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**Thermophilic co-digestion of cattle manure and food waste supplemented with
crude glycerin in induced bed reactor (IBR)**

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ABSTRACT

The aim of the present research work was to boost biogas production from cattle manure (CM) by adding food waste (FW) and crude glycerin (Gly) from the biodiesel industry as co-substrates. For this purpose, different quantities of FW and Gly were added to CM and co-digested in an induced bed reactor (IBR) at 55°C. Sonication pre-treatment was implemented in the CM + Gly mixture, applying 550 kJ/kg TS to enhance the biodegradability of these co-substrates. The best results were obtained with mixtures of 87/10/3 (CM/FW/Gly) (w/w) operating at an organic loading rate of 7 g COD/L.day, obtaining 92% COD removal, a specific methane yield of 640 L CH₄/kg VS and a methane production rate of 2.6 L CH₄/L.day. These results doubled those obtained in the co-digestion of CM and FW without the addition of Gly (330 L CH₄/kg VS and 1.2 L CH₄/L.day).

Keywords: cattle manure, food waste, crude glycerin, biogas, induced bed reactor.

1. INTRODUCTION

The use of agricultural materials such as manure, slurry and other animal and organic wastes for biogas production has significant environmental advantages in terms of heat and power production and the use of this gas as a biofuel. Biogas plants can contribute significantly to sustainable development in rural areas as well as providing farmers with new income opportunities (Directive 2009/28/EC). However, the low biogas yield of animal manure sometimes does not warrant the capital costs of farm-scale plants (Cavinato et al., 2010). The authors of the present study obtained specific methane yields of 148-185 L CH₄/kg Volatile Solids (VS) in the anaerobic treatment of dairy cattle manure and Chemical Oxygen Demand (COD) removal efficiencies of 69.7% (Marañón et al., 2001, Castrillón et al., 2002). These values are similar to those found by Amon et al. (2007), who reported a yield of 166 L CH₄/kg VS.

To enhance biogas production, pre-treatments (chemical, thermal, ultrasound or enzymatic) can be applied and/or the manure can be co-digested with other wastes to achieve synergistic effects that make the anaerobic digestion process profitable.

Our research group has already studied the co-digestion of mixtures of screened or ground cattle manure (CM) with raw glycerin (Gly) from biodiesel production, with and without pre-treatment by sonication, in batch reactors (Castrillón et al., 2011). The best results were obtained for sonicated mixtures of ground CM plus 6% Gly at thermophilic temperatures. In line with these results, studies in continuous operating mode were carried out in a continuously stirred tank reactor (CSTR) and an induced bed reactor (IBR), feeding the reactors with the sonicated mixture (Castrillón et al., 2013). The specific energy of sonication was quite low (550 kJ/kg total solids), obtaining the best results in the IBR when operating at an organic loading rate (OLR) of 6.4 g COD/L.day, with a specific methane yield of 590 L CH₄/kg VS and 89.6% COD removal.

Another waste currently used as a co-substrate is food waste (FW). Until recently, FW has not usually been source separated in Spain and, along with other household waste, forms part of the so-called “black bag” which is incinerated or disposed of in landfills. This waste can, however, be composted or anaerobically digested if properly separated from other waste. Even so, treating food waste (FW) alone by anaerobic digestion is somewhat of a challenge (El Mashad et al., 2008; Resch et al., 2011; Zhang et al., 2011) due to the hydrolysis rate, possibility as the result of the accumulation of volatile fatty acids and acidification of the reactor. In order to avoid failure of the digestion process, co-digestion with other wastes with sufficient buffer capacity is therefore necessary.

Banks et al. (2011) evaluated the feasibility of centralised pre-processing and pasteurisation of source separated household food waste followed by transport to farms for anaerobic co-digestion with dairy cattle slurry. The results obtained showed that the addition of FW improved energy yields per digester unit volume, with a corresponding increased potential for improving farm income by as much as 50%.

El-Mashad and Zhang (2010) studied the methane yield from CM and mixtures of 32% FW and 68% CM (based on volatile solids) at mesophilic temperatures in batch reactors. After 20 and 30 days of digestion, the methane yields of CM were 218 and 241 L CH₄/kg VS, respectively, while the methane yields of the CM + FW mixture were 251 and 282 L CH₄/kg VS, respectively.

