Assessing the performance of high-rate anaerobic reactors treating three-phase olive mill wastewater (OMW)

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Abstract
The aim of this study was to assess the performance of three high-rate anaerobic systems, i.e. two UASB reactors and a hybrid-UASB, operating under mesophilic conditions (37^{o}C) for the treatment of centrifuged Olive Mill Wastewater (OMW) from a Greek three-phase olive mill. The systems' performance was compared by following a feeding strategy in each reactor based on increasing the reactor's organic loading rate (OLR) by decreasing its HRT and diluting the raw (centrifuged) OMW with tap water in order to avoid phenolics accumulation in the system. The two UASB reactors were seeded with anaerobic flocculent and granular sludge respectively, whereas the hybrid-UASB reactor was inoculated with the same anaerobic granular sludge. All three reactors were tested at different OLRs. The highest COD removal and operational stability in high OLRs was observed in the UASB reactor seeded with the anaerobic flocculent sludge.

Keywords
Anaerobic digestion; high-rate anaerobic reactors; olive mill wastewater; 3-phase olive-mill; Organic Loading Rate; UASB reactor

INTRODUCTION

Olive mills are agro-industries that represent a considerable share of the Mediterranean countries economy. Olive oil extraction produces large amounts of olive mill wastewater (OMW) which poses a serious environmental risk, especially in the Mediterranean, Aegean and Marmara regions accounting for approximately 95% of the worldwide production. OMW is becoming a serious environmental problem due to its high content in organic constituents, suspended solids, phenolic compounds and their recalcitrance to biodegradation.

The majority of research conducted on OMW treatment has been focused on the use and development of anaerobic methods and bioreactors that can efficiently remove high organic loads. Among the high-rate processes that have been developed in recent years, the upflow anaerobic sludge blanket (UASB) reactor is probably the most commercially successful. In a UASB reactor, microorganisms agglutinate to form biogranules with high bioactivity and superb settleability. The microstructure of a biogranule is highly substrate dependent, leading to the formation of layered and non layered structures (Fang et al. 1995).

The present study was focused on studying the effect of OLR on the anaerobic digestion of OMW in three different high-rate configurations, after centrifugation of the raw OMW.

MATERIALS AND METHODS

Feedstock

Fresh OMW was used in the present study collected from a three-phase olive mill located in Patras (Achaia, Region of Western Greece). Because of its seasonal production and its natural tendency for fermentation, the collected OMW was stored in plastic vessels in the freezer (-18^{o}C). After unfreezing, the raw wastewater was centrifuged (15 min at 4.000 rpm followed by supernatant removal and recentrifugation under the same conditions) and the solid fraction was stored in the freezer for future treatment. The supernatant was diluted with tap water and used as feeding substrate in the reactors. The average characteristics of the raw OMW were: pH=5.15±0.04, TSS=45.08±20.97 g/L, VSS=42.97±19.57 g/L, COD=127.75±28.59 g/L, phenolics=3.28±0.09 g/L, fats & oil grease=11.02±5.06 g/L, TKN=0.93±0.26 g NH_4-N/L, ammonium N=0.08±0.02 g NH_4-N/L. The average characteristics of the centrifuged OMW were: pH=5.15±0.04, TSS=12.47±0.10 g/L, VSS=11.57±0.40 g/L, COD=92.29±4.21 g/L, phenolics=6.69±0.05 g/L, fats & oil grease=4.02±2.74 g/L, TKN=0.42 ±0.10 g NH3-N/L, ammonium N=0.04±0.01 g NH_4-N/L.

Experimental set up

Two conventional UASB reactors (UASB-F, UASB-G) were designed and constructed using plexiglass, with 7.4/8.3 L (UASB-F) and 6/6.2 L (UASB-G) working and total volume respectively, in accordance with the work of Lettinga and Hulshoff (1991). A hybrid-UASB (HUASB-G) reactor was designed and constructed with working and total volume of 6 L and 6.2 L, respectively. The main technical difference between the UASB reactors, were the plastic biomass carriers (K5 carriers) which were packed in the upper part of the HUASB-G reactor, instead of the GLS separator, preventing biomass washout and accumulating biomass on them. The K5 carriers with a protected surface of 800 m^2/m^3 were kindly provided by Anoxkaldness (Lund, Sweden).

