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SOLUBILITY, BIODEGRADABILITY, AND METHANE PRODUCTION CAPACITY OF WASTE SLUDGE PRETREATED BY THERMAL PRE-HYDROLYSIS, ELECTRO-OXIDATION AND BIOAUGMENTATION

Patricia Catalina López Vargas
Departamento de Ciencias Básicas,
Universidad Autónoma Metropolitana-Azcapotzalco
Ciudad de México, México

María Teresa Castañeda Briones
Departamento de Ciencias Básicas,
Universidad Autónoma Metropolitana-Azcapotzalco
Ciudad de México, México

Jorge Estrada Meléndez
Instituto Politécnico Nacional, Sección de Estudios de Posgrado e Investigación-ESIA UZ IPN
Ciudad de México, México

Marisol Espinoza-Castañeda
Departamento de Ciencias Básicas,
Universidad Autónoma Metropolitana-Azcapotzalco
Ciudad de México, México

Abstract: Waste sludge from wastewater treatment plants is a problem due to their high production, treatment, and management. The aim of this research was to compare the effect of three pretreatments on residual sludge to increase the degree of solubilization and the specific production of methane in subsequent anaerobic digestion. Thermal prehydrolysis (TP) of the sludge at 90 °C was performed for 90 minutes, electrooxidation of the sludge (EOP) using platinum electrodes with a current intensity of 400 mA for 45 minutes and the addition of 9 mL of specific strains of acidogenic bacteria, bioaugmentation pretreatment (BP). The degree of solubilization was increased in 34.7, 28.4 and 0.9 % with TP, EOP, and BP respectively. The efficiencies obtained for TP were 83.8 % for chemical oxygen demand reduction (COD), 79.3 % for volatile solids (VS) and a methane efficiency of 61.7 CH_4/g VS. Likewise, the EOP pretreatment showed efficiencies of 65.1 % in COD, 59.8 % in VS and a value of 16.1 CH_4/g VS; and in the pretreatment by bioaugmentation BP was obtained 59.6 % in removal of COD, 49.3 % for VS and an efficiency of 2.3 CH_4/g VS.
Keywords: Anaerobic digestion; thermal prehydrolysis; electrooxidation; bioaugmentation; biogas production.

INTRODUCTION

For efficient methane production and rapid anaerobic degradability of waste sludge from Wastewater Treatment Plants (WWTP), it is important to have a balance in the degree of reaction of the different stages involved which are hydrolysis, acidogenesis, acetogenesis and methanogenesis (Meegoda et al., 2018).

The speed of the anaerobic degradation process is limited by the speed of the slower stage, which depends on the composition of each residue. For residues in which the organic matter is in the form of particles, the limiting phase is hydrolysis, this limitation makes the process times on the order of weeks (Carrere et al., 2008).

Anaerobic digestion has been used not only for stabilization of waste sludge, but also for energy production from biogas rich in methane, which has a potential of 0.31 m^3/kg of total solids (TS), it is estimated that each cubic meter of biogas produced by anaerobic digestion generates 1.6 kilowatt-hours (Mao et al., 2015; Meegoda et al., 2018). Tests have also been carried out on the effect of the addition of specific strains of bacteria to the anaerobically digested sludge by performing the so-called vat foot, which consists of replicating larger volumes of acclimatization to obtain an inoculum rich in young cells (Bagi et al., 2007; Foladori et al., 2010). The advantage of using a pure inoculum is the efficiency in the production of such biogas and the avoidance of the presence of undesirable microorganisms. In this regard, various treatments have been used to eliminate those bacteria that do not favor the production of biogas and thus maintain in greater proportion those that do (Sandoval et al., 2009).

Thermal prehydrolysis (TP) is one of the pretreatments with the best results, it allows the partial solubilization of organic matter, the sanitation of the sludge and reduces the viscosity, with a subsequent improvement in the management and use of the sludge (Bougrier et al., 2008). In this sense, most research has focused on performing thermal pretreatment at high temperatures ranging from 120 °C to 200 °C at different times ranging from 30 to 60 minutes; however, there are two significant disadvantages for these conditions, the first being the high energy levels required and the second that at temperatures above 180 °C there is the formation of non-biodegradable refractory compounds (Ruiz, 2013). Therefore, the application of heat treatment at low temperatures (<100 °C) is an alternative that is being investigated to overcome these drawbacks.