To the best of our knowledge, little research has been carried out on the co-digestion of cattle manure and food waste supplemented with crude glycerin. According to the good results obtained in the thermophilic digestion of sonicated mixtures of CM and Gly in an IBR, the aim of the present research was to study the thermophilic anaerobic co-digestion of a previously sonicated mixture of CM and Gly, but also adding FW as another co-substrate. The reactor was operated with different mixtures of the three

wastes in order to determine the best conditions to obtain maximum biogas production and a digestate with good characteristics for its subsequent use as a fertilizer.

2. MATERIALS AND METHODS

2.1. Materials

Cattle manure was collected in 20-L plastic bottles from the cesspit of a dairy farm with 120 livestock units after stirring the contents of the cesspit and was then ground in the laboratory and stored at 4°C (for no more than 3 weeks).

The food waste was collected from a local retirement home, where the organic fraction of municipal solid waste was collected separately.

The crude glycerin was obtained from a local industrial plant which produces biodiesel from used vegetable oil. The plant, which has a production capacity of 4,000 tonnes/year, recycles and transforms used vegetable oils originating from the northwest of Spain and from the Canary Islands.

2.2. Equipment employed

The manure was ground using a domestic triturator and the food waste using a STR-2000 triturator.

The ultrasonic equipment used was a Hielscher UPS 400S (400 W, 24 kHz). A specific energy of 550 kJ/kg TS was applied to the mixture of cattle manure and glycerin.

Anaerobic co-digestion of CM supplemented with FW and Gly was carried out in an IBR reactor (Hansen and Hansen, 2005), a type of sludge bed reactor designed to treat high solid content substrates (up to 12%). The IBR used in this research was made of

PVC and presents three modifications with respect to the patent: (1) the side outlet for biogas is in the upper part of the reactor to avoid proximity of the biogas outlet to the effluent outlets (outflow and recirculation); (2) the feed was introduced through two side inlets (near the bottom), instead of one side inlet, with the aim of upgrading the mixture of substrate with the biomass inside the reactor; and (3) there are three side outlets for sampling, two of which are arranged vertically, while the third is located at the bottom of the reactor. These outlets enabling the taking of samples at different heights and the determination of the variation in solids (volatile and fixed) inside the reactor in order to know when to purge the system. The useful volume up to the triphasic separator was 19 L (Castrillón et al., 2013). The operating temperature was kept constant at $55 \pm 1^\circ\text{C}$ by means of an external water jacket.

2.3. Analytical methods

The parameters analysed to characterise the three types of waste and to monitor the performance of the reactors were: pH, total solids (TS), volatile solids (VS), chemical oxygen demand (COD), total nitrogen (TN), ammonium nitrogen ($\text{NH}_4\text{-N}$), total phosphorus (TP) and volatile fatty acids (VFA). Furthermore, the following parameters were determined in the glycerin by an external laboratory: water content, ash, methanol, glycerin, soap, glycerol ester and sodium.

Samples from the reactors (digestate) and from the biogas were taken twice a week to monitor the biodegradation process. COD was determined in accordance with Method 5220 D (closed reflux, colorimetric method) of the Standard Methods for the Examination of Water and Wastewater (APHA, 1998) using a Visible-UV Perkin Elmer Lambda 35 spectrophotometer. $\text{NH}_4\text{-N}$ was determined by titration with boric acid after distillation using a Foss Tecator Kjeltec 2200 Auto Distillation system. TN and TP were

determined by ion chromatography (861 Advanced Compact IC 2.861.0010) after their transformation into nitrates and phosphates, respectively, by digestion under pressure with H₂O₂ and formic acid in a microwave oven (Milestone Ethos 1 Advanced Microwave Digestion Labstation). Volatile fatty acids (VFA) were determined by gas chromatography using a Perkin Elmer AutoSystem XL system, equipped with a FID detector. The volume of gas produced was measured daily using a HI-TEC F101D thermal effect mass gas flow apparatus equipped with an electronic totalizer. All gas volumes reported are corrected to standard temperature and pressure (0°C, 101.3 kPa). The methane and carbon dioxide contents of the biogas were determined on an Agilent gas chromatograph using a TCD detector and a Porapack N packed column plus a molecular sieve, employing the following temperature ramp: starting temperature 35°C (1.5 min), increasing up to 55°C at a rate of 1.5°C/minute.

