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Advances in membrane operations for water purification and biophenols recovery/valorization from OMWWs

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ABSTRACT

Recovery of biomolecules from waste represents one of the most important challenges for sustainable resource exploitation.

An innovative process design for water recovery and polyphenols encapsulation from olive mill wastewaters (OMWWs) has been investigated combining conventional pressure-driven processes (microfiltration (MF) and nanofiltration (NF)) and relatively new membrane operations (osmotic distillation (OD) and membrane emulsification (ME)).

After the removal of suspended solids by an acidification/MF step, OMWWs were processed by NF in order to obtain water from the permeate side and a concentrated polyphenolic solution from the retentate side.

The NF retentate was dewatered by OD and the concentrated polyphenolic stream was encapsulated in a water-in-oil emulsion by ME.

The integrated membrane system resulted efficient in all the operation units. Indeed, relatively high fluxes, with respect to literature data, were obtained in both MF and NF steps (60 and 7 L/m^2 h, respectively); in addition high polyphenols rejections (%) were measured for the NF membrane. The concentration of the NF retentate by OD produced an enriched fraction of low molecular weight polyphenols according to a concentration factor of 7. This fraction is formulated by the ME process for the production of a W/O emulsion with an encapsulation efficiency of 90%.

According to the process mass balance, related to the treatment of 1000 L of OMWWs, 1463 g of phenolic compounds (85% of the initial phenolic content) and 800 L (80% of the initial volume) of purified water can be recovered, respectively.

1. Introduction

Resource valorisation from waste represents one of the most important challenges for sustainable industrial processes. Limited resources and increasing interest in the use of bioactive compounds play an important role in the development of sustainable waste management practices. Several physical, chemical and biotechnological approaches, have been attempted to valorise OMWWs [1] resulting in an environment protection with zero discharge.

Membrane processes such as ultrafiltration (UF) and nanofiltration (NF) have been successfully used for over three decades in food and beverage industries for the treatment of several fluids and agricultural wastewaters [2]. Interesting applications have been developed for the separation, concentration and recovery of high-added value compounds, including pectin and polyphenols, from agro-food byproducts [3,4].

Possibility to operate at room temperature, small area-requirement, no phase change and chemical additive use, low investment and maintenance costs, high efficiency and low specific energy consumption are key advantages of membrane technology over conventional separation processes in many wastewater treatment processes [5].

The combination of different pressure-driven membrane processes for the recovery of polyphenols from OMWs has been largely investigated [6–11]. Integrated systems in which membrane operations have been combined with other separation techniques such as supercritical fluid extraction [12] and chromatographic separations have been also proposed [13] for the recovery of antioxidants from OMWWs. By analysing patent publications in this field, Takaç and Karakaya [14] concluded that the future direction of the processes for the recovery of antioxidant compounds from

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OMWWs is presumably toward the use of membranes in a sequential design.

Membrane emulsification as encapsulation method has important potential in the valorization of recovered polyphenols for their application not only as food additives or as nutritional supplements, but also as active cosmetics or as drugs bioactive nutrients. The method allows the production of monodispersed droplets with controlled size in mild shear conditions [15,16]. The plethora of health benefits of biophenols coupled with the benefit demonstrated by the encapsulation justifies the possible additional cost related to the use of this emerging technology. Several limitations are reported in the use of these valuable natural compounds: (i) according to the route of administration, their efficiency depends on their bioavailability and integrity; (ii) during food products processing, distribution or storage or after oral administration, their instability in certain pH conditions or in the presence of enzymes or other nutrients, limits their activity and potential health benefits; (iii) for topical use, their sensitivity to environmental factors, including physical, chemical, and biological conditions (i.e. oxidation after light exposition) leading to the progressive appearance of a brown colour and/or unwanted odours with a considerable loss in activity; (iv) many polyphenols have an unpleasant taste which must be masked before their incorporation in food products or oral medicines [17]. Therefore, the use of encapsulated biophenolic compounds instead of free compounds (such as biophenols rich not-encapsulated solution and/or dry extracts powders) is a suitable method to protect these valuable natural compounds by maintaining their structural integrity until the consumption or the administration, mask its taste, increase the bioavailability. Among the existing encapsulation methods, membrane emulsification has been demonstrated as effective and reliable technique for the encapsulation of bioactive compounds [18–20].