Among the oxidative chemical pretreatments, the use of electrooxidation processes (EOP) has been reported, which improve the characteristics of the sludge prior to its conditioning or anaerobic digestion. Some of the advantages of EOP are: improvements in sludge dehydration (Yuan et al., 2010); reduction in volatile solids (VS) of sludge with short pretreatment times (15 to 30 minutes) in ranges from 0.5 to 2.0 A, a reduction in retention time of the subsequent anaerobic process from 23.5 to 17.5 days (Song et al., 2010) and reduction of organic matter concentration by 27 %, as well as modification of the physical characteristics of the sludge (Barrios et al., 2015).

This study aims to optimize anaerobic degradation and methane production to reduce the costs associated with the treatment and disposal of sewage sludge and obtain the most renewable energy from these by-products of wastewater treatment. To do this, three pretreatments were initially applied, one chemical by electrooxidation, one thermal with medium temperatures and one biological with the addition of isolated and specific microorganisms to digest the sludge generated in a WWTP, as well as a combination of these pretreatments to know their effectiveness together and with the aim of increasing the removal efficiency of VS and chemical oxygen demand (COD), as well as obtaining a greater amount of biogas rich in methane in the subsequent anaerobic digestion of them.

MATERIALS AND METHODS

Pretreatments were performed on a sample of sewage sludge from a municipal wastewater treatment plant. The sample consisted of a mixture in proportion (1:1) of primary residual sludge and secondary residual sludge for use as a substrate for subsequent anaerobic digestion.

The first pretreatment called TP consisted of heating the sludge mixture for 90 minutes at a temperature of 90 °C, for which Erlenmeyer flasks with a capacity of 500 mL and electric heating grills were used.

In the second pretreatment called EOP, an electrophoretic batch tank with a capacity of 300 mL was used, using platinum electrodes with a working area of 7.5 cm^2, at 15 cm of distance. The electrodes were connected to a power source with a capacity of 400 mA (BIO-RAD Power Pack 3000), which supplied direct current for 45 minutes at 97 V, 400 mA and 39 W, current density 53.3 mA/cm^2.

For the third pretreatment called bioaugmentation pretreatment (BP) 9 mL of pure inoculums composed of acidogenic and acetogenic bacteria that were obtained by isolating microbial populations following the methodology used by Sandoval et al. (2009) for groups of glucose fermentation bacteria and lactate (GFB), for propionate acetogenic bacteria, format, and ethanol (PAFEB).

With these three different pretreatments and their combinations between them, 8 different tests were performed, using a blank without pretreatment to sludge **(WP)**, **TP**, **EOP**, **BP**, TP followed by EOP **(TEP)**; TP followed by BP **(TBP)**; EOP followed by BP **(EBP)** and finally the combination of TP plus EOP plus the addition of BP **(TEBP)**.

At the beginning and at the end of the tests, the Total Oxygen Chemical Demand (tCOD) was measured using a closed reflux technique using an HACH DRB200 digester, and an HACH DR/890 colorimeter was obtained for reading; The soluble COD (sCOD) was also measured, and centrifugation at 6000 rpm/5 min was performed prior to digestion.

The degree of solubilization (DS) of organic compounds was calculated according to Equation (1) (Vigueras et al., 2013).

$$DS = \frac{sCOD\ treated - sCOD\ crude}{tCOD\ crude - tCOD\ crude} * 100 \ldots\ldots\ldots(1)$$

Where: DS = degree of solubilization expressed in percentage, sCOD treated = mg sCOD/L with pretreatment, crude sCOD = mg sCOD/L without pretreatment, crude tCOD = mg COD/L without pretreatment.

