

# Performance and kinetic evaluation of the anaerobic digestion of two-phase olive mill effluents in reactors with suspended and immobilized biomass

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## Abstract

A lab-scale study was conducted on the mesophilic anaerobic digestion of two-phase olive mill effluents constituted by the mixture of the wash waters derived from the initial cleansing of the olives and those obtained in the washing and purification of virgin olive oil. The digestion was conducted in two continuously stirred tank reactors, one with biomass immobilized on Bentonite (reactor B) and other with suspended biomass used as control (reactor C). The reactors B and C operated satisfactorily between hydraulic retention times of 25.0 and 4.0 days and 25.0 and 5.0 days, respectively. Total chemical oxygen demand (TCOD) efficiencies in the ranges of 88.8–72.1% and 87.9–71.2% were achieved in the reactors with immobilized and suspended biomass, respectively, at organic loading rates of between 0.86 and 5.38 g TCOD/l/d and 0.86–4.30 g TCOD/l/d, respectively. On comparing both reactors for the same OLRs applied, it was observed that the reactor with support was always more efficient and stable showing higher TCOD, SCOD removal efficiencies and lower VFA/alkalinity ratio values than those found in the control reactor. A mass (TCOD) balance around the reactors allowed the methane yield coefficient,  $Y_{G/S}$ , to be obtained, which gave values of 0.31 and 0.301 CH<sub>4</sub>/g TCOD<sub>removed</sub> for reactors B and C, respectively. The cell maintenance coefficients,  $k_m$ , obtained by means of this balance were found to be 0.0024 and 0.0036 g TCOD<sub>removed</sub>/g VSS d, respectively. The volumetric methane production rates correlated with the biodegradable TCOD concentration through an equation of the Michaelis–Menten type for the two reactors studied. This proposed model predicted the behavior of the reactors very accurately showing deviations lower than 10% between the experimental and theoretical values of methane production rates.

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**Keywords:** Two-phase olive mill effluents; Anaerobic digestion; Mesophilic temperature; Kinetics; Performance

## 1. Introduction

Olive oil extraction is among the most traditional agriculture industries in Spain and it has always been, and is still, of primary importance for the economy of this country, as it has 30% of the world's oil production.

The traditional three-phase manufacturing process of olive oil usually yields an oily phase (20%), a solid residue (30%) and an aqueous phase (50%), the latter arising from the water content of the fruit. Such water, combined with that used to wash and process the olives, make up the so-called “olive mill wastewater” (OMW) and also contains soft tissues from olive pulp and a very stable oil emulsion [1]. This three-phase process generates a total volume of traditional OMW of around 1.25 l/kg of olive processed.

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During the last 10 years the manufacture of olive oil has undergone important evolutionary changes in the equipment used for the separation of the olive oil from the remaining components. The latest development has been the introduction of a two-phase centrifugation process in which a horizontally mounted centrifuge is used for a primary separation of the olive oil fraction from the vegetable solid material and vegetation water. The oil then requires washing, to remove residual impurities, before finally being separated from these wash waters in a vertical centrifuge. Therefore, the new two-phase olive oil mills produce three identifiable and separate waste streams compared to two streams in earlier three-phase mills. These are the wash waters from initial cleansing of the fruit; the wash waters from the secondary centrifuge and the aqueous solid residues from the primary centrifugation. The number of plants being installed taking advantage of the new technology is increasing exponentially [2]. As well as offering process advantages they also reduce the water consumption of the mill. The introduction of this technology was carried out in the 90% of the Spanish olive oil factories [3]. Therefore, the new two-phase olive mill effluents (TPOME) constitute the mixture of the wash waters generated during the initial washing of olives (effluent 1) and the wash waters from the secondary centrifuge generated during the purification of virgin olive oil (effluent 2), the total volume of TPOME generated being around 0.251/kg of olives processed.

Previous research works have shown the suitability of anaerobic digestion process for separate treatment of these individual liquid effluent streams. A study of anaerobic digestion process of wastewaters from the washing of olives prior to the oil production process (effluent 1) carried out in a fluidised bed reactor, with biomass immobilized on sepiolite, showed TCOD removal efficiencies in the range of 50–90% when evaluated at organic loading rates (OLR) in the range of 0.46–2.25 g TCOD/l/d and using influent substrate concentrations of 4.5 g TCOD/l/d [4]. A similar reactor configuration was used for mesophilic anaerobic treatment of wastewaters from washing of virgin olive oil with a TCOD content of 3.5 g/l [5]. The bioreactor worked satisfactorily for HRTs in the range of 1.1–5.0 d, and eliminated more than 92% of the initial TCOD in all instances [5]. A laboratory-scale hybrid anaerobic reactor, the bottom third of which was occupied by a sludge blanket, the upper two-thirds by submerged clay rings, was also used for treatment of wash waters derived from the purification of olive oil (effluent 2) [6]. The reactor operated under mesophilic conditions at different substrate concentrations and HRTs in the range from 0.20 to 1.02 days. TCOD removal efficiencies of more than 89% were achieved at an OLR of 8.0 g TCOD/l/d. OLR was gradually increased from 2.6 to 7.1 g TCOD/l/d within 16 days, but the anaerobic reactor

performance did not change significantly. This hybrid reactor was operated under varying influent TCOD concentrations to monitor the response of the system to both high and low strength wash waters. The system could tolerate OLRs as high as 17.8 g TCOD/l/d with an average TCOD removal efficiency of 76.2% [6].

Considering the changes in the characteristics of the new TPOME compared to traditional three-phase OMW and previous experiments dealing with the anaerobic treatment of each one of the above-mentioned effluents separately, the aim of this work was to carry out an evaluation of the performance and kinetics of the anaerobic digestion process of the final TPOME, constituted by the mixture of the wash waters derived from the initial cleansing of the olives and those obtained in the washing and purification of virgin olive oil, both generated by the new two-phase olive oil manufacturing process. This study was carried out in laboratory-scale suspended and immobilized cell bioreactors operating in continuous mode.

## 2. Materials and methods

### 2.1. Equipment

Two anaerobic reactors with a working volume of 1 l equipped with magnetic stirrings at  $160 \text{ min}^{-1}$  and placed in a thermostatic chamber at  $35^\circ\text{C}$  were used. The reactors had an upper settling zone designed to minimize active biomass losses. The reactors were fed daily by means of external feeders and liquid effluents were removed on a daily basis through hydraulic seals, comprising 25 cm liquid columns, designed to prevent air from entering the reactors and biogas from leaving. These reactors have been described in detail elsewhere [7].

The methane volume produced in the process was measured using 4 l Mariotte reservoirs fitted to the reactors. Tightly closed bubblers containing a NaOH solution (3 M) to collect the  $\text{CO}_2$  produced in the process were intercalated between the two elements. The methane produced displaced a given volume of water from the reservoirs, allowing ready determination of the gas [7].

### 2.2. Inoculum

The reactors were inoculated with methanogenically active biomass obtained from a full-scale anaerobic digester treating brewery wastewater. The characteristics and features of the anaerobic sludge used were: total solids (TS), 38.5 g/l; mineral solids (MS), 13.6 g/l; volatile solids (VS), 24.9 g/l; total suspended solids (TSS), 33.8 g/l; mineral suspended solids (MSS), 10.4 g/l; and volatile suspended solids (VSS), 23.4 g/l. These

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