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High rate anaerobic filter with floating supports for the treatment of effluents from small-scale agro-food industries

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ABSTRACT

The performance of four laboratory-scale upflow anaerobic filters (UAFs) of about 10 L effective volume packed with small buoying polyethylene media was investigated for the treatment of wastewater discharged from various small-scale agro-food industries with different composition and concentrations viz. synthetically prepared low strength (~1.9 g COD/L), fruit canning (~10 g COD_/L), winery (~20 g COD_/L) and cheese-dairy (~30 g COD_/L) wastewaters. For the low strength wastewater, HRT was the limiting parameter with a minimum of 4 h corresponding to a maximum OLR of 12 g COD/L.d. For the high concentrated substrates, OLR was the limiting parameter, which was influenced by the nature of the substrates treated and not by the concentration of the substrates. Indeed, the UAF treating winery wastewater (20 g COD/L) had reached the highest OLR of 27 g COD/L.d with 80% COD removal efficiency, while that of the other two reactors treating fruit canning and cheese-dairy wastewaters were comparatively lower (19 and 17 g COD/L.d respectively). At the end of the experiments, the total quantities of VSS inside the reactors were high (200–342 g), indicating that the low-density polyethylene support used in this study appears to be a good colonisation matrix to increase the quantity of biomass in the reactor. The packing medium had a dual role in the retention of the biomass that is entrapment of biomass within the support and filtration of the biomass in suspension to some extent. This result was confirmed by the specific biomass activity values, which were very close to that of suspended biomass. The efficiency of liquid mixing was good, even if the biomass matrix represented up to 70% of the reactor volume for the reactor fed with fruit canning wastewater.

Keywords: UAF; Anaerobic digestion; Agro-food industrial wastewaters; Packing media; Biomass activity

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1. Introduction

Small-scale agro-industries play a major role in the economy and at the same time they remain one of the most crucial industrial generators of water pollution and consumers of fossil fuel energy [1,2]. The adoption of anaerobic digestion technology for the treatment of a wide range of agro-food industrial wastewaters has been largely favoured by the development of reactors, which permit effective biological solids retention. Retention of the active biomass within the reactor in advanced anaerobic systems is achieved either by the development of a granular sludge or by the use of inert support materials for the biomass entrapment or attachment. The fixed bed reactor or anaerobic filter is a bioprocess in which the bacteria colonize in and around certain materials, which increase the usable surface area for the bacterial growth [3]. This kind of reactor allows a higher volume load and is suitable for wastewater treatment of high organic material content.

A research work was carried out to determine the suitability and stability of an upflow anaerobic filter (UAF) with small buoying polyethylene supports for the treatment of wastewater discharged from various small-scale agro-food industries with different nature of sub-strates and feed concentrations (between 1.9 and 30 g COD_t/L) at mesophilic conditions (33±1°C). The present work aims to evaluate: (i) the maximum treatment capacity according to the various substrates; (ii) the hydrodynamics inside the reactors; (iii) the quantity of biomass inside the reactor and the specific biomass activity.

2. Materials and methods

2.1. Laboratory scale reactors

The treatment of agro-food industrial wastewaters such as low strength, fruit canning, winery and cheesedairy by UAFs was carried out using four double-walled laboratory-scale reactors designated as R_1 , R_2 , R_3 and R_4 respectively, at different operating conditions. A schematic diagram of the laboratory scale UAF used in this study is shown in Fig. 1. Each reactor had an effective volume of 10 L and was equipped with a hot water jacket to maintain a mesophilic temperature of 33±1°C. The carrier material used was small buoying polyethylene packing media, which are cylindrical in shape (29 mm high and 30/35 mm diameter) and baffled with 16 compartments. Reactors were filled with these media entities to a height of about 75 cm (about 80% of working volume of the reactor). The density and specific area of the media were 0.93 and 320 m²/m³ respectively. The reactors were fed in an upflow mode with low strength (R_1) , fruit canning (R_2) , winery (R_3) and concentrated cheese-dairy (R_{λ}) wastewaters with concentrations of about 1.9, 10, 20 and 30 g COD/L respectively. All the



Fig. 1. Schematic diagram of a laboratory scale UAF.

reactors were equipped with a continuous internal recirculation system from top to bottom of the reactor at the rate of 10 L/h. Recirculation was done mainly to eliminate the possibility of high organic loading close to the feed port and to achieve better contact of wastewater and sludge. However, for the reactor R_1 treating low strength wastewater, recirculation was stopped on day 47, mainly to minimize the solids washout at lower HRT. All the reactors were charged with anaerobic sludge (10% by volume) collected from a methanogenic digester treating distillery vinasse. The concentration of volatile suspended solids (VSS) in the sludge was estimated to be around 21 g/L.

2.2. Characteristics of the wastewater used

2.2.1. Low strength wastewater

The UAF (R_1) was fed with a COD_t of 1.9±0.2 g/L (made up of peptone, 1.09 g/L; meat extract, 0.75 g/L; NaCl, 0.48 g/L; starch, 0.36 g/L; cellulose, 0.12 g/L; CaCl₂.2H₂O, 0.04 g/L; K₂HPO₄, 0.19 g/L) mainly to represent low strength wastewater discharged from agro-food industries such as sugar mill effluents or wash water from dairy industry. pH of the synthetically-prepared feed was in the range of 6.3–6.5.

