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Anaerobic treatment of winery wastewater in moving bed biofilm reactors

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ABSTRACT

The treatment of winery wastewater in two anaerobic moving bed biofilm reactors (MBBR) (R9 and R30) with low-density polyethylene carriers differing in size, shape, structure, and specific surface area was investigated. The carrier packed in R9 was bioflow 9 with diameter of 9 mm, height of 7 mm, density of 0.84 g/cm^3, and specific surface area of 800 m^2/m^3, while the carrier filled in R30 was bioflow 30 with diameter of 29–35 mm, height of 29 mm, density of 0.94 g/cm^3, and specific surface area of 320 m^2/m^3. Both reactors were run in parallel for 232 days. A maximum organic loading rate (OLR) of 29.59 g COD/L day with more than 80% soluble chemical oxygen demand (COD) removal efficiency was achieved in R9 at hydraulic retention time (HRT) of 1.55 days, whereas a maximum OLR of 18.43 g COD/L d with 80% soluble COD reduction was attained in R30 at HRT of 2.49 days. Biogas production of each reactor was increased and strongly correlated with its OLR. The experimental results showed that R9 performed better than R30 in achieving OLR and in attaching biomass, implying that the performance of the anaerobic MBBR was enhanced by an increase in the specific surface area of the carrier used. Both the reactor performances can be promoted by short-term supplementation of some essential trace metals needed in anaerobic digestion of winery wastewater. The study provided not only a good basis for comparing the effect of packing material to the efficiency of anaerobic digestion system, but also an approach on how to optimize the performance by short-term dosing of trace elements.

Keywords: Anaerobic treatment; Winery wastewater; Moving bed biofilm reactor; Carrier media

1. Introduction

Anaerobic digestion technology has been widely used for treating a variety of high-strength industrial wastewaters [1] due to its low nutrient requirement, less sludge production, and energy recovery in the form of methane. Some drawbacks suffered in the technology such as slow growth bacteria, long hydraulic retention time (HRT), and process instability is overcome by the high-rate anaerobic concept based on anaerobic granular sludge and biofilm systems. The typical granular sludge systems are the upflow anaerobic sludge bed (UASB) and the expanded gran-

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ular sludge bed (EGSB) reactors [2], and typical biofilm systems are the anaerobic fixed bed, the anaerobic fluidized, and expanded bed reactors [3]. The anaerobic biofilm process makes the high-rate anaerobic treatment process as an attractive option over the conventional anaerobic treatment methods.

Winery and distillery wastewater is characterized by high-organic matter with chemical oxygen demand (COD) of 15–60 g/L and acidic pH value [4,5]. Since the organic component in the wastewater is highly biodegradable, many anaerobic digestion processes [6–18] have been used in the pretreatment of the wastewater before it is charged into the natural water system or aerobic posttreatment system.

Moving bed biofilm reactors (MBBR) is a highly effective biological treatment process, which incorporates the benefits of both the activated sludge process and a biofilm reactor by filling freely moving carrier elements into its compartment, which provides a large specific surface area for biomass attachment [19–22]. The movement within the reactor is carried out by aeration under aerobic conditions and by a mechanical stirrer or biogas agitation under anaerobic conditions. MBBR has the advantage of low head loss, no filter channeling, no need of backwashing, and biomass recycling. Biofilm carrier element is an important component of MBBR. The characteristics of the carrier material such as the specific area, filling fraction (volume of carrier in empty reactor), surface roughness, porosity, strength, and durability determine the capability of biomass attachment and the treatment efficiency of MBBR. A variety of biofilm carriers with different size, shape, specific surface area, and material were used in MBBR, including polyethylene (PE) and polypropylene (PP) cylindrical rings and tube chips [23–29], polyurethane (PU) foam [30–32], PU-activated carbon [33], polyvinyl alcohol (PVA) gel [34], light-expanded clay aggregate [35], cigarette filter rods [36], diatomaceous earth [37], and ceramic granules [38], etc. To date, aerobic MBBR has been widely used in urban, industrial, and agricultural wastewater treatment under different conditions, while only a few anaerobic MBBRs have been used in the treatment of milk permeate [28], sulfate-containing wastewater [29], sisal leaf waste leachate [39], landfill leachate [40], thermomechanical pulp (TMP) white water [41], high strength wastewater constructed with molasses [42], etc.

