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ORIGINAL PAPER

Improving Anaerobic Co-digestion of Sewage Sludge with Thermal Dried Olive Mill Wastewater

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Abstract

Anaerobic co-digestion of olive mill wastewater (OMW) and sewage sludge (SS) is a promising technique in terms of biogas production and quality. An OMW mixture was subjected to pre-treatment (thermal drying), which can reduce the total COD by 72.8% of 10% OMW mixture. OMW mixtures at concentrations of 2, 5, and 10% were tested. OMW addition can improve biogas production if the mixture exceeds 5% (v/v) concentration in the feed. The digestion of SS produced 31 Nm^3/Kgvs and with an addition of OMW increased biogas production in the range of 0.337–0.42 Nm^3/Kgvs (2–10% v/v in the feed), where the best addition was 10% with 0.42 Nm^3/Kgvs of biogas. VS removal was estimated, it was over 55.2% of 10% OMW about 2.2 times of digestion of SS. Also, COD removal efficiency exceeded 72.2% of 10% OMW which is 1.2 times of 100% SS.

Keywords Anaerobic co-digestion · Olive mill wastewater · Sewage sludge · Pre-treatment · VS removal · COD reduction

Introduction

In the daily operation a wastewater plant, a significant quantity of activated and primary sludge is collected through sequential treatment processes. Sludge management contributes to decreased environmental impacts, in addition to lowering the total cost of wastewater treatment up to 60% [1]. Given the significant organic matter existing in the generated sludge, most efforts have been directed towards the anaerobic digestion (AD) process [2].

As a result, the AD process presents the most suitable solution to minimize the quantity of sludge generated through treatment processes, and to improve energy efficiency. In particular, using co-digestion technique of primary or secondary sludge with other organic waste has been proposed to enhance biogas and methane production [3–5]. Indeed, implementation of these studies was recommended at several wastewater treatment plants, using existing anaerobic digester infrastructure at those plants [6–8].

Agro-industries such as olive oil production represent a considerable share of the economy in Middle East countries. The oil can be extracted through three different processes: traditional pressing, three-phase system and two-phase system. Traditionally pressed and three-phase centrifugation systems produce oil, a semi-solid waste and olive mill wastewater (OMW). However, the composition of the by-products of OMW contain more organic pollutants and phenols [9–11], and thus cause serious risks, in terms of water poisoning and environmental impact. Currently, the most common method for eliminating OMW is through evaporation in storage ponds. Among several options currently available for OMW treatment ''such as: aerobic process, chemical oxidation, flocculation,..'' [11–15] and then can be used in the AD process [16, 17]. However, the main purposes of the AD process is to produce energy, moreover to stabilize the material. Table 1, shows a summary of characteristics of OMW.

Previous studies have shown that the digestion of one substance alone may lead to the accumulation of Volatile Fatty Acids) VFA) combined with high organic loading rate (OLR), which inhibits methanogenesis and destabilizes the digestion process [22, 23]. The AD process inhibition may associate

Statement of Novelty: This paper introduces a new technique to enhance the production of biogas and increase the efficiency of COD removal. This technique is to digest sewage sludge and dried olive mill waste in different concentration, BMP tests were used to investigate and improve the effect of these co-substrate on the co-digestion process and on the performance of anaerobic digestion in mesophilic conditions. Where the olive mill waste was dried thermally through solar energy.

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Table 1 Reported composition values of olive mill waste

Substrate	pH	COD (g/l)	BOD (g/l)	Total solids (g/l)	VS (g/l)	Phenols (g/l)	References
OM wash water	6.0	2.735	NA	0.456	NA	0.291	[18]
Three-phase OMW	5.14	68.78	17.12	49.14	NA	5.06	[19]
Three-phase OMWW	5.0	131	41	83.3	54.9	6.8	[20]
Two-phase OMWW	4.89	21.5	NA	16.7	14.0	0.06	[21]

with a certain load of organic acids and long-chain fatty acids. According to most studies, the co-digestion of two or more kinds of substances leads to improved biogas production in the terms of quantity and quality [3, 4, 7, 18].

