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Short Communication

# Treatment of screened dairy manure by upflow anaerobic fixed bed reactors packed with waste tyre rubber and a combination of waste tyre rubber and zeolite: Effect of the hydraulic retention time

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**Abstract**

Two laboratory-scale anaerobic fixed bed reactors were evaluated while treating dairy manure at upflow mode and semicontinuous feeding. One reactor was packed with a combination of waste tyre rubber and zeolite (R1) while the other had only waste tyre rubber as a microorganism immobilization support (R2). Effluent quality improved when the hydraulic retention time (HRT) increased from 1.0 to 5.5 days. Higher COD, $BOD_5$, total and volatile solids removal efficiencies were always achieved in the reactor R1. No clogging was observed during the operation period. Methane yield was also a function of the HRT and of the type of support used, and was 12.5% and 40% higher in reactor R1 than in R2 for HRTs of 5.5 and 1.0 days, respectively. The results obtained demonstrated that this type of reactor is capable of operating with dairy manure at a HRT 5 times lower than that used in a conventional reactor.


*Keywords:* Upflow anaerobic fixed bed reactor; Waste tyre rubber; Zeolite; Hydraulic retention time; Removal efficiency

## 1. Introduction

Anaerobic digestion of animal waste is a valuable alternative for reducing environmental problems derived from intensive animal breeding and for obtaining biogas, a low cost source of energy (Ong et al., 2000; Hill et al., 2001; Keshtkar et al., 2001; Ihara et al., 2006). Interest in anaerobic digestion of dairy manure has increased in the last few years due to the reduction in odours of stored and land applied manure (Güngör and Karthikeyan, 2008).

During the digestion process, the decrease in concentrations of manure volatile acids reduces odour. In addition, the byproduct of the process, the biogas, constitutes a source of energy that can be collected and used. Dairy wastes are usually treated by anaerobic digestion in conventional digesters with hydraulic retention times higher than 15 days (Keshtkar et al., 2003; Chachkhiani et al., 2004; Giesy et al., 2005). In conventional digesters like batch, complete mixed and plug-flow reactors, the hydraulic retention times (HRTs) approach the values of the retention times of microorganisms or solid retention times (SRTs). Therefore, in order to prevent the washout of microorganisms, high volumes of reactor are required with respect to the volume of waste to be processed. With the aim of reducing the reactor volumes, different alternatives for microorganism retention such as sludge recycling or immobilization, 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 types of waste in the past few years because of its capacity for retaining the microorganisms attached to the support. Therefore, the hydraulic retention time can be considerably reduced. In addition, AFBRs are easy to acclimatize and may

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overcome influent variations without process failure. Different authors have studied the application of anaerobic fixed bed reactors for treating cattle and dairy waste (Wilkie, 2005; Giesy et al., 2005). 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 anaerobic fixed bed reactor design is the selection of 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 like polyvinyl-chloride sheets, needle-punched polyester, glass, rasching rings, tyre rubber and other materials have been used for the attachment and growth of anaerobic biomass (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 Forster, 2002; Michaud et al., 2002; Melidis et al., 2003; Rovirosa et al., 2004; Yang et al., 2004). In all cases, anaerobic fixed bed reactors containing supports with high porosity 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 tyre 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 (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 to reduce the inhibitory effect of ammonia produced during the anaerobic decomposition of proteins, aminoacids and urea (Milán et al., 2001, 2003; Montalvo et al., 2005, 2006; Tada et al., 2005). The experimental results obtained demonstrated 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 anaerobic digestion of these types of waste without the addition of zeolite.

Based on the literature, the present paper dealt with the utilization of waste tyre rubber with and without zeolite as support medium in upflow anaerobic fixed bed reactors when treating dairy manure. The effect of HRT on the process stability, organic matter (COD, $BOD_5$, TS and VS) removal efficiencies and methane yield was also assessed.

## 2. Methods

### 2.1. Characteristics of the waste used in the experiments

The waste used in the experiments was obtained from a dairy unit situated near the laboratory of Industrial Materials, Department of Physics, National University, Heredia, Costa Rica, by mixing the raw manure with an equal volume of tap water to simulate the conditions of the dairy effluents. The characteristics and features of the raw manure and the waste obtained after dilution and used in the experiments are summarized in Table 1. The cows were fed with a mixture of grass, banana peels and barley.