At the end of the pretreatments, anaerobic digestion of the sludge was carried out. Triplicate tests were performed using WP as substrate, and the combinations of sludge pre-treated by TP, EOP and BP. To do this, serological bottles with a capacity of 100 mL were used, 80 mL of sludge sample was inoculated by 20 mL of anaerobic granular biomass, then gassed with N_2 and CO_2 in a ratio of 80:20 and sealed with rubber plugs, which allowed the use of hypodermic needles to measure excess gas and to be able to take samples. The bottles were kept at room temperature for a period of 30 days. Figure 1 shows the experimental design and the graphical representation of the contents of each bottle. The parameters analyzed during the anaerobic digestion period were total COD by closed reflux technique, pH by potentiometer, volatile solids (VS) by gravimetry, biogas production by displacement of water column. These parameters were measured every third day and biogas composition at the end of the stabilization stage only.

RESULTS AND DISCUSSION

The comparison of results between the different treatments was performed in terms of COD reduction efficiency, as reduction efficiencies and biogas production and was complemented with the analysis of the behavior of the pH variables, solubility, and composition of biogas.

In Figure 2 the percentages of solubility after pretreatment of the sludge in each test can be seen.

TP and EOP increased the amount of sCOD in all trials due to cell rupture and intracellular material release. It can be seen that the highest DS was obtained in the TPE, by applying the combination of thermal pretreatment followed by electrooxidation pretreatment, with a 45 % increase in sludge solubility. TP and EOP also showed a notable increase in solubility (34.7 % and 28.4 % respectively. On the other hand, BP presented a very low degree of solubility. However, the results of this research are lower than reported by Vigueras et al. (2013) using an autoclave at 120 °C for 15 minutes obtained 38 % on the solubilization of sewage sludge.

CHEMICAL OXYGEN DEMAND

Table 1 presents the statistical summary of the chemical oxygen demand response variable COD, which was evaluated for the performance of organic and inorganic matter reduction in the different treatments.

The behaviour of the COD reduction is shown in Figure 3. All treatments showed a decrease in this parameter; the best results that presented the highest load eliminated with an average reduction efficiency were obtained with the TEP with 87.1 %. In general, all trials where thermal pretreatment was performed obtained the highest percentages of reduction (TP 83.5 %, TBP 85.1 %, TEBP 83.8 %), Trials where electrooxidation was used also presented a significant reduction (EOP 65.1 %, EBP 71.3 %), while BP showed a lower average reduction of 59.6 % and WP crude sludge the lower reduction of 51.1 %.

VOLATILE SOLIDS

Table 2 shows the statistical summary of the response variable VS, which was evaluated for the performance of the reduction of organic matter in the different treatments.

Figure 1. Experimental design with 8 different combinations of pretreatments used in this research.

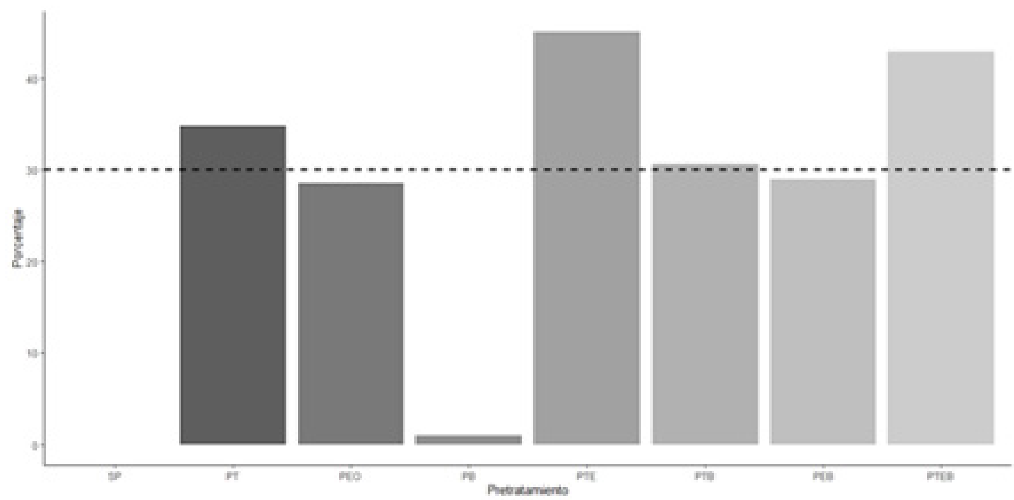


Figure 2. Percentages of solubility after pretreatment of sludge.

