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Review

From laboratory- to industrial-scale plants: Future of anaerobic digestion of olive mill solid wastes

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HIGHLIGHTS

- Scale-up of anaerobic digestion (AD) with olive mill solid waste (OMSW) is reviewed.
- The feasibility of pretreatment processes were compared at different operating scales.
- Techno-economic challenges are associated with the scaling-up of mono-AD of OMSW.
- CoAD with manure could improve year-round process stability in larger-scale plants.

GRAPHICAL ABSTRACT

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ABSTRACT

In this review, the main properties of olive mill solid waste, the primary by-product of olive oil production, and its feasibility as a feedstock for anaerobic digesters operating at laboratory-, pilot- and industrial-scales are discussed in detail. Nutrient addition and thermal pretreatments were found to have the potential to address the challenges arising from the high carbon-to-nitrogen ratio, the low pH, and the high concentration of phenolic compounds. Furthermore, anaerobic co-digestion with different organic feedstocks has been identified as one of the most promising options to solve the aforementioned problems and the seasonality nature of olive waste, while improving the efficiency of anaerobic treatment plants that operate throughout the whole year. The insights generated from this study show co-digestion with wastes from animal farming to be the most environmentally and economically sustainable method for improving anaerobic digestion processes with olive mill solid waste.

1. Introduction

According to the most recent estimations, the worldwide production of olives stands at about 23 million tons per year, of which about 20 million tons are used in the production of olive oil, and the remaining 3 million tons are allocated to the production of table olives (Di Giacomo and Romano, 2022). With 69 % of the world's production, the European Union is the largest producer of olive oil, with most of its production

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being concentrated in 4 member states, namely Spain, Italy, Greece, and Portugal (Di Giacomo and Romano, 2022; Directorate-General for Agriculture and Rural Development, 2022). The olive oil manufacturing process is marked by notable quantities of residues within a relatively short period of time (October – March), which must be managed efficiently from both economic and environmental perspectives (Aliakbarian et al., 2011; Christoforou and Fokaides, 2016). Depending on the technology employed for olive oil extraction, the quantity and chemical composition of solid and fluid residues produced are different. Over the past few decades, most olive mills have used three-phase (3P) and two-phase (2P) centrifugal procedures. At the conclusion of each process, the systems produce three distinct fractions: a solid residue called olive cake, olive pomace (OP), or olive mill solid waste (OMSW), the oil, and a liquid fraction called olive mill wastewater (OMWW). During the 3P extraction, a large amount of water is added throughout the centrifugation process, resulting in a huge volume of wastewater (Fig. 1(a)). The failure of the development of an efficacious and cost-effective approach for OMWW treatment has led olive mill operators to prefer the "eco-friendly" 2P method, avoiding the introduction of additional water during the centrifugation steps (Manthos et al., 2023). However, the amount of OMSW from the 2P process is estimated to be approximately four-fold the amount of oil generated. As can be seen in Fig. 1(a), which schematically compares the 3P and 2P centrifugation processes, around 900 kg of OMSW per ton of processed olives result from 2P system utilization (Alburquerque et al., 2004; Cappelletti et al., 2014). Particular interest in the 2P method has been shown in areas where water resources are limited and/or wastewater discharge needs to be minimized. This will support the achievement of the Sustainable Development Goal (SDG) target 6 on improving water quality by reducing pollution, eliminating dumping, and minimizing the release of hazardous chemicals and materials. Moreover, the 2P centrifuge is less complex, and it has reduced energy consumption and operating costs (Borja et al., 2006). Recently, the results obtained from the work of Restuccia et al. showed that the 2P extraction process is more sustainable than the 3P

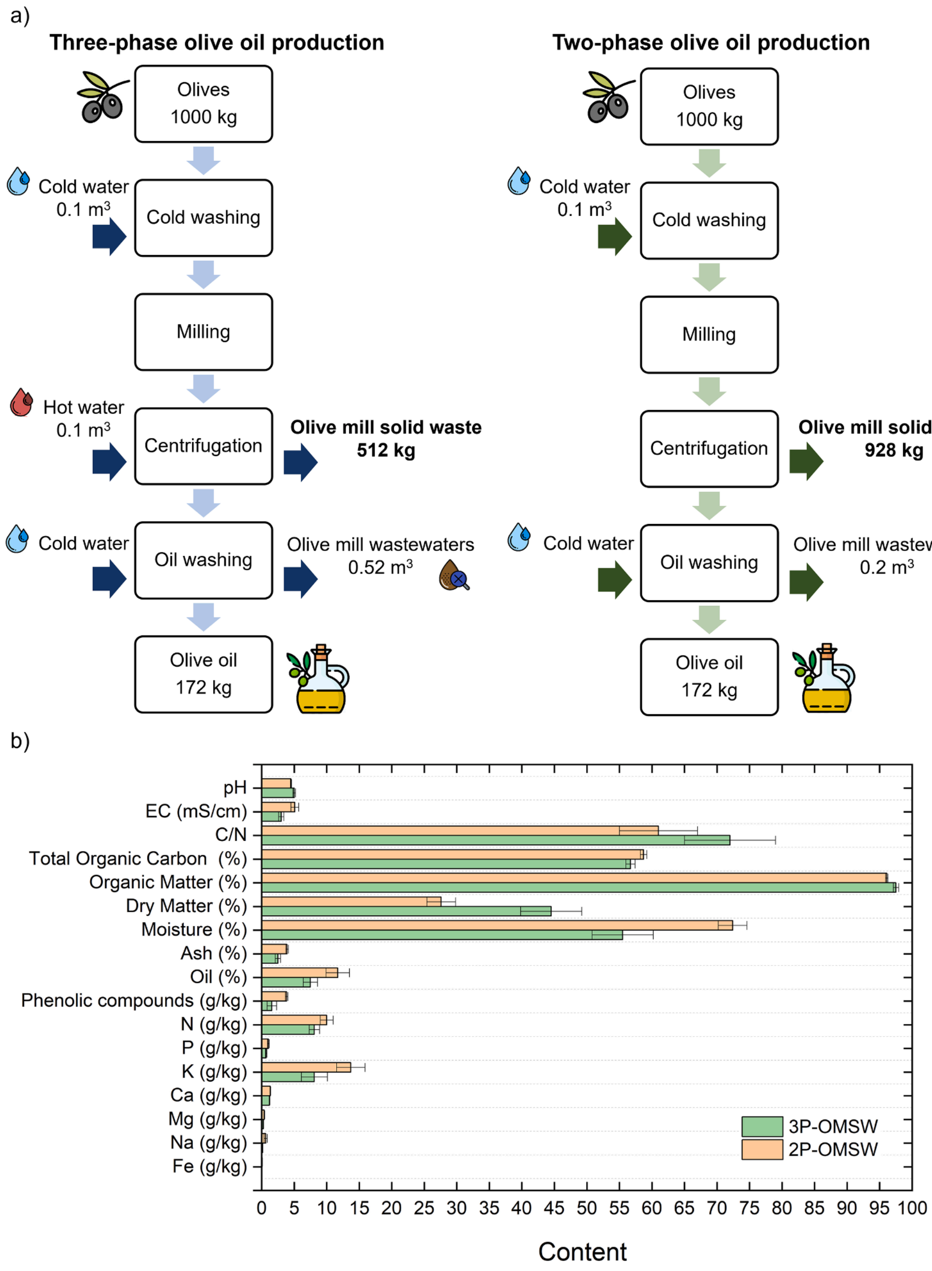

Fig. 1. (a) Comparison of the three- and two-phase centrifugation systems for olive oil extraction (adapted from (Cappelletti et al., 2014)). (b) Main characteristics and components of the olive mill solid waste from two-phase (2P-OMSW) and three-phase (3P-OMSW) centrifugation systems (adapted from (Černe et al., 2023)).

one, following the life cycle assessment (LCA) methodology in all nine impact categories considered (Restuccia et al., 2022). It is reasonable to foresee that the 2P systems will rapidly replace the 3P plants, implementing the recycling of the aqueous phase (i.e., OMWW). In this way, the only residue from the oil industry will be a solid material, the OMSW.

1.1. Olive mill solid waste properties and current managing practices

The comprehensive physico-chemical characterization of olive solid wastes is a crucial aspect to foresee and assess its efficiency and potential application as an energy resource. The chemical composition of olives and OMSW depends on several factors, such as their variety, the degree of ripeness at the time of harvest, and the duration and conditions of temporary storage (Di Giacomo and Romano, 2022). The structure of OMSW primarily comprises a combination of pulp and skin of the olive fruit, alongside a minimal quantity of residual oil. The main characteristics of the OMSW are a low pH (4.7–5.2) (Rouvalis and Iliopoulou-Georgudaki, 2010) and a high carbon-to-nitrogen (C/N) ratio (Černe et al., 2023), while the structural composition is 45–75 % water, 18–25 % cellulose and hemicellulose, and 8–15 % lignin (Fig. 1(b)). The moisture content of OMSW is highly variable depending on the particular olive oil extraction technology employed, i.e., 45–55 % for the 3P and 55–75 % for the 2P systems (Restuccia et al., 2022; Vlyssides et al., 2004).

The majority of oil mills still treat their wastes in open ponds to evaporate water and then spread the residues on potentially cultivable lands, thereby posing significant environmental risks (Justino et al., 2012; Sellami et al., 2008). This practice is probably due to the relatively small size of most of the olive processing sites and their scattered distribution across the territories. However, the process is land-consuming and not economically attractive, it generates unpleasant odors, methane, and sulfur oxides emissions, and is frequently responsible for vermin proliferation (Orive et al., 2016). Furthermore, national legislations that vary across the Mediterranean countries set limits to the quantity of olive mill wastes that can be discharged into agricultural soil (Serrano et al., 2021; Tamborrino et al., 2021). This is due to the high concentration of phenols, low pH, and phytotoxicity of residues that might have negative effects on the physico-chemical properties of soil, the microorganisms, and the groundwater quality (Stoyanova et al., 2017). Animal feeds have also been formulated using OMSW. However, since phenolic compounds serve as digestive enzyme inhibitors, OMSW shows little nutritional value while having a high fiber concentration (Dermeche et al., 2013). To date, the combustion of OMSW for energy generation has emerged as the most widespread option for its valorization (Christoforou and Fokaides, 2016). Nevertheless, the high moisture content coupled with the significant energy requirements associated with combustion may jeopardize its potential as a comprehensive solution for OMSW valorization (Messineo et al., 2020). Moreover, a recent research has shown that incorporating dried OMSW in coal-burning power facilities has resulted in unanticipated issues due to the tendency of the OMSW to form cohesive clusters, commonly referred to as “caking” (Williams et al., 2017). Therefore, there is an imperative need to enhance the possibilities of OMSW valorization, considering both techno-economic and environmental perspectives.

1.2. Anaerobic digestion: Opportunities and challenges with olive mill solid waste

The anaerobic digestion (AD) process has been widely recognized as a highly effective and ecologically friendly biochemical process for the generation of bioenergy from organic biomasses, with the production of biogas and digestate. The constituents of biogas are primarily methane (CH_4) in a percentage ranging from 50 % to 75 %, alongside carbon dioxide (CO_2) and minor constituents including ammonia (NH_3), siloxanes, H_2, N_2, and O_2, among others (Liberti et al., 2019). Application of biogas are direct utilization as a fuel, deployment in co-generation systems to produce both electricity and heat, and upgrading through the process of purification to extract methane for resale as a form of natural gas (“biomethane). The AD process is marked by a sequence of biochemical conversions, comprising hydrolysis, acidogenesis, acetogenesis, and methanogenesis, with each step being brought about by distinct groups of bacteria in the absence of oxygen (Fezzani and Cheikh, 2010). The source of the inoculum is a central aspect, as it defines the initial activity of the bacterial community and establishes the balance of microorganisms that will control substrate digestion kinetics (De la Rubia et al., 2018; Yan et al., 2023). In particular, the source of the inoculum affects the methane yield, the timing of the process, and its stability (Ge et al., 2016). Numerous substances can be used as an inoculum, including digestate from other AD processes, sludge from wastewater treatment, effluent from the treatment of manure, or landfill leachate (De la Rubia et al., 2018). The digestate, being rich in nutrients and micronutrients , can be employed as a fertilizer for agronomic purposes, with the primary focus on enhancing soil fertility and increasing the quality of plant growth, in addition to offering an ecological alternative growing medium or nutrient suspension for hydroponic vegetable cultivation (Fig. 2) (Aboelfetoh et al., 2022; Alburquerque et al., 2012; Fantozzi and Buratti, 2009).

AD can be conducted either in a single step (single reactor) or in separate steps (two or three apparatuses). In particular, in the two-stage system, the acidogenesis is separately conducted in a reactor, while methanogenesis is carried out in a second vessel fed with the effluent from the first one. The main purpose of separating the two phases is to improve specific gas production and to optimize the degradation of the organic substrate by selecting and enriching different bacteria and archaea in each separate process stage. However, multi-stage reactors are often described as uneconomical solutions, due to the increment of the capital costs of the biogas plant (Liberti et al., 2019). Due to their simple designs and ease of operation, semi-continuously and continuously stirred tank reactors (sCSTRs and CSTRs) are popular configurations in both research and industrial settings since they allow greater homogeneity of process variables such as substrate concentration, pH, and temperature (Mahata and Das, 2022). Whereas only a few studies have been conducted on thermophilic processes that operate above the temperature threshold of 55 °C, anaerobic treatment of OMSW has predominantly been investigated in the mesophilic temperature ranges, typically between 32 and 40 °C (Gunay and Karadag, 2015). However, AD can be hindered by several characteristics of OMSW, such as low pH values, high salinity, high content of lignocellulosic biomass, high C/N ratio (which would slow down the microbial growth rate and, therefore, all substrate transformation reactions into biogas), and high concentration of polyphenols and other compounds with strong antibacterial activity, such as vanillin and hydroxymethylfurfural (De La Lama et al., 2017; Li et al., 2018; Serrano et al., 2019a; Siciliano et al., 2016). It is noteworthy that the degradation products of lignin are generally phenolic compounds, including vanillic acid, syringic acid, ferulic acid, and p-coumaric acid, which have different but strong antimicrobial mechanisms of action against bacteria (Contardi et al., 2021; Rincón et al., 2013). In the case of OMSW, they end up contributing to the polyphenolic content of the olive residue during the hydrolysis and degradation steps of the matrix. One of the most significant challenges of AD with OMSW is its high seasonality (October – March), with olive production varying considerably from year to year. The small size of most of the mills and their spread-out distribution in the territory hamper the feasibility of larger centralized treatment plants (Messineo et al., 2020; Orive et al., 2021). On the other hand, the high installation costs as well as the seasonality of the produced wastes often discourage olive oil producers from setting up their own AD plants (Messineo et al., 2020).

