



Co-digestion of swine excreta associated with increasing levels of crude glycerin

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ABSTRACT - The objective of this research was to evaluate the performance of anaerobic co-digestion of swine excreta associated with increasing doses of crude glycerin and different hydraulic retention times (HRT). A completely randomized design was adopted in a 3 × 4 factorial arrangement composed of three HRT (10, 17, and 24 days) and four crude glycerin doses (0, 5, 10, and 15 g/100 g of total solids [TS]), with four replications per treatment. The assessed parameters were: biogas production potential, reductions of volatile solids (VS), chemical oxygen demand (COD), and most probable number of total and thermotolerant coliforms. The biogas production per added VS presented quadratic effect at 17 and 24 days of HRT, with ideal doses of 5.5 and 5.9 g of crude glycerin/100 g TS, respectively. There was no difference among glycerin doses at 10-day HRT for VS reductions; however, at HRT of 17 and 24 days, there were differences, with greater reduction of 61.1% for 5 g of crude glycerin/100 g TS at 24-day HRT. The COD reduction values showed an effect among retention times, in which the 24-day HRT provided the best results. Reductions in coliforms were greater than 99%, with no difference among treatments. Addition of 5 to 6 g of crude glycerin/100 g TS with a 24-day HRT is more effective in biogas production and reduction of VS, COD, and coliforms from co-digestion of swine excreta.

Key Words: biodiesel, biogas, manure, waste

Introduction

The increasing production of biodiesel has generated millions of tons of crude glycerin per year. Biodiesel is converted through transesterification reaction whereby crude glycerin is generated in approximately 10%. In addition, crude glycerin contains some impurities in its composition, such as water, methanol, and non-glycerol organic material, which gives it a low commercial value (Larsen et al., 2013).

The search for alternatives to use this by-product is important in order to avoid the occurrence of environmental and economic problems. Recent studies on anaerobic co-digestion of glycerol and excreta from animal production have aimed to reduce the impact of excess crude glycerin and increase the potential of this process. This alternative seems to be very promising, since it allows the reduction of the hydraulic retention time (HTR) spent to achieve the

same biogas production generated by using only wastes (Siles et al., 2009).

According to Castrillón et al. (2013), crude glycerin is readily degradable, possibly due to high carbon, which is favorable to the anaerobic co-digestion. Therefore, the product might improve biogas yield up to 380% in swine waste substrates (Astals et al., 2012).

The ideal ratio of crude glycerin added to swine waste and excreta must be estimated with the aim of achieving results that indicate increases in production of biogas and methane, as well as significant reductions in organic constituents and pathogens, which, in turn, improves the biofertilizer quality. Thus, this research was conducted to determine the anaerobic digestion performance in semi-continuous digesters with hydraulic retention times of 10, 17, and 24 days and inclusion of crude glycerin at the levels of 0, 5, 10, and 15 g/100 g total solids (TS) in the substrates.

Material and Methods

The study was carried out in Dourados, MS, Brazil. The climate of this region, according to the Köppen classification, is a Cwa mesothermal humid, with average annual precipitation and temperatures ranging from 1250 to 1500 mm and 20 to 24 °C, respectively.

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The experimental design was completely randomized, in a 3×4 factorial arrangement, with three HRT (10, 17, and 24 days) and inclusion of crude glycerin at four levels (0, 5, 10, and 15 g of crude glycerin/100 g TS) and four replications per treatment (48 experimental units).

Semi-continuous digesters were loaded with excreta collected from a commercial swine production and crude glycerin obtained from biodiesel industry, both from the region of Dourados, MS. Swine excreta were collected directly by scraping the floor of the stalls with swine in the finishing phase.

Total solids content was established at 2 g/100 g TS for the influent composition by mixing excreta, crude glycerin, and water, considering also the TS content in crude glycerin. Glycerin was stored in sealed containers until dilution with excreta to compose the daily load of the digesters.

According to analyses performed by the Paraná State Institute of Technology, swine excreta and crude glycerin were characterized at the beginning of the experimental period as follows: crude glycerin composition: 3.63 g/100 g moisture, 39.3 g/100 g glycerol, 4.75 g/100 g methanol, 47.3 g/100 g fatty acids, 2 g/100 g crude protein, 12.1 mg/kg sodium, 372.28 mg/kg potassium, 68.25 mg/kg calcium, 15.15 mg/kg magnesium, 171.63 mg/kg phosphorus, <0.4 mg/kg cadmium, <4.0 mg/kg lead, 1.880 g O₂ L⁻¹ chemical oxygen demand (COD), and pH 8.9; collected excreta composition: 28.32 g/100 g TS, which were 80.25 g volatile solids (VS)/100 g TS. The average value of COD was 794.1 g O₂ per kg of excreta. The most probable numbers of total and thermotolerant coliforms were 5.7×10^{10} and 1.4×10^{10} , respectively.