2.2.2. Fruit canning wastewater

Another UAF (R_2) was operated with fruit canning wastewater. Raw pineapple juice was diluted in the ratio of 1:15 using tap water to represent a high-strength soluble fruit canning wastewater. COD, of the wastewa-

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ter was in the range of 9.0–11.6 g/L. The substrate was supplemented with nutrients such as NH_4Cl and $NH_4H_2PO_4$ to attain COD:N:P of 400:7:1 in the wastewater as suggested by Henze and Harremoes [4]. The major carbohydrate constituents in the wastewater were the simple sugars: sucrose, glucose and fructose and the two major acids were citric and malic. pH of the feed ranging from 3.5 to 4 was adjusted to 6–6.5 prior to feeding using sodium hydrogen carbonate (NaHCO₃).

2.2.3. Winery wastewater

The feed of the UAF (R₃) i.e. high strength winery wastewater was prepared by diluting red wine (11% alcohol content) with tap water in the ratio of 1:10. Average total and soluble COD of the wastewater prepared were found to be 20.2±0.9 g COD_t/L and 18.5±0.5 g COD_s/L. Total suspended solids (TSS) were found in the range of 1.4 g/L and the pH was about 5.5. pH of the feed was maintained at around 6-6.5 using NaHCO₃. The feed was supplemented with nutrients such as NH₄Cl and NH₄H₂PO₄ to maintain COD:N:P of 400:7:1 in the wastewater. Ethanol is the primary constituent of winery wastewater.

2.2.4. Concentrated cheese-dairy wastewater

An acidic cheese whey (with pH of about 4 and concentration of about 60 g COD_t/L) taken from a middlesize cheese factory was diluted with tap water in the ratio of 1:2 to simulate the effluents of cheese dairies without whey recovery. Whey contains lactose (5%), proteins (1%), lipids (0.2%), ash (0.6%) and water (94%), along with most of the minerals present in milk and watersoluble vitamins [5]. An UAF (R₄) was fed with complex cheese dairy wastewater of COD_t varying between 23 and 40 g/L and particulate COD (COD_p) between 1.2 and 5.7 g/L. pH of the feed was adjusted to 6–6.5.

2.3. Analytical methods

The performance of the UAFs was evaluated by monitoring total (COD_t) and soluble (COD_s) chemical oxygen demands, total suspended solids (TSS), volatile suspended solids (VSS) and alkalinity according to the Standard Methods [6] both at inlet and outlet of the reactors. Volatile fatty acids (VFAs) were measured using gas chromatograph with a flame ionization detector GC 8000 (Fison instrument) and an automatic sampler AJ 800 (Fison instruments), nitrogen being the carrier gas (335 kPa).

Towards the end of experiments, residence time distribution (RTD) studies were performed using sodium chloride (25 mL of 300 g NaCl/L) as a tracer in these reactors. The tracer solution was injected as a pulse in the influent stream and the evolution of the electrical conductivity, linked to the changes in the NaCl concentration [C(t)], was continuously measured online every 30 s at the outlet of the reactor using an electrical conductivity probe. This technique applied to fixed bed bioreactors is commonly described in the literature for biological reactors [7,8], and the analysis of tracer response curve is well established [9]. Although NaCl is not the best tracer for studying the mixing in a biological reactor, it was selected because it is simple to use. The aim of this part of the research was limited to assess the overall efficiency of the mixing of the liquid.

At the end of the experimental study, all the reactors were emptied to quantify the amount of volatile solids inside the reactors. The biomass inside the reactors was categorized into two groups, namely entrapped/attached biomass and the free biomass. Free biomass included both the settled biomass and the non-attached biomass. Supports were removed in batches of 1 L (which consisted of approximately 24 pieces of polyethylene media) starting from the top of the reactor (near the outlet) to the bottom (above the sludge bed). The supports were placed in aluminium foils and oven dried for 24 h to estimate the total solids. Oven-dried solid samples were scrapped out from the supports and ignited at 550°C for 2 h to estimate the volatile solid content. Whereas, the TSS and VSS of free biomass were determined according to the Standard Methods [6]. The specific biomass activity was estimated using the method described by Thanikal et al. [3].

2.4. Experimental strategy

UAFs were fed with wastewaters of different composition and concentrations, representing various agro-food industrial effluents. Four UAFs were set-up to treat (i) R_1 low strength, (ii) R_2 – fruit canning, (iii) R_3 – winery and (iv) R_4 – concentrated cheese dairy wastewaters. These reactors were operated for a total period of 174, 185, 180 and 197 days respectively. UAFs R₁, R₂, R₃ and R₄ were initially fed at a low OLR of 0.4 g COD/L.d and an extended HRT of 4.5, 24, 48 and 77 d, respectively. OLR was then progressively increased in steps by 20–30% once or twice a week provided that COD_c removal efficiency remained above 80%. A COD removal efficiency of 80% was considered as the threshold level in the present study for the operation of the reactors as previously described by Thanikal et al. [3]. Efforts were made to maintain constant influent COD concentration, while the OLR was gradually increased by decreasing the hydraulic retention time (HRT). Maximum OLR corresponding to 80% COD removal was thus ascertained for each reactor. At this stage, a pulse of NaCl as tracer was injected along with the feed and its concentration in the effluent was monitored in order to analyze the hydrodynamics of UAFs. At the end of the experiments, all the reactors were dismantled and all the supports were removed in order to quantify the biomass inside the reactors.

