Dairy Wastewater Treatment by Anaerobic Fixed bed Reactors from Laboratory to pilot-scale plant: A case study in Costa Rica Operating at Ambient Temperature

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ABSTRACT:The effect of hydraulic retention time in a range from 1.0-5.5 days was evaluated in a laboratoryscale anaerobic fixed bed reactor packed with a hybrid material composed of tire rubber and zeolite. Under these conditions, COD removal efficiencies varied from 28.3% to 82.1%, respectively. Over the more than 6 months of operation, no clogging was observed. The results obtained demonstrated that this type of reactor was capable of operating with dairy waste at a hydraulic retention time 5 times lower than that used in a conventional digester. Based on the laboratory-scale experimental results obtained, a pilot-scale plant was designed. The pilot plant was installed in "Cot de Oreamuno" near the city of Cartago, Costa Rica.Biogas produced in the pilot-scale anaerobic plant was used for the generation of electricity on the farm. In this case, a COD removal efficiency of 63.6 % was achieved in the full-scale anaerobic plant at a hydraulic retention time of 3 days, this value being comparable with that obtained at laboratory-scale.

Key words: Dairy wastewater, Anaerobic, Fixed bed reactors, Costa Rica, Ambient temperature

INTRODUCTION

Anaerobic digestion of animal waste is the best technology for improving the environmental problems derived from animal breeding. In addition, biogas, which helps reduce fossil fuel consumption (Parson, 1986; Hobson, 1992; Uemura, 2010), is obtained. Interest in the anaerobic digestion of dairy manure has increased in the past few years due to the reduction in odors of stored and land applied manure. During the digestion process, bad odors and organic matter concentrations are reduced and a source of renewable energy (biogas) is produced. Dairy wastes are usually treated by anaerobic digestion in conventional digesters with hydraulic retention times (HRTs) higher than 15 days (Giesy et al., 2005) because these HRTs must be higher than the retention times of microorganisms or solid retention times (SRTs) in order to prevent the washout of microorganisms. Therefore, high reactor volumes are required with respect to the volume of waste to be processed. With the aim of reducing reactor volumes, different alternatives for microorganism retention, such as sludge recycling or immobilization on a support, have been developed.

Anaerobic fixed bed reactors (AFBRs) are based on the principle of the immobilization of microorganisms on a support. This type of reactor has been successfully and widely applied for the treatment of different wastes due to its capacity for microorganism retention on the support and, therefore, the hydraulic retention time can be considerably reduced. In addition, AFBRs are easy to acclimatize and can overcome influent variations or shock loads without process failure. Moreover, the construction, operation and maintenance costs of the AFBRs are lower than those required for other high-rate reactors. The effluent of the AFBRs contains few suspended solids, eliminating the need for the separation or recycling of the solids and so the biological system recovers to the conditions present before the reactor was stopped more quickly. These characteristics make the AFBR extremely useful for the treatment of high and medium strength wastewaters (Zinatizadeh et al., 2007; Rajakumar and Meenambal, 2008). Different authors have studied the application of anaerobic fixed bed reactors for treating cattle and dairy wastes (Lo et al., 1983; van den Berg and Kennedy, 1983; Lo, 1984a and

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b; Sánchez and Rodríguez, 1992; Venkataraman *et al.*, 1992; Wilkie, 2005; Giesy *et al.*, 2005). This type of wastewater is characterized by its high organic and polluting load (Rico *et al.*, 2009; Mobarak-Qamsari *et al.*, 2012). Good results were achieved in all cases with COD removal efficiencies in the range of 50% to 80% and biogas production rates in the range of 0.8 to 4.7 volumes per volume of reactor per day at hydraulic retention times in the range of 5-1 days.