The objective of this study is to compare the performances of two anaerobic MBBRs with different PE carriers to treat winery wastewater, and to conclude how big the effects of carrier types on the performances of anaerobic MBBRs.

2. Materials and methods

2.1. Reactor and carrier

The two upflow anaerobic MBBRs (R9 and R30) of similar size with an internal diameter of 23.8 cm and a height of 89 cm were fabricated in this study (Fig. 1). Both reactors were made of Plexiglas cylinder with a working volume of 32.9 L, and immersed in a thermostat water bath where temperature was controlled in the range of 31–39°C during the operation days. The wastewater in influent tank was fed into each reactor with a Masterflex peristaltic pump, and mechanically mixed with a stirrer. Inside each reactor, a submerged Superma centrifugal pump was installed at its bottom

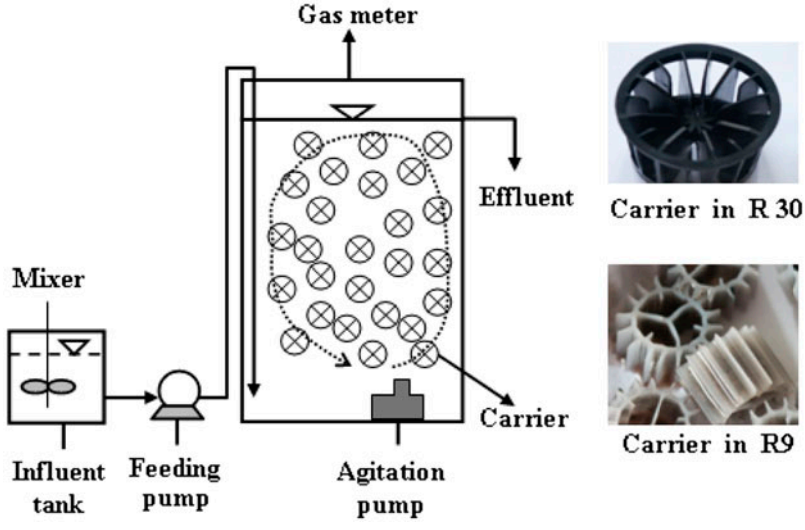

Fig. 1. Experimental setup used in the study.

for liquid mixing. The liquid mixing regime was 2–8 times per hour, and each mixing just lasted 1.25 min. Biogas produced from each reactor was connected to a gas meter for measuring biogas production.

Both reactors were filled with different kind of small floating PE carriers for biomass attachment. The volumetric filling fraction of carrier in each reactor took 66% its empty working volume. The carrier filled in R9 was bioflow 9, while the carrier in R30 was bioflow 30 (Fig. 1). Bioflow 9 had the filigree structure, with small size (9 mm in diameter and 7 mm in height), low density (0.92 g/cm^3), and great specific surface area (800 m^2/m^3). Bioflow 30 had a more open design compared to bioflow 9, with large size (29–35 mm in diameter and 29 mm in height), high density (0.94 g/cm^3), and low specific surface area (320 m^2/m^3). The carrier movement inside each reactor was carried out by the liquid mixing with the submerged agitation pump.

2.2. Seed sludge and wastewater

Each reactor was seeded with anaerobic sludge of 15 L from an anaerobic fixed bed reactor for the winery wastewater treatment. Suspended solids (SS), volatile suspended solids (VSS), and VSS/SS ratio of the seed sludge were 36.8, 20.36, and 0.55 g/L, respectively.