The proportion of water, crude glycerin, and excreta varied according to the treatment (Table 1).

Table 1 - Swine excreta, water, crude glycerin, and glycerol amounts added daily to digesters loaded with different levels of crude glycerin doses and hydraulic retention times (HRT)

Crude glycerin (g/100 g TS)	HRT (days)	Excreta (g)	Water (g)	Crude glycerin (g)	Glycerol (g)
0	10	254	3558	0	0
0	17	185	2591	0	0
0	24	123	1720	0	0
5	10	241	3568	4.08	1.60
5	17	175	2598	2.97	1.16
5	24	116	1724	1.97	0.77
10	10	248	3904	8.91	3.48
10	17	153	2414	5.51	2.15
10	24	114	1793	4.09	1.60
15	10	233	3915	13.37	5.23
15	17	144	2420	8.26	3.23
15	24	107	1798	6.14	2.40

TS - total solids.

During the experimental period, biogas production and potential were analyzed by considering the added and reduced amounts of VS and COD, and the most probable number (MPN) of total and thermotolerant coliforms.

Biogas production was determined by daily measurements of vertical displacement of gasometers, and then, this value was multiplied by their cross-sectional area to estimate the biogas volume. Adjustments of biogas volume at 1 atm and 20 °C were applied according to the literature (Caetano, 1985). Potentials of biogas production were calculated by dividing the production outputs by the amount of TS, VS, and COD added to the digesters. Levels of TS, VS, COD and MPN were monitored by following the methodology described by APHA (2005).

Results obtained during a four-week experimental period were subjected to analysis of variance at $\alpha = 0.05$ by the F test, considering the HRT and crude glycerin doses as sources of variation. Orthogonal contrasts were used to assess the effects of linear and quadratic order of the retention times and levels of glycerin. Crude glycerin and HRT effects were assessed through regression analysis by using SAEG 9.1 software.

Results and Discussion

Regression analysis showed a negative linear effect ($P < 0.05$) on potential per grams of added TS, VS, and COD at 10-day HTR. However, with HRT of 17 and 24 days, a quadratic effect was observed due to increasing levels of crude glycerin (Figures 1, 2, and 3).

The results of reduction of biogas production with the shortest HRT indicate that the amount of energy available from crude glycerin was probably not used effectively by microorganisms in a 10-day hydraulic retention time.

The highest values of potential for solids were achieved using 5 g crude glycerin/100 g TS in the composition of the influent of the digesters, with ideal inclusions of crude glycerin of 5.4 and 5.7 g/100 g TS, and 5.5 and 5.9 g/100 g TS for VS added at 17- and 24-day HRT, respectively (Figures 1 and 2).

The potential results of TS and VS were superior to those reported by Miranda et al. (2012), who evaluated biogas yield from bench digesters loaded with excreta from initial, growing, and finishing phases of swine fed diets based on corn and sorghum with a 30-day HRT. In comparison, with no inclusion of crude glycerin, our percentage values were greater by 58, 10, and 25% for TS; and 71, 17, and 41% for VS at 10-, 17-, and 24-day HRT respectively. The potential obtained in the current study

was greater possibly because of the type of digester used, which was a semi-continuous feeding digester.

Regression models of all three HRT, according to the levels of crude glycerin on the potentials of biogas production per gram of COD added, demonstrated similar behavior to the potentials regarding TS and VS (Figure 3). The lowest biogas potential per COD added (0.19 L) with crude glycerin inclusion was achieved by digesters operated with 10 days of HRT. At 17 days of HRT, a regression curve equation indicated that the highest results were reached with inclusion of 5.9 g/100 g TS. However, the ideal level of inclusion was 6.8 g/100 g TS crude glycerin at 24 days of HRT, which also presented mean values 30% higher

than 10-day HRT and 20% higher when compared with 17-day HRT.

An increase of 62.7% in the potential per added COD was achieved with inclusion of 5 g of crude glycerin/100 g TS when compared with the treatment without crude glycerin at 17 days, while at 24 days of HRT, with 5 g of crude glycerin/100 g TS inclusion the increase in the potential per added COD was 67.7%.

Astals et al. (2012) obtained lower potential increase by using semi-continuous digesters with HRT of 20 days and better control of external effects (continuous stirring at 60 rpm and controlled temperature). These authors reported potential of 0.013 L biogas. COD^{added}⁻¹ without adding crude glycerin to swine residue and 0.020 L biogas. COD^{added}⁻¹ by adding 4 g of crude glycerin/100 g TS compared with the total volume of the digester, resulting in a lower potential increase of approximately 53.8%.

