Improving the recovery of phenolic compounds recovery from olive mill wastewater by using activated spent coffee grounds

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During olive-oil production, significant amounts of wastes rich in phenolic components and degradation-resistant organic matter with antimicrobial and phytotoxic properties are generated, rendering these wastes a severe environmental hazard. Unfortunately, since there is an absence of an explicit regulation regarding olive mill waste discharge, commonly the management of this liquid waste known as “Olive Mill Wastewater”- OMW consists of its disposal in water recipients (Paraskeva and Diamadopoulos, 2006). Aiming to minimize this environmental threat, the scientific community has focused its efforts on the development of novel techniques regarding OMW depollution. Among them, adsorption is considered to be the most effective and low-cost technique for the recovery of value-added components from OMW (Elgarahy *et al* 2019; Rahmanian *et al* 2014; Singh *et al* 2008). However, the prohibitive cost of commercial adsorbents, as well as the need for their regeneration led researchers to the investigation and optimization of the adsorption process using novel, low-cost biosorbents originating from food industry by-products (Ververi and Goula, 2019).

The aim of the present study is the recovery of phenolic components from OMW using as a biosorbent, coffee industry by-products. Spent coffee grounds (SCG) are the primary solid by-product generated during brewing of coffee powder (Zuorro and Lavecchia, 2012). SCG is an inexpensive solid waste, which is abundant after coffee beverage production. According to the International Coffee Organization, total consumption of coffee exceeded 9.9×10^6 kg worldwide in 2021. Given the fact that 1 ton of green coffee beans can generate up to 650 kg of SCG and 1 kg of coffee beverage corresponds to 2 kg of wet SCG, it can be concluded that the need to exploit this by-product is urgent (Murthy and Naidu, 2012; Mata *et al* 2018).

Primarily, OMW was collected from a three-phase mill in Lesvos island and filtered. SCG were dried at 45 °C for 24 h to reach a moisture content of about 3% w/w and were used for the extraction of phenolic compounds. After extraction, solid residue of the filtration was dried. Batch adsorption experiments took place in order to study 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 determine the optimum adsorption conditions. The maximum adsorption yield observed was 45.44% after 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. This value can be considered relatively low, however, in comparison with other methods that have been studied for OMW depollution, it can be characterized as satisfactory, especially due to the fact that SCG used were not thermally or chemically treated.

In order to improve total phenols uptake, chemical and thermal activation of the biosorbent is investigated. To study the effect of thermal activation on adsorption yield, the biosorbent was heated in an oven at 100, 150, 200, and 250 °C for 2 h. As far as the chemical activation is concerned, three different methods were investigated using sodium hydroxide solution (NaOH), methanol, and coating with milk proteins. Specifically, in the case of NaOH activation, the biosorbent was stirred with sodium hydroxide solution (NaOH, 2 M) for 24 h at room temperature with a solid to liquid ratio of 2 g/33 mL and then at 45 °C for 2 h. Afterwards, filtration took place and the solid residue washed with distilled water and dried at 80 °C for 4 h. On the other hand, biosorbent was also activated using acidic methanol as described by Memon et al (2009). Specifically, 9 g of adsorbent was suspended in 633 mL of 99.9% methanol to which 5.4 mL of concentrated 0.1 M hydrochloric acid (HCl) was added. The solution was continuously stirred at 60 °C for 24 h. Then the solid residue was isolated, washed with distilled water, and dried at 40 °C for 24 h. Finally, another innovative technique for the increase of adsorption capacity of SCG was investigated. Especially, the biosorbent was coated with milk proteins and batch adsorption experiments at optimum conditions were repeated in order to determine the possible improvement of adsorption efficiency.

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