3. Results and discussion

3.1. Example of the results obtained

An example of the results obtained from the UAF treating winery wastewater is presented in Fig. 2. The reactor was operated for about 6 months. During the start up period (initial 14 days of operation), the COD, and COD_c concentrations in the effluent remained quite high, at a low OLR of 0.4 g COD/L.d (Fig. 2). These values then decreased gradually from 2.7 to a minimum of 0.8 g COD₄/L and from 2.1 to 0.4 g COD₄/L resulting in COD₄ removal efficiency of around of 96%. Perusal of the data given in Fig. 2 reveals a decreasing trend in COD removal with gradual increase in OLR. In this experiment, the OLR was increased to a maximum of 35 g COD/L.d corresponding to a removal efficiency of about 70% and 74% for COD, and COD, respectively (Fig. 2). As the COD, removal efficiency of 80% was considered as the threshold level for the operation of the reactor, the UAF treating winery wastewater should not be operated at OLR higher than 27 g COD/L.d and HRT lesser than 19 h. For the lowest OLR, the influent COD was efficiently converted to VFA-COD leaving only 0.3 g/L as non VFA-COD, which represents the non-biodegradable fraction of the effluent. The efficiency of this conversion, i.e. non VFA to VFA, decreased on increasing OLR beyond 15 g COD/L.d. TSS concentration at outlet varied widely with the change in OLR and ranged between 0.3–1.6 g/L. Up to 100 days of operation, the escape of TSS increased (to a maximum of 1.6 g/L) with the increasing OLR (up to 16.5 g COD/L.d). From day 100 onwards, the escape of solids in the effluent was stabilized to an average value of 0.82± 0.09 g/L. VSS was found to be between 69 and 78% of TSS. Initially, the media was not colonized and with time the biomass colonized in and around the media. Beyond 100 days, the reduction in the biomass washout may probably be linked to a better filtration capacity due to the entrapment of solids within the supports. A similar trend was observed in the other three reactors

but with different threshold limits (detailed data not shown).

3.2. Design parameters and comparison of reactor performance

Table 1 summarises the main experimental results obtained at steady state conditions for the four different substrates with concentrations ranging from 1.9 to 30 g COD/L. The minimum COD_s at outlet measured during these experiments were very low for all the wastewaters indicating that their biodegradability was very high with 94% of COD_s removal efficiency for the most diluted (i.e. low strength) wastewater and more than 98% for the 3 most concentrated wastewaters (effluent data, Table 1).

On comparing the behaviour of the different reactors, it was observed as expected, that with the increase in the organic matter concentration of the substrates (i.e. from 1.9 to 30 g/L), the values of minimal HRT were also increased from 4 h for the low strength wastewater to 1.6 days for the wastewater with 30 g COD/L. For low strength wastewater with concentration less than or around 2 g COD/L, the HRT becomes the limiting factor and it seems that 4 h is the minimum HRT that can be used for the design of UAF with the floating media used in this study.

For higher concentrations, the OLR is the controlling parameter, which can be used in the design of the volume of anaerobic reactors. The results obtained with three different substrates at 10, 20 and 30 g COD/L (Table 1) showed that the maximum OLR corresponding to a COD removal of 80%, which can be used in the design of UAF with the polyethylene supports were in the range of 17– 27 g COD/L.d.

There was no relationship found between the initial concentration of the wastewater and the maximum OLR obtained. The nature of the compounds in the influent seemed more important than its concentration. In case of UAF R_3 treating winery wastewater at feed COD_t of 20 g/L, COD_c removal of ≥80% was achieved at OLR



Fig. 2. Temporal variations of OLR and COD removal efficiencies for the reactor R₃ treating winery wastewater.

Synopsis of experimental values obtained at steady-state conditions for the treatment of different substrates in UAFs