2.4 Experimental methods

Bearing in mind the results obtained in previous studies (Castrillón et al., 2011; Castrillón et al., 2013), co-digestion of mixtures of ground cattle manure, crude glycerin and ground food waste was carried out at 55°C. The analysed mixtures, expressed as CM/FW/Gly in mass fraction, were 94/2/4, 87/10/3, 83/15/2, 82/15/3 and 90/10/0. Prior to co-digestion, the mixture of CM and Gly was pre-treated by sonication, applying a specific energy of 550 kJ/ kg TS. This energy was chosen in line with previous results obtained by the authors (Castrillón et al., 2013) and by Elbeshbishi et al. (2011). After the pre-treatment, FW was added and stirring was applied for 3-5 minutes to obtain a homogeneous mixture.

The reactor used in this study was previously used to digest CM supplemented with Gly (94/6, w/w) at 55°C, obtaining a methane yield of around 600 L CH₄/kg VS and a

methane production rate of 2 L CH₄/L.day, operating at an OLR of 6.44 g COD/L.day with a recirculation rate of 1 (Castrillón et al., 2013). At the end of this prior study, food waste was added to the digester at a ratio of 94/2/4 (CM/FW/Gly), the OLR in this case being 6.92 g COD/L.d. After reaching the steady state with this mixture, the ratios of co-substrates were varied while trying to maintaining a similar OLR in the reactor. The digester was fed once a day. The hydraulic residence time (HRT) was 20 days in all experiments.

The pH remained stable throughout the digestion process and there was no need to add alkalinity to the digester. The simultaneous presence of ammonia and bicarbonate in the digester results in the formation of a buffer system (Li et al., 2011).

3. RESULTS AND DISCUSSION

3.1. Characterization of the waste

Table 1 shows the characteristics of the three types of waste used in this study. The methanol and pure glycerin contents of the crude glycerin were 7.8% and 46.4%, respectively. The COD of crude glycerin is very high, around 1250 g/kg. Siles et al. (2009) reported an average COD of acidified glycerin of 1010 g/kg. As the C/N ratio of glycerin is around 248, this waste constitutes a suitable co-substrate for anaerobic digestion of nitrogen-rich waste, such as cattle manure and food waste, with a C/N ratio of around 15.

The ground cattle manure used in the experiments had a high water content (90.2%). Total COD values averaged 52.7 g/kg and volatile solids 50.9 g/kg, representing 52% of total solids. Nitrogen was present in both the organic and ammonium forms, the average

values of NH_4^+ -N and total N being 1.4 g/kg and 1.8 g/kg, respectively. The C/N ratio ranged around 15.7.

The food waste had a water content of 76.8% and an acidic pH (5.8). Volatile solids presented average values of 232 g/kg (95% of total solids), similar to those found by Zhang et al. (2007) and Neves et al. (2009). Nitrogen was present in both organic and ammonium forms, 3.2 g/kg and 1.4 g/kg, respectively, the organic nitrogen being higher than in cattle manure. The C/N ratio for food waste ranged around 15.4, similar to the values obtained by Han and Shing (2004) and Zhang et al. (2007), 14.7 and 14.6, respectively.

3.2. Co-digestion of manure, food waste and crude glycerin

Cattle manure was the major component of all the mixtures. According to the provisions of the Spanish Manure Digestion Plan, aimed at promoting manure slurry treatment both in centralized and individual plants in order to reduce methane emissions, energy producers may benefit from funding. However, in order to obtain the maximum subsidy, the co-substrates which may be added to enhance biogas production should not exceed 20%; i.e. the amount of cattle manure in the mixture should be $\geq 80\%$.

As already stated, previous research was carried out adding Gly to CM, obtaining the best results for 6% Gly under thermophilic conditions. In the present study, FW was also added, starting from 2% and subsequently increasing the proportion to 10% and 15%. The amount of glycerin was varied from 4% to 0%.

