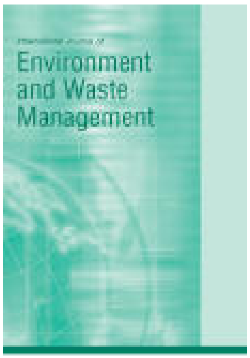



**International Journal of Environment and Waste Management**

ISSN online: 1478-9868 - ISSN print: 1478-9876
https://www.inderscience.com/ijewm

# Batch and semi-continuous anaerobic codigestion of Olive mill wastewaters and diluted poultry manure: a laboratory scale optimal ratio evaluation

Imane Qarraey, Abd-Essamad Moujanni, Aaziz Ouatmane

**DOI:** 10.1504/IJEWM.2024.10061192

**Article History:**

| | |
|---|---|
| Received: | 19 September 2020 |
| Last revised: | 24 May 2021 |
| Accepted: | 29 June 2021 |
| Published online: | 09 January 2024 |

# Batch and semi-continuous anaerobic codigestion of Olive mill wastewaters and diluted poultry manure: a laboratory scale optimal ratio evaluation

Imane Qarraey*, Abd-Essamad Moujanni and Aaziz Ouatmane

Department of Biology, Sciences and Technology,
Faculty Sultane Moulay Slimane University,
Box 523 Beni Mellal, Morocco
Email: imane.ka99@gmail.com
Email: moujanni01@gmail.com
Email: ouatman67@yahoo.fr
*Corresponding author

**Abstract:** The anaerobic codigestion of olive mill wastewater (OMWW) and poultry manure (PM) was investigated in order to determine the appropriate mixture ratio with a focus on total and OMWW organic load contribution. Codigestion was first explored through batch tests using a combination of four PM dry basis ratios (1 to 4%) and four volumetric OMWW ratios (0 to 15%). Thereafter, codigestion was attempted at semi-continuous mode, at batch identified appropriate PM ratio, using a gradual OMWW increase up to 20%. Results showed that the maximum BMP was recorded with PM monodigestion with biogas peak of 410.48 mL/gVS at 5.38 gVS/L. Under the semi-continuous conditions, increasing OMWW fed ratio improved biogas yield up to total feed of 1.57 gVS/L.d achieving a maximum production of 215.85 ± 23.56 mL/gVS.d. The codigestion of OMWW and PM was possible up to a critical OMWW VS contribution of 40% to 50%.

**Keywords:** anaerobic codigestion; biogas; OMWW; optimal mixture; poultry manure.

**Reference** to this paper should be made as follows: Qarraey, I., Moujanni, A-E. and Ouatmane, A. (2024) 'Batch and semi-continuous anaerobic codigestion of Olive mill wastewaters and diluted poultry manure: a laboratory scale optimal ratio evaluation', *Int. J. Environment and Waste Management*, Vol. 33, No. 1, pp.1–13.

**Biographical notes:** Imane Qarraey works as a PhD student with the Bioenergy, Agroressource, and Environment Team, Faculty of Science and Technology, Sultane Moulay Slimane University, Box 523 Beni Mella, Morocco. He received his Bachelor's in Quality Control in the Food Industries in 2013 and Master's in Quality Management in the Agro-Food Industries in 2015 at Sultan Moulay Slimane University, Morocco.

Abd-essamad Moujanni is a PhD Environmental Engineering, Bioenergie, Agroressource, and Environment Team, Science and technology Faculty, Sultane Moulay Slimane University, Box 523 Beni Mella, Morocco. He received his Bachelor's in Sciences and Techniques in Control of Food Industries Quality in 2012 and Master's in Sciences and Techniques in Management of Food Industries Quality in 2014 and finally, he received his PhD in Environmental Engineering in 2020.

Aaziz Ouatmane holds a National Doctorate in Soil Science in 1995 and PhD in Waste Management in 2000 from the Cadi Ayyad University – Marrakech. After joining the Faculty of Science and Technology of Beni Mellal in 1996, he occupied the position of the Head of the Life Sciences Department between 2005 and 2008 and coordinated various applied research projects in environmental sciences and sustainable development. He is particularly interested in the agronomic and energy recovery of waste and effluents from the agri-food industries and is currently the Head of Bioenergy Research team at the same faculty.

## 1 Introduction

Olive growing has a great economical interest and is one of the most highly developed crops in Morocco, with a superficies of 1,050.500 ha in 2016 (Maazouz, 2016). Due to increasing demand of the internal and external markets for olive oil, the olive industry has also undergone significant development in recent decades. During 2017, the national olive oil production was assessed to be140,000 tons. About 5.76% of this production is currently supplied by Beni Mellal region, which focuses on 748 olive Mill plants. The extraction process of olive oil generates tremendous quantities of high phytotoxic OMWW that have a negative impact on the environment, especially soil and water. The most common, cheapest, and most viable system of OMWW management has been its disposal into open evaporation ponds. However, several studies had shown that the long-term storage of OMWW effluents leads to their transformation into partially dry and toxic sediments or sludge, in which potentially toxic compounds are concentrated together in organic matter which becomes more recalcitrant (Kavvadias et al., 2017; El Gnaoui et al., 2020; Sáez et al., 2020).

Although olive wastewater was intensively studied and several treatment procedures including physical, chemical, biological, or combined technologies have been recommended, few processes are currently used at full scale due to economic constraints. Among all the studied treatments, the anaerobic process is regaining consideration by researchers as one of the main promising treatments since it offers the possibility to produce green energy and break down toxicological compounds contained in these wastewaters and could allow environmental safety disposal (Ghanam et al., 2013). Compared to most foods and agro-industrial, OMWW effluents are highly rich in dissolved organic carbon with COD value up to 200 g/L, and are consequently considered as an important and attractive resource of bioenergy (El Gnaoui et al., 2020; Thanos et al., 2020).

