemical (methanol, sodium hydroxide, and protein-coating) activation on the adsorption efficiency of SCG was investigated. Generally, the utilization of activated SCG for OMW phenolics adsorption has not been extensively examined, particularly concerning mild thermal and milk protein-coating activation. Additionally, the alterations in adsorption efficiency was correlated with modifications in the physicochemical properties of SCG, including microstructure, pore size, surface area, and presence of functional groups. Furthermore, Fourier-transform infrared (FT-IR) spectroscopy was employed to detect alterations in chemical groups and bonding arrangements within the spectra of both raw and thermally or chemically activated SCG samples, both before and after batch adsorption experiments. As a part of a comprehensive study, high-performance liquid chromatography (HPLC) analysis was performed to determine and compare the phenolic profiles of OMW samples before and after the adsorption trials, while the identification and quantification of the aforementioned pure phenolics were also performed.

References

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