ParametersLow strengthFuit canningWineryCheese-dairy wastewater (R)WineryCheese-dairy wastewater (R)Influent dats(R)(R)(R)(R)(R)Influent dats1.9±0.20±12±0.93±3.1Soluble COD (g/L) at feed1.75±0.29.4±0.618.5±0.52±2.8Soluble COD (g/L) at feed0.25±0.051.2±0.131.42.6±0.6VSS concentration (g/L) at feed00.25±0.051.2±0.131.42.6±0.6Reactor operating data-0.73±0.11.051.6±0.4Reactor operating data1219400.151.8±0.52±2.8IHRT (h) corresponding to 80% CODs removal efficiency4121940OLR (g COD/L, d) corresponding to 80% CODs removal 12192.717efficiency1.183.65.05.0Kaverage CODs (g/L)0.033.24.78.8Average CODs (g/L)0.121.20.823.4Average TSS concentration (g/L)0.121.20.823.4Bitmass inside the reactor at the end of the experiment124800.93.4TSS of free biomass [i.e. settled +non-attached biomass] (g)103124800.93.8TSS of free biomass [i.e. settled +non-attached biomass] (g)871086691TSS in the reactor (g)2012033.21.765VSS OS firee biomass [i.e. settled +non-attached biomass] (g)8					
Influent data Influent data Total COD (g/L) 1.9±0.2 10±1 20±0.9 30±3.1 Soluble COD (g/L) at feed 1.75±0.2 9.4±0.6 18.5±0.5 28±2.8 TSS concentration (g/L) at feed 0.25±0.05 1.2±0.13 1.4 2.6±0.6 VSS concentration (g/L) at feed - 0.73±0.1 1.05 1.65±0.4 Reactor operating data - - 1.9 40 OLR (g COD/L, d) corresponding to 80% CODs removal efficiency 4 12 19 40 OLR (g COD/L, d) corresponding to 80% CODs removal efficiency 4 12 19 40 Average CODs (g/L) 0.015 1.8 3.6 5.0 Average CODs (g/L) 0.30 3.2 4.7 8.8 Average CODs (g/L) 0.12 1.2 0.82 3.4 Biomass inside the reactor at the end of the experiment - - 1.5 1.5 TSS of free biomass [i.e. settled +non-attached biomass] (g) 109 124 80 109 TSS in the reactor (g) <t< th=""><th>Parameters</th><th>Low strength wastewater (R1)</th><th>Fruit canning wastewater (R2)</th><th>Winery wastewater (R3)</th><th>Cheese-dairy wastewater (R4)</th></t<>	Parameters	Low strength wastewater (R1)	Fruit canning wastewater (R2)	Winery wastewater (R3)	Cheese-dairy wastewater (R4)
Total COD (g/L) 1.9±0.2 10±1 20±0.9 30±3.1 Soluble COD (g/L) at feed 1.75±0.2 9.4±0.6 18.5±0.5 28±2.8 TSS concentration (g/L) at feed 0.25±0.05 1.2±0.13 1.4 2.6±0.6 VSS concentration (g/L) at feed - 0.73±0.1 1.05 1.65±0.4 Reactor operating data - - 0.73±0.1 1.05 1.65±0.4 Reactor operating data - - 0.73±0.1 1.05 1.65±0.4 Reactor operating data - - 12 19 40 OLR (g COD/L, d) corresponding to 80% CODs removal efficiency - - - 5.0 Average CODs (g/L) 0.15 1.8 3.6 5.	Influent data				
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TSS concentration (g/L) at feed 0.25 ± 0.05 1.2 ± 0.13 1.4 2.6 ± 0.6 VSS concentration (g/L) at feed $ 0.73\pm0.1$ 1.05 1.65 ± 0.4 Reactor operating data $ 0.73\pm0.1$ 1.05 1.65 ± 0.4 HRT (h) corresponding to 80% CODs removal efficiency 4 12 19 40 OLR (g COD/L. d) corresponding to 80% CODs removal efficiency 12 19 27 17 efficiency 12 19 27 17 Effluent data at 80% CODs removal efficiency 1.15 1.8 3.6 5.0 Average CODs (g/L) 0.15 1.8 3.6 5.0 Average CODs (g/L) 0.30 3.2 4.7 8.8 Average TSS concentration (g/L) 0.12 1.2 0.82 3.4 Biomass inside the reactor at the end of the experiment 124 80 109 TSS on the supports (g) 178 282 319 272 TSS of free biomass [i.e. settled +non-attached biomass] (g) 109 124 80 109 TSS in the reactor (g) 287 406 399 381 Entrapped VSS inside the supports (g) 113 234 225 178 VSS of free biomass [i.e. settled +non-attached biomass] (g) 87 108 66 91 Total VSS in the reactor (g) 200 342 291 269 No. of supports 205 201 203 198 VSS/TSS [Entrapped solids] (%) 63 83 71 <td>Soluble COD (g/L) at feed</td> <td>1.75±0.2</td> <td>9.4±0.6</td> <td>18.5±0.5</td> <td>28±2.8</td>	Soluble COD (g/L) at feed	1.75±0.2	9.4±0.6	18.5±0.5	28±2.8
VSS concentration (g/L) at feed – 0.73±0.1 1.05 1.65±0.4 Reactor operating data – 0.73±0.1 1.05 1.65±0.4 Reactor operating data – 0.73±0.1 1.05 1.65±0.4 HRT (h) corresponding to 80% CODs removal efficiency 4 12 19 40 OLR (g COD/L. d) corresponding to 80% CODs removal 12 19 27 17 efficiency 12 19 27 17 Effluent data at 80% CODs removal efficiency 4 1.8 3.6 5.0 Average CODs (g/L) 0.15 1.8 3.6 5.0 Average SC concentration (g/L) 0.12 1.2 0.82 3.4 Biomass inside the reactor at the end of the experiment – – – – Entrapped TSS on the supports (g) 178 282 319 272 TSS of free biomass [i.e. settled +non-attached biomass] (g) 109 124 80 109 TSS of free biomass [i.e. settled +non-attached biomass] (g) 87 108 66 91 Total VSS in the reactor (g) 200 342 291	TSS concentration (g/L) at feed	0.25±0.05	±0.05 1.2±0.13 1.4		2.6±0.6
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HRT (h) corresponding to 80% CODs removal efficiency 4 12 19 40 OLR (g COD/L. d) corresponding to 80% CODs removal 12 19 27 17 efficiency 12 19 27 17 Effluent data at 80% CODs removal efficiency 0.15 1.8 3.6 5.0 Average CODs (g/L) 0.10 1.2 0.82 3.4 Average TSS concentration (g/L) 0.12 1.2 0.82 3.4 Biomass inside the reactor at the end of the experiment 12 19 272 272 TSS of free biomass [i.e. settled +non-attached biomass] (g) 109 124 80 109 TSS in the reactor (g) 287 406 399 381 Entrapped VSS inside the supports (g) 113 234 225 178 VSS of free biomass [i.e. settled +non-attached biomass] (g) 87 108 66 91 Total VSS in the reactor (g) 200 342 291 269 No. of supports 205 201 203 198 VSS/TSS [Entrapped solids] (%) 63 83 71 65 </td <td>Reactor operating data</td> <td></td> <td></td> <td></td> <td></td>	Reactor operating data				
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efficiency Effluent data at 80% CODs removal efficiency Average CODs (g/L) 0.15 1.8 3.6 5.0 Average CODt (g/L) 0.30 3.2 4.7 8.8 Average TSS concentration (g/L) 0.12 1.2 0.82 3.4 Biomass inside the reactor at the end of the experiment 272 TSS of free biomass [i.e. settled +non-attached biomass] (g) 109 124 80 109 TSS in the reactor (g) 287 406 399 381 Entrapped VSS inside the supports (g) 113 234 225 178 VSS of free biomass [i.e. settled +non-attached biomass] (g) 87 108 66 91 Total VSS in the reactor (g) 200 342 291 269 No. of supports 205 201 203 198 VSS/TSS [Entrapped solids] (%) 63 83 71 65 Specific activity (g COD/g of VSS. d) 0.60 0.56 0.93 0.63	OLR (g COD/L. d) corresponding to 80% CODs removal	12	19	27	17