One of the most important aspects in the design of an anaerobic fixed bed reactor is selecting an adequate support material. A variety of natural materials such as smooth quartzite pebbles, shells, granite stones, cinder, volcanic stones, zeolite, wooden blocks, brick ballast and synthetic materials such as polyvinyl-chloride sheets, needle-punched polyester, glass, raschig rings, waste tire rubber and other materials have been used for the attachment and growth of anaerobic biomass (Sánchez and Roque, 1987; Borja et al., 1996; Tay et al., 1996; Gourari and Achkari-Begdouri, 1997; Reyes et al., 1999; Show and Tay, 1999; Jawed and Tare, 2000; Picanco et al., 2001; Nikolaeva et al., 2002; Ahn and Foster, 2002; Mihaud at al., 2002; Melidis et al., 2003; Rovirosa et al., 2004; Yang et al., 2004). In all cases, anaerobic fixed bed reactors containing high porosity supports have shown better efficiencies than reactors filled with non-porous supports. It has also been reported that the organic matter removal efficiency in fixed-bed reactors is directly related to the characteristics of the support materials used for the immobilization of anaerobes. Waste tire rubber has also been used as support media in the treatment of piggery waste, distillery waste and sewage with considerable removal efficiencies, showing the suitability of this material as a support medium in anaerobic fixed bed reactors (Borja at al., 1996; Reyes et al., 1999; Nikolaeva et al., 2002). Natural zeolite has also been used for anaerobic microorganism immobilization in anaerobic digesters treating cattle and piggery waste, as well as for reducing the inhibitory effect of ammonia produced during the anaerobic decomposition of proteins, amino acids and urea (Borja et al., 1993; 1994; 1995; Milán et al., 2001 and 2003; Montalvo et al., 2005; Tada et al., 2005; Montalvo et al., 2006). The experimental results obtained demonstrate that the addition of zeolite enhanced the anaerobic digestion of cattle and piggery waste by increasing the kinetic constant values with respect to the values obtained in the anaerobic digestion of this type of waste without the addition of the support. Based on the literature reviewed, the subject of this paper dealt with the use of a new hybrid material composed of waste tire rubber and zeolite as microorganism support in the anaerobic digestion of dairy wastewater with experiments from laboratory to pilot-scale. The experimental results obtained in the pilot-scale anaerobic reactor installed in Costa Rica were also reported and discussed. All experiments were conducted at ambient temperature.

MATERIALS & METHODS

The wastewater used in the study was collected from a dairy unit located in "Cot de Oreamuno", Cartago, near the laboratory of Industrial Materials, Department of Physics, National University, and Heredia, Costa Rica. The cows were fed with a mixture of grass, banana peels and barley. The waste was collected during the washing period of the dairy floors. The characteristics and features of the wastewater used are presented in Table 1. The reactor consisted of an acrylic cylinder 48 cm high and 36 cm in diameter. The total volume of the reactor was 26 litres. The volume occupied by the support was 18 liters while the free volume was 16 liters. The reactor operated in up flow mode and the wastewater was added in a semi continuous regime. The reactor operated at ambient temperature and no effluent recirculation was applied. The raw waste was introduced into the reactor through a glass cylinder with a conical bottom and a valve at the inlet. Two pipes 2.5 cm in diameter were used for influent feeding and effluent extraction. A pipe for the outlet of biogas was situated at the top of the reactor. The biogas produced was collected in a gas holder floating in a solution of 10% (v/v) NaOH to remove CO₂, which allowed the measurement of methane gas production. Fig. 1 shows a schematic diagram of the laboratory-scale reactor used. The reactor was packed with one hundred pieces of a novel hybrid material composed basically of tire rubber and zeolite. Each piece of tire rubber, used for the preparation of the hybrid support, was 12.0 cm long, 7.1 cm wide and 0.3 cm deep. The surface contact area of each piece of support was 97.1 cm², therefore the total contact area

 Table 1. Characteristics and features of the dairy wastewater used in this study

Parameter	Waste	
	Average value	Standard deviation
COD (g/L)	39	7
BOD (g/L)	17	4
TS (g/L)	48	8
VS (g/L)	38	8
TVFA (mequiv./L)	69	10
Alkalinity (mequiv./L)	203	25
ρ (TVFA/Alk.)	0.34	0.05
pН	7.2	0.3

* TS: total solids; VS: volatile solids; TVFA: total volatile fatty acids; ñ: TVFA/Alkalinity ratio (mequiv. acetic acid/mequiv. CaCO₂)



Fig. 1. Schematic diagram of the laboratory-scale anaerobic fixed bed reactor

Table 2. Characteristics of the zeolite used in the laboratory-scale and pilot-scale experiments

Component	Composition % (W/W)
SiO ₂	66.6
Al_2O_3	12.2
Fe_2O_3	2.1
CaO	3.2
MgO	0.8
Na ₂ O	1.5
K ₂ O	1.2
IW*	11.0
Total	98.6

*IW: ignition wastes

was 9,710 cm². The average volume of each piece of support was 22.1 cm³ and the apparent density was 1.3 g/cm³. Each piece of tire rubber was fixed with and joined by 5.3 g of natural zeolite 1mm in size by means of inert silicone, amounting to a total of 530 g of zeolite. Table 2 shows the characteristics of the zeolite fixed and stuck to the support media (tire rubber).

