Anaerobic Co-digestion of Cattle Manure and Sewage Sludge: Influence of Composition and Temperature

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***Abstract*-This paper presents the study of co-digestion of two types of organic wastes: cattle manure and sewage sludge from waste water treatment stations. Co-digestion of sewage sludge and cattle manure has the advantage of sharing processing facilities, unifying management methodologies, reducing operating costs and dampening investment and temporal variations in composition and production of each waste separately.**

The aim of this work was to select suitable operating conditions (both composition and temperature) of anaerobic co-digestion process of cattle manure and sewage sludge to optimize the process in the biogas generation. The batch tests have been developed at mesophilic and thermophilic conditions to determine the anaerobic biodegradability of three different mixtures of cattle manure and sewage sludge, both in static or stirring conditions.

The obtained experimental results indicate that the anaerobic biodegradability of raw sludge and cattle manure mixtures is more efficient at thermophilic conditions since a greater elimination of organic matter with a greater methane yield is obtained. The most efficient process corresponds to the mixture with 25% v/v of cattle manure and 75% v/v of raw sludge with values of 62% and 75.7% of COD and DOC removals, respectively and methane yields of 2200 mL CH_4/g COD_r and 306 ml CH_4/gVS_r, presenting a period of starting of 12 days. Also, it is verified that a higher amount of cattle manure in the mixture means a higher alkalinity and a greater percentage of methane in biogas. The biodegradability validation tests at stirring conditions confirm the kindness of the thermophilic process.

Keywords- Co-Digestion; Biodegradability; Cattle Manure; Sewage Sludge; Mesophilic; Thermophilic

I. INTRODUCTION

The generation of excess sludge is the main problem faced by wastewater treatment plants in urban areas (WWTP). Approximately 0.5 to 2% of treated water becomes necessary to manage sludge at a cost of over 50% of the costs of operation of the WWTP. Currently, the most widely used treatment process for the recovery of the sludge is anaerobic digestion of sludge at different temperature conditions [1, 2].

Moreover, the intensification of farming has led to an increase in the generation of potentially polluting cattle manure. This intensification of animal production started during the fifties and, in essence, means the concentration of animals per unit area and the increased use of inputs [3]. Until 1980, in Europe there was a sustained increase in production that accompanied the increase in demand for livestock products [4]. This new scenario, in turn, led to various regulations in order to reduce the environmental impact of these new practices by limiting the number of animals per unit area. Anaerobic digestion of manure allows the sustainable management of this waste and also generates biogas with high energy content.

In general, anaerobic digestion is the most widely used technology for treating organic wastes. Interest in this technology has focused on an increase in process efficiency and a reduction in investment and operation costs [5]. The mesophilic temperature regime (30-40 ºC) has long been adopted for anaerobic digestion. However, it has been reported that the removal efficiency of organic matter, methane production as well as the inactivation of pathogens of the thermophilic digestion (50-60 ºC) at low retention times, was higher than the mesophilic digestion [6, 7]. Despite this, the use of thermophilic anaerobic digestion has been limited as a result of some disadvantages, such as poor supernatant quality, poor sludge dewaterability and poor process stability related to chronically high propionate concentrations [8, 9].

On the other hand, there are biodegradable wastes from different sources that have low potential for biogas production due to its low content of organic matter or its low biodegradability. In these cases, the anaerobic co-digestion is presented as a successful methodology that combines several biodegradable organic substrates able to increase the production potential of biogas per kilogram of mixture removal, both at mesophilic and at thermophilic conditions [8]. The main advantage of co-digestion is to harness the synergy of mixtures and compensate for the deficiencies of each of the substrates separately. It also produces an improvement in the balance of nutrients in the substrate (C:N:P = 300:5:1), decreases the contents of seasonal waste and generates higher production of biogas. Also, presents economic advantages from the sharing of equipment and costs [10].

Environmental problems related to the use of cattle manure and reducing the impact of these problems, as well as the endeavor to further utilize the nutrients contained by manure, have promoted the development of new manure treatment methods. One alternative is the anaerobic co-digestion with others organic wastes such as sewage WWTP sludge. Anaerobic digestion has many economic and environmental benefits apart for those associated with energy production [11, 12, 13].