Variable	Units	Average	σ	media	minimum	maximum	n	% Reduction
WP	mgO_2/L	962.0	191.2	954	508	1275	15	51.1
TP	mgO_2/L	403.3	279.9	253	168	1125	15	83.5
EOP	mgO_2/L	771.2	223	786	401	1185	15	65.1
BP	mgO_2/L	882.8	176.7	865	465	1260	15	59.6
TEP	mgO_2/L	339.8	289.3	196	124	1129	15	87.1
TBP	mgO_2/L	455.8	283.9	342	102	1198	15	85.0
EBP	mgO_2/L	582.2	256.9	545	241	1293	15	71.3
TEBP	mgO_2/L	494.3	329.5	321	128	1175	15	83.8

Table 1. Chemical oxygen demand evaluated in the treatments WP, TP, EOP, BP, TPE, TBP, EBP and TEBP.

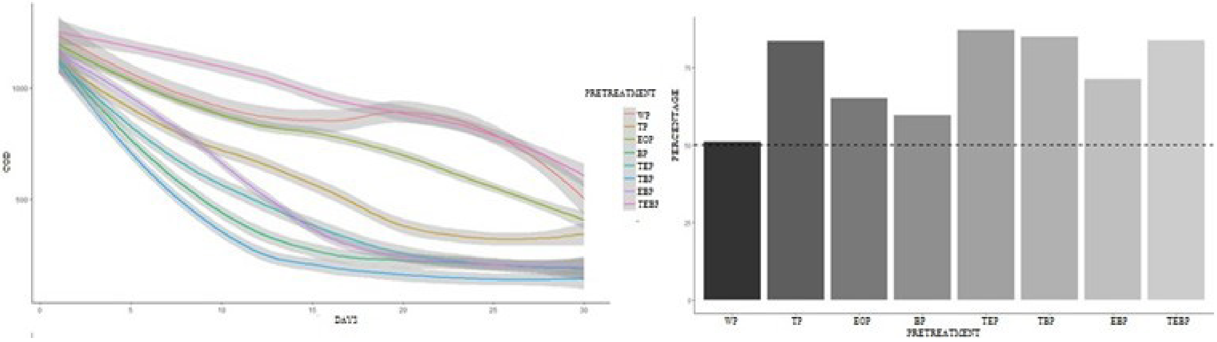


Figure 3. Behavior of COD evaluated in treatments WP, TP, EOP, BP, TEP, TBP, EBP and TEBP.

Variable	Units	Average	σ	media	minimum	maximum	n	% Reduction
WP	g/L	1.48	0.31	152	0.98	1.95	10	45.0
TP	g/L	1.18	0.52	1.34	0.35	1.84	10	79.3
EOP	g/L	1.15	0.32	1.14	0.65	1.77	10	59.8
BP	g/L	1.39	0.30	1.38	0.86	1.89	10	49.3
TEP	g/L	0.81	0.47	0.58	0.31	1.65	10	79.1
TBP	g/L	1.00	0.46	0.84	0.51	1.86	10	71.3
EBP	g/L	1.25	0.35	1.27	0.76	1.82	10	55.0
TEBP	g/L	0.92	0.41	0.88	0.38	1.85	10	75.6

Table 2. Volatile solids evaluated in treatments WP, TP, EOP, BP, TEP, TBP, EBP and TEBP.

Secondary sludge, because of its higher content of VS, allows larger removals than primary sludge. The typical reduction of VS in an anaerobic mixed sludge digester (primary plus secondary) range from 45 % to 60 %. Figure 4 shows the behavior in the reduction of VS during treatments WP, TP, EOP, BP, TEP, TBP, EBP and TEBP.

The TP applied to the primary and secondary sludge mixture benefited the high elimination of VS, this can be seen in the removal efficiencies achieved in all trials where TP was used. The greatest reduction was observed in TEP (79.3 %) and TP (79.1 %). The lowest reduction of VS was shown in WP crude sludge with 45 %, EOP showed removals of 60 % on average and BP removals of 49 %.

pH

The pH varied between 7.60 and 6.3 after performing the pretreatments, remaining within the optimal range of 6.0 to 8.5 for the development of biological degradation processes (Lennin et al., 2009). Figure 5 shows the behavior of this parameter during the digestion stage in each of the treatments.