The laboratory-scale reactor was inoculated with 1.5 liters (9.4 % of the effective volume of the reactor) of well digested anaerobic sludge obtained from a laboratory-scale plug-flow anaerobic digester working at 60 days of hydraulic retention time. The characteristics of the inoculum used were: total solids (TS), 6 %; volatile solids (VS), 65 % of the TS; and a pH of 7.8.

Once the inoculums was added to the reactor, the reactor volume was completed with tap water and the feeding was started up with the addition of progressive

volumes of diluted waste of 0.5 L/d for the first 60 days, 0.8 L/d for the next 40 days, 1.6 L/d for the next 30 days and, finally, 3.0 L/d for the last 30 days of this period. The daily volume added was changed when the variation of effluent characteristics was at a minimum and daily methane production was practically constant according to the recommendations in the literature (Michaud *el al.*, 2002).

Five experimental runs corresponding to five different values of hydraulic retention time (*HRT*) were carried out in order to evaluate the effect of this parameter on the process performance. The values of *HRT* assessed were: 5.5, 4, 3, 2 and 1 day corresponding to runs 1, 2, 3, 4 and 5, respectively. With these values of *HRT*, the reactor operated at volumetric organic loading rates (*OLR*) of 4.4, 6.0, 8.0, 12.0 and 24.0 g COD/ L/d, respectively. The reactor was fed at semi continuous mode by adding the corresponding volumes once a day. This feeding procedure was

selected taking into account the results achieved in recent studies of anaerobic digestion of dairy waste using UASB reactors, which demonstrated that reactors operating at intermittent mode improved the efficiency of the biological conversion by reducing the accumulation of organic matter in the sludge bed through a higher methanization with a high feed less period (Coelho et al., 2007). The volumes of waste added to the reactor were: 2.9, 4, 5.3, 8 and 16 L/d for runs 1, 2, 3, 4 and 5, respectively. Steady-state conditions were assumed when the methane yield (Y_{μ}) remained practically constant through time. Once the $Y_{\rm M}$ remained constant, sampling of influent and effluent was initiated. Each experimental run had duration of 4-5 times the corresponding HRT. During the experiments, the temperature in the reactor varied in the range of 22-26 °C (ambient temperature).

During the study, samples of influents and effluents of the reactor were analyzed three times a week. The samples were analyzed in triplicate and the following parameters determined: chemical oxygen demand (COD), biochemical oxygen demand (BOD_s), total solids (TS), volatile solids (VS), alkalinity (Alk.), total volatile fatty acids (TVFA) and pH. These determinations were carried out according to the Standard Methods for the Examination of Water and Wastewater (APHA, 1997). Methane gas production was determined every day by measuring the gas volume collected in the gas holder. The volume collected in the gas holder was considered to be made up mainly of methane, as CO₂ was removed by the solution of 10% (v/v) NaOH. The methane gas volumes were corrected at standard temperature and pressure (STP) conditions.

RESULTS & DISCUSSION

Table 3 summarizes the experimental results obtained for the different HRTs assayed in the laboratoryscale reactor. As can be seen, an increase in the HRT brought about an improvement in the effluent quality due to the decrease in the COD, BOD₅, TS and VS concentrations. Hence, the process performance appears to be directly related to the HRT. An increase in the HRT would result in a decrease in the wastewater linear velocity through the support, improving the mass transfer from the liquid to the biofilm and, therefore, favoring process performance (Smith et al., 1996; Elmitwalli et al., 2000). The concentration of TVFA in the effluent at a HRT of 1 day was higher in comparison with that observed in the influent because of the hydrolysis of complex organic matter. At HRTs higher than 1 day, the effluent TVFA concentration decreased, achieving a minimum value at a HRT of 5.5 days, due to the use of volatile organic acids for methane production. Because of organic matter decomposition in anaerobic conditions, the effluent alkalinity increased as the HRT increased. Given that the buffering capacity of the experimental systems was found to be at favorable levels with excessive alkalinity present at all *HRTs*, the stability of the process and efficiency of methanogenesis were hardly affected. The experimental data obtained in this work showed that a level of alkalinity in the range of 204-228 meq CaCO₃/L was sufficient to prevent the pH from dropping to below 6.8 at *HRTs* in the range of 1.0-5.5 days. In addition, the pH values in the reactor were always higher than 6.8, showing a slight increase with increased *HRTs*. These pH values were always higher than the lower limit of the optimum pH range which has been reported for anaerobic processes (Fannin, 1987).