This paper reports on anaerobic co-digestion of two types of organic wastes: cattle manure and sewage sludge from wastewater treatment stations. This technology is an attractive option to improve the yields of the anaerobic digestion of wastes due to the positive synergisms established in the digestion medium, a fact that increases the economic viability of the biogas plants. The main advantage of this technology-based system is an improved methane yield created by the supply of additional nutrients to the mixture.

However, there is little or no information about the co-digestion of cattle manure and sewage sludge, even those are wastes with important environmental problems [14, 15, 16].

The objective of this work was to select the operational conditions of anaerobic co-digestion process of sewage sludge and cattle manure to enhance the biogas generation in the process (CH_4). The batch tests have been developed at mesophilic and thermophilic temperatures to determine the anaerobic biodegradability of three different mixtures of cattle manure and sewage sludge, both in static or stirring conditions.

II. MATERIAL AND METHODS

A. Characterization of Wastes

Cattle manure was collected in a semi-intensive breeding farm located in El Puerto de Santa María-Cádiz (Spain). The mixed sludge (50% homogeneous mixtures of sludge from primary and secondary treatment) came from the sewage treatment plant in Arcos de la Frontera, Cádiz (Spain).

Initially the following parameters were measured: pH, Total and Volatile Solids (TS and VS), Total Organic Carbon (TOC), Total and Soluble Chemical Oxygen Demand (COD_T and CODs), alkalinity, ammonium (NH_4^+), Total Kjeldahl Nitrogen (TKN) and volatile fatty acids (VFA).

Table I summarizes the physical and chemical characterization of cattle manure (CM) and sewage sludge (SS). The manure has a higher organic loading and higher solids content while the slurry has a higher concentration of nitrogen. The C/N is higher for manure (high in organic matter content) than for the sludge (high in N), where feasible co-digestion of both residues.

TABLE I PHYSICO-CHEMICAL CHARACTERIZATION OF COW MANURE (CM) AND SEWAGE WWTP SLUDGE (SS)

	Cattle Manure		Sewage Sludge	
PARAMETER	**Average**	**Standar Deviation**	**Average**	**Standar Deviation**
pH (upH)	6,37	0,91	6,81	0,12
Total Solids (%TS)	18,24	0,54	1,43	0,29
Volatile Solids (% VS)	83,37	0,49	69,73	3,67
Ammonium (mg NH_3-N/kg)	74,62	50,09	34,84	12,59
Total Kjeldhal Nitrogen (% dry weight)	1,82	0,09	8,59	1,18
Total COD (COD_T, g O_2/L)	98,21	3,62	15,71	2,34
Soluble COD (CODs, g O_2/L)	64,15	13,81	0,59	0,30
Alcalinity (mg $CaCO_3$/L)	3475,00	318,20	1030,00	115,96
Total Organic Carbon (mg/l)	19235,00	975,81	117,99	8,12
C/N ratio	26,57	nd	4,71	nd
Total acidity (mg Acetic/L)	nd	nd	45,4	nd

B. Design of Mesophilic and Thermophilic Anaerobic Biodegradability Batch Tests of Cattle Manure-Sewage Sludge

1) Design of Static Tests:

The tests were conducted in glass reactors of 500 mL total volume (450 mL working volume) with an outlet for collecting the biogas produced in Tedlar bags (Figure 1). Different cattle manure-sewage sludge mixtures were labeled as follows (expressed in volume/volume):

- Mix 1: 25% cattle manure - 75% sewage sludge (25CM-75SS);
- Mix 2: 50% cattle manure - 50% sewage sludge (50CM50SS);
- Mix 3: 25% cattle manure - 75% sewage sludge (75CM-25SS).

Figure 1 Experimental equipment used in the development of the biodegradability tests

The inocula used were from two separate effluents from mesophilic and thermophilic reactors in operation in the Research Group. The inoculation rate was 10% of total digester volume. All tests were carried out in batch and in duplicate. Also, two control tests were incorporate consisting of 225 mL (50% of total volume) and thermophilic or mesophilic inoculum, respectively. Prior to the starting, inert nitrogen gas was recirculated to the headspace.