Effluent data at 80% CODs removal efficiency 0.15 1.8 3.6 5.0 Average CODs (g/L) 0.30 3.2 4.7 8.8 Average TSS concentration (g/L) 0.12 1.2 0.82 3.4 Biomass inside the reactor at the end of the experiment 124 80 109 TSS of free biomass [i.e. settled +non-attached biomass] (g) 109 124 80 109 TSS in the reactor (g) 287 406 399 381 Entrapped VSS inside the supports (g) 113 234 225 178 VSS of free biomass [i.e. settled +non-attached biomass] (g) 87 108 66 91 Total VSS in the reactor (g) 200 342 291 269 No. of supports 205 201 203 198 VSS/TSS [Entrapped solids] (%) 63 83 71 65 Specific activity (g COD/g of VSS. d) 0.60 0.56 0.93 0.63	efficiency				
Average CODs (g/L) 0.15 1.8 3.6 5.0 Average CODt (g/L) 0.30 3.2 4.7 8.8 Average TSS concentration (g/L) 0.12 1.2 0.82 3.4 Biomass inside the reactor at the end of the experiment 178 282 319 272 TSS of free biomass [i.e. settled +non-attached biomass] (g) 109 124 80 109 TSS in the reactor (g) 287 406 399 381 Entrapped VSS inside the supports (g) 113 234 225 178 VSS of free biomass [i.e. settled +non-attached biomass] (g) 87 108 66 91 Total VSS in the reactor (g) 200 342 291 269 No. of supports 205 201 203 198 VSS/TSS [Entrapped solids] (%) 63 83 71 65 Specific activity (g COD/g of VSS. d) 0.60 0.56 0.93 0.63	Effluent data at 80% CODs removal efficiency				
Average CODt (g/L)0.303.24.78.8Average TSS concentration (g/L)0.121.20.823.4Biomass inside the reactor at the end of the experiment </td <td>Average COD_s (g/L)</td> <td>0.15</td> <td>1.8</td> <td>3.6</td> <td>5.0</td>	Average COD _s (g/L)	0.15	1.8	3.6	5.0
Average TSS concentration (g/L) 0.12 1.2 0.82 3.4 Biomass inside the reactor at the end of the experiment Entrapped TSS on the supports (g) 178 282 319 272 TSS of free biomass [i.e. settled +non-attached biomass] (g) 109 124 80 109 TSS in the reactor (g) 287 406 399 381 Entrapped VSS inside the supports (g) 113 234 225 178 VSS of free biomass [i.e. settled +non-attached biomass] (g) 87 108 66 91 Total VSS in the reactor (g) 200 342 291 269 No. of supports 205 201 203 198 VSS/TSS [Entrapped solids] (%) 63 83 71 65 Specific activity (g COD/g of VSS. d) 0.60 0.56 0.93 0.63	Average CODt (g/L)	0.30	3.2	4.7	8.8
Biomass inside the reactor at the end of the experiment282319272Entrapped TSS on the supports (g)178282319272TSS of free biomass [i.e. settled +non-attached biomass] (g)10912480109TSS in the reactor (g)287406399381Entrapped VSS inside the supports (g)113234225178VSS of free biomass [i.e. settled +non-attached biomass] (g)871086691Total VSS in the reactor (g)200342291269No. of supports205201203198VSS/TSS [Entrapped solids] (%)63837165Specific activity (g COD/g of VSS. d)0.600.560.930.63	Average TSS concentration (g/L)	0.12	1.2	0.82	3.4
Entrapped TSS on the supports (g)178282319272TSS of free biomass [i.e. settled +non-attached biomass] (g)10912480109TSS in the reactor (g)287406399381Entrapped VSS inside the supports (g)113234225178VSS of free biomass [i.e. settled +non-attached biomass] (g)871086691Total VSS in the reactor (g)200342291269No. of supports205201203198VSS/TSS [Entrapped solids] (%)63837165Specific activity (g COD/g of VSS. d)0.600.560.930.63	Biomass inside the reactor at the end of the experiment				
TSS of free biomass [i.e. settled +non-attached biomass] (g)10912480109TSS in the reactor (g)287406399381Entrapped VSS inside the supports (g)113234225178VSS of free biomass [i.e. settled +non-attached biomass] (g)871086691Total VSS in the reactor (g)200342291269No. of supports205201203198VSS/TSS [Entrapped solids] (%)63837165Specific activity (g COD/g of VSS. d)0.600.560.930.63	Entrapped TSS on the supports (g)	178	282	319	272
TSS in the reactor (g) 287 406 399 381 Entrapped VSS inside the supports (g) 113 234 225 178 VSS of free biomass [i.e. settled +non-attached biomass] (g) 87 108 66 91 Total VSS in the reactor (g) 200 342 291 269 No. of supports 205 201 203 198 VSS/TSS [Entrapped solids] (%) 63 83 71 65 Specific activity (g COD/g of VSS. d) 0.60 0.56 0.93 0.63	TSS of free biomass [i.e. settled +non-attached biomass] (g)	109	124	80	109
Entrapped VSS inside the supports (g) 113 234 225 178 VSS of free biomass [i.e. settled +non-attached biomass] (g) 87 108 66 91 Total VSS in the reactor (g) 200 342 291 269 No. of supports 205 201 203 198 VSS/TSS [Entrapped solids] (%) 63 83 71 65 Specific activity (g COD/g of VSS. d) 0.60 0.56 0.93 0.63	TSS in the reactor (g)	287	406	399	381
VSS of free biomass [i.e. settled +non-attached biomass] (g) 87 108 66 91 Total VSS in the reactor (g) 200 342 291 269 No. of supports 205 201 203 198 VSS/TSS [Entrapped solids] (%) 63 83 71 65 Specific activity (g COD/g of VSS. d) 0.60 0.56 0.93 0.63	Entrapped VSS inside the supports (g)	113	234	225	178
Total VSS in the reactor (g) 200 342 291 269 No. of supports 205 201 203 198 VSS/TSS [Entrapped solids] (%) 63 83 71 65 Specific activity (g COD/g of VSS. d) 0.60 0.56 0.93 0.63	VSS of free biomass [i.e. settled +non-attached biomass] (g)	87	108	66	91
No. of supports 205 201 203 198 VSS/TSS [Entrapped solids] (%) 63 83 71 65 Specific activity (g COD/g of VSS. d) 0.60 0.56 0.93 0.63	Total VSS in the reactor (g)	200	342	291	269
VSS/TSS [Entrapped solids] (%) 63 83 71 65 Specific activity (g COD/g of VSS. d) 0.60 0.56 0.93 0.63	No. of supports	205	201	203	198
Specific activity (g COD/g of VSS. d) 0.60 0.56 0.93 0.63	VSS/TSS [Entrapped solids] (%)	63	83	71	65
	Specific activity (g COD/g of VSS. d)	0.60	0.56	0.93	0.63