The anaerobic flocculent sludge was obtained from the anaerobic CSTR digester of the city of Patras' WWTP. The UASB-F reactor was seeded with the flocculent sludge. The main characteristics of this sludge were: pH=7.57, TSS=26.58 g/L, VSS=15.90 g/L, TS=28.12 g/L, VS=16.59 g/L. The anaerobic granular sludge, consisting of granules uniform in size (1–3 mm), was obtained from a full scale UASB reactor treating dairy wastewater. The granular sludge was used for the inoculation of UASB-G and HUASB-G. The main characteristics of the granular sludge were: pH=7, TSS=59.15 g/L, VSS=33.26 g/L, TS=62.43 g/L, VS=35.57g/L. All reactors were operated under mesophilic conditions (37 °C).

During start-up the reactors were seeded up to the 1/3 of their active volume with the aforementioned anaerobic inoculums, they were filled with tap water and started their operation at a constant flow with an upflow velocity of 1 m/h. Samples were withdrawn periodically during their operation and chemical analyses were performed.

Analytical methods

pH measurements were carried with an Orion 3-Star (Thermo scientific), while total and volatile suspended solids, TS, VS, dissolved and total COD, TKN, ammonium nitrogen, total and ortho – phosphates were determined according to *Standard Methods* (APHA, 1995). For the determination of carbohydrates, L-tryptophan, sulfuric and boric acid were added to the samples, and were then measured colorimetrically at 520 nm (Joseffson, 1983). Phenolic compounds were determined according to the Folin–Ciocalteu method (at 760 nm). Biogas production was measured separately by a tailor made hybrid gas meter configuration, based on the description given by Angelidaki et al (1991). Biogas composition and volatile fatty acids (VFA) analysis were performed throughout the experimentation period in both systems as reported by Dareioti et al. (2010).

RESULTS AND DISCUSSION

The operating conditions and the performance of the reactors during OMW treatment under various OLRs are presented in Tables 1 and 2. In all operational phases the pH of the reactors was maintained in an average value of 6.8-7.2. $NaHCO_3$ was also added in some cases in order to sustain the alkalinity of reactors above 4 g/L. In the UASB-F reactor, granulation has not been observed (optical observation). According to De Vrieze et al. (2012) *Methanosarcina* sp., are characterized as heavy duty methanogens and are able to achieve stable growth even at low HRT, high OLR and high levels of some inhibitors. The stability of the process could be correlated with the predominance of methanogenic species in the reactor, of the *Methanosarcina* genus, as a result of the characteristics of the OMW. However further investigation has to be made on the microbial ecology of the reactors via molecular techniques.

Table 1. Operating conditions and average performance of the system seeded with flocculent sludge.

	UASB-F					
Phases	*A*	*B*	*C*	*D*	*E*	*F*
HRT (d)	9	9	9	9	6	4
OLR (g COD/L_R d^{-1})	0.95	1.32	2.2	4.14	6.20	9.47
COD removal (%)	65±2.6	76±1.4	73±4.5	70±5.3	63±7.0	49±7.0
Biogas Production Rate (L_B/L_R d^{-1})	0.14±0.05	0.28±0.06	0.49±0.11	1.15±0.21	2.07±0.32	2.34±0.21
CH_4 content (%)	59.10±6.75	64.58±1.36	57.59±4.96	56.67±4.75	51.61±2.69	52.04±3.51
Yield (L CH_4 g^{-1} COD converted)	0.20±0.03	0.17±0.03	0.17±0.06	0.23±0.04	0.25±0.03	0.29±0.02
Phenols removal (%)	80±7.8	78±3.9	72±8.8	56±11.3	52±8.6	29.4±3.9

Table 2. Operating conditions and average performance of the systems inoculated with the granular sludge

	UASB-G		HUASB-G		
Phases	*A*	*B*	*A*	*B*	*C*
HRT (d)	9	9	9	9	9
OLR (g COD/L_R d^{-1})	4.21	2.46	4.21	2.46	4.21
COD removal (%)	32±12.7	44±15.2	32±6.3	52±12.0	59±5.5
Biogas Production Rate (L_B/L_R d^{-1})	0.91±0.25	0.58±0.11	1.01±0.23	0.88±0.08	1.32±0.13
CH_4 content (%)	34.07±8.20	45.27±4.42	35.93±5.53	50.92±3.17	45.33±1.62
Yield (L CH_4 g^{-1} COD converted)	0.21±0.07	0.24±0.03	0.27±0.08	0.32±0.02	0.26±0.02
Phenols removal (%)	69±14	71±11	46±14	44±16	57±13