2) Design of Tests with Stirring Conditions:

The co-digestion processes were performed on 2 batteries of 4 stirred reactors to promote biomass-substrate contact and benefit biodegradation. The reactors used had a working volume of 1700 mL for the treatment mesophilic and 2700 mL for thermophilic treatment (Table II).

TABLE II VOLUMES OF WASTE AND INOCULA USED FOR THE ASSEMBLY OF THE REACTORS

	Mesophilic reactors	**Thermophilic reactors**
Total reactor volume (mL)	2000	3000
Working reactor volume (mL)	1700	2700
Inoculum (mL)	170 (10% working volume)	270 (10% working volume)
Cattle manure (mL)	382,5 (22,5% working volume)	607,5 (25% volumen restante)
Sewage Mixed Sludge (mL)	1147,5 (67,5% working volume)	1822,5 (67,5% working volume)
Control reactors (mL)	850 (50% working volume)	1350 (50% working volume)

The reactors were filled with the mixture 25% cattle manure and 75% of WWTP sludge and thermostated at 35 °C and 55 ºC under mesophilic or thermophilic conditions, respectively (Figure 2). The cattle manure is accepted as an average density of 800 kg/m^3 [16]. The inoculation rate was, in all cases, 10% of working volume. The tests were developed at batch conditions and in duplicate. Also, four control tests were incorporate consisting of 225 mL (50% of total volume) and thermophilic or mesophilic inoculum, respectively. Prior to the starting, inert nitrogen gas was recirculated to the headspace.

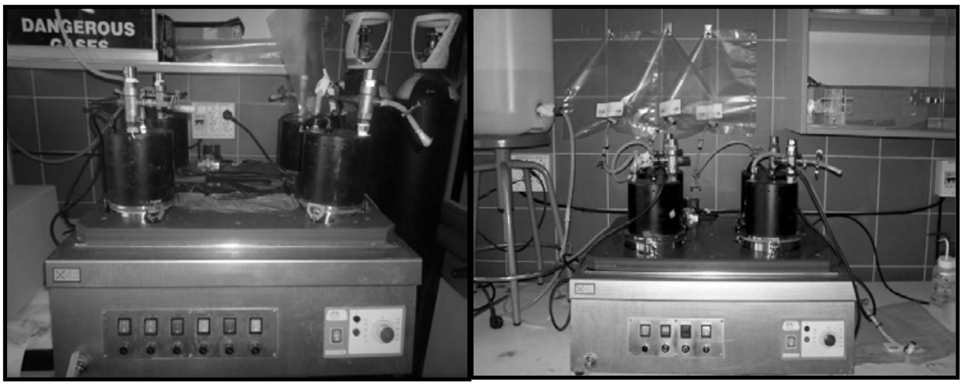


Figure 2 Experimental equipment used in the stirred biodegradability tests:mesophilic (left) and thermophilic (right)

Reactors have an outlet for collecting the biogas produced in Tedlar bags. The analyses of volume and gas composition were performed every 24 hours, using a gas flow meter for determining the volume generated, and a gas chromatograph for analysis of its composition.

In all studies (tests with and without agitation), a weekly pH control was performed to prevent the inhibition of methanogenic microorganisms, maintaining it at values close to neutrality (pH 7) by adding sodium hydroxide (NaOH 10M). The volume and composition of biogas were measured every 48 hours.

When biogas (methane) generation stopped (indicating the end of the degradative process), we proceeded to dismantle and analyzed the composition of the content of each reactor: pH, Total and Volatile Solids (TS and VS), Total Organic Carbon (TOC), Chemical Oxygen Demand and Total Soluble (COT_T and CODs), alkalinity, ammonium (NH4-), Total Kjeldahl Nitrogen (TKN) and volatile fatty acids (VFA). The frequency of analysis was each 48 hours at baseline, decreasing the frequency of the same as the process is stabilized, ending with a weekly analysis frequency.

3) Analytical Techniques Used:

The analytical techniques used are described in Standard Methods for the Analysis of Water and Wastewater [17].

III. RESULTS AND DISCUSSION

A. Biodegradability Tests

Tables III-A and III-B show the initial and final values of the different physicochemical parameters analyzed in samples from both thermophilic and mesophilic tests. The values shown are averages of the two replicas made.