However, several factors are well highlighted as severely compromising the performance and stability of the anaerobic digestion of OMWW and are unfavourable to the development of anaerobic bacteria, particularly the high concentration of phenol compounds and the low amount of nitrogen and high C/N ratio (Zarkadas et al., 2019). To overcome recalcitrance and unsuitability of OMWW to anaerobic digestion, many authors recommended codigesting of OMWW with other wastes and nitrogen-rich sources to counterbalance their low nitrogen content (Fezzani and Cheikh, 2010). Usually, anaerobic codigestion is a cost-effective waste treatment method, in which two different types of organic waste are mixed and processed together in a single facility (Alatriste-Mondragón et al., 2006). The reason for using a mixture of two different wastes in the anaerobic codigestion process is to take advantage of the abundance of a specific compound in one type of waste to compensate for its shortage in the other type of waste, and therefore increasing biodegradability and methane production (Alatriste-Mondragón et al., 2006; Moujannı et al., 2018). Anaerobic digestion of crude OMW had lower treatment efficiency in terms of elimination of organic compounds, production of biogas, and process stability, which could be attributed to the toxicity of phenols to bacteria methanogens and lack of nitrogen (Al Afif and Linke, 2019). For these reasons, the codigestion of OMWW has been proposed to improve the performance of anaerobic digestion, by mixing OMWW with other substrates rich in nitrogen, mainly animal manure, and agro-industrial residues. The Codigestion of OMWW and chicken manure are theoretically quite attractive because of the possibilities of nutrient compensation between these two wastes but also of the possible dilution of the toxic constituents, in this case, ammonium and phenol. However, the choice of co-management ratios is generally based on various parameters, either on volumetric ratios, COD load, total VS or simply the proportions of territorial availability of PM and OMWW (Rabii et al., 2019).

The objective of the present study is to determine the appropriate OMWW/PM mixing ratios with a focus on the identification of the corresponding inlet concentration range regarding the main key anaerobic parameters. The study was therefore based on Batch test using a large mixing ratio range and then at semi-continuous mode based on identified suitable start-up and feeding ratios. To allow maximal anaerobic microbial adaptation and organic compound biodegradation, the semi-continuous test was operated at a gradual OMWW increased ratios instead of using separate OMWW ratios tests. Acclimation was also shortened by using inoculum prepared from lab OMWW and landfill leachates codigestion study.

## 2 Materials and methods

### *2.1 Raw material origin and characteristics*

All used wastes in this work were sourced locally. PM material was provided by a small local poultry farm, it consisted mainly of chicken dropping and feathers with moisture content around 37 %. To avoid eventual moisture and biodegradation associated errors, the whole collected quantity of PM wastes was air-dried at lab temperature and then screened to remove gross feathers. After mechanical homogenisation, samples were retired for analysis determination and the Batch experiment, the rest was stored in a sealed plastic sampling bag at 4°C until use for subsequent digestion test (semi-continuous experiment). OMWW was collected from an olive oil production plant using traditional discontinuous three phasic extraction processes. During the day of wastes reception, an aliquot of 500 ml was used for analysis, and the rest was kept at 4 °C and was used for digestions tests. The basic composition and characteristics of the used OMWW and PM are reported in Table 1. Inoculum suspension was taken from a lab experiment of leachates and OMWW codigestion. It presents a neutral pH (7.5) and low volatile solids content (0.79% dry basis).

**Table 1** Physicochemical characteristics of used OMWW and PM raw material

| *Substrate* / *Parameters* | *OMWW* | *PM* |
|---|---|---|
| Water content (%) | 95 ± 0.28 | 37 ± 0.21 |
| Total solids | 83.2 ± 7.80 g/L | 26.34 ± 0.89% (wet basis) |
| pH | 5.16 ± 0.11 | 7.15 ± 0.07 |
| Alkalinity (mg $CaCO_3$/L) | 1587 ± 53.03 | 5524 ± 142.83 |
| Conductivity (ms/cm) | 28.52 ± 1.57 | 7.32 ± 0.04 |
| COD | 96 ± 3.18 g/L | Nd |
| TNK | 0.42 ± 0.07 g/L | 3.22*± 0.15 |
| Ammonium ($NH+_4$) | 0.11 ± 0.06 g/L | 1.54*± 0.33 |
| Total phenols | 8.63 ± 0.26 g/L | Nd |
| Volatile solids (VS) | 79.15 ± 4.70 g/L | 44.4*± 0.17 |

Notes: *% (dry basis), nd : not determined.

### *2.2 Batch codigestion and Biochemical methane potential determination*

To identify appropriate batch codigestion ratios of PM and OMWW, four loads of PM (1%, 2%, 3%, and 4% dry basis) and four volumetric ratios of OMWW (0%, 5%, 10%, and 15%), were used. The anaerobic digestion was accomplished in 1.5-liter bottles with a working volume of 1.3 liters. The combination of each mixture was prepared by diluting the corresponding weigh of air-dried PM and OMWW volume and 100 ml inoculum with deionised water to 1.3 liters. Total and OMWW volatile solids loads were ranging from 3.58 to 46 gVS/L and 3.95 to 11.87 gVS/L respectively. To compensate for the low alkalinity of OMWW, the pH of all mixtures was adjusted to a neutral value (7), and then the reactors were flushed with N2 for 3–4 min to ensure anaerobic conditions, and were incubated at a temperature-controlled room in a water bath at 32°C. To maintain suitable digestion, reactors were stirred 2 min twice each day based on a preliminary test. A blank reactor with only deionised water and inoculum was incubated at the same conditions. Biogas production was measured three days interval at a standard temperature of 0°C. The digestion was maintained for a period of 50 days until no biogas production.