The UASB-F reactor has operated for about 250 days with maximum OLR of 9.47 gCOD/$L_R d^{-1}$. It achived stable operation, the methane yield was maintained near the theoritical values and had the higher efficiency than the other two. Among the reactors seeded with the granular sludge, higher OLRs were applied to the HUASB-G reactor reaching a stable operation in an OLR of 4.21 g COD/L_R d^{-1}. Moreover, the mean methane content remained below 50% throughout the whole operation period. The UASB-G reactor failed to maintain stable operation in an OLR 4.21 g COD/L_R d^{-1} and a rapid increase in the acetic acid concetration was observed, with a constant increase in the propionic acid which is an indicator of process imbalance. Biogas production rate increased but finally dropped to low values. The operating conditions under the different OLRs and the Biogas Production Rate (BPR) during the phases are presented in Figure 1.

Among VFAs that existed in the reactors effluent, acetic and propionic acid were the dominant ones. During the transistion period between the various phases (Figure 2), a rapid accumulation of VFAs was observed, in all reactors. The rest of the VFAs' (butyric, iso-butyric, valeric, iso-valeric, caproic acid) were also determined and their sum were below 300 mg/L, 1500 mg/L and 800mg/L in all phases in UASB-F, HUASB-G and UASB-G reactor respectively.

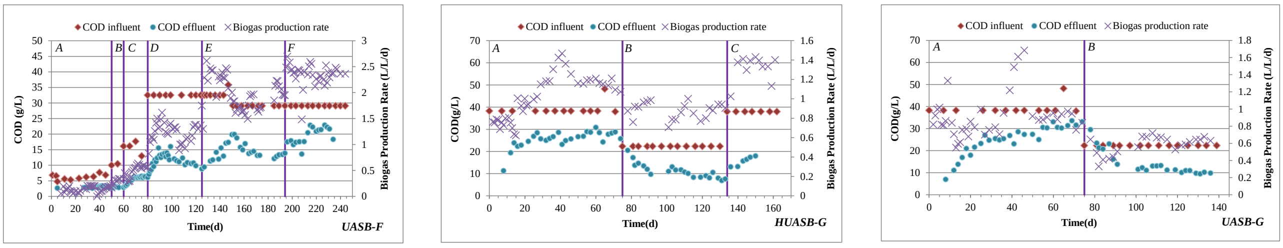

Figure 1. Profile of the COD concentration in the influent and effluent of the reactors, and the Biogas Production Rate under the different operational conditions.

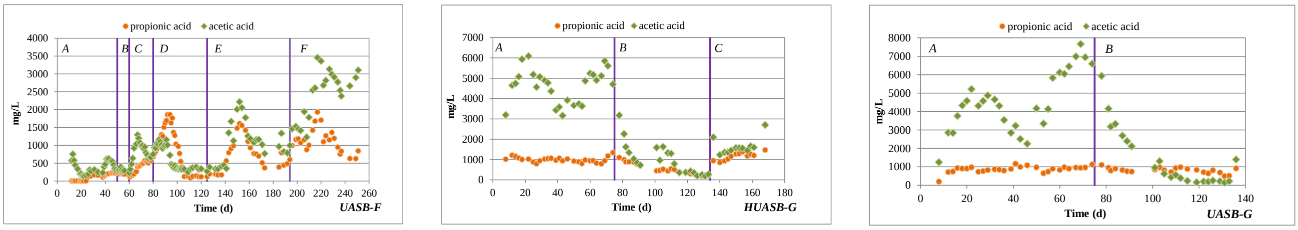

Figure 2. Profile of the VFAs concentration in the effluent of the reactors under the different operational conditions.

CONCLUSIONS

In this study three high-rate reactors were tested regarding their treatment efficency of OMW. The UASB-F reactor achieved a stable operation in higher OLRs compared to the HUSB-G and UASB-G reactors. During the operation of UASB-F for about 250 days no sludge granulation was observed. On the other hand, well developed granules which were used to inoculate HUSB-G and UASB-G lost their structure (optical observation) as well as the performance of the reactors remained in low values. Further investigation has to be made on the effect of phenolic compounds on anaerobic granules structure.

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