A sustainable phenolic compound extraction system from olive oil mill wastewater

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1. Introduction

The reduction of waste by-products along the supply chain allows food manufacturers to achieve a double benefit in terms of disposal costs and environmental impact abatement.

Such aspects have a direct positive effect on the profitability of business, especially when these by-products can be reused in the production of foods with health benefits (Hyde et al., 2001). Growing interest in healthy food has allowed several researchers to work on upgrading and improving the methods for reusing or recycling by-products deriving from different food processes (Bräutigam et al., 2014). In Mediterranean countries, the olive processing industry generates the most important agro-industrial wastewaters. In particular, Olive Oil Mill Wastewater (OOMW) is a secondary product of the olive oil extraction process, containing soft tissues of the olive fruit and the water used in the various stages of the oil extraction treatment. OOMW (from three-phase solvent extraction systems) is a recalcitrant dark brown effluent, with a distinctive odour, an acid pH (4.0–5.5) and high conductivity (6000–16,000 $\mu$S/cm). Furthermore, OOMW presents high values for most pollution parameters (Ouzounidou et al., 2010): $\text{BOD}_{2}:40–95$ g/L, $\text{COD}:50–180$ g/L, $\text{LD}:50$, toxicity for fish: 8.7% and it contains large amounts of suspended solids and high concentrations of polycyclic aromatic compounds, e.g. simple phenols and flavonoids, or polyphenols (0.5–24 g/L). Hence, if not recycled, OOMW constitutes a serious environmental problem because it represents a significant polluting waste (Zagklis et al., 2013). The olive fruit is very rich in phenolic compounds but, since the phenolic content of the olive fruit has a greater solubility in water than in fatty acid, only 2% of the their total amount passes during the oil phase, while the remaining is lost in the OOMW (approximately 53%) and in the pomace (approximately 45%) (Rodis et al., 2002). The extraction and the recycling of the biologically active compounds from OOMW may turn a polluting residue into a source of natural antioxidants, object of growing interest in pharmaceutical and food industries. A wide range of phenolic compounds was identified in Extra Virgin Olive Oil (EVOO) and OOMW, including phenolic alcohols, secoiridoids derivatives, phenolic acids, lignans, and flavonoids (Artajo et al., 2007). In particular, the potential...
innovative use of EVOO by-product is related to their richness in hydroxytyrosol and its derivatives (Servili et al., 2011a). The European Regulation EU 432/2012 declares that olive oils which contain at least 5 mg of hydroxytyrosol and its derivatives (e.g. oleuropein complex and tyrosol) per 20 g can be considered a healthy food. In order to uphold this claim, information should be given to the consumer regarding the beneficial effect that could be obtained with a daily intake of 20 g of olive oil. However, the quantity of hydroxytyrosol and its derivatives in Extra Virgin Olive Oil (EVOO), as well as in OOMW, is affected by several agronomic factors (such as cultivar, ripening stage, geographic and genetic origin of olive fruit, olive trees irrigation) (Caruso et al., 2014; Marra et al., 2016) and technological aspects (such as oil extraction conditions during crushing, malaxation and EVOO separation) (Ange rosa et al., 2004). Hence, in certain conditions, olive oil would not fit the requirements of the European Regulation EU 432/2012. In particular, several olive cultivars are genetically characterized by a low fruit phenolic content and consequently the corresponding EVOOs cannot guarantee some health advantages. Valls et al. (2015) have demonstrated that EVOO enriched with phenolic compounds can offer additional health benefits compared with a standard EVOO with moderate polyphenol content. In particular, the addition of polyphenols could improve the sensory characteristics of EVOO while preserving the characteristic varietal flavour (Servili et al., 2011a) and ensuring an optimal intake of polyphenols through the habitual diet (Suárez et al., 2009). Thus, the enrichment of EVOO is a way of increasing its health-promoting properties whilst consuming the same or less fat. In this context, OOMW treatments, that allow phenol extraction, may lead to economic benefits (García-Castello et al., 2010). These treatments are mainly based on biological degradation (anaerobic and aerobic) (Beltran et al., 2008) or on advanced oxidation process (Adhoum and Monser, 2004), as well as on various combinations of these processes (Khouri et al., 2006). It should be noted that the efficiency of the process, the complexity and the costs involved might vary significantly. High costs are often the main reason for not adopting these OOMW treatment methods (El-Abbassi et al., 2014).