3.2.1. Removal efficiencies

The physicochemical characteristics of the reactor influent for the different operating conditions are given in Tables 2, 3 and 4. These tables also include the characteristics of

the effluent once steady-state conditions were achieved in the reactor (constant biogas production and constant effluent COD and VS) for each mixture.

The best results were obtained for CM/FW/Gly mixtures of 94/2/4 and 87/10/3, whose organic loading rates (OLR) were similar, 6.92 - 6.99 g COD/L.day (Table 2). In both cases, COD removal efficiencies were around 92%, though VS removal was slightly higher for the mixture with a higher proportion of food waste (87/10/3), 86.7% versus 83.2%. VFA concentrations in the final effluents were very low (\leq 87 mg/L), as can be seen in Table 2.

Increasing the proportion of food waste in the mixture from 10% to 15% while maintaining a similar OLR in the digester due to the reduction in CM and Gly (2%) led to only a slight decrease in COD and VS removals (Table 3), but to a considerable reduction in methane yield. When maintaining the same amount of FW (15%) while increasing the amount of Gly (3%), the OLR increased to 7.79 g COD/L.day and the methane yield doubled, although COD and VS removals were only slightly higher (88.2% and 83.7%, respectively).

Comparing the results obtained using the same amount of crude glycerin in the mixture (87/10/3 versus 82/15/3), higher organic matter removals were obtained when a smaller amount of food waste was introduced into the reactor, 92% COD removal and 86.7% VS removal versus 88.2% COD removal and 83.7% VS removal, respectively.

Finally, when Gly was not added to the mixture of CM and FW (90/10), this did not lead to improve the removal of organic matter (88.2% COD, 82.5% VS) even though the binary mixture presented a lower OLR (5.53 g COD/L.day) than the ternary mixtures (Table 4).

3.2.2. Methane production

Figures 1 and 2 show the evolution of the specific and volumetric methane productions throughout the study, while Table 5 shows the methane yields obtained in the co-digestion of the different mixtures.

The specific methane yield for the 87/10/3 and 94/2/4 mixtures was 640 L CH₄/kg VS in both cases, although the volumetric production rate was slightly higher in the mixture with a higher FW content: 2.57 L CH₄/L.day versus 2.43 L CH₄/L.day.

Increasing the proportion of food waste in the mixture from 10% to 15% while maintaining a similar OLR in the digester due to the reduction in CM and Gly led to a major decrease in methane yield (170 L/kg VS versus 640 L/kg VS).

For the same amount of FW in the mixture (15%), the production of methane increases when increasing the amount of glycerin added (360 L/kg VS). However, the methane yield was even higher when the food waste was 10% (640 L CH₄/kg VS).

According to the results obtained, it can be concluded that 15% FW in the feed to the digester results in an excessive contribution of organic matter from this FW that need to be hydrolysed and an increase in the concentration of VFA in the effluent (Table 3).

However, 10% FW could constitute an acceptable amount in the mixture when operating at an OLR of around 7 g COD/L.day (3.8 g VS/L.day).

The production of methane when Gly was not added to the mixture of 90% CM and 10% FW was 330 L CH₄/kg VS and 1.18 L CH₄/L.day, approximately half that obtained with the 87/10/3 mixture. Similar results were obtained by Zhang et al. (2012). These authors studied the anaerobic co-digestion of CM and FW in batch and in semi-continuous mode, obtaining a specific methane yield of 388 L/kg VS in batch operations and 317 L/kg VS in semi-continuous operations.

The addition of small amounts of Gly to CM and FW co-substrates always produces an increment in methane yield. This is a consequence of the much higher biodegradability of glycerin compared to that of cattle manure. However, as is well known, only small amounts of glycerin can be added as a supplement in anaerobic co-digestion, the maximum amount depending on the composition of the crude glycerin (content in methanol, glycerin, esters, soaps, Na or K). In previous research, the optimum amount of crude glycerin to supplement cattle manure so as to enhance biogas production in anaerobic digestion was found to be 6% (w/w) when operating at 55°C (Castrillón et al. 2011). In this respect, when crude glycerin (6% w/w) was added to CM, the biogas yield increased to 590 L CH₄/kg VS (Castrillón et al., 2013). Amon et al. (2006) also found this amount of glycerin to be the optimum to be added to a mixture of 54% pig manure, 31% maize silage and 15% maize corns. The methane yield in the co-digestion at 38-40°C increased from 335 L/kg VS to 439 L/kg VS.