From a wastewater treatment point of view, anaerobic digestion of OMW offers an excellent approach because co-digestion of sewage sludge with OMW provides the necessary nutrients and buffer capacity. In order to improve the OMW digestion, the process requires a pre-treatment operation using a physicochemical or biological stage to decrease the organic load and the concentration of potential inhibitors of AD [11, 24], this can be acomplished through co-digestion with sewage sludge (SS). Also, co-digestion of SS–OMW improves C/N ratio and pH value. The biogas quality is based on an improved composition of the effluent to be ready for the digestion process, since the co-substrates are usually complementary to the major waste in most cases, or due to an increased volatile solid ratio with enough humidity. According to previous studies, that biogas produced in an anaerobic co-digestion is significantly increased when a mixture of wastes is used, compared to only one source of influent [25–27].

Co-digestion investments involve increasing the annual cost due to the expense transporting raw material, especially that which is combined with WTP. The investments required to improve logistics costs [22] take in consideration that OMW accumulation is a seasonal issue that needs to factor in storage in addition to transportion. Some studies recommended that to resolve cost issue, process such as biomass densification ‘‘drying process’’ [28–30] be used. These procese would also improve the ratio of volatile solid (VS) and increase in the production of biogas.

This work reports on the use of dried OMW in co-digestion with SS, and investigates the quantity and quality of biogas production by adding dried OMW in different concentration ratios of 2–10% v/v to SS. This technique has been applied by other authors by implementing an aerobic process to dry organic substrates, achieving high rates of substrate degradation and biogas production [15].

Methodology: Materials and Methods

Feedstock

Sewage sludge (SS) was obtained from the secondary sludge of a wastewater treatment plant of the city of Irbid (Actual load about 8635.1 m^3/d and hydraulic load outside 8276.9 m^3/d), Jordan. SS was stored at 4 °C until use. The samples used in the BMP tests were prepared according to UNI 5667-13/2000. OMW was extracted from a two-phase oil extraction that belonged to the olive harvest season of 2016 and pre-treated thermally. It was dried at a temperature of 100°C to reduce the volume to 30% of the initial volume, then it was stored and frozen at 4°C. Co-substrates were homogenized using a manual shaking and were prepared on a volume (v/v) basis. Raw materials were analyzed (physicochemical characteristics - proximate analysis) through TGA (Thermographic Analyzer), to obtain the TS (Total Solid), VS (Volatile Solid), ASH, F.C (Fixed Carbone) and U (Humidity), thus were determined according to the standard methods as described [5]. The characteristics of the feedstock are summarized in Table 2.

Experimental Setup

The biomethane potential (BMP) tests were performed in closed vessels and repetition of the tests in order to obtain reliable results. The vessel essentially consists of 1 l realized in Boro-silicate glass and has an input hole to introduce and extract the substrate and a major neck to collect the biogas generated in the process.

The biogas volume generated during the experiment is measured using a 3 l tank which used to determine the volume of biogas generated during the AD process. Daily biogas production was recorded by using the volumetric water displacement method. In this method, produced biogas enters through a tube placed in a conical flask with a manual stopper connected with an airbag to collect

Table 2 Composition of sewage sludge (SS) and olive mill wastewater (OMW)

Substrate	U (%)	TS (%)	VS (%)	Ash (5)	F.C (%)	pH
SS	95.2 ± 0.34	3.82 ± 5	2.60 ± 4.2	0.98 ± 2	0	7.1 ± 0.2
OMW	85 ± 0.36	11.8 ± 3.1	5.10 ± 1.5	0.82 ± 2.3	1.0 ± 1.8	5.2 ± 0.1

the biogas. A flask installed at the center of tank. The quality of biogas was investigated and analyzed by GC as described Alrawashdeh et al. [5]. The vessels operated under mesophilic conditions (35 ± 2° °C) via water bath surrounding the vessels. The substrate inside the reactor was homogenized by manual shaking, allowing for complete mixing. Biogas in excess was vented to avoid pressurized conditions.