### 2.2. Characteristics of the laboratory-scale anaerobic fixed bed reactors

Two laboratory-scale anaerobic fixed bed reactors (AFBR) were used in the experiments. The reactors consisted of acrylic cylinders 48 cm high and 36 cm in diameter. The total volume of the reactors was 26 l. The support media had an apparent volume of 18 l while the free volume of each reactor was 16 l. Therefore, the ratio of free to apparent volume was 0.89. The reactors operated in upflow mode and the waste was intermittently fed in a semicontinuous regime. The raw waste was introduced into the reactors through a glass cylinder with a conical bottom and a valve at the inlet to the reactors. Two pipes of 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 each reactor. The biogas produced was collected in gas holders floating in solutions of 10% (v/v) NaOH to remove $CO_2$, which allowed the measurement of methane gas production.

### 2.3. Characteristics of the support media

Both reactors were packed with one hundred pieces of waste tyre rubber. Each piece of support is 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 $cm^2$, hence the total contact area was 9710 $cm^2$. The average volume of each piece of support was 22.1 $cm^3$ and the apparent density was 1.3 g/$cm^3$. The reactor named R1 received the above-mentioned pieces of tyre rubber, but with 5.3 g of natural zeolite 1 mm in size fixed to each piece of tyre rubber by means of inert silicone for a total amount of 530 g of zeolite. The characteristics of the zeolite fixed to the support medium were:

Table 1
Characteristics and features of the fresh manure and waste used in the experiments

| Parameter | Fresh manure | | Waste | |
|---|---|---|---|---|
| | Average value | Standard deviation | Average value | Standard deviation |
| COD (g/l) | 77 | 12 | 39 | 7 |
| $BOD_5$ (g/l) | 35 | 7 | 17 | 4 |
| TS (g/l) | 95 | 17 | 48 | 8 |
| VS (g/l) | 75 | 15 | 38 | 8 |
| TVFA (meq/l) | 131 | 28 | 69 | 10 |
| Alkalinity (meq/l) | 390 | 46 | 203 | 25 |
| ρ (TVFA/Alk.) | – | – | 0.34 | 0.05 |
| pH | 7..3 | 0.4 | 7.2 | 0.3 |

$SiO_2$, 66.6%; $Al_2O_3$, 12.2%; $Fe_2O_3$, 2.1%; CaO, 3.2%; MgO, 0.8%; $Na_2O$, 1.5%; $K_2O$, 1.2%; and ignition wastes, 11.0%. The reactor named R2 was packed with one hundred pieces of tyre rubber only, as was previously described.

### 2.4. Characteristics of the inoculum used in the experiment

Each reactor was inoculated with 1.5 l (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 pH: 7.8.

### 2.5. Procedure for reactor acclimatization

Once the inoculum was added to each reactor, the reactor volume was completed with tap water and the feeding was started up with the addition of diluted manure, the characteristics of which are given in Table 1. The waste was added at increasing flow-rates, starting with a flow-rate 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 increased when the variation of effluent characteristics was at a minimum and daily methane production was practically constant according to literature recommendations (Michaud et al., 2002).

### 2.6. Experimental procedure

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 in both reactors. 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 reactors 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 reactors were fed at semicontinuous mode 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 operated in an 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 feedless period (Coelho et al., 2007). Therefore, the volumes of waste added to the reactors were: 2.9, 4, 5.3, 8 and 16 l/day for runs 1, 2, 3, 4 and 5, respectively. Steady-state conditions were assumed when the methane yield ($Y_M$) remained practically constant through time. Once the $Y_M$ remained constant, sampling of influent and effluent was initiated. Each experimental run had a duration of 4–5 times the corresponding HRT. During the experiments, the temperature in the reactors varied in the range of 22–26 °C.

### 2.7. Sampling and analysis

During the steady-state conditions, samples of influent and effluents of the reactors were analysed three times per week. The samples were analysed in triplicate and the following parameters were determined: chemical oxygen demand (COD), biochemical oxygen demand ($BOD_5$), total solids (TS), volatile solids (VS), alkalinity (Alk.), total volatile fatty acids (TVFA) and pH. These determinations were carried out according to 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 $CO_2$ was removed by the solution of 10% (v/v) NaOH. The methane gas volumes were corrected at standard temperature and pressure conditions.