Wastewater used in this experiment came from 100 m^3 storage tank of raw wine distillery wastewater. The characteristic of winery wastewater was listed in Table 1. Both reactors were fed with the wastewater one from running day 1 to day 49, and fed with the wastewater two between day 50 and day 232. The wastewater one was characterized by the low COD, volatile fatty acids (VFA), SS and VSS, and high pH value, while the wastewater two was characterized by the high COD, VFA, SS and VSS, and low pH value.

Several specific trace elements were discontinuously added into the feed wastewaters of both reactors in attempt to accelerate the anaerobic digestion of winery wastewater during the experiment. R9 was only supplemented with 50 mg/L Fe, 10 mg/L Ni, and 10 mg/L Co from day 178 to day 181. R30 was added with 50 mg/L Fe, 10 mg/L Ni, and 10 mg/L Co from day 178 to day 181, with 50 mg/L Fe from day 210 to day 213, with 50 mg/L Fe and 10 mg/L Ni from day 214 to day 217, with 50 mg/L Fe,10 mg/L Ni, 10–20 mg/L Co, 2 mg/L Cu, 20 mg/L Zn, and 0.8 mg/L Mo from day 218 to day 225, respectively. The added trace elements were in the forms of $FeCl_2{\cdot}4H_2O$, $NiCl_2{\cdot}6H_2O$, $CoCl_2{\cdot}6H_2O$, $Na_2Mo_4{\cdot}2H_2O$, $CuCl_2{\cdot}2H_2O$, and $ZnSO_4{\cdot}7H_2O$.

2.3. Analytical methods

The routine laboratory tests included pH value, soluble COD, VFA, biogas production and its composition, SS, and VSS. The pH value and biogas production were measured everyday but weekends. Soluble COD, VFA, and biogas composition were tested three times a week.

The pH value was measured immediately after sampling with a WTW 537 pH meter. Soluble COD was determined using a colorimetric method with Hach DR/2010 direct reading spectrophotometer.

The VFA was measured using a gas chromatograph (GC 8100 Fisons Instruments) with a flame ionization detector (FID), coupled with an automatic sampler (AS 800 Fisons Instruments). The silica capillary column type in the gas chromatograph was ECTM-1000 (Alltech) with length 15 m, internal diameter 0.53 mm, and film thickness 1.2 μm. The injector and detector temperatures were 250 and 275°C, respectively. Carrier gases were N_2 (25 kPa), H_2 (50 kPa, 30 mL/min), and air (100 kPa, 300 mLmin). All

Table 1
Characteristics of winery wastewater

Composition	Wastewater one	Wastewater two
Total COD (g/L)	16.68	45.55
Soluble COD (g/L)	15.52	44.18
Acetate (g/L)	3.16	8.01
Propoinate (g/L)	2.32	3.97
Iso-butyrate (g/L)	0.11	0.01
Butyrate (g/L)	1.00	2.42
Iso-valerate (g/L)	0.10	0.39
Valerate (g/L)	0.81	0.01
pH value	5.75	3.62
SS (g/L)	2.06	3.27
VSS (g/L)	1.13	2.46

samples were compared to the standards in the range from 0.25 to 1 g/L for acetic, propionic, butyric, isobutyric, valeric, and iso-valeric acids. The volume of sample injected for VFA measurement was 1 μL.

Biogas production of R9 was measured with a wet test gas meter, and biogas production of R30 was monitored by using a simple water displacement gas meter, where a counter registered a unit after a certain volume of the solution containing Na_2SO_4 and H_2SO_4 was displaced [43]. Biogas composition was analyzed using Shimadzu GC-8A gas chromatography with catharometer, coupled with Shimadzu CR-3A integrator. Two stainless steel columns were used in chromatography, one was Hayesep column packed with silica gel (80–100 mesh) for separation of CO_2 (2 m in length and 3.175 mm in diameter), and another packed with molecular sieve 5 Å (80–100 mesh, 2 m in length and 3.175 mm in diameter) for CO_2, N_2, O_2, and H_2 separations. Carrier gas was argon (300 kPa). Oven, detector, and injection temperatures were 30, 100, and 100˚C, respectively.