Co-digestion (coAD) could be an effective strategy to reduce the C/N ratio of OMSW (up to optimal values of 20–30), while simultaneously rising nutrient levels, reducing acidity, and mitigating the concentration

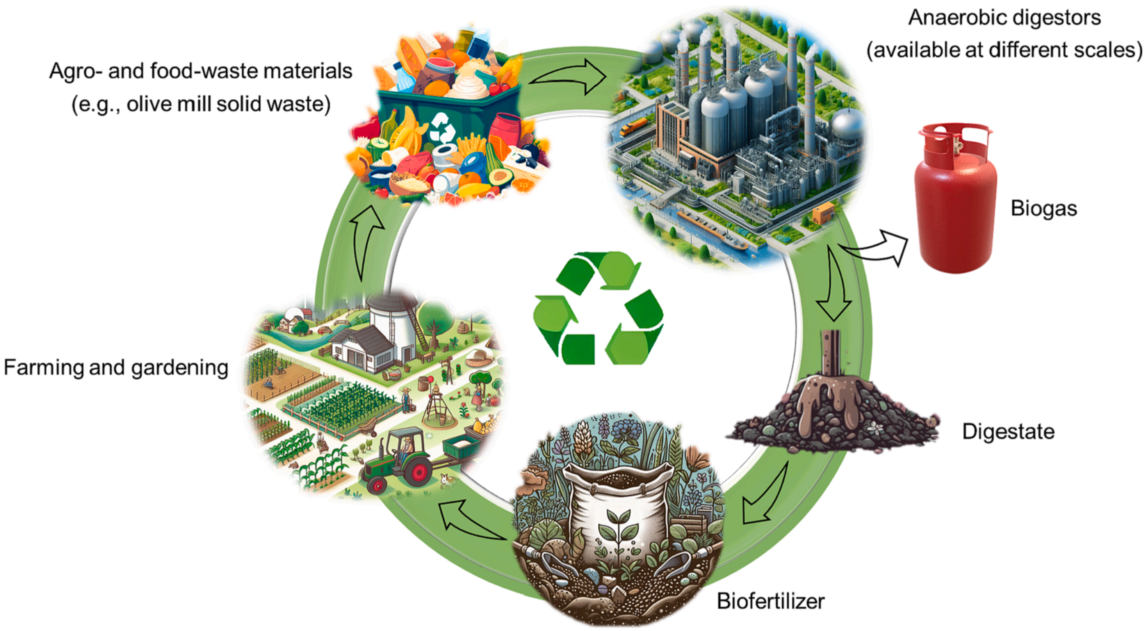

Fig. 2. Schematic process diagram of anaerobic digestion. It contributes to the implementation of circular economy concepts.

of inhibitory substances, including polyphenols, in the reactor (Fernández-Rodríguez et al., 2022; Li et al., 2018). Moreover, most of the co-substrates introduced in AD reactors are waste from food and animal farming activities or sludge from wastewater treatment and are available year-round. Undeniably, even if the practical application of coAD mainly depends on the presence or absence of such co-substrate at a given site, coAD with different biomasses mixed at optimum ratios could represent a technologically and economically viable solution for OMSW treatment.

2. Aim of the review and literature analysis methodology

This review aims to critically analyze the recent research advancement in the field of AD of OMSW. It complements other reviews on the topic by giving a comparison of laboratory-, pilot-, and industrial-scale studies, with a focus on pretreatment applied, co-feedstocks fed, and methane production values obtained. In the present review, data were selected from published research articles (2000–2023) using Google Scholar, PubMed, Scopus, and Science Direct databases. The forced keywords “anaerobic digestion” and “olive mill solid waste”/“olive pomace”/“olive cake” were used for the search. Articles with consistent

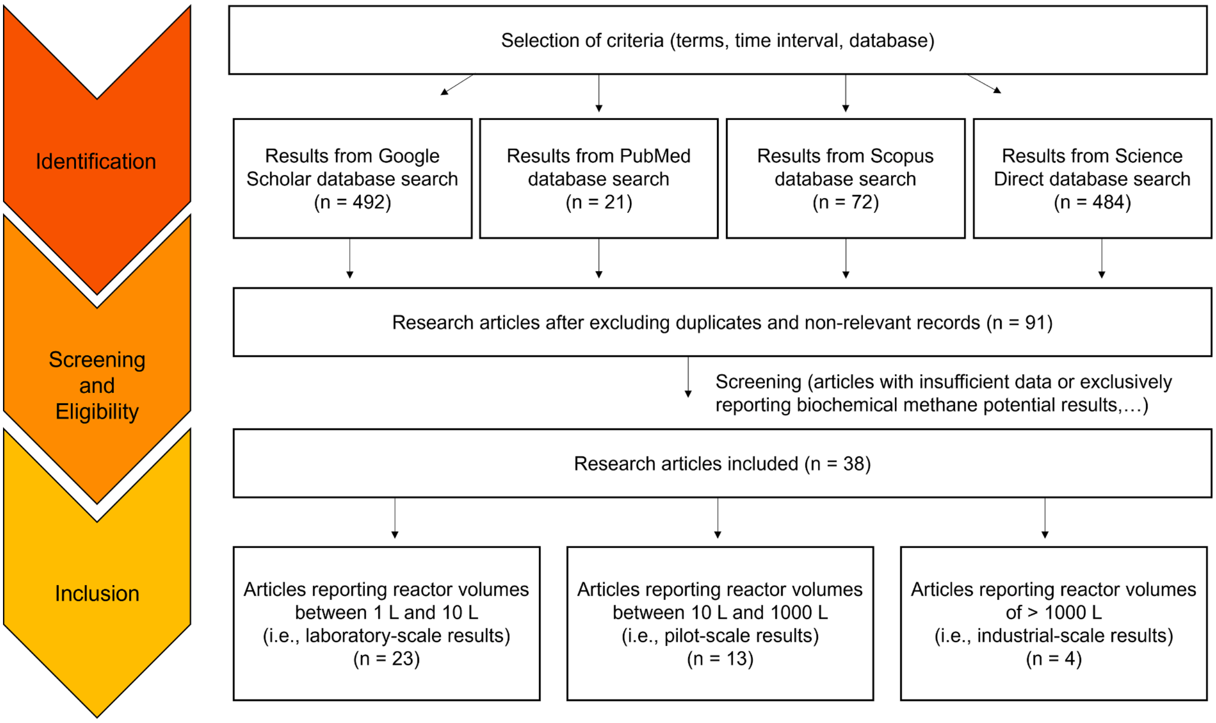

Fig. 3. Flowchart outlining literature search and articles selection process for the present systematic review.

and clear data were considered and are here described in chronological order in their pertinent section. Fig. 3 shows the flowchart of the literature search strategy for this systematic review and the subsequent division of articles into groups for further analysis. Tables in this review report the most relevant parameters related to AD of OMSW, including the optimized operational parameters and biogas/methane production. For Tables reporting results from mono-AD experiments, the inoculum source is specified, being a central element affecting the efficiency and yield of biomethane in such processes (Evranos and Demirel, 2015). When specified in the original article, the type of olive oil production process (2P or 3P) is indicated. Papers that exclusively reported biochemical methane potential tests were excluded, due to the limit of applicability of their results to pilot- and industrial-scale plant processes. It is known from the literature that the current methodology of these tests does not have the capability to evaluate the adaptation potential of the anaerobic microbial community to inhibitory compounds across a prolonged time frame (Koch et al., 2020).

Within the context of AD research, the word “pilot-scale” might be confusing. Such systems must possess the ability to undergo scaling-up procedures without necessitating substantial design modification and incurring a drop in performance functionality. Therefore, the following volume-based criterion was chosen for this review paper: pilot-scale reactors have sizes ranging from 10 to 1000 L, whilst laboratory-scale digesters have reactor volumes ranging from 1 to 10 L (Bird et al., 2022; Rodriguez-Perez et al., 2018; Zhang et al., 2020). The structure of this review follows the objective outlined above. Specifically, each paragraph chronologically presents the main findings of AD experiments performed at laboratory- (paragraph 3), pilot- (paragraph 4), and industrial-scale (paragraph 5). Research needs and future perspectives are then discussed in paragraph 6.

3. Laboratory-scale anaerobic digestion of olive mill solid waste

Different studies have been conducted to evaluate the potential benefits and limits of AD processes of olive mill solid wastes on a laboratory scale (i.e., with reactor volumes less than 10 L). Typically, experiments were conducted to determine the optimal conditions and parameters, such as organic loading rate (OLR) and hydraulic retention time (HRT), for the degradation of the substrate, production of biomethane, removal of chemical oxygen demand (COD), and volatile solids (VS), while simultaneously ensuring the stability of the process (Tables 1, 2). Among the first to study the process of AD with OMSW were Tekin and Dalgiç in 2000, who explored the biogas generation from slurry obtained by blending OMSW with water (Tekin and Dalgıç, 2000). The highest biogas productivity was shown to be 0.70 $L_{biogas}/(L_{reactor}*d)$, corresponding to a methane productivity of 0.62 $L_{CH4}/(L_{reactor}*d)$. A subsequent, more detailed study was carried out by Borja et al. (Borja et al., 2002), who examined the digestibility of OMSW in a CSTR using four different influent substrate concentrations ranging from 0.9 to 15.0 $g_{COD}/(L_{reactor}*d)$, corresponding to 20–80 % OMSW. The findings indicated that, while methane productivity marginally dropped when the OLR was raised, the volume of methane daily produced grew linearly. In particular, at an OLR of 12.02 $g_{COD}/(L*d)$ and a HRT of 12.5 days for the most concentrated substrate (OMSW 80 %), COD and VS removal efficiencies of 89 % and 91 % were achieved, respectively, with a maximum methane productivity of 2.12 $L_{CH4}/(L*d)$. A follow-up study by the same authors using different OMSW concentrations, including 100 % OMSW, showed that an excess increase in influent substrate concentration led to process failure by lowering pH and increasing the ratio of total volatile fatty acids (VFAs) to total alkalinity (Borja et al., 2004). Lastly, the authors demonstrated that the methanogenic phase predominated at high HRT values, whereas the hydrolysis and acidogenic stages did so at HRT shorter than 20 days (Borja et al., 2005).

In 2006 a research study was conducted by Rincón et al. on the anaerobic digestion of 2P-OMSW in CSTRs (Rincón et al., 2006) at OLRs lower than 3 $g_{COD}/(L*d)$. The authors observed a positive correlation between loading rate and methane production: for instance, by increasing the OLR from 0.75 to 3.00 $g_{COD}/(L*d)$, methane productivity increased from 0.16 $L_{CH4}/(L*d)$ to no less than 0.66 $L_{CH4}/(L*d)$, while the corresponding COD removal efficiency only decreased from 97 % to 96 %, with a methane yield essentially unaffected by OLR. In a further research published in 2008, the same authors, looking at the impact of HRT and OLR on CSTR performance (Rincón et al., 2008a), confirmed the above linear correlation between methane productivity and OLR (higher than in the previous study) and found COD removal efficiencies ranging from 77 % to 97 %. The highest methane productivity (1.7 $L_{CH4}/(L*d)$) was attained at OLR of 9.2 $g_{COD}/(L*d)$ and HRT of 17 days, while the mean methane yield was found to be 0.24 $L_{CH4}/g_{COD\ removed}$.

Pretreatments are widely employed in AD studies, particularly in the case of hardly biodegradable substrates. Controlling the formation of VFAs without affecting the methanogenic activity of bacteria can be made possible by using the first acidogenic phase as a pretreatment for the second methanogenic one. In particular, in the hydrolytic-acidogenic reactor, a more solubilized OMSW is obtained with a high concentration of VFAs and a high percentage of acetic acid as the principal precursor of methane (Rincón et al., 2008b). The methanogenic stage of two-stage AD of 2P-OMSW was investigated by three different research-groups (Koutrouli et al., 2009; Rincón et al., 2009; Stoyanova et al., 2017). In particular, Rincón et al. (Rincón et al., 2009) observed a methane productivity of 3.24 $L_{CH4}/(L*d)$ at an OLR of 20 $g_{COD}/(L*d)$ and 5 days HRT, corresponding to a methane yield as high as 0.27 $L_{CH4}/g_{COD\ removed}$. The second-step methane-producing reactor developed by Koutrouli and coworkers proved successful when operating at HRTs of 20, 15, and 10 days, with maximum methane productivity of 1.13 $L_{CH4}/(L*d)$ at 10-day HRT and an OLR of 5.26 $g_{COD}/(L*d)$, but it failed at HRT of 5 days, due to an increase in volatile fatty acids concentration (Koutrouli et al., 2009). The two-stage process described by Stoyanova et al., which combined a first thermophilic acidification stage and a second mesophilic methanogenic one, proved to be highly effective in OMSW AD (Stoyanova et al., 2017). Their experimental setup comprised a 1-L gas-tight plastic chamber for the first stage (4 days HRT) and a 6-L CSTR for the second methanogenic stage, with 150 days HRT. Such value is much higher than those reported by other studies and would have required higher reactor volumes or a reduction of the necessary HRT, as pointed out by the authors themselves. The methanogenic process remained stable at OLR of 1.56 $g_{VS}/(L*d)$, and the buffering capacity of the system was twice as high compared to the one-stage fermentation, without the supplement of additives. However, the methane yield of the process (0.2 $L/g_{COD\ removed}$) was slightly lower than that observed by Rincón et al. (0.27 $L/g_{COD\ removed}$).

The effects of physical, chemical, and biological pretreatments of OMSW on its anaerobic digestibility and biogas production have been investigated as well, even if their environmental and economic aspects are frequently overlooked. De La Lama et al. suggested a heating step at 120 °C for 180 min as a pretreatment to increase OMSW biodegradability (De La Lama et al., 2017). By applying such conditions, the lowest lignin and hemicellulose contents were found in the residue. Other positive consequences include sanitation and a decrease in viscosity. However, it is often reported that the application of high temperatures leads to larger expenses and to the formation of recalcitrant compounds (Carrere et al., 2016). An OLR of 4.5 $g_{VS}/(L_{reactor}*d)$ ensured maximum methane productivity (1.72 $L_{CH4}/(L*d)$), 39.8 % higher than that obtained from untreated OMSW at the same OLR. At OLRs between 1.5 and 2.2 $g_{VS}/(L*d)$, Al Afif and Linke tested the AD of 3P-OMSW in an 8-L CSTR (Al Afif and Linke, 2019) after treatment with the enzyme mixture MethaPlus®, which aids in the breakdown of cellulose into glucose, cellobiose, and oligomers. While enzymatic pretreatments often demand low energy and can be easily implemented in large-scale reactors, their cost and substrate specificity are their weak points for AD applications. Al Afif and Linke reported that the methane output increased by 10 % under thermophilic conditions, but after 70 days of digestion (higher than most of the reported studies), the accumulation of

Table 1

Laboratory-scale mono-digestion of OMSW.