## 3. Results and discussion

### 3.1. Effect of the HRT on the process stability

Table 2 shows a summary of the experimental results obtained at different values of HRT in the two reactors studied (R1 and R2). As can be seen, an increase in the HRT brought about an improvement in the effluent quality due to the decrease in the COD, $BOD_5$, TS and VS

Table 2
Characteristics of the effluents obtained for the different HRTs used in the two reactors studied

| | R1 | | | | | R2 | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | HRT (d) | | | | | HRT (d) | | | | |
| Parameter | 1 | 2 | 3 | 4 | 5.5 | 1 | 2 | 3 | 4 | 5.5 |
| COD (g/l) | 28 ± 5 | 21 ± 4 | 15 ± 3 | 9 ± 2 | 7 ± 2 | 30 ± 6 | 23 ± 5 | 17 ± 5 | 11 ± 4 | 9 ± 3 |
| $BOD_5$ (g/l) | 7 ± 2 | 6 ± 2 | 5 ± 1 | 3 ± 1 | 2 ± 1 | 8 ± 2 | 7 ± 2 | 6 ± 2 | 5 ± 1 | 3 ± 1 |
| TS (g/l) | 41 ± 7 | 33 ± 6 | 26 ± 5 | 21 ± 5 | 15 ± 3 | 44 ± 8 | 37 ± 8 | 31 ± 7 | 25 ± 5 | 19 ± 5 |
| VS (g/l) | 29 ± 6 | 22 ± 5 | 17 ± 4 | 13 ± 4 | 8 ± 3 | 31 ± 7 | 24 ± 6 | 19 ± 5 | 15 ± 4 | 10 ± 4 |
| TVFA (meq/l) | 73 ± 10 | 55 ± 7 | 47 ± 6 | 42 ± 6 | 40 ± 5 | 77 ± 10 | 56 ± 9 | 54 ± 9 | 45 ± 8 | 43 ± 8 |
| Alkalinity (meq/l) | 206 ± 25 | 210 ± 24 | 216 ± 24 | 223 ± 24 | 228 ± 25 | 204 ± 26 | 208 ± 25 | 210 ± 25 | 212 ± 26 | 218 ± 26 |
| ρ (TVFA/Alkalinity) | 0.35 ± 0.04 | 0.26 ± 0.03 | 0.22 ± 0.03 | 0.19 ± 0.03 | 0.18 ± 0.02 | 0.38 ± 0.04 | 0.27 ± 0.04 | 0.26 ± 0.03 | 0.21 ± 0.03 | 0.20 ± 0.02 |
| pH | 6.6 ± 0.4 | 6.7 ± 0.5 | 6.7 ± 0.5 | 6.7 ± 0.5 | 6.7 ± 0.6 | 6.5 ± 0.5 | 6.5 ± 0.5 | 6.5 ± 0.5 | 6.6 ± 05 | 6.6 ± 05 |

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, favouring the process performance (Elmitvalli 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 higher HRTs, the effluent TVFA concentration decreased, achieving a minimum value at a HRT of 5.5 days due to the utilization of the volatile organic acids for methane production. Because of the 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 favourable 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 show that a level of alkalinity in the range of 204–228 meq/l as $CaCO_3$ is sufficient to prevent the pH from dropping to below 6.5 at HRTs in the range of 1.0–5.5 days. In addition, the pH values in both reactors were always higher than 6.5, showing a slight increase with increased HRTs. These pH values were higher than the lower limit of the optimum pH range that has been reported for anaerobic processes (Fannin, 1987).

The TVFA/alkalinity ratio ($\rho$) can also be used as a measure of process stability and as an index of 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 favourably without the risk of acidification. As can be seen in Table 2, the values of this ratio remained constantly lower than 0.5 in all runs for both reactors 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. The comparison of effluent characteristics of R1 and R2 showed that the reactor that used zeolite and tyre rubber as a microorganism support (R1) produced higher quality effluents when compared with the reactor that only used tyre rubber as support (R2). This corroborated the theory that the combination of zeolite and tyre rubber as a bacterial immobilization support enhances the course of the stabilization process.