Experimental Procedure

All the vessels were filled up to 20% of their volume with different mixtures of SS–OMW prepared as recommended in the similar studies. Three mixtures were utilized 98% SS + 2% OMW, 95% SS + 5% OMW, and 90% SS + 10% OMW in addition, one vessel of 100% SS and were prepared on a volume basis, in order to investigate the production of biogas of the SS–OMW co-digestion and mono-digestion of SS. Initially, the vessels of BMP test were inoculated with anaerobic sludge which contained 15.2 g/l TS, 5.9 g/l VS and 19.2 g/l COD.

The AD tests were carried out in vessels operating in semi-continuous mode; where a given volume of digested sludge was extracted daily from the vessel by a syringe, then immediately the same volume of the mixture was introduced back into the vessel so that the substrate was remained at constant-volume.

The vessels for the BMP test were operated under steady-state conditions characterized by stable digestion process and the values of pH were constant throughout the test. Biogas production was analyzed for each vessel and the composition of this biogas was detected. Samples of influent and effluent were analyzed (proximate analysis) for TS, VS, ASH, F.C and U, also, the value of pH and COD. Tables 3 and 4 summarize of the mixture characteristics and operating parameters. Proximate analysis were carried out through Thermographic Analysis (TGA), the COD was detected through Nanocolor kits and spectrophotometer (Macherey–Nagel).

Biogas yield was monitored on a daily basis by the water volumetric method. Produced biogas was sampled using gas-tight syringes. Biogas was sampled and analyzed by a gas chromatograph (490 micro GC, Agilent Technologies, Santa Clara, CA, USA), biogas samples were collected in gas-tight syringes via vessel rubber stoppers. GC was used to analyze CH_4, CO_2, CO, C_2, O_2 and N_2. The GC consists of a capillary column and thermal detector, where the column injector and detector were operated at 80, 100 and 180 °C. Helium and Argon were used as a carrier gas with a flow rate of 10 ml/min. Analyses of samples were carried out in duplicate.

Table 3 Concentration of raw materials in each mixture

Vessel	pH	Substrate (g)	SS (%)–OMW (%)
1 and 2	6.6 6.4	160 g SS 3.3 g OMW 21.5 g Inoculum	98–2
3 and 4	6.12 6.28	160 g SS 8 g OMW 32 g Inoculum	95–5
5 and 6	5.9 6.0	160 g SS 17 g OMW 23 g Inoculum	90–10
7 and 8	6.6 6.4	100 g SS	100–0

Results and Discussion

Figure 1 presents biogas production ($Nm^3/Kgvs$) for mixtures of 2, 5 and 10% OMW, compared to the daily biogas production of SS (0% OMW). Moreover, the daily biogas production was found to increase as the concentration of OMW increased. As shown in Fig. 1, a high biogas production rate was present on day 15 till day 40 for all tests due to bacterial adaptation and optimum conditions of AD process. Moreover, there is a significant increase in daily biogas yield when 5% OMW–95% SS and 10% OMW–90% SS is used as co-mixture in the digestion process. Daily production of all vessels has shown a repeated pattern of degradation that occurred in two periods during the test. However, the 2% OMW and 100% SS have lower methane content than 5 and 10% OMW. In the case of 100% SS, it has the lowest yield of biogas, and by tracing the biogas production, it has a high concentration of CO_2 production.