The literature mentions that the main problem that occurs during the start-up is the acidification of the reactor (Callejas et al., 2019). In all trials where pretreatment with biological inoculum was used, acidification occurred; in the BP values of 5.35 were reached, in the TBP of 5.09, the EBP showed low values of 4.79 and finally in the TEBP the minimum values of 4.50 were found on day 27. However, this acidification did not occur in the trials in which the addition of micro-organisms was not performed. The pH variation was lower in WP crude sludge, from 7.13 to 6.24, followed by TP from 6.89 to 6.05, EOP with a range of 6.5 to 6.28.

BIOGAS

The statistical summary of the volume of biogas generated during treatments is presented in Table 3.

Table 4 shows the composition of biogas.

Once the digester was stabilized, the biogas generated in the process (volume and composition of biogas as a function of the eliminated VS) was quantified with respect to the feed load. Municipal anaerobic digesters produce methane with a range of 0.75-1.0 m^3/kg of volatile degraded solids under mesophilic conditions. Figures 6 and 7 show the results of biogas production and its efficiency in methane production.

The results of biogas production from the evaluated sludge show that in the trials without pretreatment there was a significant production of methane of 56.2 % with a yield of 28 mL CH_4/gVS removed; in TP trials the production was higher than for untreated trials showing 79 % methane in biogas and 61.7 CH_4/gVS removed. In TEP trials, methane production was very high, 84.9 % and 60.6 CH_4/gVS removed. For the EOP, 60.9 % methane was obtained, but a yield of 16.1 CH_4/gVS removed. Finally, for bioaugmentation tests, very low methane percentages of 6.4, 10.15, 8.35 and 1.9 % were obtained for BP, TBP, EBP and TEBP respectively, which was reflected in low biogas yields. This is because the acidification conditions that occurred inhibited almost completely the methanogenic bacteria that inhabited the inoculum sludge, which was reflected in the decrease in methane production.

These results show that methane production from untreated sludge samples requires a longer digestion period to biotransform biodegradable organic matter to methane. Thermal pretreatments proved to be the best in terms of reduction of COD, VS and in the production of biogas rich in

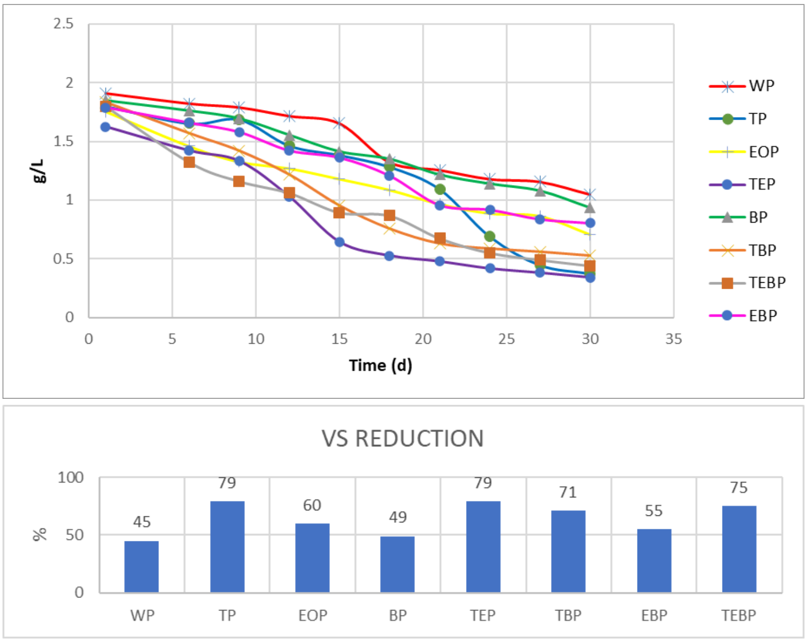


Figure 4. Behavior of volatile solids evaluated in treatments WP, TP, EOP, BP, TEP, TBP, EBP and TEBP.

