Factorial design of phenolic extraction process from two phase olive mill waste

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Abstract

Throughout the last decades, the olive oil extraction process has shifted from the traditional stone press extraction method to the continuous three-phase decanter and finally to the two phase system. Using this new technology, the olive paste is separated into two phases: olive oil and the II phase olive mill waste (OMW). The latter is a semi-solid waste with high moisture content 60-80% and a texture that makes its treatment and transportation very difficult. Moreover, OMW has a strong unattractive odor, an extremely high degree of organic pollution (COD values can reach up to 150 g/L) and a high organic load (Roig et al., 2006). It also contains lipids, organic acids and polyphenols (Vlyssides et al., 2017). In some cases, traces of pesticides, derived from the olive fruit cultivation, are also present.

Up to now, more than 50 different phenolic compounds have been identified in OMW (Torrecilla, 2010). Oleuropein, an ester of hydroxytyrosol and the elenolic acid glucoside, is the most abundant polyphenol present in many varieties of olives (Montedoro et al., 1993). Polyphenols seem to be responsible for the phytotoxic and antimicrobial effect of this waste and, in high concentrations, are potentially dangerous for the environment and human health (Torrecilla, 2010). All these characteristics of OMW create a clear need for the development of more effective methodologies in order to reduce its toxic potential before its re-use in agriculture.

In this study, a method for the extraction of phenolic compounds from two-phase OMW is proposed and a factorial design experiment has been carried out in order to optimize the extraction of three selected phenolics: oleuropein, tyrosol and hydroxytyrosol. To an initial amount of waste, concentrated sulfuric acid is added, followed by stirring for about 45 minutes at a temperature of 60 °C. The solution is then cooled down to 20°C and centrifuged at 4000 rpm. The supernatant containing the phenolics is filtered to remove suspended solids and total phenolic charge (TPC) is measured. The factorial design experiments were carried out with a variation of four factors (water/solid ratio, temperature, time and H_2SO_4 concentration) in order to optimize the extraction of the main phenolic compounds contained in OMW.

At the optimized conditions a scale up experiment has been conducted in which the supernatant solution of phenolics after filtration is purified by ultra-filtration membranes of specific molecular weight cut off (MWCO) to remove residual oil or other solid particles. The permeate solution is then added to a suitable amount of resins which absorb the phenolic components. After stirring and after the supernatant becomes clear and adsorption complete, the resins are dried at a temperature of 60°C for 24h to remove moisture. Methanol is finally added to the resins and vigorously shaken to remove the phenolics and the final solution is led to an evaporation unit to remove the solvent. The resulting concentrate contains a very high concentration of phenolics. At each stage of the experimental process, the pH and total phenolic charge (TPC) are monitored. In the final concentrate, each phenolic compound is quantitatively and qualitatively measured by HPLC/DAD.

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