In this study conventional membrane operations, such as microfiltration (MF) and nanofiltration (NF), and relatively new membrane operations, such as osmotic distillation (OD) and membrane emulsification (ME), were combined in order to produce biophenols formulations of interest for food or nutraceutical applications from OMWWs. The performance of the investigated membranes was evaluated in terms of fluxes, polyphenols and TOC rejection (%) and encapsulation efficiency (%) in the selected operating conditions. A mass balance of the process was carried out on the basis of water and biophenols recovered processing the OMWWs by the innovative sequential design.

2. Materials and methods

2.1. Olive mill wastewaters

Olive mill wastewaters used in this work were obtained from the 3-phase centrifugation process and were collected in different times of year. They were supplied by Olearia San Giorgio (San Giorgio Morgeto, Italy)coming from the biologic olive oil production without using of toxic pesticides. In order to reduce the suspended content, OMWWs were pre-treated according to the procedure reported in the previous work [21]. This method permits to completely remove suspended solids and therefore reducing fouling phenomena during the membrane filtration.

2.2. Integrated membrane system

OMWWs were treated according to the combination of membrane operations depicted in Fig. 1. The different steps investigated were: microfiltration (MF), nanofiltration (NF), osmotic distillation (OD) and membrane emulsification (ME). The membranes used and their main characteristics are reported in Table 1.

2.2.1. Microfiltration set-up

MF experiments were performed by using a pilot unit equipped with a feed tank of 100 L, a centrifuge pump (LOWARA, CEA 120/5), a stainless steel housing to accommodate the tubular membrane module and a permeate tank. Two manometers located before and after the membrane module allow to measure the inlet and outlet pressure. The transmembrane pressure (TMP) value was regulated by a pressure control valve on the retentate side. The feed flow rate (Q_f) was measured by a digital flowmeter; the permeate flow rate was automatically measured and registered by a mass flow controller. The feed temperature was controlled by a heat exchanger fed with tap water.

MF experiments were carried out according two different operative modes: batch concentration and feed-and-bleed. In the batch concentration configuration the retentate was returned to the feed reservoir while the permeate was collected separately: this configuration was used in order to define the highest volume reduction factor (VRF) in combination with a suitable productivity of the MF process. The feed-and-bleed configuration was investigated to simulate the operative mode usually used for continuous operation when a higher recovery rate must be obtained. According to this configuration, the permeate was removed from

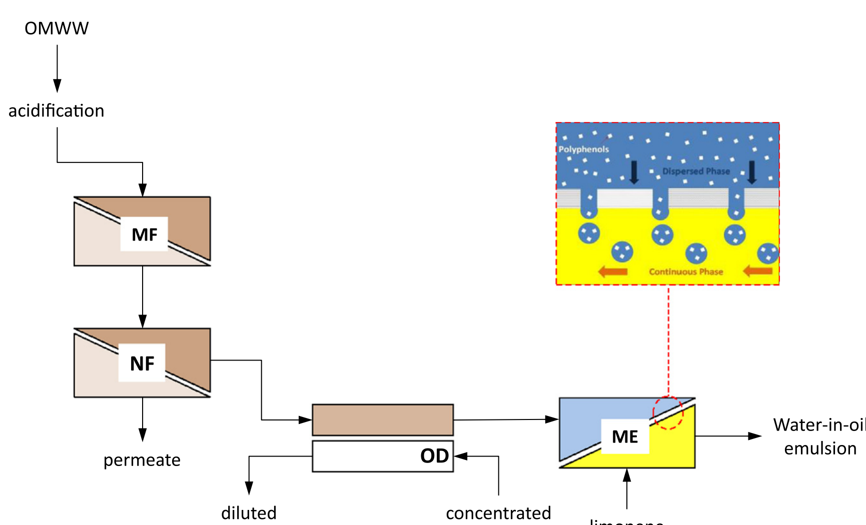

Fig. 1. Schematic representation of investigated membrane processes.