### *2.3 Biogas kinetic assessment*

Kinetic of biogas production in Batch condition was assumed that had correspondence to the specific growth rate of methanogenesis bacteria in digester (Syaichurrozi et al., 2013). The cumulative biogas production for all Batch reactors was then monitored by fitting the experimental data to the Gompertz kinetic model using the following equation (1):

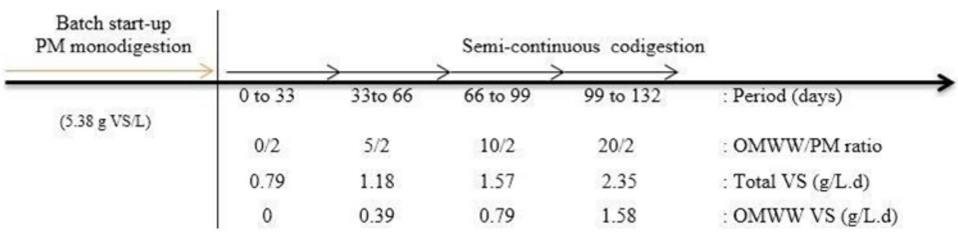
$$P = A\, exp\left\{-exp\left[\mu\, .\, e\, /\, A\right](\lambda - t) + 1\right\}$$

where $A$ represent the biogas production potential (mL/gVS), $\mu$ the maximum biogas production rate (mL/gVS.d) and $\lambda$ the minimum time required to produce the biogas (days).

## 2.4 Semi continuous codigestion experiment

The semi-continuous test was conducted using a lab reactor equipped with an emerged device that serves for both effluent withdrawal and influent feeding. The used reactors had the same total and working volume as for the Batch test. At the first step, the reactor was operated for 33 days at Batch-start-up mode using PM monodigestion at the best organic load based on Batch results, which corresponded to the mass load of 2% PM and 5.38 gVS/L. Thereafter, the operating conditions were changed to the semi-continuous mode during which the reactor was feed first with only 2% PM suspension. After steady-state achievement, the fed was changed to OMWW/PM mixture with a gradual increase of OMWW volumetric ratio from 0% to 20%. Each ratio was run for 33 days digestion period as described in Figure 1. During the digestion period, the reactor was mixed 5 min twice a day at 5 hours interval: a first mixing to enhance digestion and a second one to allow reactor homogenisation during discharging and feeding operation. The hydraulic retention time was set to 20 days with an intermittent feed of 3 days. The temperature was maintained at 37°C using a water bath. The produced biogas was measured by water displacement method at three days interval and the discharged effluents were continuously analysed for pH and volatile fatty acids (VFAs) concentration monitoring.

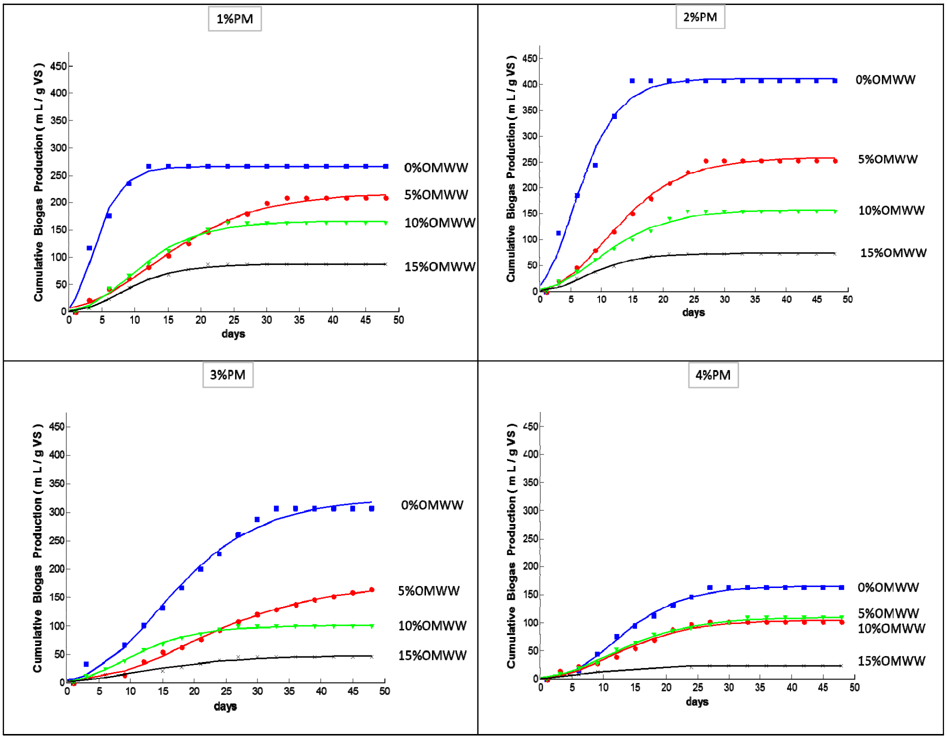
**Figure 1** Semi-continuous codigestion operational conditions




## 2.5 Analytical methods

All the analytical determinations were done according to the Standard Methods for the Examination of Water and Wastewater (Rodier et al., 2009). Water content and total solid were determined by drying samples at 105°C for 24h, Volatile solids content (VS) was determined after drying samples at 105°C for 72h and ignition overnight at 550°C. Total nitrogen (TNK) and ammonium were determined by kjeldhal method (AOAC, 1997). pH and electrical conductivity were determined by Hanna pH metre and conductivity metre. Phenol content was determined using Singleton method and al 1999 quoted by Wolfe et al. (2003) (Rodier et al., 2009) and chemical oxygen demand (COD) was measured by spectrophotometer after a total digestion with $H_2SO_4$ and potassium dichromate at 150°C for 2 h. VFA concentration was determined by titration with 0.1 M sodium hydroxide, and alkalinity was determined by acid titration using 0.02 N of hydrochloric acid (Rodier et al., 2009). Biogas production was measured by water displacement column at atmospheric pressure and 25 ± 3°C controlled room temperature.