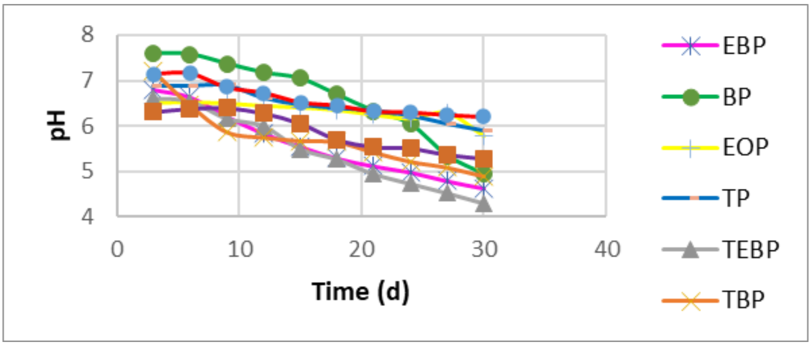


Figure 5. Behavior of pH in treatments EBP, BP, EOP, TP, TEBP, and TBP.

Variable	Units	Average	σ	media	minimum	maximum	n
WP	mL/d	16.1	8.7	15	3	32	15
TP	mL/d	27.6	21.6	24	0	63	15
EOP	mL/d	19.7	13.1	13	3	42	15
BP	mL/d	11.2	10.5	6	0	30	15
TEP	mL/d	23.8	19.3	20	0	56	15
TBP	mL/d	19.6	16.8	13	0	46	15
EBP	mL/d	19.4	7.7	20	3	35	15
TEBP	mL/d	20.6	20.5	12	0	56	15

Table 3. Volume of biogas generated during treatments WP, TP, EOP, BP, TEP, TBP, EBP and TEBP.

Biogas	Nitrogen		Carbon Dioxide		Methane	
Sample	Quantity [mmoles]	Quantity [%]	Quantity [mmoles]	Quantity [%]	Quantity [mmoles]	Quantity [%]
WP	0.001145	9.75	0.010565	34.05	0.013855	56.2
TP	0.000525	2.05	0.00483	18.95	0.020165	79
EOP	0.00057	1.85	0.011545	37.2	0.018915	60.95
BP	0.00145	7	0.017985	86.6	0.00132	6.4
TEP	0.000455	1.9	0.003175	13.2	0.02048	84.9
TBP	0.00062	3.2	0.016955	86.65	0.001985	10.15
EBP	0.000595	2.85	0.01754	88.8	0.001645	8.35
TEBP	0.000405	2.35	0.01623	95.75	0.000225	1.9

Table 4. Biogas composition generated in the treatments WP, TP, EOP, BP, TEP, TBP, EBP and TEBP.

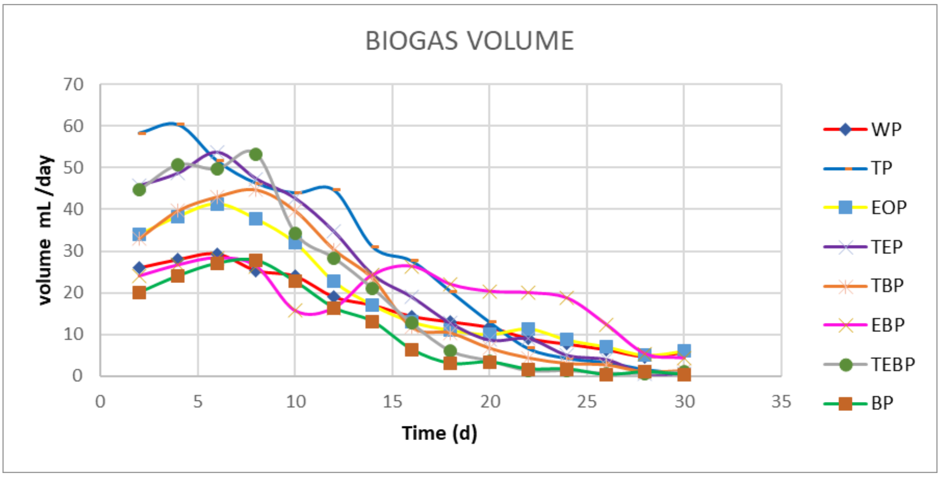
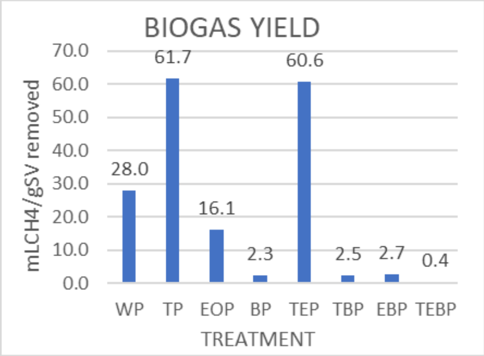