Volume of reactor (L)	Type of reactor	Substrate	Incolum source	Pretreatment	HRT (days)	OLR	COD removal efficiency (%)	VS removal efficiency (%)	Methane production	Methane yield	Reference
1	sCSTR	OMSW	LL	–	20	–	65	–	0.62 L/(L*d)	0.07 L/g_{COD}	(Tekin and Dalgıç, 2000)
2	sCSTR	2P-OMSW	DAD	Steam explosion (200 °C 5 min)	–	1.0 g_{VS}/(L*d)	–	–	0.16 L/(g_{VS}*d), 0.4 $g_{COD\text{-}CH4}$/(g_{COD}*d)	–	(Serrano et al., 2019a)
2	sCSTR	2P-OMSW	DAD	Thermal (170 °C 1 h)	25	1.0 g_{VS}/(L*d)	–	85	0.17 L/(g_{VS}*d)	–	(Serrano et al., 2019b)
6	sCSTR	OMSW	DAD	Organosolv with water and ethanol	6	2.6 g_{VS}/(L*d)	73	53	1.83 L/(L*d)	–	(Paz et al., 2023)
1	CSTR	2P-OMSW	DAD	–	12.5	12.0 g_{COD}/(L*d)	89 (80 % OMSW)	91 (80 % OMSW)	2.12 L/(L*d)(80 % OMSW)	0.30 L/$g_{COD\ removed}$ (20 % OMSW)	(Borja et al., 2002)
1	CSTR	2P-OMSW	DAD	–	50	3.76 g_{COD}/(L*d)	–	–	2.25 L/(L*d)	0.35 L/g_{COD}	(Borja et al., 2004)
1.5	CSTR	2P-OMSW	DAD	–	16.6	–	31	–	0.94 L/(L*d)	–	(Borja et al., 2005)
2	CSTR	2P-OMSW	DAD	–	–	3.0 g_{COD}/(L*d)	96	–	0.66 L/(L*d)	0.26 L/$g_{COD\ removed}$, 0.29 L/$g_{VS\ removed}$	(Rincón et al., 2006)
2	CSTR	2P-OMSW	DAD	–	17	9.2 g_{COD}/(L*d)	77	–	1.70 L/(L*d)	0.24 L/$g_{COD\ removed}$	(Rincón et al., 2008a)
1.8	CSTR	2P-OMSW	DAD	Acidification	5	20.0 g_{COD}/(L*d)	61	56	3.24 L/(L*d)	0.27 L/$g_{COD\ removed}$	(Rincón et al., 2009)
2	CSTR	2P-OMSW	DAD	Thermal (120 °C 180 min)	74	4.5 g_{VS}/(L*d)	72	–	1.72 L/(L*d), 0.38 L/(g_{VS}*d)	–	(De La Lama et al., 2017)
3	CSTR	2P-OMSW	DAD	Hydrogenogenic reactor	10	5.26 g_{COD}/(L*d)	53	–	1.13 L/(L*d)	0.14 L/kg_{COD}	(Koutrouli et al., 2009)
6	CSTR	2P-OMSW	DAD	Acidification	150	1.5 g_{VS}/(L*d)	–	–	–	0.20 L/g_{VS}, 0.20 L/$g_{COD\ removed}$	(Stoyanova et al., 2017)
8	CSTR	3P-OMSW	EM	Enzymes	70	2.0 g_{VS}/(L*d)	–	–	–	0.30 L_{biogas}/g_{VS} (CH_4 50–58 %)	(Al Afif and Linke, 2019)

2P, two-phase centrifugal process; 3P, three-phase centrifugal process; COD, chemical oxygen demand; CSTR, continuously stirred tank reactor; DAD, digestate from previous AD processes; EM, effluent of the treatement of animal manure; HRT, hydraulic retention time; LL, landfill leachate; OLR, organic loading rate; OMSW, olive mill solid waste; sCSTR, semi-continuously stirred tank reactor; VS, volatile solids.

Table 2
Laboratory-scale co-digestion of olive mill solid waste.

Volume of reactor (L)	Type of reactor	Substrate	Pretreatment	HRT (days)	OLR	COD removal efficiency (%)	VS removal efficiency (%)	Methane production	Methane yield	Reference
1	Batch-fed	2P-OMW (67 %) + cow manure (33 %)	–	45	–	–	–	–	0.34 L/g_{VS} (55 °C), 0.24 L/g_{VS} (35 °C)	(Aboelfetoh et al., 2022)
2.4	Batch-fed	OMSW (30 %) + Organic fraction of municipal solid waste (70 %)	–	170	–	83	81	0.03 L/(L*d)	0.04 $_{CH4\text{-}COD\ removed}$/ g_{VS}	(Ağdağ, 2011)
5	Batch-fed	3P-OMW	–	126	–	83	–	0.24 L_{biogas}/(L*d) 70–77 % CH_4	–	(Fezzani and Cheikh, 2008)
1	Batch-fed	3P-OMSW + cow manure	Enzymes	40	–	–	–	–	0.13 L/g_{VS} (3:1 mix), 0.18 L/g_{VS} (with enzymes to the 1:1 mix)	(Al Afif and Amon, 2019)
2.5	Batch-fed	OMSW (50 %) + wastewater sludge (50 %)	Ultrasounds or microwaves (on sludge)	30	–	–	51 – 54	0.05 L/(L*d) (ultrasounds), 0.06 L/(L*d) (microwaves)	0.26 L/g_{VS} (ultrasounds), 0.32 L/g_{VS} (microwaves)	(Aylin Alagöz et al., 2015)
5	sCSTR	2P-OMSW (60 %) + cow manure (40 %)	–	40	–	–	–	0.34 L/(L*d)	–	(Rubio et al., 2022)
6	sCSTR	OMSW (40 g) + wastewater sludge (75 mL)	Organosolv with water and ethanol (on OMSW)	6	2.5 g_{VS}/(L*d)	92	87	0.37 L_{biogas}/(L*d) 65–70 % CH_4, 0.23 L_{CH4}/(g_{VS}*d)	–	(Paz et al., 2023)
2.5	CSTR	OMSW (50 %) + sewage sludge (50 %)	5 % KOH	20	1 g_{VS}/(L*d)	–	43	–	0.22 L/g_{VS}	(Elalami et al., 2020b)
6	CSTR	2P-OMSW (57 %) + chicken manure (42 %)	–	193	1.5 g_{VS}/(L*d)	–	–	–	0.17 L/g_{VS}, 0.17 L/$g_{COD\ removed}$	(Stoyanova et al., 2017)
6	CSTR	2P-OMSW (4.3 %) + food waste (95.7 %)	10 % NaOH (on OMSW)	28.7	4.6g_{COD}/ (L_{sludge} *d)	–	–	–	0.45 L/g_{VS_OMSW}	(Al-Mallahi et al., 2016)

2P, two-phase centrifugal process; 3P, three-phase centrifugal process; COD, chemical oxygen demand; CSTR, continously stirred tank reactor; HRT, hydraulic retention time; OLR, organic loading rate; OMW, olive mill waste; OMWW, olive mill wastewater; OMSW, olive mill solid waste; sCSTR, semi-continuously stirred tank reactor; VS, volatile solids.

VFAs was the first indicator of a process failure. In 2019, Serrano et al. conducted steam explosion pretreatments at a temperature of 200 °C for 5 min to assess the digestibility of dephenolized OMSW for a period of 275 days (Serrano et al., 2019a). According to the authors, steam explosion allows higher breakdown (i.e., hydrolysis) of the lignocellulosic structures compared to other thermal treatments. However, poor enzymatic digestibility has been documented when steam explosion is performed under extremely harsh circumstances, which is often related to the production of furans (from xylose and glucose breakdown) and phenolics (from lignin degradation) (Mabutyana and Pott, 2021). AD was found to be stable at an OLR of 1 $g_{VS}/(L*d)$ with a methane productivity of 0.16 $L_{CH4}/(g_{VS}*d)$. In the same year, the same researchers described an alternative approach for OMSW thermal pretreatment involving the application of pressurized steam at a temperature of 170 °C and a pressure of 0.85 MPa for 60 min (Serrano et al., 2019b), which gave comparable results. The stability of AD of thermally pretreated and dephenolized OMSW, at an OLR of 1 $g_{VS}/(L*d)$, enabled a methane productivity of 0.17 $L_{CH4}/(g_{VS}*d)$; however, due to the accumulation of VFAs, the system was unable to operate at an OLR of 2 $g_{VS}/(L*d)$, resulting in the complete failure of the process. Recently, Paz et al. highlighted the positive effect of organosolv extraction on improving the suitability of OMSW as an AD substrate (Paz et al., 2023). In particular, such pretreatment (50 % (v/v) ethanol/water mixture and 0.5 % (w/v) H_2SO_4 as a catalyst in a stainless-steel autoclave reactor) was able to convert the lignin fraction of the material into a more accessible form to the AD microbial communities (i.e., it led to an increase in carbohydrate content). The AD of 60 g feeding of treated OMSW into a 6-L sCSTR ensured a value of 1.83 $L_{CH4}/(L*d)$ at an OLR of 2.60 $g_{VS}/(L*d)$, higher than that obtained by De la Lama et al. with thermal pretreatment (1.72 $L_{CH4}/(L*d)$ at an OLR of 4.5 $g_{VS}/(L*d)$ (De La Lama et al., 2017).

In conclusion, looking at methane production (normalized to reactor volume and experiment duration), it is possible to notice that without any pretreatment Borja et al. in 2004 obtained the highest methane productivity (2.25 $L_{CH4}/(L*d)$) and Rincón et al. the highest value using a two-stage AD system (3.24 $L_{CH4}/(L*d)$) (Borja et al., 2004; Rincón et al., 2009). It has to be noted that throughout the years those authors always used as the inoculum source a methanogenically active biomass from a laboratory-scale AD reactor processing OMWW. Such inoculum is already acclimatized to the higher polyphenols content and low pH values characteristic of olive by-products, and it is likely to allow a faster and more efficient digestion of the substrates. Among the various pretreatments proposed, Paz et al. and De La Lama et al. obtained a value of 1.83 $L_{CH4}/(L*d)$ and 1.72 $L_{CH4}/(L*d)$ with organosolv pretreatment or by treating OMSW at 120 °C for 180 min, respectively (De La Lama et al., 2017; Paz et al., 2023). It is interesting to notice that both research groups used as the inoculum source the anaerobic sludge from industrial-scale AD reactors.

3.1. Laboratory-scale anaerobic co-digestion of olive mill solid waste

Several studies reported successful coAD of OMSW with municipal organic wastes, agro-industrial wastes, and animal manures (Fig. 4(a), Table 2). Each kind of residue has different characteristics regarding pH, C/N ratio, and presence of trace elements or toxic substances, allowing to combine two or more of them to reach the optimum nutrient composition for AD. Fezzani et al. aimed to optimize the proportions of OMWW and OMSW for coAD in 5-L digesters at 37 °C (Fezzani and Cheikh, 2008). The results indicated that the optimal amount of OMSW was about 56 g_{TS}/L_{OMWW}, which allowed to almost triple biogas production (0.24 L/(L*d)) compared to OMWW alone (0.09 L/(L*d)) and almost duplicate soluble COD (sCOD) removal efficiency (83 % vs. 45 %). However, the start-up time of steady biogas production was not significantly changed. It has to be noted that, coming from the same organic source (i.e., olives), a decrease in operational and traveling costs for AD plant operators is expected with these types of feedstocks. On the other hand, the high seasonality of OMWW and OMSW might hamper the feasibility of scaling up this type of coAD process. In 2011 Ağdağ found that the co-digestion of OMSW with the organic fraction of municipal solid waste (OFMSW) at a ratio of 30:70 (“run 1”) was more effective than using a feedstock composed of 30:70 (OFMSW:OMSW, “run 2”) (Ağdağ, 2011). The maximum cumulative methane gas production was recorded as 17.6, 13, and 9.7 L in control (only OFMSW), run 1 and run 2 reactors, respectively, at the end of the 170-day digestion period, corresponding to methane productivity of only 0.04, 0.03, and 0.02 $L_{CH4}/(L*d)$, respectively. Subsequently, Aylin Alagöz et al. found that the methane yield from the mono-digestion of OMSW was 0.18 L_{CH4}/g_{VS}, whereas the co-digestion with wastewater sludge yielded approximately 0.21 L_{CH4}/g_{VS} (Aylin Alagöz et al., 2015). Moreover, compared to coAD with untreated wastewater sludge, the microwave (30 min at 175 °C and 2000 kPa) and ultrasonic (20 kHz, 70 % amplitude, and 200 W of supplied power) pretreatments applied to sludge prior to coAD resulted in an additional 52 % and 24 % increase in methane production, respectively. Consequently, the former pretreatment of sludge allowed the co-digestion process to achieve the highest methane yield (0.32 L_{CH4}/g_{VS}) and productivity (0.06 $L_{CH4}/(L*d)$). It is worth mentioning that while both ultrasound and microwave pretreatments have a high electricity demand, ultrasound methods are usually more easily scalable than microwave ones (Carrere et al., 2016).

Alkaline pretreatments of organic materials have been found to facilitate the solubilization of various components, including sugars, polyphenols, and lipids, and to reduce their lignin content (Elalami et al., 2020a). Chemical pretreatments have usually a low energy demand but can be extremely costly. The pretreatment with NaOH has been extensively studied and documented in the literature, making it the most commonly employed technique in this regard. The investigation conducted by Al-Mallahi and colleagues in 2016 aimed to determine the optimal parameters for coAD of NaOH-pretreated 2P-OMSW with shredded and 50 % diluted (by weight) food waste in four distinct mixing ratios (Al-Mallahi et al., 2016). The goal of this study was to conduct a simulation of the AD process occurring in a plant transitioning from mono-digestion of food waste to co-digestion with OMSW during the period of olive oil production. As previously mentioned, this is of particular importance for the scalability, costs, and start-up of an AD plant, given the high seasonality of OMSW. The authors noted that the use of 10 % NaOH for 6 days to pretreat OMSW resulted in a methane yield as high as 0.45 $L_{CH4}/g_{VS\ OMSW}$ and pH stability across the experimental runs with food waste. Even though the 20 % NaOH-pretreated OMSW ensured the maximum sCOD value and an 11 % higher methane yield, pH adjustment was required. The use of KOH rather than NaOH may potentially mitigate the negative effects of these pretreatments on the application of digestate as a soil fertilizer thanks to its lower soil salinization potential (Bolzonella et al., 2018; Elalami et al., 2020b). The ability of KOH alkaline pretreatment to enhance methane production was assessed by Elalami et al (Elalami et al., 2020b). Specifically, compared to the untreated mixture, the KOH pretreatment (5 % TS basis, at 25 °C for 2 days) increased methane production by 15 % in the 1:1 blend of OMSW and wastewater (or “sewage”) sludge (0.22 L/g_{VS}) by significantly hydrolyzing the hemicellulose-like and cellulose-like fractions contained in the biomass. Moreover, the digestates (originating from untreated and KOH-pretreated residues) were characterized according to their physico-chemical and agronomic properties. KOH pretreatment reduced the TS and VS content (due to methane improvement) and increased the ammonium content (due to higher protein degradation rates) in digestates, which led to significant impacts on the dry mass of vegetables grown with such biofertilizers. Recently, the coAD of OMSW (pretreated with organosolv extraction, as described earlier) and wastewater sludge was also analyzed by Paz et al., who reported a methane production of 0.23 $L_{CH4}/(g_{VS}*d)$, and the highest volatile solids removal so far (87 %) for laboratory-scale coAD of OMSW (Paz et al., 2023).