**Figure 2** Cumulative biogas production of OMWW/PM ratios where experimental data are given in symbols and simulated plots in line (see online version for colours)



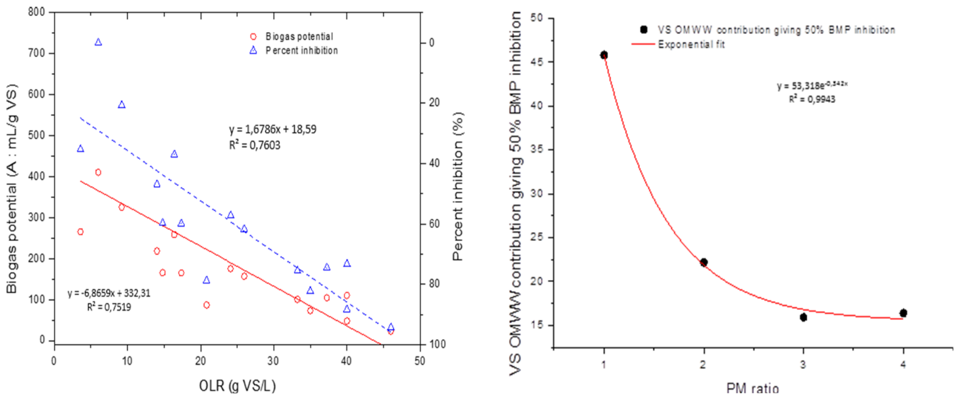

## 3 Results and discussion

### 3.1 Batch optimal mixture ratios

The yields and potential of biogas according to the batch tests and the modified Gompertz equation are illustrated in Figure 2 and Table 2. The experimental and simulated results showed a good fit with strong correlation coefficients. Ratios comparison showed that the highest biogas yield was achieved for PM monodigestion. It was noticed that both the low (1% PM) and high PM load (4% PM) give poor biogas yield. The peak performance was recorded for 2% PM with biogas yield of 410.48 mL/gVS and daily biogas production rate of 36.85 mL/gVS.d. In terms of organic load, appropriate PM monodigestion was possible in the range of 5.38 to 9.19 gVS/L (2 to 3% of PM). The corresponding TNK and ammonium inlet ranges were 0.32 to 1.34g/L and 0.154 to 0.632 g/L, respectively. These results are close to that reported by (Farrow et al., 2017) which found an increase in biogas yield of 30% (470 – 607 L/kgVS) during the batch digestion tests of PM.

For the current study, the decrease in biogas production observed at a high PM load (4% PM), could be attributed to an overload effect and/or to ammonium accumulation which generally represent the main potential inhibitors for the anaerobic digestion of PM as indicated in the literature (Sampaio et al., 2011; Markou, 2015). Even if the overall entry ammonium concentration was kept at a relatively low level, the increase in VS increases the load in protein compounds and this can lead to an accumulation of ammonium and free ammonia up to an inhibitory threshold, as reported by other authors (Khoufi et al., 2015).

**Figure 3** (a) plot of biogas production potential (A), and inhibition % versus initial organic load, and (b) OMWW volatile solids contribution giving 50% bmp inhibition versus pm ratio for the whole batch tests (all OMWW/PM ratios) (see online version for colours)




(a) (b)

Codigestion results showed that the increasing of OMWW volumetric ratio for each PM ratio affect negatively the anaerobic digestion process. Besides, it was noticed that biogas yield decreased with increasing either OMWW or PM ratio. The recorded BMP varied from a maximum of 258.8 at a low mixing ratio to a minimum of 23.38 mL/gVS when combining high ratio. Acceptable yields with a value in the range of 218.21 to 258.8 mL/g VS were possible at the lowest mixing ratios: 1 to 2% PM and 5% OMWW. On the opposite, at high PM ratio, the addition of OMWW even at low proportion resulted in strong inhibition of the codigestion process. Interestingly, the plot of total VS versus BMP showed a significant linear negative correlation regardless of the digestion ratios [Figure 3(a)]. These results suggest that total inlet VS was the main codigestion key parameter and that codigestion inhibition could be attributed mainly to overloading effect and acid accumulations. Similar conclusion was reported by other authors for which the organic load may influence considerably the codigestion process and could led to fast and unsuitable accumulation of acids being the main digestion inhibitor independently of wastes Characteristics (Sampaio et al., 2011; El Gnaoui et al., 2020). Based on maximum BMP, the threshold of 50% inhibition was observed at total VS of 10 to 20 gVS/L depending on PM ratio [Figure 3(b)]: it corresponded to OMWW VS contribution of 45.80%, 22.17%, 16.42%, and 16.37% for 1, 2, 3, and 4% PM ratio respectively as reported in Figure 3b. Therefore, the inhibitory effect of OMWW was clearly amplified by PM load increase, resulting in an obvious negative synergetic effect. These results disagree with those of (Khoufi et al., 2015) according to which codigestion stimulated anaerobic digestion and allowed optimal Batch codigestion condition at 70% OMWW and 30% PM mixing ratio. Similarly (Gannoun et al., 2016) shown that the codigestion of OMWW with SW at different mixing ratios of 40:60 and 50:50 (OMW: SW), improves the methane yield and revealed excellent stability of the digester, even at higher organic charges. The negative effect of OMWW on codigestion was however highlighted by many others studies in good agreement with the current study. The addition of OMWW to cattle manure (Rubio et al., 2019), and to activated sludge (Alrawashdeh and Al-Essa, 2019) caused inhibition of methanogenes and reactor failure. It was also reported that OMWW VS contribution over 40% led to reverse and negative effects and reduce significantly OMWW and PM codigestion performance (Li et al., 2015). The synergetic negative effect between OMWW and PM at high organic load could be attributed to negative interaction involving both overloading effect, phenol and ammonium specific effect. These finding are in agreement with that found by (Astals et al., 2014).