The aforementioned values are similar to the limiting concentrations of crude glycerin in digestion with livestock waste obtained by other authors, such as Amon et al. (2006), who used batch digesters with a mixture of swine manure, corn silage, and canola flour and obtained similar results, indicating as optimal dose an addition of 65 g of crude glycerin/100 g TS to the mixture.

Holm-Nielsen et al. (2008) conducted a study with an OnLine Near Infrared Spectroscopy (NIR) monitoring system to measure volatile fatty acids (VFA) generated in the fermentation process of organic waste from the food industry and pure glycerol (99.55 g/100 g TS) in semi-continuous digesters at 53 °C. When the glycerol concentration was increased from 3.5 to 6.55 g/100 mL

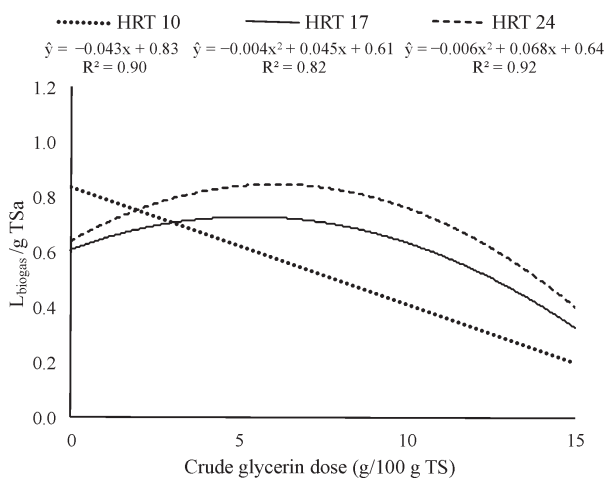


Figure 1 - Regression models for potential of biogas production per gram of total solids (TS) at hydraulic retention times (HRT) of 10, 17, and 24 days and crude glycerin doses of 0, 5, 10, and 15 g/100 g total solids.

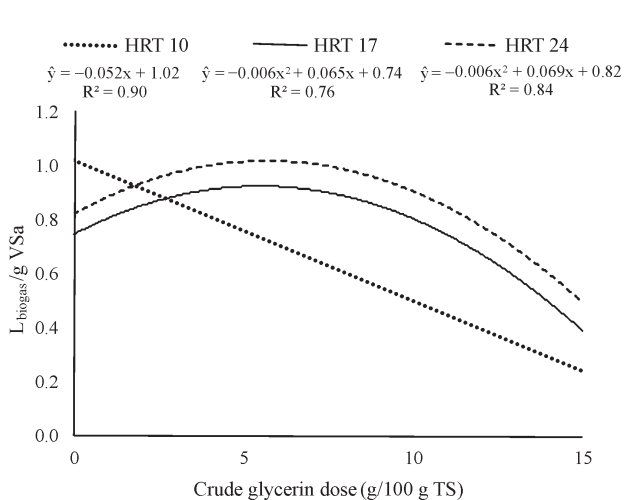


Figure 2 - Regression models for potential of biogas production per gram of volatile solid (VS) at hydraulic retention times (HRT) of 10, 17, and 24 days and crude glycerin doses of 0, 5, 10, and 15 g/100 g total solids (TS).

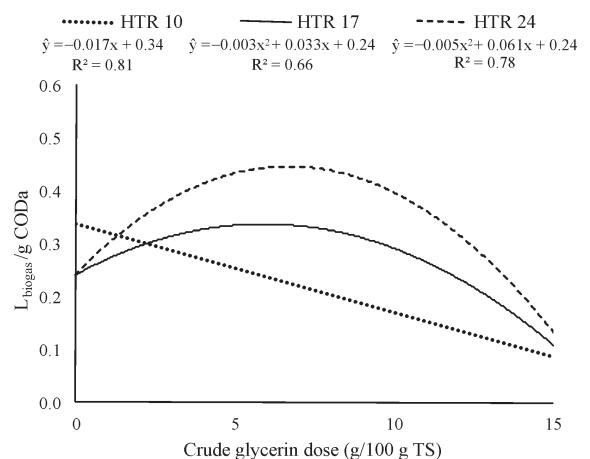


Figure 3 - Regression models for potential of biogas production per gram of chemical oxygen demand (COD) at hydraulic retention times (HRT) of 10, 17, and 24 days and crude glycerin doses of 0, 5, 10, and 15 g/100 g total solids (TS).

of the total volume of the digester, the production of VFA was increased, which caused an organic overload due to an inhibition of methanogenic phase, ending the process in complete collapse.