The TVFA/alkalinity ratio (ρ) can also be used as a measure of process stability and as an index of the acid base equilibrium of the process (Fannin, 1987). When this ratio is less than 0.4-0.5 the process is considered to be operating favorably, without risk of acidification. As can be seen in Table 3, the values of this ratio remained constantly lower than 0.4 in all runs for the reactor showing that process failure did not occur, in spite of the short *HRTs* used in this study. In addition, the value of ρ also decreased as the *HRT* rose showing that the stability of the anaerobic process tended to increase when the *HRT* increased.

Taking the experimental values of influent and effluent COD into account, COD removal efficiency was calculated as follows:

$$E = [(COD_I - COD_E)/COD_I] \times 100$$
(1)

where *E* is COD removal efficiency (%), COD_I is the influent COD and COD_F is the effluent COD.

Table 4 shows a summary of the efficiencies calculated on the basis of COD values. The efficiency in the reactor increased as the *HRT* increased, achieving a maximum value (82.1%) at a *HRT* of 5.5 days. The COD removal efficiency was a non-linear function of the *HRT*. Therefore, the increase in the efficiency (*E*) diminished progressively with the increase in the *HRT*. The experimental value pairs of COD removal efficiency (*E*) and *HRT* can be adjusted to the following exponential rise to maximum function:

$$E = E_{max} \left[1 - \exp(-k (HRT)) \right]$$
⁽²⁾

where *E* and E_{max} are the COD removal efficiency (%) at a specific *HRT* and the maximum removal efficiency (%) at an infinite *HRT* respectively, and *k* is an overall parameter or condition-specific coefficient. By fitting the above mentioned (*E*, *HRT*) pair values to the proposed equation by non-linear regression using the SigmaPlot 11.0 software, the following values for the parameters of equation (2) with their 95% confidence limits were obtained: $k = 0.32 \pm 0.04$ days⁻¹ and $E_{max} = 90 \pm 4$ %. The last value indicates that the dairy wastewater contains a fraction of about 10% of recalcitrant organic

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			HRT (d)		
Parameter	1	2	3	4	5.5
COD (g/L)	28 ± 5	21 ± 4	15 ± 3	9 ± 2	7 ± 2
$BOD_5 (g/L)$	7 ± 2	6 ± 2	5 ± 1	3 ± 1	2 ± 1
TS (g/L)	41±7	33 ± 6	26 ± 5	21 ± 5	15 ± 3
VS (g/L)	29 ± 6	22 ± 5	17 ± 4	13 ± 4	8 ± 3
TVFA (mequiv/L)	73 ± 10	55 ± 7	47 ± 6	42 ± 6	40 ± 5
Alkalinity (mequiv./L)	206 ± 25	210 ± 24	216 ± 24	223 ± 24	228 ± 25
ρ*	0.35 ± 0.04	0.26 ± 0.03	0.22 ± 0.03	0.19 ± 0.03	0.18 ± 0.02
pH	6.8 ± 0.4	6.9 ± 0.5	6.9 ± 0.5	6.9 ± 0.5	6.9 ± 0.6

 Table 3. Characteristics of the effluents obtained (mean values ± standard deviations) for the different HRTs

 used in the laboratory-scale reactor

 $*\rho = (TVFA/Alkalinity)$

Table 4. Effect of *OLR* and *HRT* on the COD removal efficiency (*E*, %) in the laboratory-scale AFBR

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OLR (kg COD/m ³ d)	HRT(d)	E (%)
4.4	5.5	82.1
6.0	4	76.9
8.0	3	61.5
12.0	2	46.2
24.0	1	28.2

matter, which is not degradable by anaerobic digestion. The low values of the confidence limits of the two parameters and of the standard error of estimate (2.31) as well as the high value of the determination coefficient of the regression ($R^2 = 0.9956$) demonstrate the good fit of the experimental values to the proposed model. This model would allow predicting the efficiency values for a specific theoretical *HRT* value.