**Table 2** OMWW/PM mixture characteristic and the corresponding Modified Gompertz

| | *Codigestion parameters* | | | | | *Gompertz data* | | |
|---|---|---|---|---|---|---|---|---|
| *PM(%)* | *OMWW (%)* | *Total VS (g/L)* | *inlet TNK (g/L)* | *Inlet ammonium (g/L)* | $R^2$ | *A:mL/gVS* | *μ :mL/gVS.d* | *λ:(Days)* |
| 1 | 0 | 3.58 | 0.32 | 0.154 | 0.998 | 265.56 | 38.69 | 0.70 |
| | 5 | 14.00 | 0.34 | 0.159 | 0.997 | 218.21 | 8.63 | 2.60 |
| | 10 | 17.32 | 0.36 | 0.164 | 0.997 | 164.8 | 10.27 | 2.81 |
| | 15 | 20.75 | 0.38 | 0.17 | 0.994 | 86.95 | 6.65 | 2.64 |
| 2 | 0 | 5.38 | 0.64 | 0.308 | 0.993 | 410.48 | 36.85 | 1.04 |
| | 5 | 16.36 | 0.66 | 0.313 | 0.998 | 258.80 | 13.79 | 3.47 |
| | 10 | 26.00 | 0.68 | 0.318 | 0.996 | 157.0 | 8.77 | 2.21 |
| | 15 | 35.00 | 0.70 | 0.324 | 0.998 | 72.82 | 5.40 | 1.89 |
| 3 | 0 | 9.19 | 0.96 | 0.462 | 0.996 | 325.6 | 12.85 | 4.39 |
| | 5 | 24.14 | 0.98 | 0.467 | 0.998 | 175.53 | 5.261 | 6.09 |
| | 10 | 33.25 | 1.00 | 0.472 | 0.998 | 100.6 | 5.73 | 2.09 |
| | 15 | 40.00 | 1.02 | 0.478 | 0.986 | 47.877 | 1.91 | 1.78 |
| 4 | 0 | 14.76 | 1.28 | 0.616 | 0.998 | 165.24 | 9.59 | 4.76 |
| | 5 | 37.25 | 1.30 | 0.621 | 0.993 | 104.73 | 5.31 | 3.59 |
| | 10 | 40.00 | 1.32 | 0.626 | 0.997 | 109.82 | 5.37 | 3.06 |
| | 15 | 46.00 | 1.34 | 0.632 | 0.995 | 23.381 | 1.66 | 1.71 |

## *3.2 Semi continuous experiment*

### *3.2.1 Biogas yield*

Anaerobic digestion at semi-continuous mode is based on intermittent cyclic discharging and bioloading of processed biomass and raw biomass at a given period. It is commonly more convenient than batch mode since it prevents the accumulation of potential inhibitor and maintains overall favourable conditions for bacterial growth (Park et al., 2018). According to recent work, (Chan et al., 2018), intermittent feeding may improves codigestion performance, particularly through active COD and VFA metabolism. Chicken manure monodigestion, had shown that intermittent feeding at 7 days interval and 10% volume removing/loading ratio was 33.5% more efficient in terms of methane yield compared to Batch process (45 days periodic mode) (Baltrėnas and Kolodynskij, 2020). Moreover, mixing chicken manure with other waste and increasing the amount of newly loaded bioloading from 10% to 15% was recommended. For the current study, the results of the codigestion OMWW and PM in semi-continuous mode using a short intermittent interval are illustrated in Figure 4 and Table 3. During the batch start-up period, the average daily biogas yield was 95.17 mL/L of the reactor. When the operating conditions changed to semi-continuous mode based on an intermittent 3-day supply with 23% of bioloading, the process led to a significant increase in biogas yield. For the monodigestion of PM at an organic load of 0.79 gVS/L.d, the average daily production of biogas gradually increased to reach 125.25 ± 17.18 mL/L.d and 158.54 ± 10.54 mL/gVS.d. Thereafter, when the reactor started to be supplied with an OMWW/PM mixture, the average production of biogas continues to increase by up to 10% of OMWW ratio to reach a stable state at the same time. At 20% ratio feed, the codigestion was destabilised and the production of biogas fell sharply. The biogas yield recorded was 244.40 ± 24.46, 338.89 ± 27.37, and 146.32 ± 16.58 mL/L.d for the mixing ratios of 5/2, 10/2, and 20/2 (OMWW/PM) respectively. In terms of VS load, codigestion had a peak performance at total VS of 1.57 gVS/L.d with strong inhibition at higher load. The maximum biogas recorded was 215.85 ± 23.56 mL/gVS.d and 338.89 ± 27.37 mL/L.d. These results are close to those reported in the literature for semi-continuous codigestion of PM /OMWW. The biogas yield is usually in the order of 0.7 ± 0.4 to 1.2 ± 0.3 L/Lreactor.d (Thanos et al., 2020), which is very comparable to our results. Similar results have also been reported by recent continuous co-management of OMWW and Food waste (FW) for which the optimal mixing ratio was 20% OMWW/80% FW at an organic load of 2.0 ± 0.1 kgVS/m-3.d – 1 (El Gnaoui et al., 2020). These findings suggest that the success of PM/OMWW codigestion could be limited to low organic load and low volume ratios of OMWW. However, a critical review of the available literature revealed that codigestion of OMWW with different types of nitrogen-rich wastes, including PM (Khoufi et al., 2015), activated sludge (Alrawashdeh and Al-Essa, 2019), and swine manure (Azaizeh and Jadoun, 2010) was also possible at very high loads. However, although increasing OLR can increase biogas yield, it has been reported to have a negative effect and decrease digestion performance due to overload and accumulation of acids (Serrano et al., 2019). Under continuous and semi- continuous supply, reactor failure is also very likely at 2 gVS/L.d as critical load (Serrano et al., 2019) that is in good agreement with our work.