In particular, comparing the methane yield of SS and the corresponding content after OMW co-mixtures, an increment of between 7.9 and 26% is observed. According to Angelidaki and Ahring, that biogas production of 5% OMW–50% manure co-digestion was 1250 ml/l/day (160 l CH_4/kg COD) [31]. Maragkaki et al. reported, that co-digestion of SS and food waste allow to increase methane content from 1.5 to 6%, corresponding to mono-digestion of the SS (ml/L/d) [15]. In another study, Gelegenis et al. has reported, that the biogas yield of 210 ml/L/day of 50% effluent of an olive mill and 50% of manure [32]. Actually, our study proved to be more efficient than these studies, where the biogas production and methane content were higher than those reported.

Figure 2 shows that the total COD removal efficiencies were 68–72.8% of co-substrates SS–OMW, while the total COD removal efficiency of mono-digestion was 61%. Actually, the higher total COD removal efficiency was obtained for 90% SS–10% OMW which conjunction with higher gas

Table 4 Characteristics of co-digestion substrates

Feedstock	pH	U (%)	TS (%)	VS (%)	F.C (%)	Ash (%)	COD (g/l)
98% SS–2% OMW	6.5 ± 0.5	95.36	4.64 ± 0.23	2.38 ± 0.33	0.02	0.97	43.7 ± 0.6
95% SS–5% OMW	6.2 ± 0.5	95.08	4.92 ± 0.61	2.48 ± 0.49	0.05 ± 0.25	1.00	28.02 ± 0.42
90% SS–10% OMW	6.0 ± 0.5	94.54	5.46 ± 0.38	2.65 ± 0.34	0.10 ± 0.1	1.05	49.0 ± 0. 51