As anticipated, coAD of animal manure with OMSW is a viable approach for improving biogas yield, while simultaneously addressing

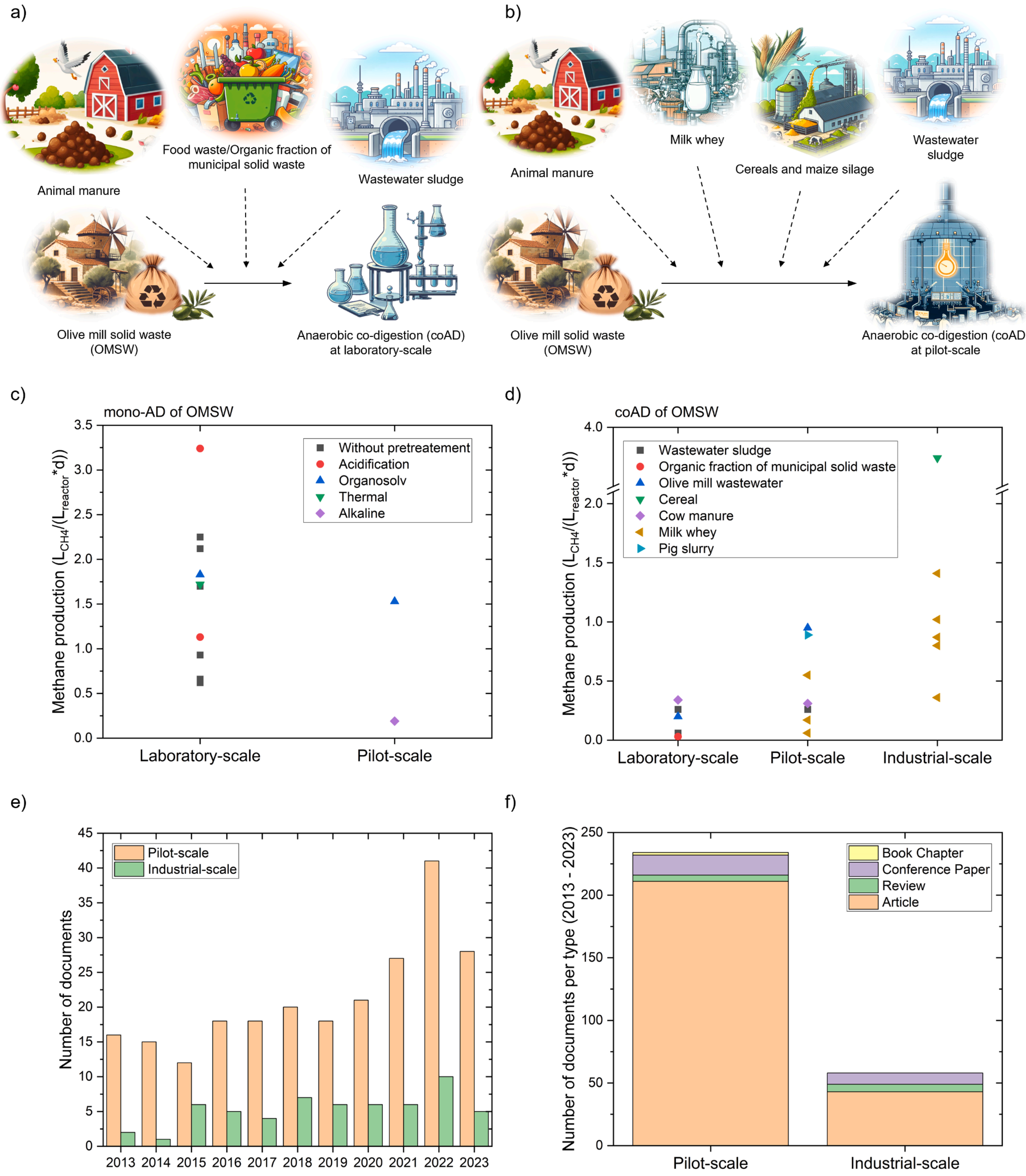

Fig. 4. Co-feedstocks tested for the co-digestion of olive mill solid waste during laboratory-scale (a) and pilot-scale (b) anaerobic digestion experiments. Methane production values obtained from various anaerobic digestion experiments carried out with different pretreatement methods on olive mill solid waste (c) or different co-feedstocks mixed with olive mill solid waste (OMSW) (d). Number of documents on anaerobic digestion (AD) performed with pilot- and industrial-scale digesters according to the Scopus database, here divided by (e) number of documents published year by year and (f) number of documents per type of publication.

the challenges associated with the management of manure wastes. With the addition of OMSW, two types of animal manure were tested and are to date reported in the literature, i.e., cow manure, also known as dairy manure or cattle manure, and chicken manure. Stoyanova et al. co-digested OMSW with chicken manure, a nitrogen-rich co-substrate able to improve the C/N ratio of OMSW as well as the buffering capacity of the mixture (Stoyanova et al., 2017). Although the maximum loading rate and process stability were both enhanced by using a co-substrate, this strategy also had certain disadvantages. The methane yield obtained at the highest OLR (0.17 L/g_{VS}) was 26 % and 15 % lower than those of single-stage and two-stage AD of OMSW alone, respectively. Ammonia inhibition caused by free ammonia nitrogen is in fact one of the major concerns for co-digestion with these waste streams (Wang et al., 2014). On the other hand, the advantages were that the HRT was 35 % lower compared to single-step digestion and that the rise in OLR to the maximum value of 1.56 g_{VS}/(L*d) at the end of coAD was achieved three times more quickly during stable digestion. Interestingly, the authors compared the trace elements (necessary for the metalloenzymes) found in the digestates retrieved from mono-AD and coAD experiments. Increments in all element levels (e.g., Mg, Ca, Fe, Co, Mn, Zn) were found when manure was added to the mixture, which would improve the performance of the digestate as a soil amendment (Wang and Lee, 2021). Al Afif et al. investigated the coAD of 3P-OMSW and cow manure in different ratios, either with or without an enzyme supplement containing cellulases, xylanases, and lipases (Al Afif and Amon, 2019). The findings showed that for 3P-OMSW, cow manure, and their combinations, the suggested retention time to achieve a high rate of biodegradation was between 20 and 30 days, and for the mixture with the addition of enzymes, it was around 40 days. Experiments at a ratio of 3:1 gave the highest methane yield (0.13 L/g_{VS}). Moreover, this parameter was increased by no less than 47 % (0.18 L/g_{VS}) when the enzymes were added to the 1:1 mixture. In a following study, Rubio et al. used a similar co-substrate and investigated the start-up stage of a sCSTR for the mesophilic treatment of a mixture of 2P-OMSW and cow manure (60:40) (Rubio et al., 2022). The biomasses were anaerobically co-digested, with a total period of 140 days required to start up and stabilize the process. During the stable period, methane productivity was 0.34 L_{CH4}/(L*d) at 40 days HRT. The experimental findings indicated that the accumulation of propionic acid played a pivotal role in hindering the progression of the methanogenic phase, thereby inducing a state of process imbalance. Lastly, the research conducted by Aboelfetoh and colleagues in 2022 aimed to identify the optimal combination of AD temperature (mesophilic and thermophilic) and 2P-OMSW to cow manure ratio in order to achieve the targeted biogas production and quality (Aboelfetoh et al., 2022). After a period of 45 days, it was observed that the methane yield increased from 0.24 L/g_{VS} to 0.34 L/g_{VS} when the temperature was raised from 35 °C to 55 °C.

In conclusion, for the laboratory-scale coAD of OMSW, it is difficult to properly assess the optimal pretreatments and feedstocks to be used in order to obtain the highest methane production. Several factors such as the availability of biomasses at the AD plant site, the cost of pretreatments, and the biomass mixing ratio all influence the final outcome and energy assessment. Several authors did not report a proper value for methane production (normalized to reactor volume and experiment duration), presenting the tests carried by Rubio et al. with OMSW and cow manure as the best-performing ones (0.34 L_{CH4}/(L*d) (Rubio et al., 2022). Moreover, it is possible to see in Table 2 that Aboelfetoh et al. reported the highest methane yield (0.34 L/g_{VS}) for a feedstock composed of OMSW and cow manure in a similar ratio (Aboelfetoh et al., 2022). It is reasonable to infer that cow manure, containing a broad range of nutrients and micronutrients (such as nitrogen, copper, manganese, and zinc), provides the required alkalinity to stabilize the AD process with OMSW (Li et al., 2021). When comparing the data obtained by laboratory-scale coAD with that of mono-digestion, it emerges that methane production values are higher in the second case (2.25 L/(L*d)), bearing in mind the advantage of using bacteria coming from AD reactors processing OMWW (Borja et al., 2004), while methane yield data seem higher for coAD experiments (0.34 L/g_{VS}) (Aboelfetoh et al., 2022). Similar findings can be found when comparing the pretreated biomass: in particular, the acidification step ensured the maximum methane production (3.24 L/(L*d))(Rincón et al., 2009) and yield (0.2 L/g_{VS})(Stoyanova et al., 2017) for the mono-digestion of OMSW, while the organosolv extraction and microwaves pretreatment resulted promising options for high methane production (0.26 L/(L*d))(Paz et al., 2023) and yield (0.32 L/g_{VS})(Aylin Alagöz et al., 2015), respectively, when co-digesting OMSW. However, few of the analyzed articles accurately report values for both categories, thus making comparison difficult. It is also important to remember that co-digestion processes are the only ones that at larger scales would ensure the operation of anaerobic digesters all-year around. In fact, without the use of a co-substrate, the amount of OMSW obtained in the few months of harvesting (mid-September-March) would not be enough to feed a large-scale anaerobic digester that operates continuously throughout the year in mono-AD mode. On the other hand, other feedstocks such as manure and slurry are available year-around at low cost and can be used to enhance the biofertilizer potential of the resulting digestates, but also dilute and store OMSW for its subsequent use as feed for coAD processes.

4. Pilot-scale anaerobic digestion of olive mill solid waste

It is important to note that while cost-effective small-scale studies play a paramount role in identifying fundamental process parameters, they often fail to mimic the rheology and the phenomena that are typically observed in pilot- and industrial-scale plants, such as foam buildup and substrate mixing problems within the reactor, resulting in a concomitant reduction in biomass conversion efficiency. Ascertaining the feasibility of translating laboratory findings to larger-scale systems and identifying potential issues are of paramount significance, otherwise laboratory results may “vanish in the haze” and not contribute to the development of applicable OMSW treatment technology. To the best of the authors’ knowledge, only two articles discussed the use of OMSW as a substrate for mono-AD carried out in pilot-scale digesters with volumes between 10 and 1000 L (Table 3). In 2016 Siciliano et al. proposed a chemical pretreatment with hydrogen peroxide (H_2O_2) under alkaline conditions (maintained by NaOH addition) for 3 h to subsequently perform 2P-OMSW AD in a 150-L sCSTR for 110 days (Siciliano et al., 2016). Polyphenols concentration was reduced by 72 % when 0.25 g of H_2O_2 were added per gram of COD at 20 °C. Moreover, the increase in the production of VFAs suggests that part of the polyphenols and other recalcitrant compounds were degraded in such a basic environment into various easily biodegradable intermediate organic compounds. Such pretreatment process led to a subsequent 77 % COD removal and a methane yield (0.33 $L_{CH4}/g_{COD\ removed}$) higher than those reported for bench-scale mono-digestion by Borja et al. (0.30 $L_{CH4}/g_{COD\ removed}$), by Rincón et al. (0.27 $L_{CH4}/g_{COD\ removed}$) for two-stage mono-digestion, and by Stoyanova et al. (0.17 $L_{CH4}/g_{COD\ removed}$) for coAD of OMSW (Tables 1 and 2) (Borja et al., 2002; Rincón et al., 2009; Stoyanova et al., 2017). Moreover, in their research, Siciliano et al. stated that proceeds of about 36.3 €/m^3_{OMSW} are achievable, which, taking into account the expense for the pretreatment, will lead to a net profit of at least 15 €/m^3_{OMSW} (considering 0.24 €/KWh). However, such analysis takes only into consideration the cogeneration of electric energy by means of biogas produced and the cost of consumption of chemical compounds (i.e., H_2O_2 and NaOH). The combination of polyphenol extraction and the subsequent AD was explored by Orive et al. in a 10-L sCSTR (Orive et al., 2021). In terms of extracting solvents, it is interesting to note that the extraction yield achieved by using for 1 h a solution of methanol, formic acid, and water was roughly five times greater than that obtained by using ethanol, formic acid, and water (solvents/2P-OMSW ratio was fixed at 1:1 (w/w)). Nevertheless, the ethanol-containing solution was preferred to perform OMSW dephenolisation due to the possible toxicity of methanol residues in the extracted materials. OMSW dephenolisation, which occurred with a yield of 55 %, had a positive impact on methanogenesis, allowing a methane productivity of 0.42 L/(g_{VS}*d) (1.53 L/(L*d)) at an OLR of 2.89 g_{VS}/(L*d) and 24 days HRT. Although in this case ethanol can be regarded as an environmentally preferable solvent, greener technologies should be explored to pretreat biomass without chemical methods. Biomethane and clean energy production cannot, in fact, come at the expense of the use of chemical solvents, as their production, utilization, and disposal methods can potentially lead to additional air, water, and soil pollution issues (Capello et al., 2007). Moreover, such a pretreatment process with solvents does not affect the solubilization of lignin and lignocellulosic components of OMSW, in contrast to what is hypothesized with thermal methods. By comparing

Table 3
Pilot-scale anaerobic digestion of olive mill solid waste.

Volume of reactor (L)	Type of reactor	Substrate	Inoculum source	Pretreatment	HRT (days)	OLR	COD removal efficiency (%)	VS removal efficiency (%)	Methane production	Methane yield	Reference
10	sCSTR	2P-OMSW	SWW	Solvent extraction of polyphenols	24	2.89 $g_{VS}/(L*d)$	85	71	1.53 L/(L*d), 0.42 L/(g_{VS}*d)	–	(Orive et al., 2021)
150	sCSTR	2P-OMSW	EM	H_2O_2 (alkaline conditions)	30	5.7 $g_{COD}/(L*d)$	77	–	0.19 L/(L*d)	0.33 L/$g_{COD\ removed}$	(Siciliano et al., 2016)

2P, two-phase centrifugal process; 3P, three-phase centrifugal process; COD, chemical oxygen demand; EM, effluent of the treatment of animal manure; HRT, hydraulic retention time; OLR, organic loading rate; OMSW, olive mill solid waste; sCSTR, semi-continuously stirred tank reactor; SWW, sludge from wastewater treatament; VS, volatile solids.

the results obtained with a solvent extraction pretreatment process at the laboratory scale by Paz et al., and the one obtained by Orive et al., it can be observed that COD and VS removal are increased when operating at pilot scales, while the biomethane productivity is lower (1.53 L/(L*d)) in comparison with the results obtained at laboratory-scale (1.83 L/(L*d)). This can be explained by the longer HRT used at the pilot scale, which can lead to a drop in pH values and compromised methane production. Furthermore, it has to be noted that the inoculum used by Paz et al. was an anaerobic sludge from a biogas production plant, while the one used by Orive et al. came from a wastewater treatment plant. Even though the methane volumetric productivity reported for OMSW by Orive et al., (1.53 L/(L*d)) was slightly lower than the average value obtained from laboratory-scale mono-AD experiments (~1.62 L/(L*d)), the one referred to the VS unit (0.42 L/(g_{VS}*d)) was higher than those obtained by De La Lama after thermal pretreatment (0.38 L/(g_{VS}*d))(De La Lama et al., 2017) and by Serrano et al. after either steam explosion (0.16 L/(g_{VS}*d))(Serrano et al., 2019a) or thermal pretreatment (0.17 L/(g_{VS}*d))(Serrano et al., 2019b) of OMSW (Table 1). Interestingly, here Orive et al. presented a detailed report on the economic feasibility of an imaginary plant that treats 8000 t of 2P-OMSW per year (Orive et al., 2021). With a capital cost of $1.74 million and operational costs of $1.46 million per year, revenues of $1.72 million per year were calculated to be achievable. Earnings from waste management and selling digestate were also included in the economic study. However, a correct market value of unpure polyphenol extracts is yet to be reported and, more importantly, the economic assessment is based on pilot-scale results (in this case in a reactor of 10 L volume).