Dealing the anaerobic codigestion of PM and OMWW which are potentially unfavourable waste, it seems that both the question of the volumetric mixing ratios, the OLR and the specific contribution of OMWW volatile solids are key parameters. By focusing on the specific effect of OMWW, the current study showed that peak performance corresponds to a volumetric ratio of 10% and an OMWW VS contribution of 50% at an organic load of 1.57 gVS/L.d. These results are in good agreement with those of (Gelegenis et al., 2007), according to which the production of biogas was slightly higher when OMW was added to the diluted PM up to a critical contribution OMWW VS of 40%. Higher volumetric ratios have recently been reported in the case of PM/OMWW codigestion (Khoufi et al., 2015). They stated that optimal codigestion in the semi-continuous jet loop reactor was achieved at a volumetric ratio of 70% OMWW/30% PM at an organic load of 9.5 gCOD/L.d and 1: 1 VS.

Semi-continuous and batch tests comparison showed that the change in the operational conditions in semi-continuous mode had a positive effect on the anaerobic codigestion of PM/OMWW. The addition of OMWW up to 10% at a PM rate of 2% improves biogas production, contrary to the batch condition. This has been attributed to the effect of the discharge and dilution of potentially produced inhibitory metabolites, particularly VFAs, as reported by (Gonçalves et al., 2012). At the same time, the failure of digestion observed at a 20% ratio confirmed the fact that the codigestion process remains very sensitive to the addition of OMWW and that the appropriate conditions were also limited to a narrow range of ratios of OMWW/PM mixture as for batch mode.

**Figure 4** Daily biogas production evolution during semi-continuous anaerobic OMWW/PM codigestion with gradual OLR increase