TABLE III-A INITIAL AND FINAL VALUES OF THE PHYSICOCHEMICAL PARAMETERS ANALYZED IN THE THERMOPHILIC TESTS (55° C)

PARAMETER	**T-25CM-75SS**		**T-50CM-50SS**		**T-75CM-25SS**	
	Beginning	**Ending**	**Beginnig**	**Ending**	**Beginning**	**Ending**
ph (upH)	5,52	7,50	5,09	7,55	5,20	7,71
Total Solids (%)	5,22 ± 0,08	3,80 ± 0,03	9,42 ± 0,07	6,29 ± 0,05	13,00 ± 0,09	8,91 ± 0,28
Volatile Solids (% ST)	76,90 ± 0,02	71,62 ± 0,08	81,83 ± 0,14	78,55 ± 7,84	79,00 ± 0,06	73,16 ± 0,35
Ammonium (mg NH3-N/kg)	109,12 ± 7,17	625,69 ± 1,43	113,85 ± 4,51	731,30 ± 1,82	257,60 ± 4,75	675,92 ± 22,97
Total Nitrogen (% s/ww)	2,94 ± 0,09	38,51 ± 20,79	1,57 ± 0,16	18,79 ± 1,54	1,74 ± 0,45	14,82 ± 2,37
Total COD (g O_2/L)	78,13 ± 4,17	35,23 ± 0,38	193,25 ± 20,09	51,70 ± 12,13	699,52 ± 20,85	63,38 ± 8,34
Soluble COD (g O_2/L)	18,01 ± 0,72	6,89 ± 0,61	40,40 ± 1,8	15,74 ± 2,65	44,46 ± 0,38	28,80 ± 1,14
Alcalinity (mg CaCO3/L)	1940 ±50,00	3980 ± 10	3250 ± 490	6160 ± 280	2100 ± 1340	10100 ± 1270
Total Organic Carbon (ppm)	4547,5 ± 139,3	1105,0 ± 5,7	10652,0 ± 1566,9	4430,5 ± 38,9	16060,0 ± 1442,5	4729,5 ± 2,1
C/N ratio	15,17	1,08	30,23	2,42	26,33	2,86
Total acidity (mg acetic/L)	1384,3	111,7	834,3	210,5	3475,4	154,5

TABLE III-B INITIAL AND FINAL VALUES OF THE PHYSICOCHEMICAL PARAMETERS ANALYZED IN THE MESOPHILIC TESTS (35° C)

PARAMETER	**T-25CM-75SS**		**T-50CM-50SS**		**T-75CM-25SS**	
	Beginning	**Ending**	**Beginning**	**Ending**	**Beginning**	**Ending**
pH (upH)	5,52	7,30	5,09	7,43	5,20	7,50
Total Solids (%)	5,22 ± 0,08	3,93 ± 0,07	9,42 ± 0,07	6,92 ± 0,11	13,00 ± 0,09	9,86 ± 0,21
Volatile Solids (% VS)	76,90 ± 0,02	71,43 ± 0,51	81,83 ± 0,14	72,60 ± 0,32	79,00 ± 0,06	72,60 ± 0,32
Ammonium (mg NH3-N/kg)	109,12 ± 7,17	649,24 ± 13,58	113,85 ± 4,51	672,48 ± 18,25	257,60 ± 4,75	785,68 ± 50,69
Total nitrogen (% s/ww)	2,94 ± 0,09	26,19 ± 9,38	1,57 ± 0,16	14,55 ± 5,74	1,74 ± 0,45	2,99 ± 0,74

Total COD Total (g O_2/L)	78,13 ± 4,17	55,07 ± 4,55	193,25 ± 20,09	52,77 ± 1,52	699,52 ± 20,85	42,34 ± 3,60
Soluble COD (g O_2/L)	18,01 ± 0,72	10,96 ± 0,23	40,40 ± 1,8	17,75 ± 0,0	44,46 ± 0,38	25,58 ± 0,0
Alcalinity (mg CaCO3/L)	1940 ±50,00	5640 ± 10,00	3250 ± 490	10050 ± 40	2100 ± 1340	19300 ± 7210
Total Organic Carbon (ppm)	4547,5 ± 139,3	3730,5 ± 16,3	10652,0 ± 1566,9	5426,0 ± 162,6	16060,0 ± 1442,5	9855,5 ± 92,6
C/N ratio	15,17	1,58	30,23	2,89	26,33	14,08
Total acidity (mg acetic/L)	1384,3	1310,0	834,3	3246,3	3475,4	2504,1

Table IV shows the values of the operating parameters of the tests under different operating conditions, which are discussed below.