The results achieved in this study highlight the major potential of this agro-industrial waste to boost biogas production in anaerobic digesters.

4. CONCLUSIONS

The addition of glycerin to cattle manure and food waste co-substrates in an IBR greatly improved the methane yield. The best results were obtained in the co-digestion of a mixture of 87/10/3 (CM/FW/Gly) operating at an OLR of 7 g COD/L.day, achieving 92.8% COD removal, methane yield of 640 L CH₄/kg VS and a volumetric methane production rate of 2.57 L CH₄/L.day (3.3 L biogas/L.day). In terms of the waste fed into the reactor, the yield was 45.3 L CH₄/kg wet waste, compared to values of 21.2 L CH₄/kg wet waste when glycerin was not added (90% CM, 10% FW).

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Captions for Tables

Table 1. Composition of crude glycerin, cattle manure and food waste used in the co-digestion experiments

Table 2. Characteristics of the influents and effluents of the digester (expressed on a fresh-weight basis) in the co-digestion of CM+FW+Gly.

Table 3. Characteristics of the influents and effluents of the digester (expressed on a fresh-weight basis) in the co-digestion of CM+FW+Gly for feeds containing 15% food waste

Table 4. Characteristics of the influent and effluent of the digester (expressed on a fresh-weight basis) in the co-digestion of CM+FW

Table 5. Comparison of the methane production for the different co-digestion mixtures

Captions for Figures

Figure 1. Specific methane yield in the thermophilic co-digestion of CM+FW+Gly

Figure 2. Volumetric methane production rate in the thermophilic co-digestion of CM+FW+Gly

Table 1. Composition of crude glycerin, cattle manure and food waste used in the co-digestion experiments

Parameter	Crude glycerin		Cattle manure	Food waste
	Average value±SD	Parameter	Average value±SD	
pH	7.8 ± 0.1	pH	7.4 ± 0.2	5.8 ± 0.4
Water (%w/w)	1.2 ± 0.1	TS (g/kg)	98 ± 0.9	232 ± 11
Ashes (%)	4.9 ± 0.1	VS (g/kg)	50.9 ± 0.6	220 ± 11
Total COD (g/kg)	1250 ± 48	Total COD (g/kg)	52.7 ± 4.5	506 ± 43
Methanol (% w/w)	7.8 ± 0.5	Total N (g/kg)	1.8 ± 0.3	3.2 ± 0.4
Glycerol (% w/w)	46.4 ± 0.2	NH ₄ ⁺ - N (g/kg)	1.4 ± 0.2	1.4 ± 0.2
Soap (% w/w)	30.3 ± 0.4	Total P (g/kg)	1.1 ± 0.1	1.5 ± 0.2
Glycerol ester (% w/w)	9.3 ± 0.2	C/N	15.7 ± 0.5	15.4 ± 0.8
C/N	248 ± 1			

Table 2. Characteristics of the influents and effluents of the digester (expressed on a fresh-weight basis) in the co-digestion of CM+FW+Gly for maximum biogas production

Parameter	94% CM+2% FW+4% Gly		87% CM+10% FW+3% Gly	
	HRT 20 days		HRT 20 days	
	6.92 g COD/L.day	3.57 g VS/L.day	6.99 g COD/L.day	3.77 g VS/L.day
Influent	Effluent	Influent	Effluent	
pH	7.8 ± 0.1	7.6 ± 0.3	7.3 ± 0.1	7.4 ± 0.2
Total COD (g/kg)	138.4 ± 18.3	10.5 ± 3.0	139.8 ± 5.3	10.1 ± 3.2
TS (g/kg)	97.4 ± 0.3	16.4 ± 0.2	103.9 ± 1.4	15.9 ± 0.9
VS (g/kg)	71.3 ± 0.5	12.0 ± 0.3	75.4 ± 1.4	10.0 ± 1.1
Acetic Ac. (mg/L)	803 ± 25	37 ± 5	727 ± 35	87 ± 4
Propionic Ac. (mg/L)	1152 ± 56	14 ± 2	1135 ± 106	n.d.
Isobutyric Ac. (mg/L)	87 ± 11	n.d.	62 ± 11	n.d.
Butyric Ac. (mg/L)	132 ± 30	n.d.	97 ± 9	n.d.
Isovaleric Ac. (mg/L)	21 ± 2	n.d.	11 ± 3	n.d.
Valeric Ac. (mg/L)	85 ± 14	n.d.	85 ± 4	n.d.

