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# Effect of aerobic pretreatment on anaerobic digestion of olive mill wastewater (OMWW): An ecoefficient treatment

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ABSTRACT

Anaerobic digestion experiments of olive mill wastewater (OMWW) without pretreatment were carried out and signs of inhibition in the biological process were observed after a time equal to 1.5 times the Hydraulic Residence Time (HRT) due to the presence of a high inhibitors concentration in OMWW. Based on these findings, OMWW was subjected to an aerobic pretreatment in order to reduce the concentration of phenolic compounds and decrease Total Chemical Oxygen Demand (TCOD), achieving a reduction of 78% and 90% of the initial polyphenols concentration and 18% and 21% TCOD reduction when the substrate was aerated for 5 and 7 days respectively.

Finally, anaerobic digestion experiments using OMWW aerated for 5 and 7 days as substrate were conducted to determine the influence of aeration time on methane yields and TCOD reduction. The results yield 5 days as the aeration time that achieves best results, given that this stabilizes the anaerobic process, can reduce the TCOD by 65% and generates almost 0.39 $m^3$ methane/kg removed TCOD.

Anaerobic digestion of OMWW will be economically feasible if the waste is pretreated by aerobic digestion, since the period of return of investment obtained under these conditions does not exceed 6 years.

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*Keywords:* Aerobic pretreatment; Anaerobic digestion; Olive mill wastewater; Economic viability; Polyphenols inhibition; Methane yield

## 1. Introduction

Spain is the largest producer of olive oil in the world. The olive oil produced in this country is extracted by the two-phase centrifugation process, which leads to the generation of solid and liquid wastes known as Two Phase Olive Mill Wastewater (TPOMW) (Barranco et al., 2004). Such residue is subjected to several treatments: firstly, bones are separated, and subsequently pomace, olive pomace oil and olive mill wastewater (OMWW) are obtained in an olive oil mill which operates in three phases. The first two substances are value-added products suitable for commercial trading, although the latter product, a highly stable emulsion composed of vegetation water, oil and soft tissues from olive pulp, continues to cause large-scale environmental problems. Nowadays, the most common method for eliminating OMWW is through evaporation in storage ponds, owing to the low investment required and the climate conditions in Mediterranean countries (Borja et al., 2006). However, this operating method has several relevant disadvantages, such as bad odour, infiltration and insect proliferation (Roig et al., 2006). The process also produces a sludge that can be used in agriculture, but its application must be controlled in order to mitigate the increase in soil salinity (López-Piñeiro et al., 2011) and its phytotoxic effects (Magdich et al., 2012). Given that the current treatment methods do not completely solve the associated environmental problems, it is crucial to investigate new waste treatment techniques that may be both effective and economically viable.

An alternative eco-efficient treatment of OMWW, based on an anaerobic treatment preceded by an aerobic pretreatment, is reported in the present manuscript.

The main purposes of anaerobic digestion (AD) of wet waste biomass are the production of energy, the recovery of the water contained in the waste and its potential use for crop

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fertirrigation and the reuse of the solid digested effluent as an organic amendment. However, according to Hamdi (1996), AD of OMWW is limited by the inhibition of methanogenic bacteria by simple phenolic compounds (Hernández and Edyvean, 2011), certain organic acids, long-chain fatty acids (LCFA), (Gonçalves et al., 2012) and polyphenols (Sayadi et al., 2000). The inhibition associated with polyphenols has been extensively studied; however, inhibitory concentrations of these compounds are difficult to be determined given the significant differences with respect to data reported in the literature due to the dispersion of anaerobic communities (different inoculums used). However, it is clear that the effect of the toxicity is related to the molecular structure of polyphenol: the inhibitory effect is found to increase as the number of hydroxyl group decreases (Akassou et al., 2010). For such reason, a pretreatment operation is required, using a physicochemical or biological stage to decrease the organic load and the concentration of potential inhibitors of AD. Among the existing physicochemical treatments, the most prominent are the following: application of electrochemical oxidation (Belaid et al., 2013), catalytic oxidation using Fenton's reagent (El-Gohary et al., 2009) and ozonation (Chedeville et al., 2009). Alternatively, most biological pretreatments use fungus species such as *Pleurotus ostreatus* (Aggelis et al., 2003; Fountoulakis et al., 2002) and *Panus trigrinus* (DÁnnibale et al., 2004) or one of several species of yeasts, such as *Candida oleophila* (Amaral et al., 2012) and *Geotrichum candidum* (Asses et al., 2009). Moreover, several authors have applied this biological pretreatment along with an aeration in order to encourage microorganisms growth, some examples could be the use of white-rot fungus *P. triginus* (DAnnibale et al., 2006) and *P. ostreatus* (Olivier et al., 2012), or another species such as *Aspergillus niger* (Hamdi et al., 1992).