### 3.2. Effect of the HRT on the process efficiency

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

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

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

Fig. 1 shows the variation of the COD removal efficiency with the HRT for reactors R1 and R2. The efficiency in both reactors increased as the HRT increased, achieving the maximum value at a HRT of 5.5 days. In reactor R1, efficiencies were always higher than in R2. In both cases, the COD removal efficiency was a non-linear function of the HRT. Therefore, the increases of the efficiency ($E$) diminished progressively with the increase in the HRT. For example, the COD removal efficiency rose from 28.2% to 46.2% for reactor R1 and from 23.1% to 41.0% for R2 when the HRT increased from 1 to 2 days, while the values of $E$ only increased from 76.9% to 82.1% and from 71.8% to 76.9% for R1 and R2, respectively, when the HRT increased from 4 to 5.5 days. Therefore, an increase of 37.5% in the reactor volume may be required to increase the efficiency for both reactors by only 5%.

On the other hand, the COD removal efficiency achieved in reactor R1 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, these operated at a HRT of 33 days and mesophilic temperature (35 °C) (Vartak et al., 1997). On the contrary, the COD removal percentages of the present study (R1) 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

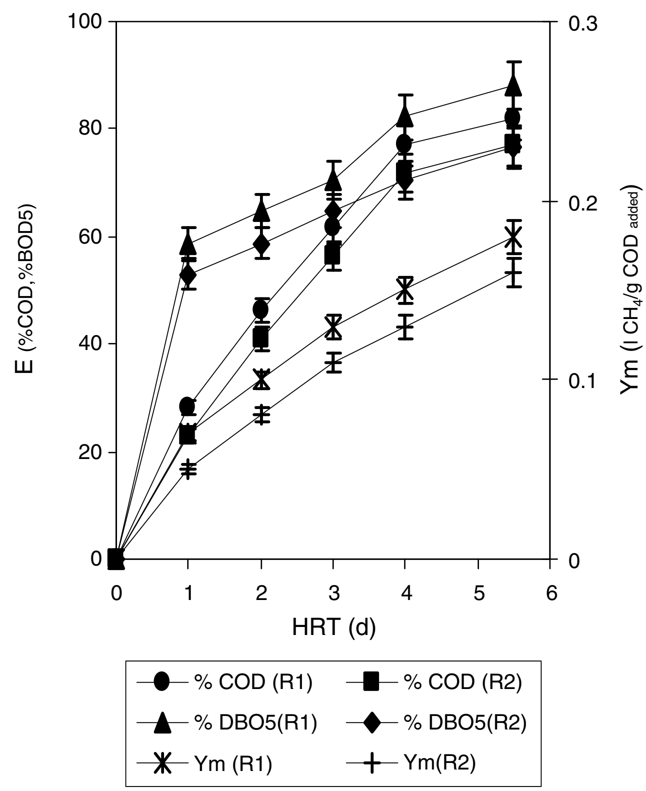


Fig. 1. Effect of the HRT on the COD, $BOD_5$ removal efficiencies and methane yield coefficients, $Y_M$, for both reactors.

(48–63%) operating with similar dairy waste at a HRT of 15 days (Demirer and Chen, 2005), with the HRT value much higher than that used in our study. In addition, the COD removal efficiency values obtained in the present work were also higher than those obtained in anaerobic batch reactor experiments treating this waste (37.9–50%) at initial COD concentrations of 53.5 g/l (Güngör and Demirer, 2004).

The $BOD_5$ removal efficiency was also calculated using the same type of equation as used for COD. Fig. 1 shows the variation of the $BOD_5$ removal efficiency with the HRT. As can be seen, the removal efficiency in $BOD_5$ increased from 58.8% to 88.2% for R1 and from 52.9% to 76.5% for R2 when the HRTs rose from 1 to 5.5 days. These removal efficiencies were also higher than those obtained in the above-mentioned AHR configuration (64%–78%) while treating dairy manure at a HRT of 15 days (Demirer and Chen, 2005). The results obtained in the present study show the high performance of the anaerobic fixed bed reactors treating dairy waste in spite of the low values of HRT (1–5.5 days) used and high OLR (24.0–4.4 g COD/l d) added to the reactors. Therefore, these results again confirm the advantages of the AFBRs in comparison with conventional digesters for which HRTs of around 20 days are needed to achieve similar removal efficiencies (Powers et al., 1997; Wilkie, 2005). Additionally, the best results obtained in reactor R1 corroborate the theory that the combination of tyre rubber and zeolite favour the anaerobic degradation process.