n.d.: not detected

Table 3. Characteristics of the influents and effluents of the digester (expressed on a fresh-weight basis) in the co-digestion of CM+FW+Gly for feeds containing 15% food waste

Parameter	83% CM + 15% FW + 2% Gly		82% CM + 15% FW + 3% Gly	
	HRT 20 days		HRT 20 days	
	6.91 g COD/L.day	3.57 g VS/L.day	7.79 g COD/L.day	3.96 g VS/L.day
Influent	Effluent	Influent	Effluent	
pH	7.3 ± 0.1	7.3 ± 0.1	7.3 ± 0.1	7.3 ± 0.1
CODt (g/kg)	138.2 ± 8.0	17.2 ± 2.6	155.9 ± 6.4	18.3 ± 6.0
TS (g/kg)	107.5 ± 1.3	25.8 ± 0.6	115.6 ± 1	25.9 ± 1.5
VS (g/kg)	71.4 ± 1.0	12.4 ± 1.0	79.3 ± 1.2	12.9 ± 1.9
Acetic Ac. (mg/L)	798 ± 42	163 ± 14	815 ± 22	153 ± 35
Propionic Ac. (mg/L)	1046 ± 48	123 ± 28	1245 ± 98	105 ± 25
Isobutyric Ac. (mg/L)	103 ± 40	72 ± 8	75 ± 2	76 ± 15
Butyric Ac. (mg/L)	105 ± 27	55 ± 2	111 ± 8	n.d.
Isovaleric Ac. (mg/L)	35 ± 12	n.d.	28 ± 9	n.d.
Valeric Ac. (mg/L)	125 ± 31	n.d.	100 ± 10	n.d.

n.d.: not detected

Table 4. Characteristics of the influent and effluent of the digester (expressed on a fresh-weight basis) in the co-digestion of CM+FW

Parameter	90% CM + 10% FW	
	HRT 20 days	
	5.53 g COD/L.day	3.36 g VS/L.day
	Influent	Effluent
pH	7.2 ± 0.1	7.3 ± 0.1
CODt (g/kg)	110.6 ± 12.2	13.0 ± 3.5
TS (g/kg)	92.6 ± 1.0	23.8 ± 1.0
VS (g/kg)	67.3 ± 1.1	11.8 ± 0.7
Acetic Ac. (mg/L)	817 ± 30	108 ± 21
Propionic Ac. (mg/L)	1405 ± 43	103 ± 11
Isobutyric Ac. (mg/L)	129 ± 12	27 ± 8
Butyric Ac. (mg/L)	137 ± 32	n.d.
Isovaleric Ac. (mg/L)	49 ± 10	n.d.
Valeric Ac. (mg/L)	139 ± 22	n.d.
n.d.: not detected		

Table 5. Comparison of the methane production for the different co-digestion mixtures

	94/2/4	87/10/3	CM/FW/Gly 83/15/2	82/15/3	90/10/0	94/0/6 ^a
g COD/L.day	6.92	6.99	6.91	7.79	5.53	6.44
L CH ₄ /kg VS	640	640	170	360	330	590
L CH ₄ /L. d	2.43	2.57	0.65	1.52	1.18	2.00
L CH ₄ /kg wet waste	45.3	45.3	13.5	28.5	21.2	36.7
% CH ₄ in biogas	76.4	77.9	65.7	68.4	68.7	65