As anticipated in the previous sections, several papers are now available on co-digestion of OMSW with other substrates to overcome some of the challenges that olive residues present during AD steps. More than a few studies reported successful coAD of OMSW with OMWW, animal residues (such as manure and whey), sewage sludge, and silage (Fig. 4(b), Table 4). It is noteworthy that different co-feedstocks require different optimized mixing ratios depending on the characteristics and composition of the raw materials, hampering the establishment of precise protocols for biogas production. As it has been already described for laboratory-scale AD, the first experiments were carried out by diluting OMSW with OMWW, which is already available in large quantities in the same operating plant. In a study conducted by Fezzani and Cheikh, the anaerobic co-digestion of OMWW and OMSW was investigated under both mesophilic and thermophilic conditions employing two 18-L sCSTRs (Fezzani and Cheikh, 2007a). The findings obtained from the mesophilic digester demonstrated that coAD occurred with an optimal methane productivity of 0.95 L/(L*d), whereas it was 0.7 L/(L*d) when OMWW was digested alone at the same conditions. On the other hand, under thermophilic conditions, methane productivity using the same feed content was twice as high (46 L/(L_{OMWW}*d) vs. 23 L/(L_{OMWW}*d)) (Fezzani and Cheikh, 2007b). Such a performance improvement may have been due to an increase in the rate of OMSW hydrolysis, resulting in more ammonium nitrogen available as well as an acceleration of both methanogenic bacteria growth and biological reactions inside the digester. Such richness in nitrogen (but also phosphorous, potassium, and calcium) in the effluents of coAD experiments makes them particularly interesting for agricultural purposes to increase soil fertility. In particular, the amount of ammonium nitrogen (NH_4^+-N), phosphorous (PO_4^--P), and potassium (K) was 200, 530, and 1380 mg/L in the effluents of OMWW digested alone and 600, 780, and 3860 mg/L in effluents of OMWW co-digested with OMSW. However, at HRT ≤ 12 days, there was a significant increase in the levels of total VFAs and COD in the effluent, and methane generation was stopped. It is widely accepted that digesters operating at a short HRT are likely to lose methanogenic bacteria that are frequently washed out before growing enough to complete the process of biogas production (Fezzani and Cheikh, 2007a). It has to be noted that, while thermophilic processes usually are more efficient, they are also more expensive than mesophilic ones. The authors subsequently used the same substrates in two 18-L mesophilic

Table 4

Pilot-scale co-digestion of olive mill solid waste.

Volume of reactor (L)	Type of reactor	Substrate	Pretreatment	HRT (days)	OLR	COD removal efficiency (%)	VS removal efficiency (%)	Methane production	Methane yield	Reference
50	Batch-fed	2P- or 3P-OMW + milk whey	–	30	–	–	28 (2P-OMW), 44 (3P-OMW)	0.06 L/(L*d) (2P-OMW), 0.17 L/(L*d) (3P-OMW)	0.04 L/$g_{VS,}$0.14 L/$g_{\Delta VS}$ (2P-OMW), 0.10 L/$g_{VS,}$ 0.25 L/$g_{\Delta VS}$ (3P-OMW)	(Battista et al., 2015)
10	sCSTR	2P-OMSW (80 %) + pig slurry (20 %)	–	24	2.72 g_{VS}/(L*d)	59	52	0.89 L/(L*d), 0.27 L/(g_{VS}*d), 0.34 L/(g_{COD}*d)	–	(Orive et al., 2016)
18	sCSTR	3P-OMW	–	24	2.34 g_{COD}/(L*d)	73	–	0.95 L/(L*d), 23 L/($L_{OMWW\ fed}$ *d)	0.30 L/g_{COD}	(Fezzani and Cheikh, 2007a)
18	sCSTR	3P-OMW	–	36	3.62 g_{COD}/(L*d)	72 (sCOD)	–	46 L/($L_{OMWW\ fed}$*d) (55 °C)	–	(Fezzani and Cheikh, 2007b)
18	sCSTR	3P-OMW	Acidification	24	6.87 g_{COD}/(L*d)	79 (sCOD)	–	40.17 L/($L_{OMWW\ fed}$*d)	0.31 L/g_{COD}	(Fezzani and Cheikh, 2010)
128	sCSTR	Cow manure (85 %) + Apple pulp (5 %) + OMSW (10 %)	–	40	2.75 g_{VS}/(L*d) or 2.65 g_{COD}/(L*d)	63		0.31 L/(L*d)	0.22 L/g_{VS}	(Riggio et al., 2015)
16	sCSTR	2P-OMSW (5 %) + sewage sludge (95 %)	Extraction of OMSW polyphenols	17	0.94 g_{VS}/(L*d)	43	27	0.26 L/(L*d)	0.28 L/g_{VS}	(Fragoso et al., 2022)
17	CSTR	Olive husk + piggery manure anaerobically digested	–	43	–	–	–	–	0.11 L/g_{VS}	(Fantozzi and Buratti, 2009)
24	CSTR	2P- or 3P-OMSW (46 %) + cow manure (8 %) + maize silage (46 %)	–	50	4.10 g_{VS}/(L *d) (2P-OMSW), 4.30 g_{VS}/(L *d) (3P-OMSW)	–	–	–	0.24 L/g_{VS} (3P-OMSW), 0.27 L/g_{VS} (2P-OMSW)	(Soldano et al., 2014)
45	CSTR	2P-OMSW (25 %) + milk whey (75 %)	–	40	3.40 g_{COD}/(L*d)	70	64	0.55 L/(L*d)	0.22 L/$g_{\Delta VS}$	(Battista et al., 2013)
75	CSTR	2P-OMSW (25 %) + cow manure (75 %)	–	21.4	5.50 g_{COD}/(L*d)	–	53 (37 °C), 54 (55 °C)	1.10 L_{biogas}/(L_{sludge}*d) (37 °C), 1.30 L_{biogas}/(L_{sludge}*d) (55 °C)	0.18 L/g_{VS} (37 °C), 0.21 L/g_{VS} (55 °C)	(Goberna et al., 2010)

2P, two-phase centrifugal process; 3P, three-phase centrifugal process; ΔVS, difference between the initial and final amount of volatile solids; COD, chemical oxygen demand; CSTR, continuously stirred tank reactor; HRT, hydraulic retention time; OLR, organic loading rate; OMW, olive mill waste; OMSW, olive mill solid waste; OMWW, olive mill wastewaters; sCOD, soluble chemical oxygen demand; sCSTR, semi-continuously stirred tank reactor; TS, total solids; VS, volatile solids.

sCSTRs arranged in series in order to conduct a comparative analysis with the conventional single-stage configuration, with a focus on key performance indicators, such as methane productivity and soluble COD level (Fezzani and Cheikh, 2010). The two-phase system operating with a HRT of 24 days in both phases allowed to achieve a methane production (40.17 L/(L_{OMWW}*d)), higher than those previously obtained with the single-stage process (23 L/(L_{OMWW} *d))(Fezzani and Cheikh, 2010). The improved qualities of the effluents generated by the acidifiers were the cause of this rise in methane production. As a matter of fact, the methanizers readily metabolized the high concentrations of VFAs present in these effluents, turning them into CH_4 and CO_2. Furthermore, the increase of both alkalinity and total nitrogen content of acidifier effluents strengthened their resistance to pH instability and shielded them from the danger of ammonium nitrogen shortage caused by methanogens. Unfortunately, comparison between these results and other studies on different scales or with different feedstocks is precluded by the chosen unit of measurement (i.e., referring to liters of OMWW added in the digester).

Animal manure is an excellent substrate for the coAD of several biomasses, including OMSW, as already mentioned in the preceding section on laboratory-scale coAD. In a 17-L CSTR, Fantozzi and Buratti examined the yields of single-stage mesophilic coAD of various matrices, namely olive husk mixed with slurries and manure from different animal species (Fantozzi and Buratti, 2009). In particular, the blend of olive husk with anaerobically pre-digested pig manure resulted in a methane yield of 0.11 L/g_{VS}. A similar co-feedstock was used by Orive et al., who conducted a study on the coAD of 2P-OMSW and pig slurry (Orive et al., 2016). Five alternative feedstock proportions, ranging from 100 % pig slurry to 100 % OMSW, were tested in a 10-L sCSTR at 37 °C, and stable pH values (7.9–8.6) were observed throughout the experiments without the addition of any chemicals or nutrients. With an OMSW:pig slurry ratio of 80:20, an OLR of 2.72 g_{VS}/(L*d), and a HRT of 24 days, a maximum methane productivity of 0.27 L/(g_{VS}*d) was recorded. The high C/N ratio of raw OMSW was lowered from 46 (which would have hindered the mono-AD process) to 38 due to the dilution with pig slurry (with a C/N ratio of 15). The outcomes showed that 2P-OMSW and pig slurry co-digestion had high process stability and higher rate of methane generation than mono-digestion of the two individual feedstocks (0.14 L/(g_{VS}*d) for 100 % pig slurry and 0.08 L/(g_{VS}*d) for 100 % OMSW). Additionally the authors included a detailed report on the economic feasibility of a future industrial-scale anaerobic plant that could treat 8750 ton of 2P-OMSW and pig slurry (80:20) per year. The results of this study are compared against those obtained from a conventional pig slurry mono-AD plant. Revenues and payback time periods were reported to be € 362,338 per year and € 210,933 per year, 6.7 and 9.2 years for coAD and mono-AD plants, respectively, the latter leading to a unprofitable scenario.

As already mentioned for laboratory-scale experiments, cow manure is another widely available co-substrate. Biogas produced by the co-digestion of 2P-OMSW and cow excreta at a ratio of 1:3 (v/v) was examined by Goberna et al. (Goberna et al., 2010). The low pH and low alkalinity of OMSW were buffered by the manure, enabling the anaerobic biodegradation of the mixture in a 75-L CSTR without any pretreatment or chemical addition. After 21 days, the ratio of VFA to alkalinity in the reactor digesting OMSW alone at OLR of 3.8 g_{COD}/(L*d) was higher than 0.8, causing acidification and inhibiting methanogenesis. The co-digestion of the mixture under mesophilic conditions resulted in more than four times higher biogas productivity (1.1 L_{biogas}/(L_{sludge}*d)) compared to the sole excreta (0.25 L_{biogas}/(L_{sludge}*d)) and a methane yield of 0.18 L/g_{VS}, while the thermophilic operation allowed further 16.6 % and 18.2 % increases in methane yield and productivity, respectively. As anticipated, further studies are needed to assess whether this increase in methane production is economically beneficial for plant operators by calculating the energy consumption required to heat the digester to thermophilic temperatures. Riggio and colleagues in 2015 investigated the coAD of OMSW, cow slurry, and apple pulp in a 128-L pilot-scale anaerobic digester under mesophilic conditions (Riggio et al., 2015). They reported that the optimal mixture for biogas and methane production was 85 % cow slurry, 10 % OMSW, and 5 % apple pulp, by volume. Operating at a HRT of 40 days and an OLR of 2.75 g_{VS}/(L*d), biogas and methane yields were found to be 0.39 L_{biogas}/g_{VS} and 0.22 L_{CH4}/g_{VS}, respectively, and the COD removal 63 %. By comparing the above two papers that described the co-digestion of cow manure and OMSW, it is easy to notice that, despite some operational differences, methane yield was quite similar: 0.22 L/g_{VS} using 10 % OMSW (Riggio et al., 2015), and 0.18 L/g_{VS} using 25 % OMSW (Goberna et al., 2010). Similar values were obtained by Soldano et al., who used a smaller amount of cow manure (8–10 %) to co-digest 2P- or 3P-OMSW (46 %) and maize silage (44–46 %) (Soldano et al., 2014). In particular, in both cases a methane yield of 0.24–0.27 L_{CH4}/g_{VS} was obtained, even if special attention to the evolution of the main parameters for AD monitoring was required. All these values are significantly higher than those obtained by Fantozzi and Buratti with pig manure and olive husk (0.11 L_{CH4}/g_{VS}) (Fantozzi and Buratti, 2009). If a comparison is made between experiments carried out with animal manure at different scales, it can be noted that the biomethane production is higher at pilot-scale (0.89 L/(L*d)) than at laboratory-scale (0.34 L/(L*d))(Orive et al., 2016; Rubio et al., 2022), similarly to what observed when using OMSW and OMWW as co-feedstocks. Accordingly, the methane yield values are slightly higher (0.18 L/g_{VS} and 0.17 L/g_{VS}) than the one reported at laboratory-scale (0.13 L/g_{VS}), even if a higher amount of OMSW was used in the mixture in the latter case.

By using different wastes from cattle farms, Battista et al. tested milk whey, an important by-product of the dairy industry, as a co-feedstock for the coAD of OMSW (Battista et al., 2013). Today the dominant use of milk whey is as animal feed or to recover lactose and whey proteins, but such valorization technologies imply high investment costs (Prazeres et al., 2012). Here, a 45-L batch reactor was used in a first attempt to co-digest OMSW and milk whey. Over time, a feed combination of 50 % (w/w) OMSW and 50 % (w/w) OMWW was diluted with water, and starting from the 40th day onwards OMWW was gradually replaced with whey. The combination had 25 % (w/w) OMSW and 75 % (w/w) whey milk. By producing up to 0.8 L_{biogas}/(L*d), the authors observed great performance in biogas generation, and the VS removal remained stable at 64 %. The present findings confirm the suitability of whey as a viable substitute for water as a diluting agent, serving also to reduce the overall levels of polyphenols present in OMSW. In a related, later investigation, it was determined which process was the most profitable in terms of methane production by comparing 2P- and 3P-OMSW combined with OMWW and milk whey (Battista et al., 2015). The mixture had around a 3:1 ratio of milk whey to olive wastes (OMSW and OMWW). Methane production and yields from 3P-OMSW in a 50-L batch digester (0.10 L_{CH4}/g_{VS}, 0.25 L_{CH4}/$g_{\Delta VS}$, and 0.17 L_{CH4}/(L*d)) were about twice the one from 2P-OMSW (0.04 L_{CH4}/g_{VS}, 0.14 L_{CH4}/$g_{\Delta VS}$, and 0.06 L_{CH4}/(L*d)), likely because 2P-OMSW has a greater moisture content than its 3P counterpart, thereby leading to a concomitant increase in the levels of polyphenols (Fig. 1(b)). The methane yield obtained with 3P-OMSW was slightly higher than the one described by the same authors in their previous paper (0.22 L_{CH4}/$g_{\Delta VS}$) (Battista et al., 2013). It is noteworthy, however, that in both investigations the addition of whey increased the acidity (since the pH of milk is usually 3–3.5) and required pH adjustment in the digesters with a NaOH solution.lab

Lastly, Fragoso and colleagues in 2022 investigated the coAD of sewage sludge sourced from a wastewater treatment plant and partially dephenolised 2P-OMSW at a concentration of 5 % (v/v) as a co-substrate in a 16-L sCSTR, with the goal of enhancing the digestibility of both substrates (Fragoso et al., 2022). The transition from mono-AD of sewage sludge to coAD with OMSW resulted in a 1.4-fold increase in COD biodegradation at a HRT of 17 days and a 39 % increase in the average daily biogas productivity (from 0.28 L/(L*d) to 0.39 L/(L*d)). Furthermore, a notable enhancement was also observed in methane-specific productivity and yield (0.26 L/(L*d) and 0.28 L/g_{VS}, respectively). These data provide further confirmation of the feasibility of converting digesters that operate year-round by feeding them with feedstocks including OMSW during their production period (October – March). Analogous values were obtained by Paz et al. who similarly used a dephenolised OMSW and sewage sludge to feed a laboratory-scale AD and obtained a methane production of 0.26 L/(L*d) (Paz et al., 2023). Moreover, the methane yield value obtained by Fragoso et al. is also comparable to the ones obtained with sewage sludge by Elalami et al. (0.22 L/g_{VS}, with KOH-pretreated biomass)(Elalami et al., 2020b) and Aylin Alagöz et al. (0.26 L/g_{VS} and 0.32 Lg_{VS} when the biomass was pretreated with ultrasounds and microwaves, respectively)(Aylin Alagöz et al., 2015), even if in the latter cases the amount of OMSW in the digester was greater (~50 %).