 \leq 27 g COD/L.d. In the case of the other two reactors treating fruit canning (10 g COD_t/L) and concentrated cheesedairy (30 g COD_t/L) wastewaters, comparatively lower OLR (i.e. maximum of 19 and 17 g COD/L.d respectively) could be applied to achieve at least 80% COD_s removal. These two reactors (R₂ and R₄) were operated for sufficiently long time at these OLRs but it was not possible to exceed these values of OLR with the condition that COD_s removal remains at least 80%. Indeed, each time the OLR was increased, a rapid decrease in the removal efficiency was observed. These loading rates appeared to be the maximum that could be applied to maintain the threshold limit for these two substrates.

For the low strength wastewater with concentration of about 1.9 g COD_t/L, the maximum OLR reached was 12 g COD/L.d with a minimum HRT of 4 h. Similar results were described by Ndon and Dague [10] in an anaerobic sequential batch reactor (AnSBR) treating dairy (non-fat dry milk) wastewater with influent COD of 1 g/L. Whereas, Halalsheh et al. [11] operated an UASB reactors treating low strength wastewater (1.6 g COD_t/L), at a comparatively lower OLR with a maximum value of 5 g COD/L.d and an HRT of 8–10 h.

For the high concentrated wastewaters, the results obtained show that the UAFs with the floating polyethylene supports could attain quite high OLRs, which are comparable with the results found in the literature. Indeed, for the treatment of fruit canning wastewater, Trnovec and Britz [12] achieved COD reductions up to 93% at OLR of 11 g COD/L.d and HRT of about 12 h using an UASB reactor. Cheng et al. [13] treated winery effluents in six winery plants using UASB reactors at a high loading rate of 15 g COD/L.d with influent COD concentration of 15 g/L and 26 h of HRT and 82% of average COD removal efficiency. In another study, Yu et al. [14] reported a maximum loading rate of 37 g COD/L.d and a HRT of 8 h with 82% COD removal efficiency for the treatment of winery effluents in anaerobic filter using string shaped plastic media. With a dairy wastewater of 10 g COD/L, maximum OLR of 10.8 g COD/L. d was reported for an anaerobic fixed bed reactor using polypropylene cascade mini-rings with COD reduction of 75% [15].