Moreover, in reactor R1, the TS and VS removal efficiencies increased from 14.6% to 68.8% and from 26.3% to 78.9%, respectively when the HRTs rose from 1 to 5.5 days. In the case of R2, the TS and VS removal efficiencies were somewhat lower, ranging from 8.3% to 47.9% and from 18.4% to 60.5% for the same range of HRT (1 to 5.5 days). A previous study (Vartak et al., 1997) reported VS removal efficiencies of up to 91% in upflow attached-film reactors with a combination of limestone and polyester as support media treating diluted dairy wastewaters but operating at a HRT of 33 days, a much higher value than those used in our study. Lower VS removal efficiencies (66%) have been achieved in baffled reactors operating with very diluted dairy wastewater (1023 mg COD/l) at a HRT of 5 days (Chen and Shyu, 1996). In addition, the results obtained demonstrate that the AFBR in upflow mode may operate with high removal efficiencies at HRTs significantly lower than those used in a conventional digester (Powers et al., 1997). With the reactors in operation for over six months, clogging of the support media was not evident in spite of the high concentration of solids contained in the influent.

### 3.3. Effect of the HRT on the methane yield

Fig. 1 shows the effect of the HRT on the methane yield ($Y_M$) for reactors R1 and R2. The value of $Y_M$ increased in the ranges of 0.07–0.18 l $CH_4$/g COD added for R1 and of 0.05–0.16 l $CH_4$/g COD for R2 when the HRT increased from 1.0 to 5.5 days. As can be seen, reactor R1 always showed the highest values for all the HRTs assayed in this study. This was significant at 95% confidence level ($P < 0.05$). Therefore, as was expected, the methane yields in the reactor with the combination of zeolite and tyre rubber (R1) were higher than those obtained in the reactor with only tyre rubber as support (R2). Specifically, the methane yield was 12.5% and 40% higher in reactor R1 than in R2 for HRTs of 5.5 and 1.0 days, respectively. Additionally, the methane yield coefficient obtained in reactor R1 at a HRT of 5.5 days (0.18 l $CH_4$/g COD added) was also higher than those obtained in anaerobic digestion of dairy waste in baffled (0.109 l $CH_4$/g COD added) and UASB reactors (0.154 l $CH_4$/g COD added) operating at a HRT of 5 days in both cases (Chen and Shyu, 1996). The values of methane yield obtained in the current study were of the same magnitude as those reported in other works of anaerobic digestion of dairy waste in fixed bed reactors with a combination of limestone and polyester as the support material (0.18 l $CH_4$/g COD added) although the latter operated at a HRT of 33 days (Vartak et al., 1997). However, higher methane yield coefficients (0.26–0.31 l $CH_4$/g VS added) were reported using completely stirred tank reactors operating with diluted (5%) manure slurry at a constant HRT of 16.2 days (Karim et al., 2005). Similar methane yield coefficient values (0.20–0.41 $m^3$/kg VS) were also obtained in the anaerobic treatment of digested cow manure after separating the liquid and solid fractions of the waste (Kaparaju and Rintala, 2008).

## 4. Conclusions

The experimental results obtained demonstrate that upflow anaerobic fixed bed reactors packed with waste tyre rubber were capable of operating efficiently at low values of HRT and high values of OLR in the treatment of dairy waste. Therefore, the volume of the reactor could be reduced five times in comparison with that used in conventional digesters without affecting the organic matter removal efficiency. The reactor packed with the combination of tyre rubber and zeolite (R1) was more efficient than the reactor with only tyre rubber as support (R2), the methane yield being 12.5–40.0% higher in R1 than in R2, when both operated at HRTs in the range of 5.5–1.0 days. This demonstrates that the combination of waste tyre rubber and zeolite was capable of improving the process performance. On the basis of results obtained, a HRT of 4 days may be considered as optimum for the design of a pilot scale anaerobic reactor for the treatment of dairy waste.

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