The SS and VSS were determined according to the Standard Method [44].

2.4. Attached solid measurement

The attached solid on carrier elements within each reactor was measured only on running days 53, 115, 207, and 232 to assess the biofilm growth during the period of the experiment. The amount of attached solids to the carrier was determined using 20 pieces of carries that were taken from the reactors. To measure total attached solids (TAS), the sampled carrier elements were dried at 105˚C in an oven for 24 h and weighed, and this value was then compared with an average weight of the fresh carrier without biomass, thus obtaining the attached solids to the carrier [45–48].To estimate the volatile attached solids (VAS), the oven-dried solid samples were scrapped from the carriers and ignited at 550˚C for 2 h. The amount of attached solids in the reactors could then be determined because the filling ratios and the number of carrier elements in the reactors were known.

3. Results and discussion

3.1. Organic loading rate and COD removal efficiency

As seen in Fig. 2, both reactors adapted very quickly to the feed of the wastewater one at the first period of 49 days. At the beginning of the period, both reactors were operated at a low organic loading rate (OLR) of about 1.3–1.5 g/L day. On running day 12, both reactors achieved a maximum soluble COD removal efficiency of more than 91% with OLR of around 1.5 g/L day, and VFA contents in both effluents were only about 0.02 g/L. With increasing of OLR, the soluble COD removal efficiencies of both reactors were decreased due to a slight accumulation of VFA within the reactors. On operation day 49, R9 attained 68.1% soluble COD removal efficiency with OLR of 3.7 g COD/L d and HRT of 4.5 days, and VAF and propionate contents in the outlet of the reactor were 2.60 and 1.05 g/L, respectively, while R30 got 73.6% soluble COD removal efficiency with OLR of 3.6 g COD/L d and HRT of 4.66 days, and VAF and propionate levels out of the reactor were 1.56 and 0.6 g/L, respectively. In general, both reactors performed comparatively well and similar in this period.

Both reactor performances became worse during the second period from day 50 to day 116 when both reactors were fed with the wastewater two, a fresh winery wastewater from wine distillery. R9 only got a soluble COD removal efficiency of 39.6–63.8% with OLR varying from 4.8 to 8.0 g COD/L d, and VFA and propionate levels out of the reactor were 2.8–10.9 and 2.04–8.95 g/L, respectively. R30 just achieved soluble COD removal efficiency of 30.6–65.7% with OLR ranging from 3.69 to 8.25 g COD/L d, and VFA and propionate contents in inlet of the reactor were 2.13–11.05 and 0.86–5 g/L, respectively. Propionate was gradually accumulated and became the major part of VFA in both reactors with OLR increasing. The poor performance of both reactors in this period was probably ascribed to VFA, especially propionate, accumulation and influent pH fluctuating in the reactors.

Both reactors stopped feeding and heating during the third operation period from day 117 to day 153 for summer vacation. Only VFA concentrations inside the reactors were monitored in this period. VFA and propionate levels on day 117 were 3.97 and 2.56 g/L for R9, 4.35 and 2.79 g/L for R30, respectively, and their levels on day 153 were 3.46 and 1.84 g/L for R9, 2.62 and 2.56 g/L for R30, respectively. The comparison of the VFA levels between these two days suggested that VFA, especially propionate, was degraded slowly under ambient temperature.