*COD of SS is 26 g/l

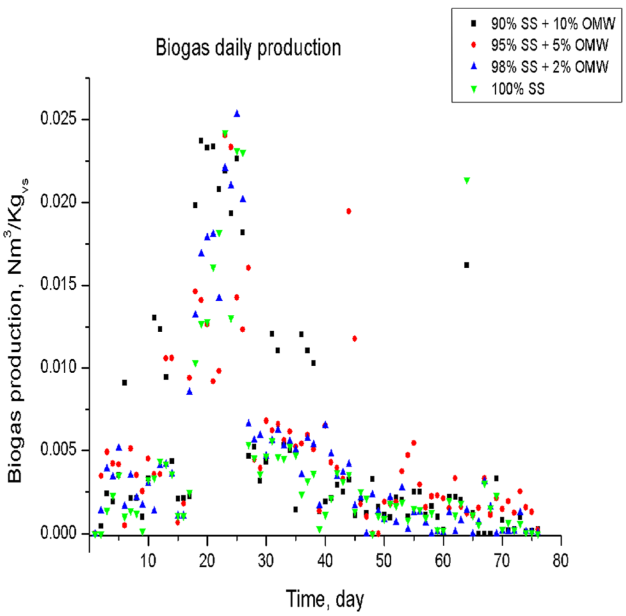

Fig. 1 Daily biogas production of all tests

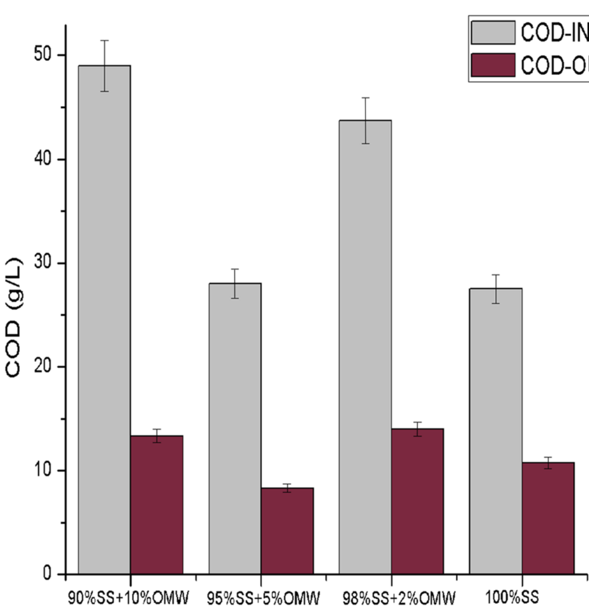

Fig. 2 Total COD removal of co-mixture and mono-substate

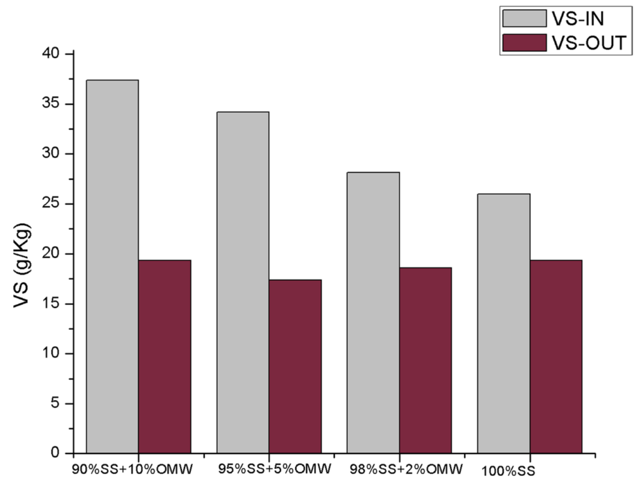

Fig. 3 VS concentration of substrates the influent and effluent

production, which involved to effective methanogenic-bacteria activity.

During the test, pH values were obtained from all tests every 3 days, pH values were stabled 7 and 8.1 without any addition of mean treatment. But we noted after venting the vessels after the increment of pressure, pH values were decreased at least 0.3. The VS values were traced for all co-substrates and mono-substrate, the result shows high VS reduction efficiency between 25.5 and 55%. Reduction efficiency of mono-substrate was the 25.5%, where the higher reduction was obtained from 90% SS–10% OMW (See Figure 3). That related to VS concentration in the co-mixture was increased when the substrate of OMW was added to the substrate of SS. The highest yield of biogas was in conjunction with the high reduction of VS. Dareioti et al. (2009) observed, that the co-digested 80% manure and 20% OMW, VS removal was obtained 34.2% corresponding to the stability of biodegradation process [25]. According to Metcalf et al. after sludge digestion, the optimum VS removal should be 30% [33].

As shown in Fig. 4, Among the different concentration of OMW, the 10% of OMW co-substrate showed the highest biogas production reduction 0.420 Nm^3/Kgvs. A biogas yield of 10% OMW agrees with the highest VS reduction and total COD efficiency removal. Comparing the same co-digestion conditions, Chu et al. reported, that removal of COD is associated with to biogas production

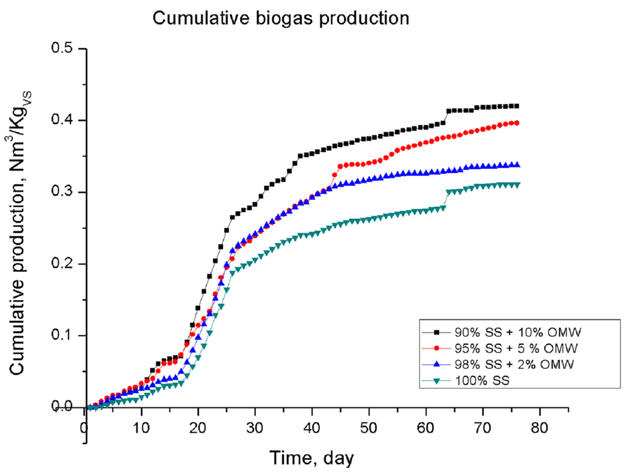

Fig. 4 Accumulative biogas production of all co-digestion and mono-digestion

Table 5 Characteristics of co-digestion and mono-digestion process

Feedstock	Biogas (Nm^3/Kg_{VS})	CH_4 (Nm^3/Kg_{VS})	VS-removal (%)	COD-removal (%)
98% SS–2% OMW	0.337	64.8	38.3	68
95%SS–5% OMW	0.396	67.1	45.6	70.4
90% SS–10% OMW	0.420	70.3	55.02	72.8
100% SS	0.310	60	25.5	61