In the thermophilic conditions, VS removal was greater in tests that have higher content of cattle manure, reaching 44% VS removal in the T-75CM-25SS test. In mesophilic range similar trends were observed, although less marked than in thermophilic conditions. Thus, the maximum VS removal was produced in M-75CM-25SS test was 38%. For the mixture M-25CM-75SS, the VS removal was aprox. 36%, and the COD and TOC removal were only 39% and 18% respectively, exceeding the values reaching in M-50CM -50SS test, with 56% CODr and 49% TOCr.

TABLE IV BIODEGRADATION YIELDS OF MESOPHILIC AND THERMOPHILIC TESTS

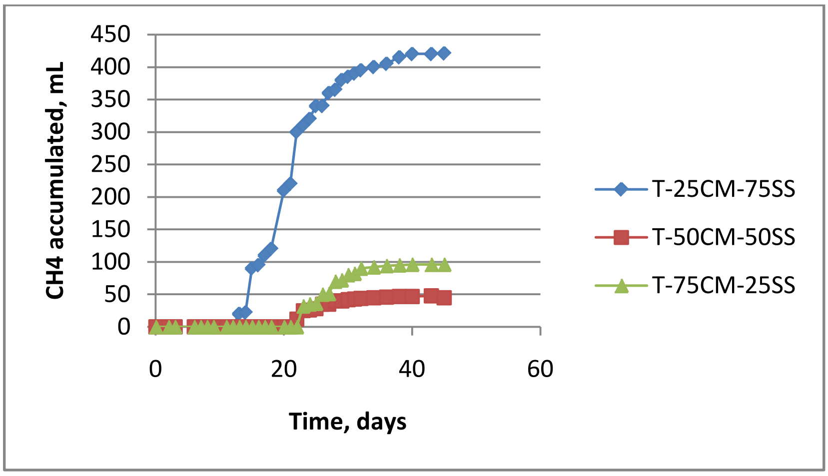
Parameter	**Mesophilic**			**Thermophilic**		
	M-25CM-75SS	M-50CM-50SS	M-75CM-25SS	T-25CM-75SS	T-50CM-50SS	T-75CM-25SS
% VS removal	35,6	34,8	38,3	37,4	35,8	43,9
CH_4 accumulated (mL)	29,6	105,6	30,3	2246,92	673,2	808,8
CH_4 yield (mL CH_4/gVSr)	4,25	8,75	1,51	306,27	54,1	35,2
Start up period (days)	12	18	18	12	21	21

pH and alkalinity values increased in all mixtures test. The higher alkalinity values were found for T-50CM-50SS test.

In relation to the VFA contents, all acids were consumed almost completely in both mesophilic and thermophilic tests. Total acid value declined more than 90-95% in the all the tests.

Table IV shows the volumes of methane accumulated reached in each essay. As shown, the value is particularly high in T-25CM-75SS test, exceeding 2 liters accumulated, and decreases significantly when increasing the amount of manure present in the sample. In general, the volumes of methane generated in thermophilic tests were higher than those achieved in mesophilic range for all mixing conditions. In order to consider the initial value of organic matter in each test and to establish appropriate comparison between different tests, the values of specific production of methane were calculated (ml CH_4 per gram of VS removed). Figure 3 shows methane yields, expressed as mL of CH_4 per gram of CODs removal, for the test at thermophilic and mesophilic conditions, respectively.

As can be seen in Figure 3, thermophilic tests were more efficient than mesophilic tests for samples with the same composition. For thermophilic tests, digester 25CM-75SS was the most efficient in terms of starting time (the generation of biogas starts at 12 days after inoculation) and in methane yield (showing 450 mLCH_4/gCODr). The difference with the other two thermophilic tests was very marked, with an advance of 10 days in start-up time and methane yield values between 5 and 10 times higher than T-50CM-50SS and T-75CM -25SS tests, respectively.



