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ORIGINAL PAPER

Selective separation of essential phenolic compounds from olive oil mill wastewater using a bulk liquid membrane

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Olive oil mill wastewater (OMWW) is very rich in phenolic compounds especially the key compounds of caffeic acid (CA), hydroxytyrosol (HTY), and tyrosol (TY). Therefore, the development of new and effective analytical and industrial methods for the separation and concentration of these valuable compounds has attracted great attention in the last decades. In this study, a selective transport and separation method for CA, HTY, and TY from OMWW samples, obtained from different olive orchards, using a new bulk liquid membrane (BLM) procedure was developed. Various factors influencing the transport efficiency such as pH of the source and receiving phases, nature and volume of the organic membrane, stirring rate, and transport time were investigated and optimized. Under optimal experimental conditions, the transport efficiencies of CA, HTY, and TY from the OMWW samples of 90.1 %, 28.4 %, and 34.9 % were obtained, respectively. Relative standard deviations (RSDs, $n = 7$) were found to be 4.1 %, 3.8 %, and 3.0 % and the limits of detection (LODs) obtained were 0.001 mg L^{-1}, 0.011 mg L^{-1}, and 0.008 mg L^{-1}, for CA, HTY, and TY, respectively.

Keywords: bulk liquid membrane, HPLC, OMWW, caffeic acid, hydroxytyrosol, tyrosol

Introduction

The olive oil production process is usually performed mechanically by pressure using a three-phase centrifugation system producing a very large volume of olive oil mill waste water (OMWW) which represents the main environmental problem of this process. On the other hand, OMWW is very rich in phenolic compounds, it has high antioxidant activity, and thus the capability of becoming the object of significant attention to pharmaceutical, cosmetic, and food industries. Many researchers have evaluated the feasibility and economic processes for the recovery of essential phenolic compounds from OMWW using different separation methods such as: solvent extraction (Ben Sassi et al., 2006; De Marco et al., 2007), ultra-filtration and inverse osmosis (Isidori et al., 2005), liquid–solid extraction (Bertin et al., 2010), and supercritical fluid extraction (Lafka et al., 2011). The classical method of Folin–Ciocalteu is a conventional colorimetric reference method for the determination of polyphenols (Mulinacci et al., 2001), which can be used only for the determination of the total content of polyphenols and it does not provide the concentration of each compound separately. On the other hand, the ethyl acetate extraction method is more convenient for the separation and determination of individual phenolic acids by GC and LC (Zafra et al., 2006). It is also frequently used to extract biophenols from aqueous matrices such as the OMWW samples (Lesage-Meessen et al., 2001; DellaGreca et al., 2004; Fki et al., 2005; De Leonardis et al., 2007; De Marco et al., 2007).

Membrane-based extraction techniques offer efficient alternatives to classical sample preparation techniques (Jönsson & Mathiasson, 1999). Membranes are barriers between two fluid phases (source and receiv-

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