The present work reports on the use of aerobic digestion as a pretreatment operation, without adding any specific species but using the microorganisms that are naturally present in OMWW, since these are acclimated to the inhibitors concentrations that exist in the waste. This technique has been applied by others authors achieving high rates of polyphenols and organic matter degradation (El Hajjouji et al., 2008; Michailides et al., 2011).

The selection of such technology is justified by the fact that it has been proved to be cheap, effective, with low associated maintenance cost and reduced construction costs given that the evaporation ponds used in the olive oil mill industry to store the OMWW can be reused.

Before the AD experiments with OMWW were carried out, previous aerobic tests were performed in order to determine the aeration time that achieves maximum reduction in polyphenols concentration of the wastes. Next, a set of AD experiments were conducted – using OMWW as substrate with and without an aerobic pretreatment stage – in order to analyze the influence of aeration time in the anaerobic process as well as to look for the aeration period which best works from both the environmental and energy perspectives. The economic viability of the construction of an industrial-scaled AD plant to treat aerated OWM is finally assessed.

## 2. Materials and methods

### 2.1. Experimental setup for aerobic pretreatment

Fig. 1 shows the main components of the aerobic reactor. It comprises a glass flask of 2 L operating volume, the reactor

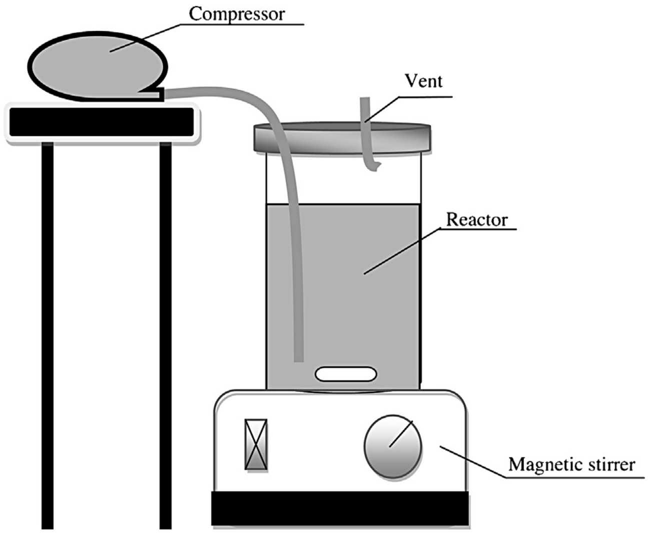


**Fig. 1 – Schematic diagram of an experimental setup used for aerobic pretreatment experiments.**

contents of which were homogenized using a magnetic stirrer. Air from a compressor was injected from the bottom of the reactor at a flow rate of 0.65 L air per litre of reactor and per minute. The temperature of the fermentation was not regulated.

The aeration process was conducted in discontinuous mode (in batch) without adding any specific aerobic bacterium, given that air injection activates the growth of aerobic microorganisms already present in the OMWW samples.

### 2.2. Experimental setup for anaerobic digestion

The anaerobic digestion experiments were carried out in a digester operating in semicontinuous mode; that is, a given volume of digested sludge was extracted daily from the reactor with a syringe and immediately the same volume of substrate was introduced, so that digestion at constant-volume configuration was guaranteed.

A scheme of the experimental setup is shown in Fig. 2, which essentially consists of a 2 L capacity glass flask, whose rim is attached to a central tube immersed in the reaction medium, and which has an input hole to introduce the substrate and an output hole to collect the biogas generated in the process. The digestion unit was submerged in a water tank and maintained at 38 °C by a thermostat equipped with a heating resistor. The substrate inside the reactor was homogenized by the continuous action of a magnetic stirrer, allowing for complete mixing.

A 5 L tank assembled to the biodigester was used to determine the volume of methane generated during the AD process. A squeeze bottle containing a sodium hydroxide solution (20% by weight) was placed between the digester and the gas tank, aimed at retaining the carbon dioxide generated during the digestion process. The methane volumes generated during the experiment displaced water in the tank, which was collected in a measuring cylinder. This way, the volume of displaced water allowed for the determination of the volume of methane generated in each experiment.

### 2.3. Preparation of substrate and start up of the anaerobic digestion process

OMWW, obtained through the treatment of TPOMW in an olive oil mill which operates in three phases, was periodically

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