Figure 6. Volume of biogas generated in the treatments WP, TP, EOP, TEP, TBP, EBP, TEBP and BP.



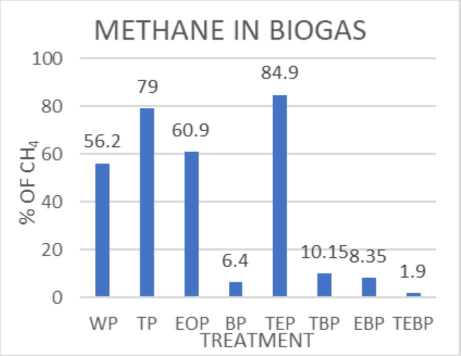


Figure 7. Biogas performance through methane efficiencies during treatments with WP, TP, EOP, BP, TEP, TBP, EBP and TEBP.

methane. This because of having decreased the particle size in a pretreatment making the organic matter more soluble, to obtain an adequate affinity of the substrate with the biomass.

CONCLUSIONS

Based on the results obtained in this research it can be concluded that the pretreatments applied to the sewage sludge met satisfactorily improving the anaerobic digestion process. The TP applied under 90 °C conditions for 90 minutes favored the hydrolysis of the macromolecules, until obtaining a degree of solubilization of 34.7 %, with the EOP applying 400 mA for 45 minutes was obtained a solubilization percentage of 28.4 %, and with the BP adding 9 mL of ACB, FGB and MET the sludge acquired 0.9 % solubilization.

COD removal efficiencies were 87.1, 85.1, 83.8, 83.5, 71.3, 65.1, 59.6 and 51.1 % for TEP> TEBP> TP> TBP> EBP> EOP> BP> WP respectively. VS removal efficiencies were 79.3, 79.1, 75.6, 71.3, 59.8, 55, 49.3 and 45 % for TP> TEP> TEBP> TBP> EOP> EBP> BP> WP respectively. The percentage increase in biogas production with respect to white WP was 71.4, 47.8, 27.9, 22.3, 21.7, 20.4 and -30 % for TP>TEP>TEBP>EOP>TBP>EBP>BP respectively. The percentage of methane in biogas was 84.9, 79, 60.9, 56.2, 10.1, 8.3.6.4 and 1.9 % for TEP>TP>EOP>WP>TBP>EBP>BP>TEBP, respectively.

In general, all the trials where TP was performed obtained the highest percentages of reduction of COD and VS, as well as the highest levels of production of biogas rich in methane. Tests using EOP electrooxidation also showed a significant reduction in COD and VS, while BP showed a lower reduction compared to WP, likewise the production of methane-rich biogas was very low in BP trials.

The best efficiencies obtained were in TP, with 83.8 % in COD reduction, 79.3 % in VS, 71.4 % in increase of biogas production, 79 % of methane in biogas and an efficiency of 61.7 mL CH_4/g VS removed, whereas in conventional digestion WP only 28 mL CH_4/gVS was removed.

This study showed that the combination of a pretreatment in anaerobic digestion of sewage sludge improves the operating time, has a low chemical requirement and is a viable alternative for the treatment of sewage sludge with high organic content.

It is recommended to analyze the effect that pretreatments have on the elimination of pathogenic microorganisms, perform a characterization of the electrodes in the EOP, performing an energy balance of the process to know if it is self-sustainable, the application of these strategies not to the production of biogas through anaerobic digestion, but to the generation of hydrogen. Once the laboratory-scale process has been studied in detail, it will be carried out on a pilot scale and optimized.

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