Both reactors restarted operation from day 154, and operated at the low OLR of 3–5 g COD/L d during the fourth running period from day 154 to day 177 for their acclimating to the wastewater two. In the period, R9 achieved soluble COD removal efficiency of 37.7–61.1% with OLR of 3.2–4.9 g COD/L d and HRT of 3.3 days, and VFA and propionate contents in its effluent were 1.5–3.5 and 1.3–1.9 g/L, respectively, while R30 obtained soluble COD removal efficiency of 35.3–53.7% with OLR of 2.8–4.6 g COD/L d and HRT of 3.47 days, and VFA and propionate concentrations in its effluent

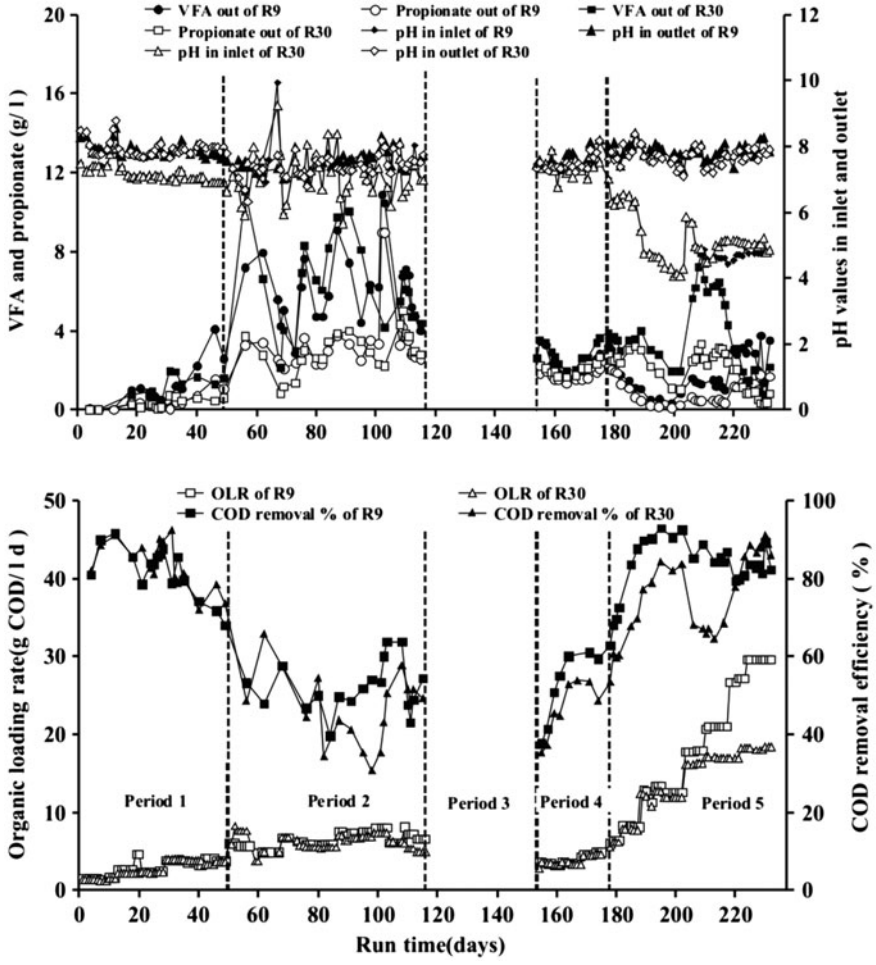

Fig. 2. OLR and COD removal efficiencies against running days of the reactors.

were 2.0–3.5 and 1.7–2.6 g/L, respectively. Both reactors still performed poorly in the period in term of the low soluble COD removal efficiency and OLR. Many studies [49–55] have proved that several specific trace elements such as iron, cobalt, nickel, tungsten, molybdenum, and zinc serve as cofactors in enzymes which are involved in the biochemistry of methane formation, and their insufficiencies may inhibit the methanogenic activity, resulting in the accumulation of VFA, especially propionate. Therefore, the poor performance of both reactors during the period may be attributed to the deficiency of certain trace elements in the both fed wastewaters.