On the other hand, the COD removal efficiency achieved in the reactor operating at a HRT of 5.5 days (82.1%) was lower than that reported in attached-film reactors with limestone gravel and polyester as supports (94%) when treating this same waste. However, this reactor operated at a HRT of 33 days and mesophilic temperature (35 °C) (Vartak et al., 1997), a HRT and operating temperature much higher than those used in the present work. On the other hand, the COD removal percentages of the present study at 4.0 and 5.5 days of HRT (76.9% and 82.1%) were higher than those achieved for an anaerobic hybrid reactor (AHR) configuration incorporating floating support media for biomass immobilization and biogas recirculation for enhanced mixing (48%-63%) operating with similar dairy waste at a HRT of 15 days (Demirer and Chen, 2005), which was much higher than those used in the study at hand.

From the data in Table 4, the methane yield values were calculated. The value of $Y_{_M}$ increased in the range of 0.07-0.18 L CH₄/g COD added when the *HRT* increased from 1.0 to 5.5 days with values of 0.10, 0.13

and 0.15 L CH₄/g COD added at *HRTs* of 2, 3 and 4 days respectively. Additionally, the methane yield coefficient obtained in the reactor at a *HRT* of 5.5 days (0.18 L CH₄/g COD added) was higher than the coefficients obtained in anaerobic digestion of dairy waste in baffled (0.109 L CH₄/g COD added) and UASB reactors (0.154 L CH₄/g COD added) operating at a *HRT* of 5 days in both cases (Chen and Shyu, 1996). Moreover, the values of methane yield obtained in the present study were of the same order of magnitude as those reported in other research works of anaerobic digestion of dairy waste using fixed bed reactors with a combination of limestone and polyester as the support material (0.18 L CH₄/g COD added), although the latter operated at a *HRT* of 33 days (Vartak *et al.*, 1997).

On the basis of the results obtained at laboratoryscale, a HRT of 3 days and OLR of 8 kg COD/m3/d can be considered as optimum for the design of a full-scale anaerobic reactor for the treatment of dairy waste. The average volume of waste to be processed in the plant considered for the design achieved a value of 2.4 m³/d with an average COD value of 39 g/L. Therefore, the average organic load used in the design was 93.6 kg COD/d. The quotient between the organic load and the organic loading rate (OLR) gives the total reactor volume. This volume was determined to be 11.7 m³, which represents the total volume of the reactor required. The empty bed volume of the reactor can be determined by the product of the HRT by the daily volume of waste to be processed (2.4 m³/d) resulting in a free volume or working volume of the reactor of 7.2 m³. The dairy farm currently has 68 milking cows and the waste was collected from the cage floor washing. The estimated quantity of manure (feces + urine) produced was 309 kg/d. The pilot plant is composed of 2 anaerobic fixed bed reactors with waste tire rubber and zeolite as support and biogas collection by 40 m³ volume plastic bag. The biogas produced during the anaerobic digestion was used to obtain the electrical energy required for cooling the milk storage tanks and sometimes for operating the milking machine.

The plant was designed in the LAMI Laboratory, Universidad Nacional de Heredia, Costa Rica. One of the anaerobic reactors consisted of a PVC cylindrical tank with 10.1 m3 total volume, 2.32 m in diameter and 3.34 m high. The other had a total volume of 1.6 m³ with a diameter of 1.13 m and was 1.63 m high. The material used in the smaller reactor construction was polyester reinforced with fiberglass, pineapple waste and banana tree waste. Both reactors operated in parallel obtaining very similar results in their operation. The main objective of the use of the smaller reactor was to check the resilience of the waste material used for its construction. Given the similarity in the results obtained, the suitability of the afore-mentioned material for constructing the smaller pilot-scale digester was demonstrated. The support media remained submerged in the liquor of both reactors by placing a structure to prevent the support flotation. Each reactor was packed with the previously mentioned hybrid material (waste tire rubber and zeolite) as microorganism immobilization supports in the same way as in the lab-scale reactor. Both reactors were inoculated with methanogenically active biomass from a plug flow anaerobic reactor located very close to the dairy farm. In order to prevent the compaction of the floating mass in the reactors and the clogging of the biogas output, a manual agitator was installed in each reactor and mixing was carried out twice a day.