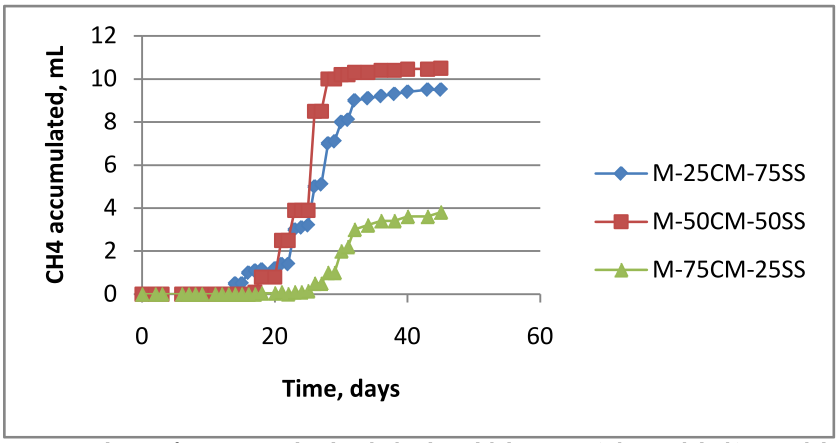


Figure 3 Evolution of CH_4 accumulated in the biodegradability tests: a) thermophilic b) mesophilic

At mesophilic conditions, digesters M-25CM-75SS and M-50CM-50SS showed a similar pattern with values close to 10 mLCH_4/CODr, which is higher than that obtained in the M-75CM-25SS test. In any case, the yields are much lower than in thermophilic assays. The starting period is 10 days for the mixture M-25CM-75SS, increasing for the other two tests.

In general, the percentage of CH_4 increased in all tests until to reach a maximum of 90% in T-50CM-50SS and T-75CM-25SS tests, suggesting that the manure concentration in the mixture increased the percentage of methane in the biogas. According to Misi and Forster [18], these high percentages of methane are attributable to the dissolution of CO_2 in the environment due to high alkalinity values in the digesters. So, the final values of alkalinity increased with the content of manure in the mixture, as shown in Table III.

Based on experimental results we can say that the thermophilic anaerobic co-digestion of cow manure and mixed-sludge treatment plant was more efficient compared to the mesophilic conditions, achieving better efficiency of organic removal and methane generation (Table IV). Highlight that the performance of the thermophilic process was several orders of magnitude larger than the mesophilic process. On the other hand, the more suitable mixing conditions for co-digestion process were 25%CM-75%SS, showing the most favorable results for all parameters associated with the consumption of organic matter and generation of biogas in thermophilic conditions and the shortest starting period (10 days).

B. Essays with Stirring Conditions

The values obtained for the initial characterization of each substrate are presented in Table V. As can be seen, there is a remarkable difference in the contents of C and N in both co-substrates. Thus, the co-digestion of these substrates can improve the C/N ratio, leading to values near the optimum range.

TABLE V PHYSIC-CHEMICAL CHARACTERIZATION OF CATTLE MANURE AND SEWAGE SLUDGE

	Cattle Manure		Sewage Sludge	
Parameter	**Average**	**Standard Deviation**	**Average**	**Standard Deviation**
TS (%)	18,24	0,54	1,43	0,29
VS (% TS)	83,37	0,49	69,73	3,67
KTN (% s/dry weight)	1,82	0,09	8,59	1,18
COD_T (g O_2/L)	98,21	3,62	15,71	2,34
COD_S (g O_2/L)	64,15	13,81	0,59	0,30
TOC (ppm)	19235,00	975,81	117,99	8,12
C/N ratio	26,57	nd	4,71	nd

Table VI summarizes the main results obtained in the tests (S-T-25CM-75SS and S-M-25CM-75SS). Results presented are related to the decrease in organic matter content (VS, COD and TOC removals), the main constituent of the substrates tested, and on the performance in methane production (CH_4 accumulated production (L/Ldigester), CH_4 yield (LCH_4/kg CODr), Specific CH_4 generation, (L CH_4/kgVSr).