During the fifth operation period from day 178 to day 232, the OLRs of both reactors were gradually increased provided that the soluble COD removal efficiency remained up to 80% or more. If the soluble COD removal efficiency of a reactor was lower than 80%, the individual or combined trace elements were discontinuously added into the fed wastewater for optimizing its performance. R9 was only fed with the trace element association containing Fe, Ni, and Co in the first three days of this period, during which the soluble COD removal efficiency of the reactor was increased by about 10%; thereafter, it performed well and stably although it was no longer fed trace elements during the 40 days that followed the three day's trace element addition, with the soluble COD removal efficiency of mostly up to 80% even though the OLR was quickly increased and pH value in influent was down to about five or less. On day 232, the soluble COD removal efficiency of R9 was 82.3% with OLR of 29.59 g COD/L d and HRT of 1.55 days, and VAF and propionate levels in its effluent were 3.5 and of 1.68 g/L, respectively. In contrast, R30 was fed with the different element associations for four times during the fifth running period, and performed unstably until after the addition of the combined trace elements containing Fe, Ni, Co, Mo, Cu, and Zn was finished. On day 232, the soluble COD removal efficiency of

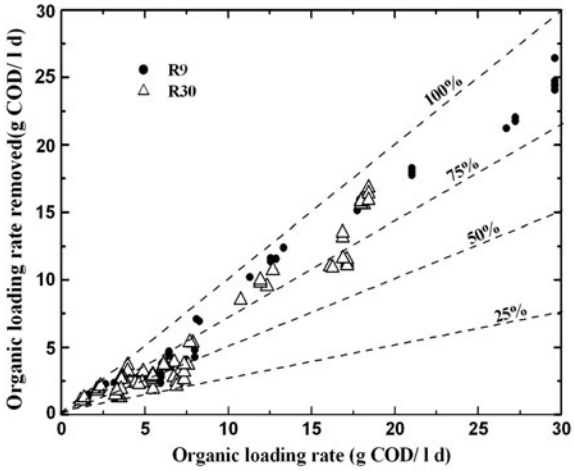

Fig. 3. Plot of OLR vs. OLR removed in the reactors.

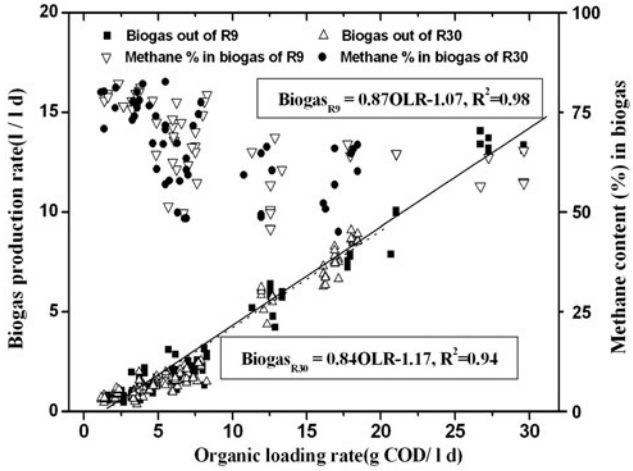

Fig. 4. Relationships between OLR and biogas production rate and methane contents.

R30 was 86.1% with OLR of 18.4 g COD/L day and HRT of 2.49 days, and VAF and propionate levels in its effluent were 2.10 and 0.8 g/L, respectively.

To identify if the fed wastewater is deficient in trace elements or not is not the objective of this study, but the fact that both reactors run well after short-term dosing some trace elements in both fed wastewaters, suggesting that inadequate amounts of trace elements were indeed present in the fed wastewater two of both reactors. In term of the OLR and COD removal efficiency, R9 performed better than R30 within 232 operation days, suggesting that the performance of the reactor was enhanced by a decrease in size and an increase in the specific surface area of the carrier used. Plot of the OLR applied against the OLR removed for both reactors showed that there did not existed peaks in Fig. 3, implying that the OLR applied and the OLR removed of both reactors can be further raised if the experiment continued.

