

Anaerobic co-digestion of olive oil pomace as a strategy for bioenergy production in the MED region

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Keywords: Anaerobic co-digestion, olive oil mill wastes, bioenergy, circular economy

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1. INTRODUCTION

Climate change and the increasing energy demand are drivers towards renewable energy sources that can contribute for a decarbonized economy. Furthermore, in a circular economy context, strategies for agro-industrial waste treatment should aim at bioenergy recovery, extraction of added-value compounds (e.g. phenolic compounds) and treated wastewater recycling.

Olive oil production is an important agro-industrial activity, with almost 2.2 million tonnes of oil produced in the European Union during 2017/2018 campaign [7]. Environmental concern regarding this activity is related to the fact that it produces large amounts of highly polluting wastes. The solid waste, pomace (OP), contains high organic matter and moisture (60-70% in two-phase system and 30-45% in three-phase mills).

Wastewater treatment plants (WWTP) produce large amounts of sewage sludge (SS) that can be used to recover bioenergy through anaerobic digestion (AD). Nevertheless, its poor biodegradability leads to low methane yields. Therefore, SS should be used in conjunction with a co-substrate with higher soluble organic matter content (co-digested) [1-4]. The optimization of energy recovery through anaerobic co-digestion (AcoD) enhances WWTP environmental performance and reduces its carbon footprint [5-6].

The aim of this work is to assess bioenergy recovery from two-phase olive mill waste through AcoD process, contributing to design a sustainable waste management solution.

2. MATERIAL AND METHODS

Olive oil mill pomace was collected during 2018/2019 campaign from a two-phase mill in Ribatejo (Portugal). Samples were stored at 4°C before characterization and pre-treatment procedures. The OP used in this study had $58.95 \pm 0.07\%$ of TS, $48.3 \pm 0.43\%$ of VS (dry basis), pH of 4.37 ± 0.42 . The OP was pre-treated by hydrolysis under alkaline conditions (0.4% sodium hydroxide solution), using a liquid to dried solid ratio of 1/10 and 24h contact time at room temperature. The liquid extract (LOP) obtained by filtration under vacuum was characterized and used for the feeding mixture.

The Sewage sludge (SS) was collected from WWTP (average flow of 53,000 m³ per day; 211,000 inhabitant's equivalent), located in Lisbon, Portugal. This sewage sludge is a mixture of primary sludge and waste activated sludge (40:60, v:v).

During the reference scenario (T0) SS was the mono-substrate, in co-digestion regime (T1) the feed was 80% SS and 20% LOP according to previous studies by the authors on the optimization of VS/TS, SCOD/TCOD and C/N ratios. Table I shows the physico-chemical characterization of the feeding during T0 and T1.

Table I: Physico-chemical characterization of the feeding during T0 and T1.

| | T0 | T1 |
|--|---------------|--------------|
| TS (gL ⁻¹) | 18.46 ± 2.04 | 19.2 ± 1.8 |
| VS (gL ⁻¹) | 13.51 ± 1.52 | 16.03 ± 1.11 |
| VS/TS (%) | 73 | 83 |
| pH | 6.70 ± 0.4 | 7.3 ± 0.37 |
| EC (mS.cm ⁻¹) | 1.51 ± 0.35 | 2.01 ± 0.61 |
| TCOD (g O ₂ L ⁻¹) | 23.23 ± 3.144 | 29.16 ± 1.35 |
| SCOD (g O ₂ L ⁻¹) | 1.18 ± 2.13 | 3.06 ± 0.11 |
| SCOD/COD (%) | 5 | 11 |
| TKN (gL ⁻¹) | 1.28 ± 0.21 | 1.02 ± 0.18 |
| C/N | 6 | 9 |

The chemical composition of the different materials used along the trials and AcoD process performance and stability was monitored based on the following parameters: pH, electrical conductivity (EC), total solids (TS), volatile solids (VS), volatile suspended solids (VSS), total chemical oxygen demand (TCOD), soluble chemical

oxygen demand (SCOD), Kjeldahl nitrogen (NK), and ammonia nitrogen (N-NH_4^+). These parameters were analysed according to the standard methods [8]. Total alkalinity (TA) was measured according to Forgács et al. [9]. Total organic carbon was determined according to Cuetos et al. [10].

AD trials were performed in a continuous stirring tank reactor (CSTR) with 11.3 L of working volume under mesophilic regime ($36 \pm 1^\circ\text{C}$). The acclimation period lasted 68 days using diluted SS. After start-up phase two different trials were developed: mono-digestion (T0) and co-digestion (T1), both with a hydraulic retention time (HRT) of 17 days. During T0 the organic loading rate (OLR) was $0.8 \pm 0.23 \text{ g}_{\text{VS}}\text{L}_{\text{Reactor}}^{-1}\text{day}^{-1}$ and for T1 the OLR was $0.94 \pm 0.12 \text{ g}_{\text{VS}}\text{L}_{\text{Reactor}}^{-1}\text{day}^{-1}$. The AD performance was accessed based on several operational parameters, such as the biogas production rate (BPR, $\text{mL}_{\text{biogas}}\text{L}_{\text{reactor}}\text{day}^{-1}$), methane content and specific methane yield (SMY, $\text{mLCH}_4 \text{ g}_{\text{VS}_{\text{added}}}^{-1}$), as well as on the efficiencies of VS removal. BPR was measured daily using a gas meter (Schlumberger, Germany) and biogas composition in terms of methane (CH_4) and carbon dioxide (CO_2) was monitored weekly. The stability of CSTR along trials was controlled by TA, digestate pH and the specific energy loading rate (SELR, day^{-1}).