The escape of the TSS from the reactor does not appear to depend on the OLR or flow rate. Indeed, reactors R_1 , R_2 and R_3 had comparatively lower TSS concentra-

tions at the outlet with average values of 0.12, 1.2 and 0.82 g/L respectively. Escape of solids was recorded to be more in R₄ treating cheese dairy wastewater. Even at an initial low OLR of 0.4 g COD/L.d, the TSS in the effluent from R₄ was observed to be 1.3 g/L. At higher OLRs, there was solids washout as indicated by the higher TSS concentration of 3.4 g/L against the initial TSS of 2.6 g/L for R_4 (Table 1). The reason for the more solids washout especially for cheese dairy wastewater is not known, but a possible explanation for it is obtained from literature wherein Malaspina et al. [16] stated that cheese whey is a quite problematic substrate to treat anaerobically because of its very low bicarbonate alkalinity, high COD concentration, a tendency to acidify very rapidly, and the tendency to produce an excess of viscous exopolymeric materials of probable bacterial origin and that can be a cause of solids washout.

Though the reactors showed effective treatment possibilities, the organic matter concentrations in the effluent remained above the discharge limits. Therefore, it is necessary to include a post-treatment stage for the effluents generated from the UAFs to comply with the limits for discharge into the environment.

3.3. Liquid mixing and reactor clogging

Before terminating the experiments, a pulse of tracer NaCl was introduced along with the feed to analyse the hydrodynamics of the UAFs viz. R_1 , R_2 , R_3 and R_4 . Details for analyzing RTD functions based on stimulus-response experiments have been described by Levenspiel [9]. The normalized concentration of NaCl [i.e. measured concentration (C_i) / initial concentration (C_0) = $E(\theta)$] in the effluent was plotted against the normalized time [i.e. time of sampling (t) / theoretical residence time (τ) = θ] and tracer response recorded as conductivity is graphically presented in Fig. 3. The results of the RTD analyses are given in Table 2.

The degree of liquid mixing inside the UAFs can be explained as follows:

 Values of dispersion coefficient (*D/uL*) for the reactors R₂, R₃ and R₄ were found to be relatively high

Table 2		
Results o	f RTD analyses	



Fig. 3. Residence time distribution curves.

 $(D/uL = 1.07 \text{ for } R_2, 1.0 \text{ for } R_3 \text{ and } 1.2 \text{ for } R_4)$ when compared to $R_1 (D/uL = 0.1)$ [Calculated from a relationship derived from analogous form of Fick's law [9], i.e.

$$\frac{\sigma^2}{\overline{t}^2} = 2\left(\frac{D}{\mu L}\right) - 2\left(\frac{D}{\mu L}\right)^2 \left(1 - e^{-\left(\frac{uL}{D}\right)}\right) \tag{1}$$

where σ^2 is the variance of the RTD curve, \overline{t}^2 is mean time of passage].

ii. The fraction of dead space in the reactor R_1 was found to be high (68% of the reactor volume) as compared to 35% for R_2 , 12% for R_3 and 14% for R_4 . [Calculated using the formula

$$\frac{V_d}{V} = 1 - v_a \mu_a \tag{2}$$

where V_d = volume of dead space in the reactor (L), V = working volume of reactor (L), v_a = fraction of tracer recovered within 2 residence times, μ_a = mean of the curve between 2 residence times].

The experimental RTD curves were compared to that of a theoretical CSTR curve (Fig. 3). It can be seen that the curves of the UAFs R_3 and R_4 fed with winery and cheese dairy wastewaters respectively, had a trend somewhat close to the CSTR. Whereas, the long tail exhibited on RTD curve for the reactor R_1 fed with low strength

Reactor	Mean \overline{t} (d)	Variance σ ² (d ²)	$\frac{\sigma^2}{\overline{t}^2}$	Dispersion number, D/uL	Dead space fraction (V _d /V) %	Experimental mean residence time, <i>t</i> _E (h)	V _L (L)	Theoretical residence time, $\tau = V_L/Q^*$ (h)
R_1	1.3	0.042	0.24	0.1	68	9.7	6.8	6
R ₂	0.137	0.014	0.75	1.07	35	3.25	3.2	5.0
R3 R4	0.550 1.14	0.21 1.0	0.72 0.77	1 1.2	12 14	13.2 27	5 5.5	13.5 27.6

 V_{I} = Volume of the liquid at the end of the study, *Q* = liquid flow rate

wastewater reflected the presence of dead zone. Reactor R₁ from day 47 onwards was operated without internal recirculation, which probably led to the development of dead space as high as 68%. While, the UAF R, fed with fruit canning wastewater, was characterised by two events (Fig. 3). The first event i.e. high peak at the beginning of the curve can be related to preferential pathways occurring within the anaerobic filter, whereas the second event (long tail at the end of the curve) can be induced by the tracer diffusion into the biofilm matrix, reflecting the presence of dead zones [8]. Experimental average residence time was only 0.65 times the theoretical residence time (τ) for R, which shows that the liquid was leaving the reactor before the expected time. The reason for this preferential pathway or channelling can be explained by the higher quantity of biomass accumulation along with the higher quantity of extracellular polymeric substances (discussed in Section 3.4). Escudie et al. [8] observed that experimental average residence time was only 0.13 times the τ , for which $E(\theta)$ went up to 22.5 in 7 years of operation for the treatment of distillery vinasse in an UAF packed with Cloisonyle® support. A similar trend of preferential pathway was also reported by Malina and Pohland [17] for treating cheese dairy wastewaters in a down-flow fixed bed reactor but with larger dead volume space of about 80%. Young and Young [18] studied the hydrodynamic characteristics of an anaerobic filter and found that the dead space was between 50 and 93% of the reactor volume. According to Ortiz-Arroyo et al. [19], when liquid containing low concentrations of fine solid impurities (such as in pine apple juice) are treated in anaerobic filters, clogging develops so that ultimately the flow becomes severely hampered.