[34], this is consistent with the literature data of comparable compositional analysis [25, 35, 36]. In light of these results of biogas yield and COD removal, it can be stated that the AD process of OMW with pre-treatment (drying process) is totally viable, as reported in the literature [11, 37]. Table 5 illustrates the accumulative production for all mixture and mono- substrate and the percentage of VS and total COD removal.

The mixture of 2 and 5% OMW showed biogas yield 0.337 Nm^3/kgs and 0.396 Nm^3/Kgvs respectively, while the biogas yield from mono-digestion of SS was 0.310 Nm^3/Kgvs. Moreover, these yields of biogas and methane content showed that as the yield increased and the VS concentration of effluent decreased, it presented the highest degradation capability.

The mean values (MV) of the pH value obtained in each test of influent and effluent acclimatization period are shown in Fig. 5. As illustrated in pH profiles of 100% SS test, pH was approximately stable between 7.3 and 8 (7.6 ± 0.1 MV), while the influent was 6.8–8 (7.3 ± 0.1 MV). Tracing pH profiles, the effluent had a maximum decrement from 8 to 7.5 due to 2% OMW addition and the maximum increment from 7.1 to 7.7 due to 10% OMW addition, with affecting the effluent pH.

Another way of assessing the performance of a digester is to examine the efficiency of the biogas production conjunction with COD removal, during the digestion process, which synchronized with VS degradation. The Fig. 6 shows the evaluation of CH_4 production for each substrate during the tests and the steady state which was reached. In light of the results, it is clear that is a relationship between the higher CH_4 yield conjunction with the higher OMW concentration in the mixture. While 100% SS digestion has lower performance (tracing pH value and rapid decrease of COD reduction and CH_4 yield). This makes sure that true organic load leads to a steady state of digestion and high reduction of pollutants.

This may be related to the fact that, as Ma et al. reported, the addition of an carbon source (such as OMW substrate), enhanced the total VS and therefore the biogas yield increased [38]. Actually, co-digestion of SS with other organic waste is highly recommended in order to improve biogas production [39, 40]. OMW is used as co-substrate since it is rich in lipid and VS and has complementary nutrients. The co-digestion of OMW–SS at ratio 10–90% is showed lower biogas production and methane content. Where the change in the mixing ratio affected microorganism activity, this result agrees with Kougias et al. Already, SS substrate is rich in microbial and thus it is easy to adapt at new mixture composition (OMW–SS) and a stepwise increase of VS which did not affect the stability of the digestion process.

Thermal drying as pre-treatment can be a very attractive process for reduction of volume in order to enhance reactor volume and increase the VS concentration. In the present study, in order to investigate co-digestion of SS–OMW effective in the biogas production and COD removal, the OMW substrate must be dry, that will improve transportation and storage issues. Therefore, for decrease the thermal energy cost which used to the dried substrate, we suggest using thermal solar energy system.

Conclusion

Olive mill waste consists of liquid and solid effluents are rich in organic pollutants. OMW has lack of nutrient balance; thus anaerobic co-digesting a mixture of dried OMW and SS could be a promising perspective on biogas production and the environment security. Pre-treatment of OMW can decrease in the initial volume and decreasing pollutants, without showing signs of inhibition during anaerobic digestion.