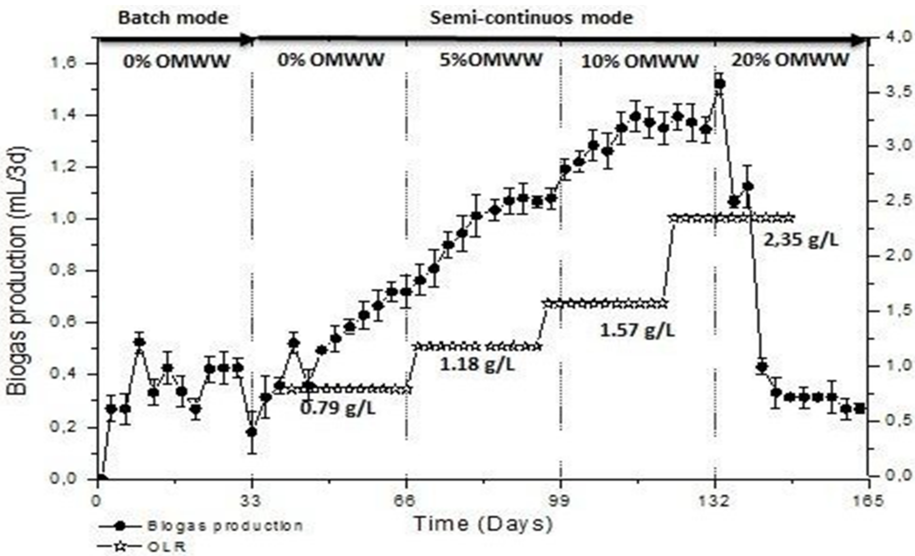


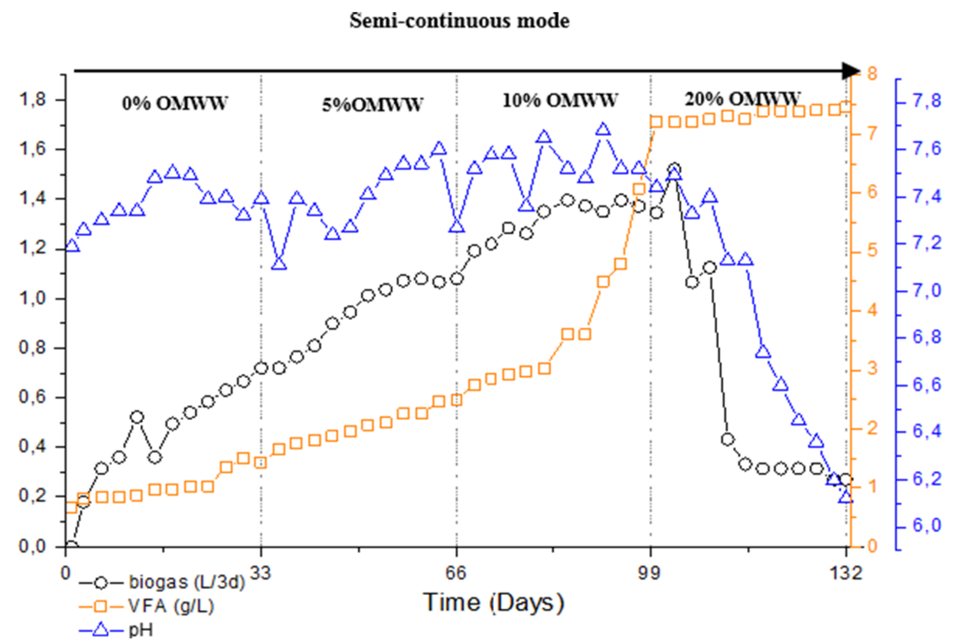

**Table 3** Biogas yield during semi-continuous experiment

| *PM/OMWW ratio* | *OLR gVS/L.d* | *OMWW VS contribution (%)* | *Average biogas yield mL/L.d* | *Average biogas productionmL/gVS $_{feeding}$.d* |
|---|---|---|---|---|
| 2/0 | 0.79 | 0 | 125.25 ± 17.18 | 158.54 ± 10.45 |
| 2/5 | 1.45 | 33.05 | 244.40 ±24.46 | 207.85 ± 20.48 |
| 2/10 | 1.95 | 50.31 | 338.89 ± 27.37 | 215.85 ± 23.56 |
| 2/20 | 3.79 | 67.23 | 146.32 ± 16.58 | 62.26 ± 7.42 |

### *3.2.2 Volatile fatty acids and pH*

VFA and pH in an anaerobic digester are generally considered to be key indicators of the progress and stability of anaerobic digestion. They are also easy to operate on a pilot scale and at the same time allow adequate monitoring. The determination of the VFAs and the pH of the outlet effluent is shown in Figure 5. Continuous pH control is important to monitor the stability of the reactor. Fermentative bacteria can function effectively over a wide pH range, that is, ie between pH 4.0 and 8.0 while methanogenic bacteria are functionally active in a pH range of 6.5 to 7.5. Low pH favours the production of volatile fatty acids, while higher pH favours the ammonia production (Kumar and Samadder, 2020). From Figure 5 the pH was relatively stable at the first stage of digestion and for a low OMWW ratio, but a slight decrease was observed when the reactor started to be supplied with 20% OMWW. The concentration of VFAs showed the same overall trend with a significant increase as the OMWW ratio increased, this can be explained by the low biodegradability of the mixture and the increasing of OLR, which providing growing conditions unfavourable to methanogenic archaea. Therefore, these results showed that the semi-continuous conditions allowed co-digestion at a relatively higher ratio of OMWW and this could be attributed to the continuous dilution of the VFAs and ammonium produced.

**Figure 5** Biogas production, pH effluent and total VFA concentrations during the semi-continuous codigestion (see online version for colours)




## 4 Conclusions

The anaerobic codigestion of OMWW and PM was studied in batch and semi-continuous mode. in the batch test, the best biogas yield was recorded for the monodigestion of PM at 2%. This charge was tested in semi-continuous mode with a gradual increase in OMWW volumetric ratio. The results prove that the optimal anaerobic codigestion could be achieved up to 10% OMWW volumetric ratio and overall loading rate of 1.57 gVS/L.d with OMWW organic load contribution up to 50%. This work led also to the conclusion that both PM and OMWW are potentials inhibitors and that their mixture may lead to synergetic negative effect at relatively low load and that particular caution should be accorded to both total and specific VS contribution for PM and OMWW anaerobic codigestion.

## References

Al Afif, R. and Linke, B. (2019) 'Biogas production from three-phase olive mill solid waste in lab-scale continuously stirred tank reactor', *Energy*, Vol.171, pp.1046–1052.

Alatriste-Mondragón, F. et al. (2006) 'Anaerobic codigestion of municipal, farm, and industrial organic wastes: a survey of recent literature', *Water Environment Research*, Vol.78, No. 6, pp.607–636.

Alrawashdeh, K.A.B. and Al-Essa, A.H. (2019) 'Anaerobic Co-digestion mill wastewater–activated sludge effect of aerobic pretreatment on the performance of omw anaerobic digestion', *Waste and Biomass Valorization*, Vol. 11, No. 9, pp.4781–4788.

AOAC (1997) 'Standard methods for the examination of water and wastewater'.

Astals, S. et al. (2014) 'Identification of synergistic impacts during anaerobic co-digestion of organic wastes', *Bioresource Technology*, Vol. 169, pp.421–427.

Azaizeh, H. and Jadoun, J. (2010) 'Co-digestion of olive mill wastewater and swine manure using up-flow anaerobic sludge blanket reactor for biogas production', *Journal of Water Resource and Protection*, Vol. 2, No. 4, p.314.

Baltrėnas, P. and Kolodynskij, V. (2020) 'Experimental research of efficiency of semi-continuous and periodic biogas production processes by using chicken manure bioloadings', *Water Environment Research*, Vol. 92, No. 5, pp.722–730.

Chan, P.C., de Toledo, R.A. and Shim, H. (2018) 'Anaerobic co-digestion of food waste and domestic wastewater – Effect of intermittent feeding on short and long chain fatty acids accumulation', *Renewable Energy*, Vol. 124, pp.129–135.

El Gnaoui, Y. et al. (2020) 'Anaerobic co-digestion assessment of olive mill wastewater and food waste: Effect of mixture ratio on methane production and process stability', *Journal of Environmental Chemical Engineering*, Vol. 8, No. 4, p.103874.

