



# Recovery of VFAs from anaerobic digestion of dephenolized Olive Mill Wastewaters by Electrodialysis



Alberto Scoma<sup>1</sup>, Felipe Varela-Corredor, Lorenzo Bertin, Carlo Gostoli, Serena Bandini\*

Department of Civil, Chemical, Environmental and Materials Engineering, Alma Mater Studiorum, University of Bologna, School of Engineering and Architecture, Via U. Terracini, 28, I-40131 Bologna, Italy

## ARTICLE INFO

### Article history:

Received 29 July 2015

Received in revised form 17 December 2015

Accepted 18 December 2015

Available online 18 December 2015

### Keywords:

Electrodialysis

VFAs

Olive Mill Wastewaters

Homopolar membranes

Biorefinery

Recovery

Fractionation

## ABSTRACT

Electrodialysis (ED) is applied for the first time as a separation-concentration step of the Volatile Fatty Acids (VFAs) enriched effluent resulting from fermentation of Olive Mill Wastewaters. The process would represent a key step of an integrated multi-purpose biorefinery scheme including the biotechnological production of polyhydroxyalkanoates from acidified bioresidues.

A feasibility study is introduced by testing separation efficiency of membranes with synthetic model solutions of different compositions (sodium acetate and NaCl, or acetic–propionic–butyric acids with NaCl) and with actual OMW (a complex mixture of sodium salts of acetic, propionic, isobutyric, butyric, isovaleric, valeric, isocaproic, caproic, eptanoic acids, NaCl and other electrolytes).

Experiments were performed at room temperature in a lab-scale 2-compartments ED stack, containing Neosepta AMX-CMX membranes, at constant current density (31 A/m<sup>2</sup>). Membrane resistance was tested by performing additional ED experiments according to a proper protocol developed in this work.

Removal of VFAs from actual OMW is feasible; no evidence of remarkable damages nor losses of membrane performance were observed after dedicated trials. Under the operative conditions investigated, VFAs removals ranged from 30% to 35%, resulting in a concentration factor between 1.2 and 1.5 with respect to the initial mother solution.

Chloride removal was remarkably high and it was greatly favoured at the beginning of operation; competition between chloride and acidic anions was remarkable as far as chloride concentration was high, whereas acidic anions transport across the membrane increased after NaCl removal overcame 50%.

Removal and concomitant concentration of VFAs were accomplished by a slight fractionation of acids with respect to the initial solution; the main outcome of which was an enrichment of acetate in the concentrate. Transport of each acidic anion across the membranes was affected by the concomitant role of concentration and diffusivity, which can shift the natural order imposed by the steric hindrance of the species.

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## 1. Introduction

Organic acids are of primary importance in our society. They are largely used for the production of foods, beverages, pharmaceuticals, plastics, resins and other biochemical or chemical products [1–3]. Volatile Fatty Acids (VFAs) are linear short-chain aliphatic monofunctional compounds, which represent suitable precursors for the production of biopolymers, reduced chemicals (esters, ketones, aldehydes, alcohols and alkanes) and biofuels [4]. Large-scale production of VFAs is conventionally achieved by chemical

synthesis, the most notable examples being acetic acid production by methanol carbonylation and propionic acid synthesis by the oxo process that involves reaction of ethylene and carbon monoxide to produce propionaldehyde, which is further catalytically oxidized. However, VFAs can be easily produced through the biotechnological exploitation of organic matrices by means of anaerobic digestion, as when operating under fermentative conditions [5–9]. Contrary to methanogenesis, energy conversion yields in fermentation are rather low and upper limits for overall VFAs concentration in acidic effluents are typically close to 20–30 g/dm<sup>3</sup> depending on the employed organic substrate, since inhibitory effects onto microbial consortia at low pH can interfere with proton gradient mechanisms [10]. Nonetheless, generation of VFA-enriched effluents is of pivotal importance to establish a carboxylate platform making use of

\* Corresponding author.

E-mail address: [serena.bandini@unibo.it](mailto:serena.bandini@unibo.it) (S. Bandini).

<sup>1</sup> Present address: LabMET, University of Ghent, Coupure Links 653, 9000 Ghent, Belgium.

## Nomenclature

### Abbreviations

ace	acetic acid
pro	propionic acid
ibut	isobutyric acid
but	butyric acid
ival	isovaleric acid
val	valeric acid
icap	isocaproic acid
cap	caproic acid
ept	eptanoic acid
VFAs	Volatile Fatty Acids

### Symbols

0	initial time
$c$	molar/mass concentration ( $\text{mol m}^{-3}/\text{kg m}^{-3}$ )
$n$	amount of substance (mol)
$n_c$	number of elementary cells
rem	cumulative removal (defined in Eq. (2))
$t$	generic time