Results show, as increasing the concentration of OMW, the biogas and methane are increasing, in addition, the

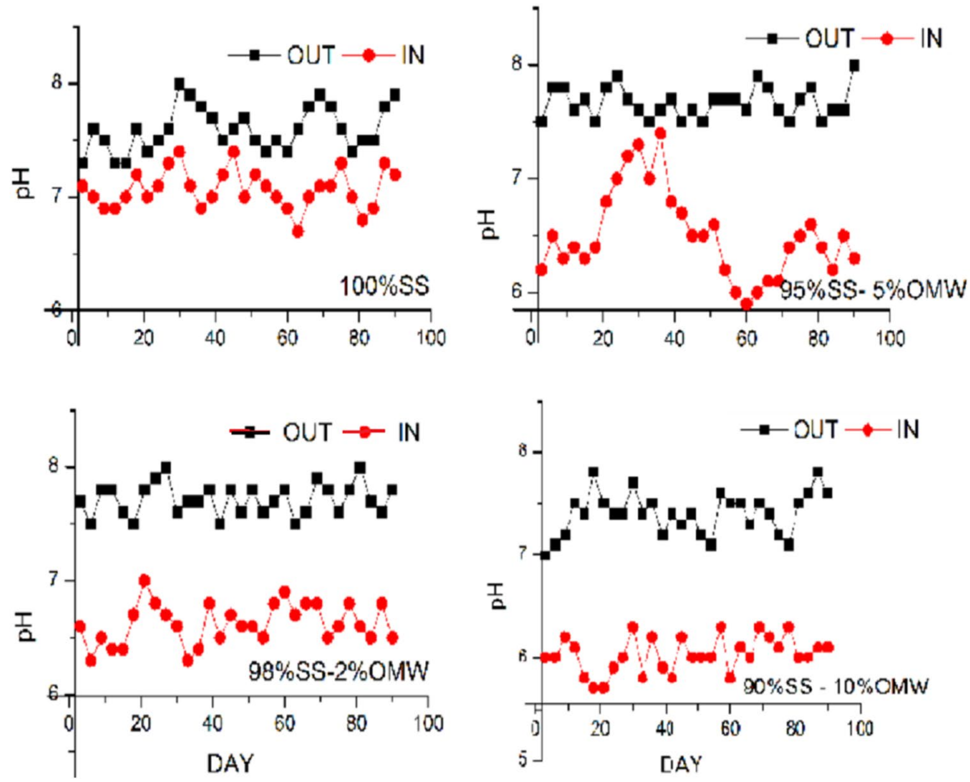

Fig. 5 pH profile of feed and effluent mixtures

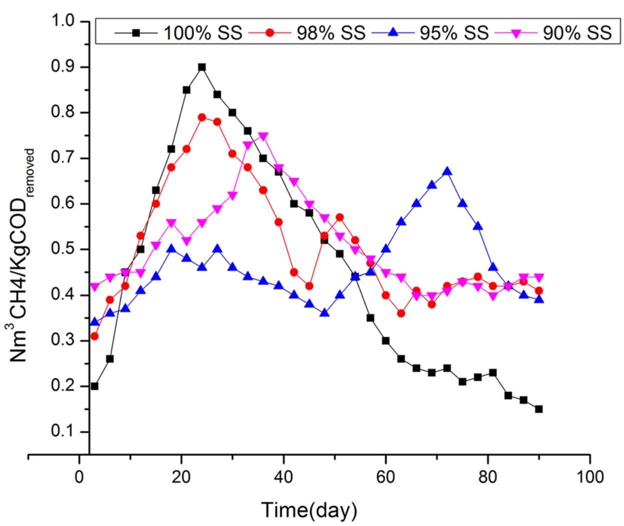

Fig. 6 Methane production VS removed TCOD

efficiency of COD and VS removal are increasing. The 10% OMW–90% SS v/v is by far the most effective, this mixture was achieved 0.42 Nm^3/KgVS of biogas production, 55.2% of VS reduction and 72.8% of COD reduction. Also, the other mixture results are very satisfactory comparing to mono co-digestion. However, the feeding rate of OMW should be low in order to avoid overloading conditions and accumulation of fatty acid which lead to inhibited anaerobic treatment.

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