3.2. Biogas production and methane contents

Fig. 4 showed that there was a good correlation between the OLR applied and the biogas production for each reactor. The volumetric biogas production rate (VBPR) of R9 varied between 0.46 and 14.06 L/L d during the whole operation days, and that was up to 13.07 L/L d on day 232. The VBPR of R9 was increased with OLR, having a slope of 0.87 L VBPR per gram COD loaded, indicating that 87% soluble COD fed to the reactor would be converted to biogas. The VBPR of R30 ranged from 0.45 to 9.09 L/L d, and that was up to 8.54 L/L d at the end of the experiment. The VBPR of R30 was also increased with the OLR, with a slope of 0.84 L VBPR per gram COD loaded, suggesting that 84% soluble COD fed to the reactor would be transformed into biogas. In term of the VBPR and the percentage of the fed soluble COD converted to biogas, R9 performed better than R30.

The biogas compositions from both reactors were somewhat similar. Generally, they were decreased with OLR increasing. The methane content of R9 ranged from 45.81 to 82.18% with mean of 68.01%, and that of R30 varied from 45.11 to 82.61% with average of 66.06%. Low methane contents in biogas from both reactors may be ascribed to overloading, which made the acidifying microorganisms being prevailed and then led to VFA accumulation.

3.3. Attached solid accumulation from R9

Fig. 5 showed that the carrier elements in both reactors were capable of attaching a considerable quantity of solids with the running days.

At the end of this experiment, TAS, AVS, TAS per carrier and AVS per carrier were 1,388.8, 951.3, 0.099 g TAS/carrier, and 0.064 g AVS/carrier for R9, 332.6, 182.6, 0.0594 g TAS/carrier, and 0.326 g AVS/carrier for R30, respectively, indicating that carrier with small size and big specific surface area had high biomass attachment that led to increase the quantity of biomass. The total SS and VSS within both reactors fluctuated with OLR and running days (Fig. 5). At the end of this experiment, the total SS and VSS were 178.8 and 108.0 g for R9, 209.7 and 104.7 g for R30, respectively. The quantity of total SS was accounted for 11.4% total solids for R9, 61.3% total solids for R30, respectively, suggesting that 88.6% total solids in R9 were on the carrier media, while 38.7% total solids in R30 were on the carrier elements. The amount of

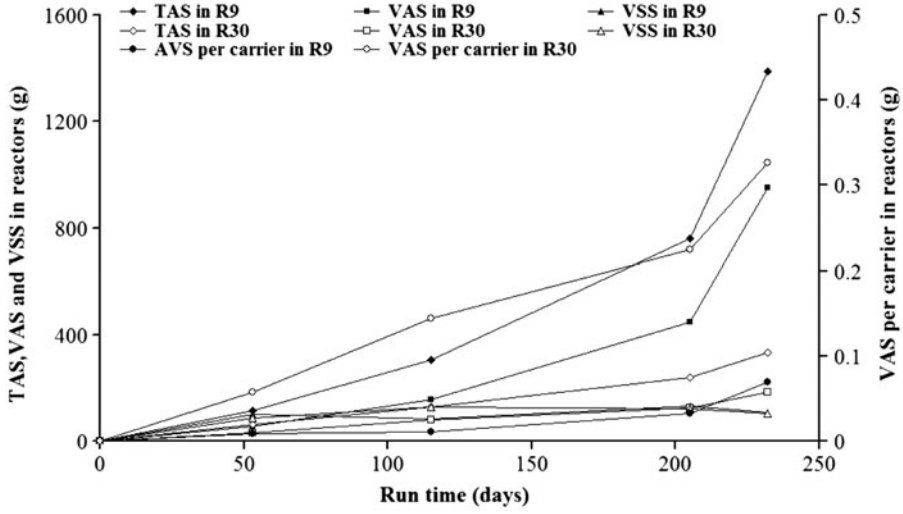

Fig. 5. Variation of attached and suspended solids with the run time of the reactors.