^aCastrillón et al., 2013

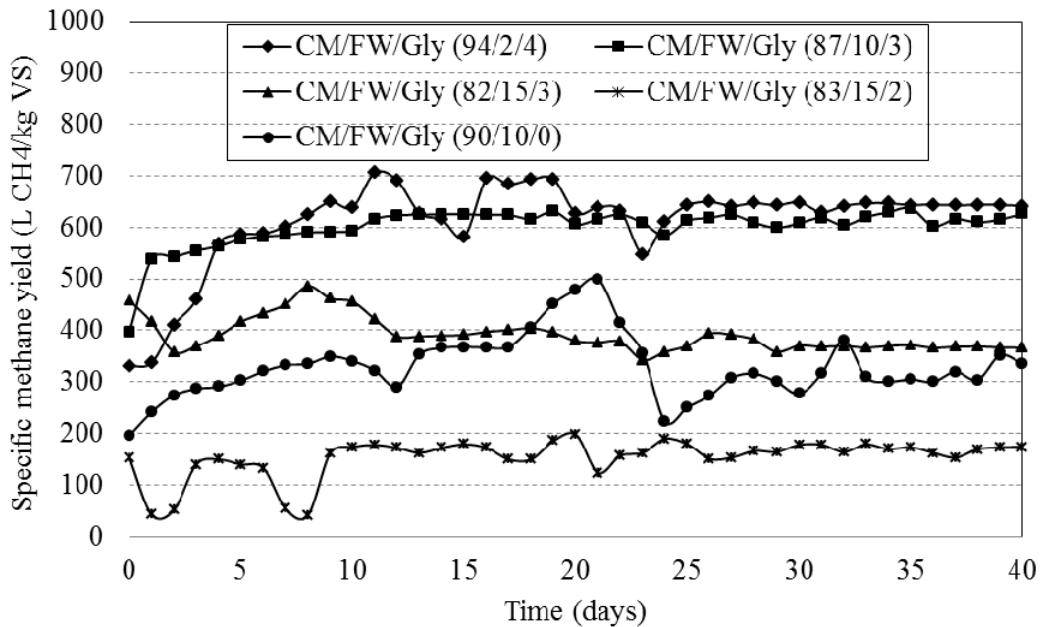


Figure 1

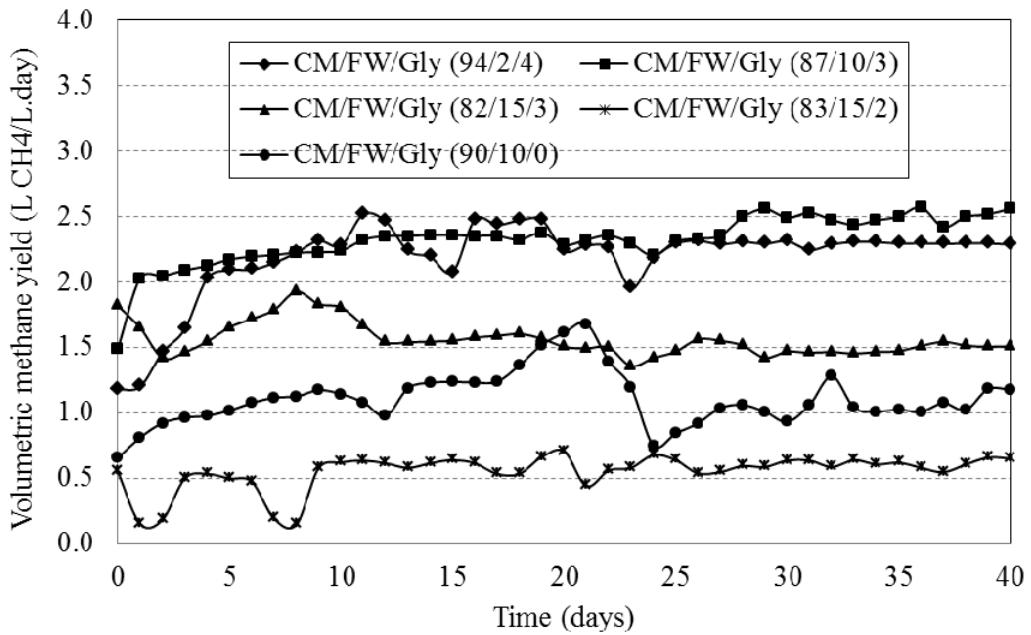


Figure 2

Highlights

- Glycerin addition double the methane yield in thermophilic anaerobic co-digestion
- Up to 93% COD removal achieved operating at 7 gCOD/L.day in an induced bed reactor
- Mixtures of 87% cattle manure, 10% food waste and 3% glycerin gave 2.6 LCH₄/L.day
- Methane content in biogas up to 78%