TABLE VI MAIN RESULTS OBTAINED IN THE CO-DIGESTION OF CATTLE MANURE AND RAW SLUDGE AT THEMOPHILIC AND MESOPHILIC CONDITIONS

Parameter	Mesophilic	Thermophilic
Start up time (days)	5	3
% VS removal	23,4	22,9
% CODs removal	54,7	50,0
% DOC removal	43,6	23,7
CH4 accumulated generation (L/Ldigester)	0,83	3,35
CH_4 yield (L CH_4/kg CODr)	28,6	320,7
Specific CH_4 generation (L CH_4/kgVSr)	56,3	215,4

Table VI shows that temperature has a strong influence on the biodegradation processes. The obtained results were very different according to the working temperature range. However, the organic load removal efficiencies (as %VS removal and %CODs removal) were very similar for test, although showed slightly higher values for mesophilic treatment. However, thermophilic treatment showed better results than mesophilic in all parameters related to the biogas generation (CH_4 accumulated production (L/Ldigester), CH_4 yield (L CH_4/kg CODr), specific CH_4 generation, (L CH_4/kgVSr).

Figure 4 shows the evolution in the volume of methane generated at thermophilic compared to mesophilic conditions.

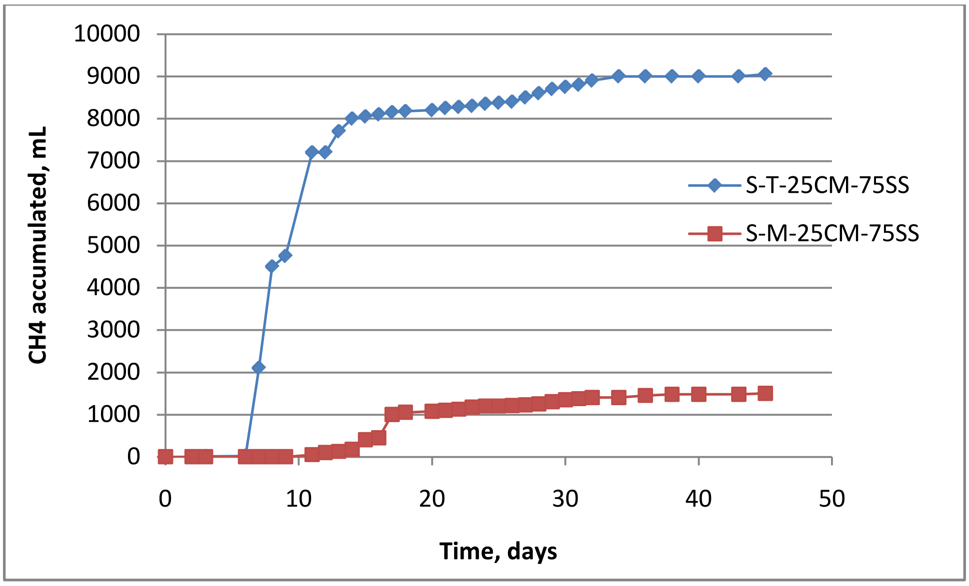


Figure 4 CH_4 accumulated for mesophilic (S-M-25CM-75SS) and thermophilic (S-T-25CM-75SS) tests

The best results were obtained for thermophilic tests, showing a greater biogas volume (3,35 L/L) with less starting time of the process (3 days).

IV. CONCLUSIONS

The co-digestion of cattle manure and mixed sewage sludge is a viable treatment at both mesophilic and thermophilic conditions. However, this co-digestion is more efficient at thermophilic than mesophilic conditions: higher organic matter removal with higher performance in methane production.

The thermophilic anaerobic co-digestion process has been more efficient for the mixture with 25% v/v of cattle manure and 75% v/v sewage sludge. The parameters evaluated to support this conclusion are based on the removal organic matter efficiency (62% and 75.7% removal of COD and TOC) and methane yield (2200 ml and 306 ml CH_4/gCODr CH_4/gVSr), presenting a start-up period of 12 days without stirring processes.

Also, it is found that a greater amount of cattle manure in the mixture involved more alkaline and a higher percentage of methane in the biogas.

Validation tests with stirring confirm the optimal composition of the mix selected thermophilic conditions allow to reach values three times higher than those obtained in mesophilic conditions for all parameters associated with the generation of biogas.

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