At the end of the study the liquid volume was observed to be around 6.8, 3.2, 5 and 5.5 L respectively for $R_{1'}$, $R_{2'}$, R_{3} and R_{4} , which means that the biomass matrix occupied a volume of about 3.2, 6.8, 5 and 4.5 L. The efficiency of liquid mixing was good for these reactors, even if the biomass matrix represented up to 70% of the reactor volume for the reactor R_{2} fed with fruit canning. The high rate of internal recirculation in anaerobic filters had a positive effect on liquid mixing.

Clogging can be explained by various parameters including the liquid mixing or amount of dispersion, fraction of dead space, channelling and quantity of biomass accumulation etc. However, the clogging in the UAF R_1 (which was operating without the internal recirculation) was comparatively more. Low value of upflow velocity (0.24 m/h) was recorded in this reactor against the upflow velocities of about 1 m/h for R_2 , R_3 and R_4 . Hence, it is recommended to have an intermittent recirculation flow in order to improve the liquid mixing inside the reactor R_1 .

Since the treatment efficiency is good, the media plugging is not a real problem at the end the experiments. However, long term investigation need to be realized to know if the hydrodynamic strengths generated both by liquid velocity and biogas flow are sufficient to maintain good macro-mixing within the fixed bed.

3.4. Biomass activity analysis

At the end of the experiments, the reactors were emptied to quantify the amount of VSS inside the reactors. The results of the biomass quantification (both entrapped/attached and free biomass) and the specific biomass activity for all the reactors are given in Table 1.

It was observed that the supports were full of biomass but the solids were not strongly attached to the surface rather they were just entrapped. Furthermore, the fraction of biomass entrapped inside the supports represented between 57 and 77% of total biomass inside the reactor (Table 1), indicating that the packing medium had a dual role in the retention of the biomass that is to say entrapment of biomass within the support and filtration of the biomass in suspension to some extent. The lowdensity polyethylene supports used in this study were able to retain between 0.7 g and 1.6 g dried solids per support and this appears to be an effective matrix for biomass retention.

The total quantity of VSS in the reactors at the end of the experiments (Table 1) were quite high with values of 200, 342, 291 and 269 g respectively for the four reactors namely R_1 , R_2 , R_3 and R_4 , with corresponding maximum OLRs of 12, 19, 27 and 17 g COD/L.d respectively, with about 80% COD removal efficiency.

It can be noted that the reactor R_2 treating fruit canning wastewater had the highest quantity of entrapped solids (342 g VSS) and a much higher fraction of VSS (83% instead of 64–71%) compared to other three reactors viz. R_1 , R_3 and R_4 . The probable reason could be the accumulation of high quantity of extracellular polymeric substances onto the polyethylene matrices. Also, the hydrodynamic (RTD) studies have shown the presence of channelling and dead zones in this reactor R_2 probably linked to the accumulated biomass [19].

It is evident from the results of specific biomass activity or specific mass loading rate that the nature of the substrates has a major influence on this parameter. For instance, the UAF (R_3) fed with winery wastewaters which contained mainly ethanol, had a much higher specific biomass activity (0.93 g COD/g of VSS per day) than the other three substrates which were quite close *viz*. low strength, fruit canning and cheese dairy (0.60, 0.56 and 0.63 g COD/g of VSS per day, respectively).

The average specific activity of total biomass in all the reactors remained comparable to the activity measured in other reactors operated with biomass in suspension showing that the supports served to entrap the biomass flocs inside the reactor. Ruiz [20] also reported higher specific activities for winery wastewaters (0.96– 1.25 g COD/g of VSS/d) compared to other substrates like slaughterhouse effluents (0.6 g COD/ g of VSS/d), sugarcane vinasses (0.67 g COD/g of VSS/d) or molasses vinasses (0.50 g COD/g of VSS/d). Comparatively lower values of specific activity could be explained by the accumulation of extracellular organic materials in the reactors treating complex substrates containing high concentrations of carbohydrates, proteins, or lipids [16]. Also, the solids present in the substrates, which are not eliminated by the anaerobic biomass, can get accumulated in the support media and explain the lower specific activity.

Finally, high quantity of biomass was retained in the UAFs using low-density polyethylene media while maintaining good specific biomass activity and it was then feasible to operate the UAF in high-rate conditions, even for the diluted wastewaters.

4. Conclusions

This study reveals that a UAF with low-density polyethylene supports was able to treat low to high strength agro-food industrial wastewaters at quite high rates. Indeed, high OLRs could be reached (12-27 g COD/L.d) even for the low-strength wastewater. Low-density polyethylene supports used in this study appears to be a good support as it was able to retain between 0.7 g and 1.6 g dried solids per support. Biomass retention in the reactor was contributed to both entrapment within the support and a filtration effect of the support on the biomass in suspension to some extent. This result was confirmed by the specific activity values, which were very close to those of suspended biomass. At the end of the experiment, the hydrodynamic (RTD) study clearly showed a partial clogging of the UAF R, which was operated without recirculation. Whereas (RTD) study for the UAFs treating high-strength wastewaters (R_2 , R_3 and R_4 with recirculation) showed that the efficiency of liquid mixing was good, even if the biomass matrix represented up to 70% of the reactor volume for the reactor fed with fruit canning wastewater.

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