VSS in both reactors was fairly similar, but the ratios of VSS to total volatile solids (VS) in both reactors were quite different. VSS in liquid phase in R9 accounted for 8.2% total VS, while that in R30 took 36.4% total VS.

The specific biomass activity was estimated with OLR in terms of g COD/day and the quantity of the total VS in the reactor [46]. On day 232, the specific biomass activity of R9 was 0.92 g COD/g VS day, and this value was less than the value of 1.19 g COD/g VS day from reactor S9 of the anaerobic fixed bed system [46], where the same kind of carriers as R9 was packed within this study; whilst the specific biomass activity of R30 was 2.11 g COD/g VS day, which was greater than 0.93 g COD/g VS day from reactor S30 of the anaerobic fixed bed system [46] filled with the same kind of carriers as R30 in this study.

The observed VS yield in R9 was 0.0575 g VS/g COD removed, which was in good agreement with the VS yield of 0.0565 g VS/g COD destroyed in the reactor S9 of the fixed bed reactor [46] and the VS yield of 0.0585 g VS/g COD removed in a downflow anaerobic packed-bed reactor [56]. The observed VS yield in R30 was only 0.0235 g VS/g COD removed, which was lower than the VS yield of 0.053 g VS/g COD destroyed in the reactor S30 of the anaerobic fixed bed reactor [46]. The maximum OLR of 29.59 g/L day obtained in R9 was mainly attributed to the greater VS.

If all the biomass in the reactors was in suspension, the bacterial density would reach to 32.2 g/L for R9, 8.7 g/L for R30, respectively, and the high-bacterial density in R9 may contribute to the tolerance to high OLR and/or short HRT.

4. Conclusions

The performances of the two anaerobic MBBRs filled with carriers of varying in size and specific surface areas were investigated using winery wastewater. Both reactors were run in parallel for 232 days. At the end of the experiment, R9 attained OLR of 29.59 g COD/L day with more than 80% COD removal efficiency at HRT of 1.55 days, and the attached solids per carrier for R9 was 0.099 g TAS/carrier, accounting for 88.6% total biomass; while R30 achieved OLR of 18.43 g COD/L d with 80% COD removal efficiency at HRT of 2.49 days, and fixed solids per carrier for R30 was 0.594 g TAS/carrier, only taking up to 38.7% total biomass.

Biogas production of each reactor was increased and strongly correlated with OLR. The reactor performance can be optimized by the short-term supplementation of trace elements essential in anaerobic digestion of winery wastewater.

The experimental results showed that R9 with small carrier of the large specific surface area performed better than R30 with big carrier of the small specific surface area in achieving the amount of OLR and in retaining the quality of attached solids, indicating that the carrier with small size and great specific surface area had high capability in biomass attachment, which resulted in the increase of the biomass quantity.

An anaerobic MBBR is very easy to fabricate and simple to operate, with a minimum mixing of carrier within the reactor, low requirement for attention, and small space for installation, etc. Therefore, the reactor would be a promoting option in high strength wastewater treatment sector.

Acknowledgments

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Symbols

AVS	—	attached volatile solids (g)
COD	—	chemical oxygen demand (g/L)
EGSB	—	expanded granular sludge blanket
FID	—	flame ionization detector
HRT	—	hydraulic retention time (h)
MBBR	—	moving bed biofilm reactor
OLR	—	organic loading rate (g COD/L d)
PE	—	polyethylene
PP	—	polypropylene
PU	—	polyurethane
PVA	—	polyvinyl alcohol
SS	—	suspended solids (g/L)
TMP	—	thermomechanical pulp
TAS	—	total attached solids (g)
UASB	—	upflow anaerobic sludge beds
VBPR	—	volumetric biogas production rate (L/L d)
VFA	—	volatile fatty acid (g/L)
VS	—	total volatile solids (g)
VSS	—	volatile solids (g/L)

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