The digesters operated in up-flow mode and the raw waste was fed once a day at the bottom of the reactors. Two pipes 10 cm in interior diameter were used for influent feeding and effluent extraction. A pipe of 5 cm diameter was situated at the top of each reactor for biogas outlet. The biogas produced was collected in a polyethylene bag with a capacity of 40 m³. The reactors were inoculated by the addition of 10 % of the operational volume of anaerobic sludge obtained from a plug flow digester in operation with similar characteristics as the inoculum used in the laboratory-scale reactor.

The results obtained during the six months of plant operation are summarized in Table 5. This table summarizes the average values obtained in the operation of both pilot-scale reactors, the standard deviations of the mean values being lower than 5% in all cases. As can be seen, the characteristics of the effluent and the methane yield were very similar to those obtained at laboratory-scale. The total average biogas production in the plant was in the range of 16.4-19.1 m³/d with an average methane concentration of 61%.

As can be observed in Table 5, average COD and TS removal efficiencies of 63.6% and 66.0% respectively were achieved in the pilot-scale anaerobic plant, operating at a low *HRT* (3 d) and high *OLR* (8 kg

Table 5.	Results obtained in the pilot-scale
	anaerobic reactors

Parameter	Effluent	Removal Efficiency(%)
COD (g/L)	14.2	63.6
TS (g/L)	16.3	66.0
pН	7.0	
Alkalinity	213	
(mequiv./L)		
Methane Yield	0.11	
(m ³ CH ₄ /kg COD		
added)		

*Values are the averages obtained in the operation of both pilot-scale anaerobic reactors taking into account that the results obtained in both cases were virtually the same (standard deviations of the mean values were lower than 5% in all cases).

 COD/m^3d). The average pH value of the effluent (7.0) was within the optimum pH range for the adequate growth of anaerobic microorganisms (Fannin, 1987). A high buffering capacity as a consequence of the high alkalinity value achieved was observed in the system, which meant that a decrease in the pH value was avoided. In comparison with other reported research works related to the anaerobic digestion of dairy wastes at full-scale, the TS removal efficiency reached in the present work (66%) was higher than that obtained in a full-scale modified plug-flow digester (Martin and Ross, 2007) after 12 months of operation (39.6%) and that obtained in a full-scale thermophilic anaerobic digester (49%) coupled with a sintered titanium cross flow ultra filter (TADU) for the separation of solids, although in the latter the HRT was 23 days (Zitomer et al., 2005). The application of a proper process thermophilic temperature (55 °C) instead of a "reduced" thermophilic range (47 °C), which is often applied in European anaerobic plants, together with the addition of certain co-substrates (agro-wastes) lead to higher biogas yield values (0.45-0.62 m³/kg VS) than those obtained in the digestion of single dairy wastes (Cavinato et al., 2010). An approximate 60% enhancement in methane yield was also observed in the co-digestion of dairy wastes with industrial confectionery wastes in a full-scale farm digester (Kaparaja et al., 2002).

The biogas produced in the plant of the present work was previously purified by means of a column packed with granular activated carbon and the concentration of hydrogen sulphide was reduced by 90 %. The purified biogas was used in an electrical power plant Generator (GENERAC 16kWat Model 005255) with 16 kW power generation capacities. This power plant produced 1.2 kW/m³ of biogas. The electricity generated using the digester biogas as fuel should be enough to double the consumption of the milking machines in a week. The cost of electricity for the dairy is 0.17 /kWh, so the annual saving using biogas would be \$1,003 while the total cost of the pilot-scale plant was \$8,000.

CONCLUSION

The experimental laboratory-scale and pilotscale results obtained demonstrate that an up flow anaerobic fixed bed reactor packed with a hybrid material composed of waste tyre rubber and zeolite was capable of operating efficiently at ambient temperatures (22-26 °C) using low values of *HRT* (3 d) and high values of *OLR* (8 kg COD/m³ d) for the treatment of dairy waste. Therefore, the volume of the reactor could be reduced five times as compared to conventional digesters without affecting the organic matter removal efficiency. In addition, the results obtained at pilot-scale were comparable to those obtained at laboratory-scale, indicating the success of the scale-up procedure.

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