3. RESULTS AND DISCUSSION

Performance parameters clearly illustrate the improvement in bioconversion during co-digestion (Table II). For example, BPR increased 59% during T1. Even more relevant is the 35% increase found in SMY, despite the 19% increase in OLR. This indicates that the bioconversion of VS to methane was more efficient. Probably this is due to improved availability of organic matter as shown by the SCOD/COD that is twice the value obtained during T0 (Table I). This aspect is very relevant as the reduced solubilisation degree of organic matter of SS is responsible for the low methane yields generally achieved. C/N is also 1.5 times higher than for T0 what also contributes to a more balanced process. Nevertheless, TA showed a slight decrease during T1 what may indicate that volatile fatty acids are accumulating and therefore alkalinity is being consumed for their neutralisation. However, during T1 average pH value of the digestate was 7.16 ± 0.07 indicating reactor stability.

Table II: Performance parameters during T0 and T1.

| Trial | T0 | T1 |
|--|-----------------|-----------------|
| OLR ($\text{g}_{\text{VS}} \text{L}_{\text{reactor}}^{-1}\text{day}^{-1}$) | 0.80 ± 0.23 | 0.94 ± 0.12 |
| BPR ($\text{mL}_{\text{reactor}}^{-1}\text{day}^{-1}$) | 596 ± 42 | 941 ± 77 |
| Methane content (% CH_4) | 63.5 ± 0.1 | 64.0 ± 0.2 |
| SMY ($\text{mL CH}_4 \text{ g}_{\text{VS}_{\text{added}}}^{-1}$) | 474 ± 29 | 638 ± 31 |
| SELR (day^{-1}) | 0.138 | 0.186 |
| TA ($\text{mg CaCO}_3\text{L}^{-1}$) | 3200 ± 95 | 2762 ± 133 |

BPR-biogas production rate; OLR-Organic loading rate;
SMY-Specific methane yield

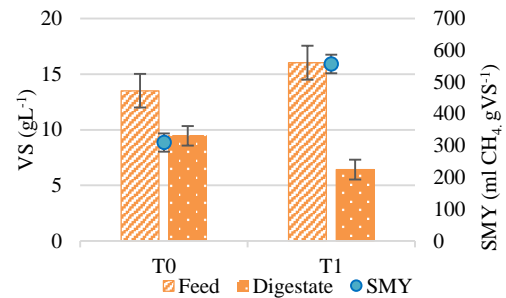


Figure 1: VS removal and SMY during T0 and T1.

During T0 the average VS removal was 30% while T1 led to 60% removal. The SMY obtained in T1 is considerably higher than the methane yields obtained by Rincón et al. [11] in biochemical methane potential (BMP) tests with pre-treated OP ($392 \pm 14 \text{ mL CH}_4 \text{ g}_{\text{VS}_{\text{added}}}^{-1}$). Serrano et al. [12] developed a semi-continuous AD process using thermally pre-treated and de-phenolized OP and for an OLR of $2 \text{ g}_{\text{VS}}\text{L}_{\text{reactor}}^{-1}\text{day}^{-1}$ obtained a SMY of $119 \pm 30 \text{ mL CH}_4 \text{ g}_{\text{VS}_{\text{added}}}^{-1}$. When they lowered the OLR to $1 \text{ g}_{\text{VS}}\text{L}_{\text{reactor}}^{-1}\text{day}^{-1}$ the SMY improved to $172 \pm 60 \text{ g}_{\text{VS}}\text{L}_{\text{reactor}}^{-1}\text{day}^{-1}$. In this study the OLR for T1 was around 1 (0.94 ± 0.12) $\text{g}_{\text{VS}}\text{L}_{\text{reactor}}^{-1}\text{day}^{-1}$ and the SMY achieved was almost four times higher, indicating an enhancement in the bioconversion process. However, the TA mentioned by Serrano et al. is $4197 \pm 400 \text{ mg CaCO}_3\text{L}^{-1}$ whereas in this study the value is 1.5 times lower. This fact may indicate that alkalinity is being consumed to neutralize volatile fatty acids, so careful monitoring should be performed to avoid reactor failing. As the OP used in this study is not de-phenolized the decrease of TA is probably related to inhibition due to the presence of phenolic compounds. In the mentioned papers AD trials were performed using the solid fraction of the pre-treated OP whereas this study used the liquid fraction (LOP). Given the better yield achieved it can be said that processing OP to obtain a liquid fraction seems to be a recommended management strategy.

4. CONCLUSIONS

The results obtained support the implementation of AcoD of SS and LOP as a strategy towards bioenergy recovery from waste. As LOP's SCOD is 33 times higher than the one from SS, the feeding mixture composition improved and therefore enhanced methane yield. Future studies can use these results to design an integrated solution aiming bioenergy recovery and valorisation of the co-product (digestate).

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