Surprisingly, the results obtained by Orive et al. for mono-digestion of OMSW on a pilot-scale (Table 3) show higher methane productivity (1.53 L/(L*d), 0.42 L/(g_{VS}*d)) when compared with co-digestion experiments (Table 4) (Orive et al., 2021). Such results are a confirmation of the necessity to remove the inhibitory compounds such as polyphenols prior to the digestion of OMSW. Moreover, the extraction of such high-added-value compounds during pretreatment steps may aid in the spread of OMSW AD and help revalorize such by-products, since these molecules can be exploited in the pharmaceutical, cosmetics, and food sectors. As an example, Orive et al. obtained 0.4 mg of hydroxytyrosol and 0.09 mg of tyrosol per gram of dry OMSW, after the extraction with ethanol, water, and formic acid. Nowadays, such pure products have a selling price of € 23700/g and € 14650/g for hydrotyrosol and tyrosol, respectively (Sigma-Aldrich). Nevertheless, in-depth investigations are still needed to assess whether polyphenols extracted and purified from OMSW can economically compete with the existing commercial ones.

5. Industrial-scale anaerobic digestion of olive mill solid waste

The industrial-scale implementation of the AD process using exclusively OMSW as biomass has yet to be documented. This can be due to the long adaptation time of the inoculum, digester instability, and more importantly limited year-round availability (Stoyanova et al., 2017). As a result, the interest of the industrial sector in this technology using such substrate has not been adequately fostered. The implementation of AD plants on an industrial scale necessitates significant investments and operational expenditures, ultimately impeding the profitability of utilizing solely olive oil mill residues. The team of Battista et al. in 2013 was the first to scale up the co-digestion of 2P-OMSW (25 % w/w) and milk whey (75 % w/w) using an industrial 2 m^3 bioreactor (Battista et al., 2013). Prior to this achievement, various fed proportions were tested in a 45-L CSTR to determine the optimal feedstock composition, as stated in the preceding section. A pulper (with a working volume of 120 L) was employed to mechanically grind and homogenize the input material. Pulping avoids the risk of the formation of recalcitrant compounds, even if it still demands high electricity consumption. Biogas productivity achieved a value (1.45 L_{biogas}/(L*d)) significantly higher than that obtained with pilot-scale operations (0.78 L_{biogas}/(L*d)), with comparable methane content (65–70 % v/v). COD and VS removal percentages were also increased by employing industrial-scale digestors (72 % vs. 64 % and 68 % vs. 64 %, respectively). Such results were also confirmed by a subsequent study from the same authors when investigating the possibility of using either 3P-OMSW or 2P-OMSW (Battista et al., 2015). The industrial-scale results indicated that AD of 3P-OMSW ensured methane yield (0.74 L/g_{VS}) and productivity (0.87 L/(L*d)) more than double that obtained using 2P-OMSW (0.29 L/g_{VS} and 0.36 L/(L*d)), likely due, as mentioned before, to the higher 2P-OMSW moisture and polyphenols contents (130 mg $_{\text{gallic acid equivalent}}$/L) compared to 3P-OMSW (25 mg $_{\text{gallic acid equivalent}}$/L). Additionally, as is well known, 3P-OMSW exhibits higher total and volatile solids concentrations, which consequently enhance the conversion of organic matter into biogas. Comparison of results from the two different volume scales (Tables 4 and

Table 5
Industrial-scale co-digestion of olive mill solid waste.

Volume of reactor (L)	Type of reactor	Substrate	Pretreatment	HRT (days)	OLR	COD removal efficiency (%)	VS removal efficiency (%)	Methane production	Methane yield	Reference
200.000	Plug-in digesters	OMSW (90 %) + crushed cereals (10 %)	–	40	5.33 g_{VS}/(L*d)	–	–	3.74 L/(L*d)	0.70 L/g_{VS}	(Tamborrino et al., 2021)
1.130 (reactor) and6.400.000 (CSTR)	Horizontal reactor and two CSTRs in series	OMSW (7.1 %) + OMWW (13 %) + maize silage (30 %) + sheep manure (7.5 %) + whey (42.4 %)	–	29 (reactor), 81 (CSTR)	3.39 g_{VS}/(L *d) (reactor), 4.33 g_{VS}/(L *d) (CSTR)	–	66 (reactor)	1.41 L/(L*d) (reactor), 0.83 L/(L*d) (CSTR)	0.43 L/g_{VS} (reactor), 0.20 L/g_{VS} (CSTR)	(Scano et al., 2021)
2.000	CSTR	2P- or 3P-OMW + milk whey	Pulper	30	0.35 kg_{TS}/(L*d) (2P-OMW), 0.39 kg_{TS}/(L*d) (3P-OMW)	–	59 (2P-OMSW), 71 (3P-OMSW)	0.36 L/(L*d) (2P-OMW), 0.87 L/(L*d) (3P-OMW)	0.29 L/g_{VS}, 0.52 L/$g_{\Delta VS}$ (2P-OMW), 0.74 L/g_{VS}, 1.07 L/$g_{\Delta VS}$ (3P-OMW)	(Battista et al., 2015)
2.000	CSTR	2P-OMSW (25 %) + milk whey (75 %)	Pulper	30	–	72	68	1.02 L/(L*d)	0.20 L/g_{VS}	(Battista et al., 2013)

2P, two-phase centrifugal process; 3P, three-phase centrifugal process; ΔVS, difference between the initial and final amount of volatile solids; COD, chemical oxygen demand; CSTR, continuously stirred tank reactor; HRT, hydraulic retention time; OLR, organic loading rate; OMW, olive mill waste; OMSW, olive mill solid waste; OMWW, olive mill wastewater; VS, volatile solid.

5) shows that methane yield, calculated in the last 15 days of the experimental campaigns, when biogas production was in a quasi-steady-state condition, was much higher in the industrial-scale plant (0.74 and 0.29 L_{CH4}/g_{VS} from 3P- and 2P-OMSW, respectively) than in the pilot-scale one (0.10 and 0.04 L_{CH4}/g_{VS} from 3P- and 2P-OMSW, respectively). Similarly to the previous study, VS removal percentages were also increased by employing industrial-scale digestors (71 % vs. 44 % from 3P-OMSW and 59 % vs. 28 % from 2P-OMSW, digested in pilot- and industrial-scale plants, respectively). In 2019 Liberti et al. tested a CSTR with the addition of an incubation reactor (where a nutrient mix is added), in which the substrate from the acidogenic digester reacts with the nutrients (Liberti et al., 2019). They employed the digestate of the primary reactor (2000 m^3), made up of 62.28 % maize, sorghum, and triticale silage, 16.26 % humid pitted pomace, and 21.55 % pig wastewater as an inoculum for the secondary digester (5000 m^3). The nutrient mixture consisted of micronutrients and macronutrients that microorganisms need for optimal growth, which however were not described by the authors due to a confidentiality agreement with the company that developed the incubator system. Moreover, the authors reported only a limited amount of information on methane generation in such an industrial plant, choosing instead to focus on the study of its economic viability. Regarding this last aspect, the authors concluded that using such a mix of nutrients in a pretreatment reactor increased methane production by 12 %, which could have significant economic advantages. The payback period can diminish to 5.8, compared to the esteem of 7 calculated for the reference situation (i.e., AD without the incubator with the nutrient mix). In a following study, Scano et al. attempted to determine the optimal parameters for coAD of a mixture of sheep manure, whey, OMSW, and OMWW (Scano et al., 2021). The authors claimed that a combination of the aforementioned agro-industrial by-products could substitute dedicated crops (e.g., maize silage and triticale) at very high replacement rates (at least 65–70 %) in an industrial-scale AD reactor. The values for the C/N ratio were reported to be 21 for the feeding mixture and 13 for the digestate, suggesting the use of the latter as a soil amendment or as a source for other energy conversion processes.

A comparative analysis of the operational performance of a 1.13 m^3 and a 6400 m^3 reactor indicated that the larger configuration can employ OLR values comparable to those of the smaller counterpart, but with lower residence time. However, the smaller plant showed yields of biogas (0.64–0.86 L_{biogas}/g_{VS}) and methane (0.30–0.43 L_{CH4}/g_{VS}) approximately 1.8 times greater than the larger one (0.35–0.4 L_{biogas}/g_{VS}, 0.20 L_{CH4}/g_{VS}). The authors attempted to explain this discrepancy by highlighting the challenge of carefully mixing the substrate inside very large reactors, which led to a decrease in the rate of biomass conversion. If a comparison is made between such results and the one obtained at the pilot scale by different authors but with similar co-feedstocks (i.e., OMSW, cow manure, and maize silage), it can be noted that similar methane yield values (0.2–0.3 L/g_{VS}) were obtained. Such comparison is an excellent hint that pilot-scale studies can guide and predict the scaling-up of treatment processes and energy output of AD with OMSW and multiple co-feedstocks.

A recent study conducted by Tamborrino et al. on coAD of olive pulp, OMSW, and 10 % of crushed cereals in two parallel 200 m^3 digesters is worth mentioning (Tamborrino et al., 2021). Both the initial hydrolysis phase and the subsequent methanogenic one that converted acids from the previous phase into methane lasted approximately 20 days. The resulting biogas contained approximately 60 % methane, corresponding to a methane yield of 0.70 L/g_{VS} and methane production of 3.74 L/(L*d), which indicates a great potential for utilizing a large share of olive oil effluents to sustain an AD facility on an industrial scale. Such high methane yield resulted very similar to the value obtained by Battista et al. with milk whey, as described above (0.74 L/g_{VS}) (Battista et al., 2015). The results of physico-chemical analyses indicated that the process stability indexes, as well as the parameters characterizing the regularity of AD kinetics (e.g., pH, the ratio between volatile organic acids, and alkalinity), were within the optimal range. Such high methane production and yields reported for industrial-scale plants by Battista et al. and Tamborrino et al. (Table 5) are higher than any other data obtained at any digestion scale. Lastly, Tamborrino et al. reported the characteristics of digestate samples with particular reference to agronomic parameters. The pH values, metals, and microelements content are largely compatible with the requirements related to digestate distribution on soil as fertilizer. In particular, the C/N final value (9.5) shows that the discharged digestate can be considered an excellent base for the composting process. It can be observed that the most studied physico-chemical but also biological pretreatments at laboratory-scale have not been applied in industrial-scale plants. This could be due to the fact that published industrial-scale data involving the use of OMSW are very scarce. Moreover, since the availability of olive residues varies seasonally, most industrial-scale studies use cereal or animal residues (e.g., whey or manure) as the primary feedstock, avoiding the need for pretreatment steps or chemical addition.

6. Research needs and future directions

To date, investigation on the AD of OMSW for biogas production has predominantly concentrated on enhancing methane yield through the identification of optimal operational parameters or the application of different pretreatment methods involving biological, physical, or chemical techniques. In the case of OMSW, a pretreatment stage before the AD process could facilitate the hydrolysis of lignocellulosic fibers by disintegrating their complex chemical structure and separating the huge amount of phenolic compounds. Findings from the scientific literature allowed to establish a maximum threshold of 600 parts per million (ppm) of polyphenols during AD processes (Tamborrino et al., 2021). Nevertheless, pretreatment procedures require a substantial amount of energy, therefore a comprehensive assessment of the energy balance must be carried out to ascertain whether the increase in methane yield compensates for energy consumption, especially in large-scale systems. It has been frequently assumed that thermal processes, using the heat release from biogas combustion, are more desirable for the energy (and economic) balance of the whole AD system than technologies that use electrical power (e.g., ultrasound methods) (Carrère et al., 2010). As a result, thermal pretreatments are already frequently implemented in full-scale AD reactors. Nonetheless, more research is needed on simple and cost-effective technologies to increase OMSW biodegradability. Alternative and green methods should be proposed, such as the one described by Shabtay et al. (2009) who grew lignin-degrading fungi on OMSW. Here the authors demonstrated the ability of an edible mushroom strain to produce a wide range of extracellular enzymes that enable it to degrade lignin, cellulose, and hemicellulose into soluble substances that can be taken up by the fungus.

Other limitations of AD of OMSW could be overcome by the addition of nitrogen (e.g., urea, NH_4Cl, and aqueous ammonia) and phosphorous (e.g., K_2HPO_4 and $(NH_4)_2HPO_4$) or trace elements in pretreatment stages, in order to compensate for their low levels in the original feedstock (Pinto-Ibieta et al., 2016). Acidity is commonly corrected with the addition of salts or hydroxides, such as $NaHCO_3$, NaOH, or $Ca(OH)_2$. However, all these chemical compounds are not considered neither environmentally nor economically friendly, with ecological risk assessment being needed especially regarding the fate of digestate. This last aspect (i.e., the characterization of the remaining digestate) is often overlooked while the fertilization value and chemical composition of digestate should be more in-depth investigated and compared. In general, few researchers have carried out economic evaluations of the application of additives or pretreatment processes for AD, especially on large-scale reactors. Achieving an optimal balance between capital and operational costs and the increment in biogas production requires a more complete and standardized approach (Ibarra-Esparza et al., 2023).

Regarding future perspectives, it is crucial to focus on replicating the most promising laboratory settings at the pilot/industrial level to investigate the technical scalability obstacles and alleviate the residual uncertainties linked to the economy of scale (e.g., transportation costs for co-feedstocks, and energy consumption). To help in designing industrial plants and predict their behavior, several computational models could be developed that take into account the complex aspects of the AD process. Some of them are already reported in the literature and are described as able to provide results close to the ones obtained with experimental trials on pilot-scale digesters (Boubaker and Ridha, 2008). In general, there is a broad need for more published data from laboratory-, pilot-, and particularly full-scale (co)AD. A comparison of methane production values ($L_{CH4}/(L_{reactor}*d)$) among results obtained with different reactor volumes is shown in Fig. 4(c, d). Several factors are affecting methane production, such as OLR, HRT, and the ratio between OMSW and the eventual co-feedstock, leading only to the possibility of a preliminary comparison. It is clear that very large discrepancies exist between research studies using similar pretreatments or co-feedstocks. As be can observed, data should not be directly projected from laboratory-scale to industrial-scale designs, especially due to the different hydrodynamic conditions. Aspects still poorly investigated are coAD of OMSW and manure at laboratory-scale level (e.g., to detect differences among various animal sources), and coAD of OMSW with the organic fraction of municipal solid waste (OFMSW) at all scale levels, being nowadays OFWSW one of the most employed substrates in full-scale AD reactors (Zamri et al., 2021).