Results for biogas production potential according to the added amounts of TS, VS, and COD indicate that 5 to 6 g of crude glycerin/100 g TS inclusion provides the best values within longer retention periods. However, when these retention periods were analyzed within each level of inclusion, there was no difference between HRT of 17 and 24 days in some of these analyses. Thus, it is possible to use 17 days as hydraulic retention time to obtain these parameters, since, from this period on, there will not be improvement in the potentials of production.

The addition of crude glycerin provided a positive linear response regarding the increase in HRT with doses of 0, 5, and 10 g/100 g TS and a quadratic effect at 15 g/100 g TS for VS reductions in digesters operated with 10, 17, and 24 days of HRT (Figure 4).

There was no difference in VS reduction among crude glycerin doses at 10 days. However, at 17 days, the doses of 0, 5, 10, and 15 g/100 g TS caused VS reductions of 38.0, 29.1, 35.4, and 52.8%, respectively.

The reductions in VS at 24 days of HRT occurred as follows: 43.5% without crude glycerin, 61.1% with addition of 5 g/100 g TS, 39.8% with 10 g/100 g TS, and 54.3% with 15 g/100 g TS. Therefore, the greatest VS reduction was attained with addition of 5 g/100 g TS crude glycerin at 24 days, but for the other doses (0, 10, and 15%), the 17-day HRT proved to be sufficient to achieve a reduction that is similar to the longest retention period studied.

In a co-digestion study of cassava wastewater with crude glycerin, Larsen et al. (2013) achieved higher

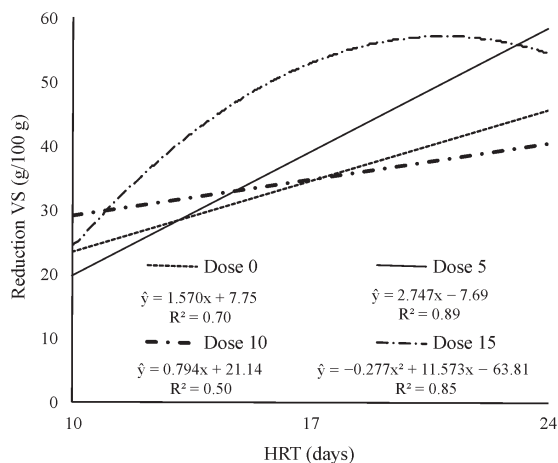


Figure 4 - Regression model for volatile solids (VS) for each crude glycerin dose as a function of hydraulic retention time (HRT).

reductions of VS with temperature set at 26 °C and HRT of 4 and 5 days. The authors obtained reductions from 90.2 to 61.5% of VS with decrease in the process efficiency as glycerin doses increased, similar to that observed in our study with 5% crude glycerin addition. Digesters supplies were based on the volumetric increase of organic load, which was of 3.05, 9.32, 14.83, and 13.59 g COD L⁻¹ d⁻¹, attained with addition of crude glycerin at 0, 2, 3, and 4 g/100 g TS, respectively. The authors also reported high reductions of COD, ranging from 96.4 to 91.5% of COD.

The determination of the best HRT to increase the reduction of VS is dependent on the amount of crude glycerin added, since, for the dose of 5 g/100 g TS, the best HRT was at 24 days, while without addition of glycerin and at 10 g/100 g TS, 17-day retention was sufficient to achieve a good reduction, and according to the regression equation, the ideal HRT for 15 g/100 g TS addition was 20.8 days.

Our findings show that for a greater VS reduction using crude glycerin in co-digestion with swine manure, the best dose is about 5 g/100 g TS with a HRT of 20 days, as reported by Castrillón et al. (2013), who obtained the greatest VS and COD reductions at 20 days by testing co-digestion of cattle waste with inclusion of 6 g/100 g TS in the solid influent.

Chemical oxygen demand reductions showed no interaction between HRT and crude glycerin doses, but presented a positive linear effect according to the HRT (Figure 5).

Retention times had an effect on COD reduction (Figure 5), presenting 41.2% reduction at 24 days, 33.6% at 17 days, and 23.0% at 10 days. Therefore, there was an improvement in COD reduction of 79.1% when HRT was increased from 10 to 24 days.