Currently, one of the most promising methods for the treatment of OOMW, considering effectiveness, environmental impact and cost, is membrane filtration (Zagklis et al., 2013). Recently a membrane filtration system was applied in an industrial scale plant to obtain a Crude Phenolic Concentrate (CPC) from OOMW (Servili et al., 2011a). Membrane technology reduces the OOMW organic load and suspended solids content (El-Abbassi et al., 2013). Microfiltration (MF) and Ultrafiltration (UF) may be used as a primary treatment step, while Nanofiltration (NF) and Reverse Osmosis (RO) for the final treatment. With ultrafiltration, only a small amount of retentate (waste) is produced (permeate is 90–95% of the volume of the feed) and very high removal of lipids is achieved. Also, the composition of the permeate can be controlled by choosing the appropriate pore size of the membrane used. This recovery system guarantees an important waste reduction because allows the production of a concentrate enriched in the phenolic fraction (CPC) and a permeate purified from phenolic compounds and a major part of the organic fraction, which could thus be also recycled in the EVOO-extraction process (Servili et al., 2011a).

According to Servili et al. (2011a), the CPC shows numerous potential uses that include recycling in the oil mechanical extraction process to obtain EVOO enriched with hydrophilic phenols for the production of functional foods, characterized by the same biological activities observed for EVOO hydrophilic phenols (Servili et al., 2011b). Despite the fact that much research has been conducted to develop new methods for phenolic compounds extraction, few economic analyses are reported in literature to evaluate the sustainability of these methodologies (Lipnizki and Field, 2001; Ruiz-Rosa et al., 2016) but none for the olive oil industry.

In our study, a commercial MF membrane filtration system was used to separate the polyphenols from the OOMW of a specific olive cultivar (Cerasuola) to enrich the olive oil of two cultivars (Nocellara del Belice and Biancolilla), with low polyphenols content (Caruso et al., 2007). In particular, the aim of our study was to demonstrate a waste product reduction derived from EVOO extraction processes and to prove the economic sustainability of the MF membrane filtration system determining the price of the enriched olive oil that permits an affordable investment. For this purpose two economical indicators, the Net Present Value and the Payback time, were analysed.

2. Materials and methods

The OOMW used in this study was collected in 2015 from an olive mill located in Calstelvetrano (TP, Italy). The main cultivars are Cerasuola (63%), Biancolilla (20%) and Nocellara del Belice (17%). The drupes were processed by the extraction system Pieralisii Leopard (Ancona, Italy) equipped with a three-phase decanter-separator system with a working capacity of 10,000 kg/h. In Table 1, the total quantity of olives from the three cultivar considered, the pomace, the wastewaters, the oil quantities, and the corresponding yields for the cultivar Cerasuola, are reported.

2.1. Olive oil mill wastewater

The prototype of the olive wastewater treatment used in this research is reported in Fig. 1. It was realized by Permeare srl (Milan, Italy) and its working capacity is about 500 L/h of wastewater.

In the mill, the vegetable water produced with a low content of material in suspension (particle size exceeding 1 mm) was accumulated in one or more tanks with a capacity of 500 L. During the load into the tank, it was necessary to add thepectolytic enzymatic preparation (Permazin ZE01). The quantity of enzyme for each treatment was assumed to be 100 g/m² of OOMW. The following membrane treatments were microfiltration, ultrafiltration and reverse osmosis, as reported in Fig. 2.

Before the ultrafiltration section, the waters pass through a membrane with a porosity of 1 μm, which allows the removal of suspended solids (microfiltration). This section is composed of 3 membranes consisting in a vessel with tubular type modules. When the treatment process starts, the pumps of the tubular section transfer the content of the tank to a work tank. The ultrafiltration section concentrates the macromolecules in the OOMW and produces the permeate, which is then concentrated through a reverse osmosis system. At the end of the ultrafiltration cycle, the concentrate is discharged from the work tank and sent to the pomace oil or to a storage tank for disposal, while the permeate is sent to the internal tank of the reverse osmosis section. This section consists of 6 modules of spiral osmosis membranes (4 “x 40”). The reverse osmosis system gathers the substances present in solutions and produces a Crude Phenolic Concentrate (CPC) with a high content in polyphenols. All the membranes were purchased from Permeare S.r.l. (Milano, Italy). The expected life of the membranes is about 5 olive oil campaigns.

2.2. Olive oil and CPC analysis

Three samples of EVOO for each cultivar considered were stored in glass bottles at 8 °C and kept in a dark environment, following the EU Regulation statements (Reg. CEE n. 2568/91).

The extraction of total polyphenol was performed following the colorimetric method of Folin-Ciocalteau (Picerno et al., 2003) and...
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