In the last ten years (2013–2023), the number of publications on pilot- and industrial-scale AD progressively increased (Fig. 4(e, f)). In particular, the last few years (2021–2023) have been the most prolific in publications. In 2022, 51 documents (41 for pilot-scale and 10 for industrial-scale AD) were recorded versus 18 in 2013 (16 for pilot-scale and 2 for industrial-scale AD). Therefore, the evolution of publication demonstrated that, nowadays, there is a progressive trend in the study of AD technology at larger-scales. This increase can be explained by a strong need for clean production of bioenergy and products, as required by the SDG agenda. However, such a trend was not detected when shifting to OMSW AD on pilot- and industrial-scales, with only 4 articles published in the last 5 years (2 on pilot-scale and 2 on industrial-scale OMSW AD). More connections are hoped between academic institutions/research centers and industries, which could boost local economies and enhance innovation through knowledge exchange. In fact, the numerous advantages offered by AD processes, as well as generous national incentives, have led to the installation of more than 18,000 anaerobic digestion plants in Europe, according to the European Biogas Association. In the latest REPowerEU scheme, the European Commission established a goal of generating 38 billion cubic meters of biomethane annually through AD by 2030. Manure will account for 33 % of all feedstocks, followed by agricultural residues (25 %), and sequential cropping (21 %) (European Commission, 2022). However, co-digestion requires more research into a variety of bio-resources and their specific blend proportions with OMSW (Kunatsa and Xia, 2022). It should be noted that, given the seasonality of OMSW production and the scattered distribution of olive mills across the territories, the best option might be provided by small pilot-scale plants. This could create opportunities for AD plant construction even in developing countries and rural areas. Additionally, the smaller plants may help to reduce concern about bad smells and loud sounds, which are more noticeable in bigger facilities (Chodkowska-Miszczuk et al., 2019). Even better, a potentially viable solution could be the conversion of existing pilot-scale AD plants treating animal and farm waste to co-digestion plants that combine those residues with olive oil residues for a short period of time. Significant economic benefits related to reduced plant operation costs can be achieved as well as an increase in process performance. Accordingly, Bacenetti et al. pointed out that it is environmentally preferred to have smaller plants using slurry and waste rather than bigger plants fed with energy crops (Bacenetti et al., 2016). More studies are needed to evaluate the environmental and economic sustainability of biogas generated through coAD of OMSW with different feedstocks, with life cycle assessment and life cycle costing studies only being recently carried out. The impacts of such systems vary mainly according to the characteristics and availability of the co-substrates. However, coAD with manure is considered the preferable treatment method due to its best performance in most environmental impact assessment analyses (Zhang et al., 2021). Recently Balcioglu et al. evaluated biogas plants fed with chicken manure, organic wastes (vegetable and slaughterhouse wastes), and crop maize silage, and found that using feedstocks with high solid content and biogas yield (i.e., organic wastes and chicken manure) led to lower environmental impacts in 15 out of 17 categories considered, including climate change (Balcioglu et al., 2022). This is feasible since the manure and slurries are a waste product of other processes, therefore there is no environmental impact related to them. Furthermore, credits for the emissions saved during their traditional treatment in open tanks were frequently associated with their AD processes (Bacenetti et al., 2016).

7. Conclusions

Primary issues of AD with OMSW are seasonality, low hydrolysis rate due to lignocellulosic materials, and presence of polyphenols. Caution must be exercised when projecting results from laboratory-scale studies straight to industrial-scale reactors. While various pretreatment strategies are being proposed, coAD with animal by-products seems the most promising solution due to its high alkalinity and the simultaneous dilution of inhibitory elements of OMSW. The comprehensive assessment of their environmental and economic benefits will help AD become a more attractive technology for managing OMSW, as it will enable the disposal of polluting waste and the production of green fertilizers and energy.

CRediT authorship contribution statement

Martina Lenzuni: Writing – review & editing, Writing – original draft, Methodology, Data curation, Conceptualization. **Attilio Converti:** Writing – review & editing, Writing – original draft, Supervision, Project administration, Funding acquisition, Conceptualization. **Alessandro Alberto Casazza:** Writing – review & editing, Writing – original draft, Supervision, Methodology, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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References

Aboelfetoh, M., Hassanein, A., Ragab, M., El-kassas, M., Marzouk, E.R., 2022. Olive mill waste-based anaerobic digestion as a source of local renewable energy and nutrients. Sustainability 14, 1402. https://doi.org/10.3390/su14031402.

Ağdağ, O.N., 2011. Biodegradation of olive-mill pomace mixed with organic fraction of municipal solid waste. Biodegradation 22, 931–938. https://doi.org/10.1007/s10532-010-9452-y.

Al Afif, R., Amon, T., 2019. Mesophilic anaerobic co-digestion of cow manure with three-phase olive mill solid waste. energy Sources Part Recovery Util. Environ. Eff. 41, 1800–1808. https://doi.org/10.1080/15567036.2018.1549157.

Al Afif, R., Linke, B., 2019. Biogas production from three-phase olive mill solid waste in lab-scale continuously stirred tank reactor. Energy 171, 1046–1052. https://doi.org/10.1016/j.energy.2019.01.080.

Alburquerque, J.A., Gonzálvez, J., García, D., Cegarra, J., 2004. Agrochemical characterisation of "alperujo", a solid by-product of the two-phase centrifugation method for olive oil extraction. Bioresour. Technol. 91, 195–200. https://doi.org/10.1016/S0960-8524(03)00177-9.

Alburquerque, J.A., de la Fuente, C., Ferrer-Costa, A., Carrasco, L., Cegarra, J., Abad, M., Bernal, M.P., 2012. Assessment of the fertiliser potential of digestates from farm and agroindustrial residues. Biomass Bioenergy 40, 181–189. https://doi.org/10.1016/j.biombioe.2012.02.018.

Aliakbarian, B., Casazza, A.A., Perego, P., 2011. Valorization of olive oil solid waste using high pressure–high temperature reactor. Food Chem. 128, 704–710. https://doi.org/10.1016/j.foodchem.2011.03.092.

Al-Mallahi, J., Furuichi, T., Ishii, K., 2016. Appropriate conditions for applying NaOH-pretreated two-phase olive milling waste for codigestion with food waste to enhance biogas production. Waste Manag. 48, 430–439. https://doi.org/10.1016/j.wasman.2015.10.009.

Aylin Alagöz, B., Yenigün, O., Erdinçler, A., 2015. Enhancement of anaerobic digestion efficiency of wastewater sludge and olive waste: Synergistic effect of co-digestion and ultrasonic/microwave sludge pre-treatment. Waste Manag. 46, 182–188. https://doi.org/10.1016/j.wasman.2015.08.020.

Bacenetti, J., Sala, C., Fusi, A., Fiala, M., 2016. Agricultural anaerobic digestion plants: What LCA studies pointed out and what can be done to make them more environmentally sustainable. Appl. Energy 179, 669–686. https://doi.org/10.1016/j.apenergy.2016.07.029.

Balcioglu, G., Jeswani, H.K., Azapagic, A., 2022. Evaluating the environmental and economic sustainability of energy from anaerobic digestion of different feedstocks in Turkey. Sustain. Prod. Consum. 32, 924–941. https://doi.org/10.1016/j.spc.2022.06.011.

Battista, F., Ruggeri, B., Fino, D., Erriquens, F., Rutigliano, L., Mescia, D., 2013. Toward the scale-up of agro-food feed mixture for biogas production. J. Environ. Chem. Eng. 1, 1223–1230. https://doi.org/10.1016/j.jece.2013.09.008.

Battista, F., Fino, D., Erriquens, F., Mancini, G., Ruggeri, B., 2015. Scaled-up experimental biogas production from two agro-food waste mixtures having high inhibitory compound concentrations. Renew. Energy 81, 71–77. https://doi.org/10.1016/j.renene.2015.03.007.

Bird, H., Heidrich, E.S., Leicester, D.D., Theodosiou, P., 2022. Pilot-scale Microbial Fuel Cells (MFCs): A meta-analysis study to inform full-scale design principles for optimum wastewater treatment. J. Clean. Prod. 346, 131227 https://doi.org/10.1016/j.jclepro.2022.131227.

Bolzonella, D., Fatone, F., Gottardo, M., Frison, N., 2018. Nutrients recovery from anaerobic digestate of agro-waste: Techno-economic assessment of full scale applications. Sustain. Waste Wastewater Manag. 216, 111–119. https://doi.org/10.1016/j.jenvman.2017.08.026.

Borja, R., Rincón, B., Raposo, F., Alba, J., Martín, A., 2002. A study of anaerobic digestibility of two-phases olive mill solid waste (OMSW) at mesophilic temperature. Process Biochem. 38, 733–742. https://doi.org/10.1016/S0032-9592(02)00202-9.

Borja, R., Rincón, B., Raposo, F., Sánchez, E., Martín, A., 2004. Assessment of kinetic parameters for the mesophilic anaerobic biodegradation of two-phase olive pomace. Int. Biodeterior. Biodegrad. 53, 71–78. https://doi.org/10.1016/j.ibiod.2003.10.002.

Borja, R., Martín, A., Sánchez, E., Rincón, B., Raposo, F., 2005. Kinetic modelling of the hydrolysis, acidogenic and methanogenic steps in the anaerobic digestion of two-phase olive pomace (TPOP). Process Biochem. 40, 1841–1847. https://doi.org/10.1016/j.procbio.2004.06.026.

Borja, R., Rincón, B., Raposo, F., 2006. Anaerobic biodegradation of two-phase olive mill solid wastes and liquid effluents: kinetic studies and process performance. J. Chem. Technol. Biotechnol. 81, 1450–1462. https://doi.org/10.1002/jctb.1563.

Boubaker, F., Ridha, B.C., 2008. Modelling of the mesophilic anaerobic co-digestion of olive mill wastewater with olive mill solid waste using anaerobic digestion model No. 1 (ADM1). Bioresour. Technol. 99, 6565–6577. https://doi.org/10.1016/j.biortech.2007.11.035.

Capello, C., Fischer, U., Hungerbühler, K., 2007. What is a green solvent? A comprehensive framework for the environmental assessment of solvents. Green Chem. 9, 927–934. https://doi.org/10.1039/B617536H.

Cappelletti, G.M., Ioppolo, G., Nicoletti, G.M., Russo, C., 2014. Energy requirement of extra virgin olive oil production. Sustainability 6, 4966–4974. https://doi.org/10.3390/su6084966.

Carrere, H., Antonopoulou, G., Affes, R., Passos, F., Battimelli, A., Lyberatos, G., Ferrer, I., 2016. Review of feedstock pretreatment strategies for improved anaerobic digestion: from lab-scale research to full-scale application. Pretreat. Biomass 199, 386–397. https://doi.org/10.1016/j.biortech.2015.09.007.

Carrère, H., Dumas, C., Battimelli, A., Batstone, D.J., Delgenès, J.P., Steyer, J.P., Ferrer, I., 2010. Pretreatment methods to improve sludge anaerobic degradability: a review. J. Hazard. Mater. 183, 1–15. https://doi.org/10.1016/j.jhazmat.2010.06.129.

Černe, M., Palčić, I., Major, N., Pasković, I., Perković, J., Užila, Z., Lukić, M., Romić, M., Zubin Ferri, T., Goreta Ban, S., Lapanje, A., Hreščak, J., Heath, D.J., Ban, D., 2023. Effect of olive-processing technology on the utilization of olive mill pomace as a soil amendment. J. Environ. Qual. 52, 610–629. https://doi.org/10.1002/jeq2.20464.

Chodkowska-Miszczuk, J., Martinat, S., Cowell, R., 2019. Community tensions, participation, and local development: Factors affecting the spatial embeddedness of anaerobic digestion in Poland and the Czech Republic. Energy Res. Soc. Sci. 55, 134–145. https://doi.org/10.1016/j.erss.2019.05.010.

Christoforou, E., Fokaides, P.A., 2016. A review of olive mill solid wastes to energy utilization techniques. Waste Manag. 49, 346–363. https://doi.org/10.1016/j.wasman.2016.01.012.

European Commission, 2022. RePowerEU: A plan to rapidly reduce dependence on Russian fossil fuels and fast forward the green transition. J Eur Union.

Contardi, M., Lenzuni, M., Fiorentini, F., Summa, M., Bertorelli, R., Suarato, G., Athanassiou, A., 2021. Hydroxycinnamic acids and derivatives formulations for skin damages and disorders: a review. Pharmaceutics 13. https://doi.org/10.3390/pharmaceutics13070999.

De La Lama, D., Borja, R., Rincón, B., 2017. Performance evaluation and substrate removal kinetics in the semi-continuous anaerobic digestion of thermally pretreated two-phase olive pomace or "Alperujo". Process Saf. Environ. Prot. 105, 288–296. https://doi.org/10.1016/j.psep.2016.11.014.

De la Rubia, M.A., Villamil, J.A., Rodriguez, J.J., Mohedano, A.F., 2018. Effect of inoculum source and initial concentration on the anaerobic digestion of the liquid fraction from hydrothermal carbonisation of sewage sludge. Renew. Energy 127, 697–704. https://doi.org/10.1016/j.renene.2018.05.002.

Dermeche, S., Nadour, M., Larroche, C., Moulti-Mati, F., Michaud, P., 2013. Olive mill wastes: Biochemical characterizations and valorization strategies. Process Biochem. 48, 1532–1552. https://doi.org/10.1016/j.procbio.2013.07.010.

Di Giacomo, G., Romano, P., 2022. Evolution of the olive oil industry along the entire production chain and related waste management. Energies 15, 465. https://doi.org/10.3390/en15020465.

Directorate-General for Agriculture and Rural Development, 2022. Producing 69% of the world's production, the EU is the largest producer of olive oil.

Elalami, D., Carrere, H., Abdelouahdi, K., Garcia-Bernet, D., Peydecastaing, J., Vaca-Medina, G., Oukarroum, A., Zeroual, Y., Barakat, A., 2020a. Mild microwaves, ultrasonic and alkaline pretreatments for improving methane production: Impact on biochemical and structural properties of olive pomace. Bioresour. Technol. 299, 122591 https://doi.org/10.1016/j.biortech.2019.122591.

Elalami, D., Monlau, F., Carrere, H., Abdelouahdi, K., Oukarroum, A., Zeroual, Y., Barakat, A., 2020b. Effect of coupling alkaline pretreatment and sewage sludge co-digestion on methane production and fertilizer potential of digestate. Sci. Total Environ. 743, 140670 https://doi.org/10.1016/j.scitotenv.2020.140670.

Evranos, B., Demirel, B., 2015. The impact of Ni, Co and Mo supplementation on methane yield from anaerobic mono-digestion of maize silage. Environ. Technol. 36, 1556–1562. https://doi.org/10.1080/09593330.2014.997297.

Fantozzi, F., Buratti, C., 2009. Biogas production from different substrates in an experimental Continuously Stirred Tank Reactor anaerobic digester. Bioresour. Technol. 100, 5783–5789. https://doi.org/10.1016/j.biortech.2009.06.013.

Fernández-Rodríguez, M.J., de la Lama-Calvente, D., García-González, M., Moreno-Fernández, J., Jiménez-Rodríguez, A., Borja, R., Rincón-Llorente, B., 2022. Integral valorization of two-phase olive mill solid waste (OMSW) and related washing waters by anaerobic co-digestion of OMSW and the microalga raphidocelis subcapitata cultivated in these effluents. J. Agric. Food Chem. 70, 3219–3227. https://doi.org/10.1021/acs.jafc.1c08100.

Fezzani, B., Cheikh, R.B., 2007a. Anaerobic co-digestion of olive mill wastewater with olive mill solid waste in a tubular digester at mesophilic temperature. Bioresour. Technol. 98, 769–774. https://doi.org/10.1016/j.biortech.2006.04.020.

Fezzani, B., Cheikh, R.B., 2007b. Thermophilic anaerobic co-digestion of olive mill wastewater with olive mill solid wastes in a tubular digester. Chem. Eng. J. 132, 195–203. https://doi.org/10.1016/j.cej.2006.12.017.

Fezzani, B., Cheikh, R.B., 2008. Optimisation of the mesophilic anaerobic co-digestion of olive mill wastewater with olive mill solid waste in a batch digester. Desalination 228, 159–167. https://doi.org/10.1016/j.desal.2007.09.007.

Fezzani, B., Cheikh, R., 2010. Two-phase anaerobic co-digestion of olive mill wastes in semi-continuous digesters at mesophilic temperature. Bioresour. Technol. 101, 1628–1634. https://doi.org/10.1016/j.biortech.2009.09.067.