Farrow, C. et al. (2017) 'Anaerobic digestion of poultry manure: process optimization employing struvite precipitation and novel digestion technologies', *Environmental Progress and Sustainable Energy*, Vol. 36, No. 1, pp.73–82.

Fezzani, B. and Cheikh, R.B. (2010) 'Two-phase anaerobic co-digestion of olive mill wastes in semi-continuous digesters at mesophilic temperature', *Bioresource Technology*, Vol. 101, No. 6, pp.1628–1634.

Gannoun, H. et al. (2016) 'Microbial community structure associated with the high loading anaerobic codigestion of olive mill and abattoir wastewaters', *Bioresource Technology*, Vol. 201, pp.337–346.

Gelegenis, J. et al. (2007) 'Optimization of biogas production from olive-oil mill wastewater, by codigesting with diluted poultry-manure', *Applied Energy*, Vol. 84, No. 6, pp.646–663.

Ghanam, J. et al. (2013) 'Response of anaerobic consortium populations to carbon / nitrogen ( c / n ) variation during anaerobic digestion of olive mill wastewater', *Journal of Biotechnology Letters*, Vol. 4, No. 1, pp.72–78.

Gonçalves, M.R., Freitas, P. and Marques, I.P. (2012) 'Bioenergy recovery from olive mill effluent in a hybrid reactor', *Biomass and Bioenergy*, Vol. 39, pp.253–260.

Kavvadias, V. et al. (2017) 'Fate of potential contaminants due to disposal of olive mill wastewaters in unprotected evaporation ponds', *Bulletin of Environmental Contamination and Toxicology*, Vol. 98, No. 3, pp.323–330.

Khoufi, S., Louhichi, A. and Sayadi, S. (2015) 'Optimization of anaerobic co-digestion of olive mill wastewater and liquid poultry manure in batch condition and semi-continuous jet-loop reactor', *Bioresource Technology*, Vol. 182, pp.67–74.

Kumar, A. and Samadder, S.R. (2020) 'Performance evaluation of anaerobic digestion technology for energy recovery from organic fraction of municipal solid waste: a review', *Energy*, Vol. 197, p.117253.

Li, K., Liu, R. and Sun, C. (2015) 'Comparison of anaerobic digestion characteristics and kinetics of four livestock manures with different substrate concentrations', *Bioresource Technology*, Vol. 198, pp.133–140.

Maazouz, S. (2016) *Le Prix Du Litre D'huile D'olive Est Passé À 45 DH* [online] https://www.agrimaroc.ma/les-faits-marquants-du-5e-forum-international-de-l-agro-pole-olivier/.

Markou, G. (2015) 'Improved anaerobic digestion performance and biogas production from poultry litter after lowering its nitrogen content', *Bioresource Technology*, Vol. 196, pp.726–730.

Moujannı, A., Qarraey, I. and Ouatmane, A. (2018) 'Anaerobic codigestion of urban solid waste fresh leachate and domestic wastewaters: biogas production potential and kinetic', *Environmental Engineering Research*, Vol. 24, No. 1, pp.38–44.

Park, J. H. et al. (2018) 'Effect of feeding mode and dilution on the performance and microbial community population in anaerobic digestion of food waste', *Bioresource Technology*, Vol. 248, pp.134–140.

Rabii, A. et al. (2019) 'A review on anaerobic co-digestion with a focus on the microbial populations and the effect of multi-stage digester configuration', *Energies*, Vol. 12, No. 6, p.1106.

Rodier, J. et al. (2009) *L'analyse De L'eau-9e* éd., *Eaux Naturelles, Eaux Résiduaires*, Eau De Mer, Dunod.

Rubio, J.A. et al. (2019) 'Mesophilic anaerobic co-digestion of olive-mill waste with cattle manure: effects of mixture ratio', *Frontiers in Sustainable Food Systems*, Vol. 3, p.9.

Sáez, J.A. et al. (2020) 'Olive mill wastewater-evaporation ponds long term stored: integrated assessment of in situ bioremediation strategies based on composting and vermicomposting', *Journal of Hazardous Materials*, Vol. 402, p.123481.

Sampaio, M.A., Gonçalves, M.R. and Marques, I.P. (2011) 'Anaerobic digestion challenge of raw olive mill wastewater', *Bioresource Technology*, Vol. 102 No. 23, pp.10810–10818.

Serrano, A. et al. (2019) 'Performance evaluation of mesophilic semi-continuous anaerobic digestion of high- temperature thermally pre-treated olive mill solid waste', *Waste Management*, Vol. 87, pp.250–257.

Syaichurrozi, I., Budiyono and Sumardiono, S. (2013) 'Predicting kinetic model of biogas production and biodegradability organic materials: biogas production from vinasse at variation of COD/N ratio', *Bioresource Technology*, Vol. 149, pp.390–397.

Thanos, D. et al. (2020) 'Enhanced biogas production in pilot digesters treating a mixture of olive mill wastewater and agro-industrial or agro-livestock by-products in Greece', *Waste and Biomass Valorization*, Vol. 11, pp.1–9.

Zarkadas, I.S. et al. (2019) 'anaerobic digestion of olive mill wastewater: focusing on the effect of nitrogen source', *Clean - Soil, Air, Water*, Vol. 47, No. 3, p.1800300.

## Nomenclature

| | |
|---|---|
| BMP | Biochemical methane potential |
| OMWW | Olive mill wastewater |
| OLR | Organic loading rate |
| PM | Poultry manure |
| VFAs | Volatile fatty acids |
| VS | Volatile solids |