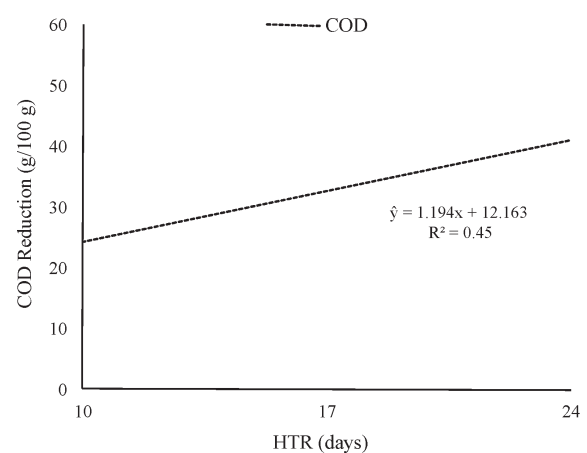


Figure 5 - Regression model for hydraulic retention time (HRT) over chemical oxygen demand reduction (COD).

Table 2 - Most probable number of total coliforms and thermotolerant coliforms and reduction percentage during anaerobic co-digestion of swine excreta under growing doses of crude glycerin and different hydraulic retention times (HRT)

Crude glycerin dose (g/100 g total solids)	HRT	Total coliforms			Thermotolerant coliforms		
		Affluent	Effluent	Reduction (%)	Affluent	Effluent	Reduction (%)
0	10	5.7×10^{10}	3.0×10^7	99.95	1.4×10^{10}	2.9×10^7	99.79
	17	5.7×10^{10}	1.3×10^6	100.00	1.4×10^{10}	4.2×10^5	100.00
	24	5.7×10^{10}	2.9×10^7	99.95	1.4×10^{10}	2.8×10^7	99.80
5	10	5.7×10^{10}	2.5×10^5	100.00	1.4×10^{10}	7.7×10^4	100.00
	17	5.7×10^{10}	3.9×10^7	99.93	1.4×10^{10}	3.9×10^7	99.72
	24	5.7×10^{10}	5.4×10^6	99.99	1.4×10^{10}	5.3×10^6	99.96
10	10	5.7×10^{10}	3.1×10^7	99.94	1.4×10^{10}	2.9×10^7	99.79
	17	5.7×10^{10}	8.2×10^5	100.00	1.4×10^{10}	4.0×10^5	100.00
	24	5.7×10^{10}	1.3×10^7	99.98	1.4×10^{10}	1.3×10^7	99.91
15	10	5.7×10^{10}	3.8×10^7	99.93	1.4×10^{10}	6.5×10^6	99.95
	17	5.7×10^{10}	1.9×10^6	100.00	1.4×10^{10}	3.4×10^5	100.00
	24	5.7×10^{10}	1.3×10^7	99.98	1.4×10^{10}	1.2×10^6	99.98

Reductions in TS, VS, and COD with the longest HRT, regardless of the dose of crude glycerin added, corroborate the results of Orrico Junior et al. (2009), who analyzed swine manure digestion with and without solid fraction separation in semi-continuous tubular digesters operated at different retention times (15, 22, 29, and 36 days) and reported the greatest reductions without solid fraction separation at 36 days (TS = 66.7%, VS = 70.3%, and COD = 81.7%), followed by 29 and 22 days and the lowest reductions at 15 days (TS = 55.6%, VS = 64.0%, and COD = 61.7%).

Regarding the average reductions in MPN of total and thermotolerant coliforms in substrates prepared with swine manure and crude glycerin (Table 2), there was neither interaction between the two evaluated parameters nor effect of doses and HRT on the results.

Orrico Junior et al. (2010) reported different results when evaluating the treatment of manure from swine fed corn and sorghum, in semi-continuous digesters at four HTR (30, 60, 90, and 120 days). The authors observed differences with regard to retention times (HRT) and obtained the best reductions in MPN of total and thermotolerant coliforms in substrates that remained for longer periods under digestion.

The similarities in the results for coliform reductions can be related to the shorter time interval among HRT employed in the current research when compared with the results shown by Orrico Junior et al. (2010), who adopted an interval of 30 days, and another study of Orrico et al. (2009) in which there was no difference among HRT with an interval of 7 days during digestion process of swine manure with and without separation of solid fraction in semi-continuous digesters operated at 15, 22, 29, and 36 days.

Reductions of more than 99.9% in MPN of coliform may be related to the type of digester. As stated by Orrico

Junior et al. (2009), tubular digesters usually have high acid production in the input of the digester, which reduces the microorganism population due to the low pH in the beginning of the process.

Conclusions

The addition of 5 to 6 g/100 g total solids of crude glycerin improves the potential of biogas production and the reduction of solids, chemical oxygen demand, and coliforms at hydraulic retention times of 17 and 24 days. However, crude glycerin inclusion is not indicated for use in semi-continuous digesters operated under similar conditions to those of this study with hydraulic retention times of 10 days or less.

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