Fragoso, R., Henriques, A.C., Ochando-Pulido, J., Smozinski, N., Duarte, E., 2022. Enhanced biomethane production by co-digestion of mixed sewage sludge and dephenolised two-phase olive pomace. Waste Manag. Res. J. Sustain. Circ. Econ. 40, 565–574. https://doi.org/10.1177/0734242X211003979.

Ge, X., Xu, F., Li, Y., 2016. Solid-state anaerobic digestion of lignocellulosic biomass: recent progress and perspectives. Bioresour. Technol. 205, 239–249. https://doi.org/10.1016/j.biortech.2016.01.050.

Goberna, M., Schoen, M.A., Sperl, D., Wett, B., Insam, H., 2010. Mesophilic and thermophilic co-fermentation of cattle excreta and olive mill wastes in pilot anaerobic digesters. Biomass Bioenergy 34, 340–346. https://doi.org/10.1016/j.biombioe.2009.11.005.

Gunay, A., Karadag, D., 2015. Recent developments in the anaerobic digestion of olive mill effluents. Process Biochem. 50, 1893–1903. https://doi.org/10.1016/j.procbio.2015.07.008.

Ibarra-Esparza, F.E., González-López, M.E., Ibarra-Esparza, J., Lara-Topete, G.O., Senés-Guerrero, C., Cansdale, A., Forrester, S., Chong, J.P.J., Gradilla-Hernández, M.S., 2023. Implementation of anaerobic digestion for valorizing the organic fraction of municipal solid waste in developing countries: technical insights from a systematic review. J. Environ. Manage. 347, 118993 https://doi.org/10.1016/j.jenvman.2023.118993.

Justino, C.I.L., Pereira, R., Freitas, A.C., Rocha-Santos, T.A.P., Panteleitchouk, T.S.L., Duarte, A.C., 2012. Olive oil mill wastewaters before and after treatment: a critical review from the ecotoxicological point of view. Ecotoxicology 21, 615–629. https://doi.org/10.1007/s10646-011-0806-y.

Koch, K., Hafner, S.D., Weinrich, S., Astals, S., Holliger, C., 2020. Power and limitations of biochemical methane potential (BMP) tests. Front. Energy Res. 8 https://doi.org/10.3389/fenrg.2020.00063.

Koutrouli, E.C., Kalfas, H., Gavala, H.N., Skiadas, I.V., Stamatelatou, K., Lyberatos, G., 2009. Hydrogen and methane production through two-stage mesophilic anaerobic digestion of olive pulp. Bioresour. Technol. 100, 3718–3723. https://doi.org/10.1016/j.biortech.2009.01.037.

Kunatsa, T., Xia, X., 2022. A review on anaerobic digestion with focus on the role of biomass co-digestion, modelling and optimisation on biogas production and enhancement. Bioresour. Technol. 344, 126311 https://doi.org/10.1016/j.biortech.2021.126311.

Li, Y., Li, L., Sun, Y., Yuan, Z., 2018. Bioaugmentation strategy for enhancing anaerobic digestion of high C/N ratio feedstock with methanogenic enrichment culture. Bioresour. Technol. 261, 188–195. https://doi.org/10.1016/j.biortech.2018.02.069.

Li, Y., Zhao, J., Krooneman, J., Euverink, G.J.W., 2021. Strategies to boost anaerobic digestion performance of cow manure: Laboratory achievements and their full-scale application potential. Sci. Total Environ. 755, 142940 https://doi.org/10.1016/j.scitotenv.2020.142940.

Liberti, F., Pistolesi, V., Mouftahi, M., Hidouri, N., Bartocci, P., Massoli, S., Zampilli, M., Fantozzi, F., 2019. An incubation system to enhance biogas and methane production: a case study of an existing biogas plant in umbria. Italy. Processes 7, 925. https://doi.org/10.3390/pr7120925.

Mabutyana, L., Pott, R.W.M., 2021. Photo-fermentative hydrogen production by Rhodopseudomonas palustris CGA009 in the presence of inhibitory compounds. Int. J. Hydrog. Energy 46, 29088–29099. https://doi.org/10.1016/j.ijhydene.2020.12.189.

Mahata, C., Das, D., 2022. Current Status and Prospects of Biohydrogen Production Process, in: Saini, J.K., Sani, R.K. (Eds.), Microbial Biotechnology for Renewable and Sustainable Energy. Springer Nature Singapore, Singapore, pp. 99–133. https://doi.org/10.1007/978-981-16-3852-7_5.

Manthos, G., Zagklis, D., Zafiri, C., Kornaros, M., 2023. Comparative life cycle assessment of anaerobic digestion, lagoon evaporation, and direct land application of olive mill wastewater. Bioresour. Technol. 388, 129778 https://doi.org/10.1016/j.biortech.2023.129778.

Messineo, A., Maniscalco, M.P., Volpe, R., 2020. Biomethane recovery from olive mill residues through anaerobic digestion: A review of the state of the art technology. Sci. Total Environ. 703, 135508 https://doi.org/10.1016/j.scitotenv.2019.135508.

Orive, M., Cebrián, M., Zufía, J., 2016. Techno-economic anaerobic co-digestion feasibility study for two-phase olive oil mill pomace and pig slurry. Renew. Energy 97, 532–540. https://doi.org/10.1016/j.renene.2016.06.019.

Orive, M., Cebrián, M., Amayra, J., Zufía, J., Bald, C., 2021. Integrated biorefinery process for olive pomace valorisation. Biomass Bioenergy 149, 106079. https://doi.org/10.1016/j.biombioe.2021.106079.

Paz, A., Solisio, C., Converti, A., Casazza, A.A., 2023. Application of organosolv technology to improve the anaerobic digestion of olive oil pomace. Ind. Crops Prod. 204, 117249 https://doi.org/10.1016/j.indcrop.2023.117249.

Pinto-Ibieta, F., Serrano, A., Jeison, D., Borja, R., Fermoso, F.G., 2016. Effect of cobalt supplementation and fractionation on the biological response in the biomethanization of Olive Mill Solid Waste. Bioresour. Technol. 211, 58–64. https://doi.org/10.1016/j.biortech.2016.03.031.

Prazeres, A.R., Carvalho, F., Rivas, J., 2012. Cheese whey management: A review. J. Environ. Manage. 110, 48–68. https://doi.org/10.1016/j.jenvman.2012.05.018.

Restuccia, D., Prencipe, S.A., Ruggeri, M., Spizzirri, U.G., 2022. Sustainability assessment of different extra virgin olive oil extraction methods through a life cycle thinking approach: challenges and opportunities in the elaio-technical sector. Sustainability 14. https://doi.org/10.3390/su142315674.

Riggio, V., Comino, E., Rosso, M., 2015. Energy production from anaerobic co-digestion processing of cow slurry, olive pomace and apple pulp. Renew. Energy 83, 1043–1049. https://doi.org/10.1016/j.renene.2015.05.056.

Rincón, B., Raposo, F., Borja, R., Gonzalez, J.M., Portillo, M.C., Saiz-Jimenez, C., 2006. Performance and microbial communities of a continuous stirred tank anaerobic reactor treating two-phases olive mill solid wastes at low organic loading rates. J. Biotechnol. 121, 534–543. https://doi.org/10.1016/j.jbiotec.2005.08.013.

Rincón, B., Borja, R., González, J.M., Portillo, M.C., Sáiz-Jiménez, C., 2008a. Influence of organic loading rate and hydraulic retention time on the performance, stability and microbial communities of one-stage anaerobic digestion of two-phase olive mill solid residue. Biochem. Eng. J. 40, 253–261. https://doi.org/10.1016/j.bej.2007.12.019.

Rincón, B., Sánchez, E., Raposo, F., Borja, R., Travieso, L., Martín, M.A., Martín, A., 2008b. Effect of the organic loading rate on the performance of anaerobic acidogenic fermentation of two-phase olive mill solid residue. Waste Manag. 28, 870–877. https://doi.org/10.1016/j.wasman.2007.02.030.

Rincón, B., Borja, R., Martín, M.A., Martín, A., 2009. Evaluation of the methanogenic step of a two-stage anaerobic digestion process of acidified olive mill solid residue from a previous hydrolytic–acidogenic step. Waste Manag. 29, 2566–2573. https://doi.org/10.1016/j.wasman.2009.04.009.

Rincón, B., Bujalance, L., Fermoso, F.G., Martín, A., Borja, R., 2013. Biochemical methane potential of two-phase olive mill solid waste: Influence of thermal pretreatment on the process kinetics. Bioresour. Technol. 140, 249–255. https://doi.org/10.1016/j.biortech.2013.04.090.

Rodriguez-Perez, S., Serrano, A., Pantión, A.A., Alonso-Fariñas, B., 2018. Challenges of scaling-up PHA production from waste streams. A Review. J. Environ. Manage. 205, 215–230. https://doi.org/10.1016/j.jenvman.2017.09.083.

Rouvalis, A., Iliopoulou-Georgudaki, J., 2010. Comparative assessment of olive oil mill effluents from three-phase and two-phase systems, treated for hydrogen production. Bull. Environ. Contam. Toxicol. 85, 432–436. https://doi.org/10.1007/s00128-010-0106-x.

Rubio, J.A., Fdez-Güelfo, L.A., Romero-García, L.I., Wilkie, A.C., García-Morales, J.L., 2022. Start-up of the mesophilic anaerobic co-digestion of two-phase olive-mill waste and cattle manure using volatile fatty acids as process control parameter. Fuel 325, 124901. https://doi.org/10.1016/j.fuel.2022.124901.

Scano, E.A., Grosso, M., Pistis, A., Carboni, G., Cocco, D., 2021. An in-depth analysis of biogas production from locally agro-industrial by-products and residues. An Italian Case. Renew. Energy 179, 308–318. https://doi.org/10.1016/j.renene.2021.07.050.

Sellami, F., Jarboui, R., Hachicha, S., Medhioub, K., Ammar, E., 2008. Co-composting of oil exhausted olive-cake, poultry manure and industrial residues of agro-food activity for soil amendment. Bioresour. Technol. 99, 1177–1188. https://doi.org/10.1016/j.biortech.2007.02.018.

Serrano, A., Fermoso, F.G., Alonso-Fariñas, B., Rodríguez-Gutiérrez, G., López, S., Fernandez-Bolaños, J., Borja, R., 2019a. Long-Term Evaluation of Mesophilic Semi-Continuous Anaerobic Digestion of Olive Mill Solid Waste Pretreated with Steam-Explosion. Energies 12, 2222. https://doi.org/10.3390/en12112222.

Serrano, A., Fermoso, F.G., Alonso-Fariñas, B., Rodríguez-Gutiérrez, G., López, S., Fernandez-Bolaños, J., Borja, R., 2019b. Performance evaluation of mesophilic semi-continuous anaerobic digestion of high-temperature thermally pre-treated olive mill solid waste. Waste Manag. 87, 250–257. https://doi.org/10.1016/j.wasman.2019.02.003.

Serrano, A., Villa-Gomez, D., Fermoso, F.G., Alonso-Fariñas, B., 2021. Is anaerobic digestion a feasible alternative to the combustion of olive mill solid waste in terms of energy production? A Critical Review. Biofuels Bioprod. Biorefining 15, 150–162. https://doi.org/10.1002/bbb.2159.

Shabtay, A., Hadar, Y., Eitam, H., Brosh, A., Orlov, A., Tadmor, Y., Izhaki, I., Kerem, Z., 2009. The potential of Pleurotus-treated olive mill solid waste as cattle feed. Bioresour. Technol. 100, 6457–6464. https://doi.org/10.1016/j.biortech.2009.07.044.

Siciliano, A., Stillitano, M.A., De Rosa, S., 2016. Biogas production from wet olive mill wastes pretreated with hydrogen peroxide in alkaline conditions. Renew. Energy 85, 903–916. https://doi.org/10.1016/j.renene.2015.07.029.

Soldano, M., Labartino, N., Rossi, L., Fabbri, C., Piccinini, S., 2014. Recovery of Agro-Industrial By-Products for Anaerobic Digestion:olive Pomace and Citrus Pulp. Proc. 22nd Eur. Biomass Conf. Exhib. 23-26 June 2014, 3 Pages. https://doi.org/10.5071/22NDEUBCE2014-1CV.3.43.

Stoyanova, E., Lundaa, T., Bochmann, G., Fuchs, W., 2017. Overcoming the bottlenecks of anaerobic digestion of olive mill solid waste by two-stage fermentation. Environ. Technol. 38, 394–405. https://doi.org/10.1080/09593330.2016.1196736.

Tamborrino, A., Catalano, F., Leone, A., Bianchi, B., 2021. A real case study of a full-scale anaerobic digestion plant powered by olive by-products. Foods 10. https://doi.org/10.3390/foods10081946.

Tekin, A.R., Dalgıç, A.C., 2000. Biogas production from olive pomace. Resour. Conserv. Recycl. 30, 301–313. https://doi.org/10.1016/S0921-3449(00)00067-7.

Vlyssides, A.G., Loizides, M., Karlis, P.K., 2004. Integrated strategic approach for reusing olive oil extraction by-products. J. Clean. Prod. 12, 603–611. https://doi.org/10.1016/S0959-6526(03)00078-7.

Wang, W., Lee, D.-J., 2021. Valorization of anaerobic digestion digestate: a prospect review. Bioresour. Technol. 323, 124626 https://doi.org/10.1016/j.biortech.2020.124626.

Wang, M., Sun, X., Li, P., Yin, L., Liu, D., Zhang, Y., Li, W., Zheng, G., 2014. A novel alternate feeding mode for semi-continuous anaerobic co-digestion of food waste with chicken manure. Bioresour. Technol. 164, 309–314. https://doi.org/10.1016/j.biortech.2014.04.077.

Williams, O., Eastwick, C., Kingman, S., Giddings, D., Lormor, S., Lester, E., 2017. Overcoming the caking phenomenon in olive mill wastes. Ind. Crops Prod. 101, 92–102. https://doi.org/10.1016/j.indcrop.2017.02.036.

Yan, Y.-J., Li, X., Lu, C.-S., Kobayashi, T., Zhen, G.-Y., Hu, Y., 2023. A review on start-up phase optimization of kitchen waste anaerobic digestion. Fermentation 9. https://doi.org/10.3390/fermentation9070603.

Zamri, M.F.M.A., Hasmady, S., Akhiar, A., Ideris, F., Shamsuddin, A.H., Mofijur, M., Fattah, I.M.R., Mahlia, T.M.I., 2021. A comprehensive review on anaerobic digestion of organic fraction of municipal solid waste. Renew. Sustain. Energy Rev. 137, 110637 https://doi.org/10.1016/j.rser.2020.110637.

Zhang, Y., Jiang, Y., Wang, S., Wang, Z., Liu, Y., Hu, Z., Zhan, X., 2021. Environmental sustainability assessment of pig manure mono- and co-digestion and dynamic land application of the digestate. Renew. Sustain. Energy Rev. 137, 110476 https://doi.org/10.1016/j.rser.2020.110476.

Zhang, L., Lim, E.Y., Loh, K.-C., Ok, Y.S., Lee, J.T.E., Shen, Y., Wang, C.-H., Dai, Y., Tong, Y.W., 2020. Biochar enhanced thermophilic anaerobic digestion of food waste: focusing on biochar particle size, microbial community analysis and pilot-scale application. Energy Convers. Manag. 209, 112654 https://doi.org/10.1016/j.enconman.2020.112654.