EF	enrichment factor (defined in Eq. (5))
$F$	Faraday constant ( $\text{C mol}^{-1}$ )
$I$	electric current (A)
$J$	molar flux ( $\text{mol m}^{-2} \text{s}^{-1}$ )
$N$	molar cumulative transport (defined in Eq. (1)) ( $\text{mol s}^{-1}$ )
$R$	universal gas constant
$T$	thermodynamic temperature (K)
$T_j$	transfer number (defined in Eq. (3))
$V$	electric potential (V)
$W$	overall specific energy requirement (defined in Eq. (8)) ( $\text{J mol}^{-1}$ )
$\alpha$	separation ratio (defined in Eq. (6))
$\varepsilon_c$	current efficiency (defined in Eq. (7))

### Subscripts and superscripts

$j$	generic specie
(1)	diluate compartment
(2)	concentrate compartment

highly impacting biowastes [11]. VFAs mixtures generated from renewable biowaste material could be used for the bioproduction of polyhydroxyalkanoates (PHAs) [35], which are microbial polyesters produced and stored by several bacteria [7]. The availability of VFA-enriched effluents would allow to feed PHA-producing processes without inducing significant dilution of the biomass.

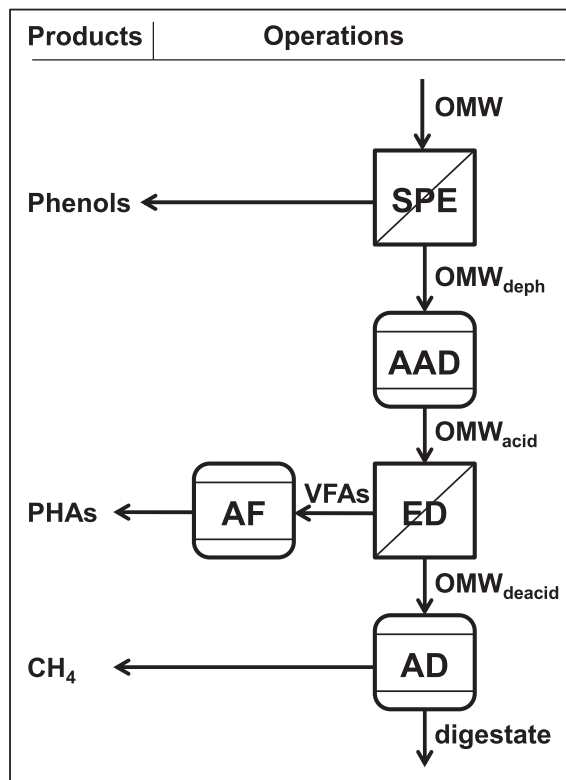
Among several biowastes investigated as renewable resources, PHAs production from acidified Olive Mill Wastewaters (OMWs) has been recently developed and described [12]. Economical sustainability of this process may benefit of an integration within a multi-purpose biorefinery scheme (Fig. 1). OMWs can be pre-treated to recover polyphenols, which are natural antioxidants typically occurring in this biowaste, by means of a solid phase extraction process (SPE). Since OMW polyphenols can exert antimicrobial activities, SPE results into a dephenolized stream undergoing a more effective and performing fermentation of OMWs [8]. Application of an Electrodialysis (ED) separation process to the acidified effluent ( $\text{OMW}_{\text{acid}}$ ) would generate a further VFA-enriched stream to be used for PHAs production in an aerobic fermentation step (AF). The exhausted liquid ( $\text{OMW}_{\text{de-acid}}$ ) produced by ED would thus represent an ideal aqueous effluent by which diluting solid biowastes in anaerobic digestion (AD) processes suitable for biomethane production. The present scheme (Fig. 1) represents only one of the many potential application which would benefit from the further refining of fermentation broths, as the availability (and feasibility) of sustainable recovery of VFAs and organics derived from biowaste management represents the main bottleneck for full development of second generation biorifineries [4,11].

The interest of membrane processes for separation and concentration of VFAs from fermentation broths is well documented in the literature [13–15]. Among membrane techniques, ED has been tested for various purposes [16–29,2,3,30–33] which were aimed at achieving different objectives related to the biotechnological production of VFAs, such as:

- Continuous removal of VFAs to increase the bioreactor productivity [17–24,2,3].
- VFAs concentration [17,18,25–29].
- VFAs demineralization [26].
- Conversion of sodium salts of VFAs into the corresponding acids [18,19,33].

- Fractionation of the VFAs mixtures [12,13,29].

Feasibility of VFAs removal has been established and improvements of bioreactor productivity have been confirmed. That could be achieved by ED operating in different modes: high removals are



**Fig. 1.** Schematic flow sheet of the proposed overall process. Effluents: Olive Mill Wastewater (OMW), dephenolized Olive Mill Wastewater ( $\text{OMW}_{\text{deph}}$ ), acidified Olive Mill Wastewater ( $\text{OMW}_{\text{acid}}$ ), VFA-enriched effluent (VFAs), deacidified Olive Mill Wastewater ( $\text{OMW}_{\text{deacid}}$ ). Operations: solid phase extraction of polyphenols (SPE), VFAs production by anaerobic acidogenic digestion (AAD), VFAs concentration by Electrodialysis (ED), PHAs production by aerobic fermentation (AF), biomethane production by anaerobic digestion (AD).

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