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BIOGAS PRODUCTION BY ANAEROBIC DIGESTION OF WASTEWATER FROM PALM OIL MILL INDUSTRY

PRODUCCIÓN DE BIOGÁS MEDIANTE DIGESTIÓN ANAEROBIA DE AGUAS RESIDUALES PROVENIENTES DE LA INDUSTRIA PALMERA

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ABSTRACT

The environmental impact caused by the fossil fuel use encourages society to look for new sources of renewable energy, such as biodiesel. During the last years, palm oil production has dramatically increased in Colombia, since it is the main raw material for biodiesel production. As consequence of the process, palm oil mill effluents with high content of pollutants are released to the environment. Since these effluents have physicochemical characteristics that make them suitable for the production of biogas by anaerobic digestion of residual water, this research evaluates the production of methane using wastewater as substrate from a Colombian palm oil mill. Anaerobic digestion experiments were conducted in batch mode to evaluate the influence of pH and inoculum to substrate ratio, by using two differents inoculums. It was found that the most suitable inoculum was a mixture of 1:1 v/v urban Wastewater Treatment Plant (WWTP) anaerobic sludge/pig manure at a ratio 2 g Volatile Solids (VS) inoculum/g VS substrate, which presented the highest accumulated methane production, reaching 2740 mL methane (0.343 m^3 CH_4/kg VS) without neutralizing pH.

Keywords: *Wastewater, Anaerobic digestion, Palm oil, Methane.*

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RESUMEN

El impacto ambiental generado por el uso de combustibles fósiles, incentiva a la sociedad a buscar nuevas fuentes de energía renovables tales como el biodiesel. En Colombia, la materia prima más utilizada para producir biodiesel es el aceite de palma, con lo que su producción ha aumentado drásticamente en los últimos años, generando efluentes con alta carga contaminante para el medio ambiente como consecuencia del proceso. Dado que las características fisicoquímicas de estos efluentes son propicias para la producción de biogas mediante digestión anaerobia, este trabajo evalúa la producción de metano a partir de agua residual de una empresa extractora de aceite de palma colombiana. Se realizaron experimentos de digestión anaerobia en modo batch para evaluar la influencia del pH y la relación inóculo/sustrato utilizando dos inóculos diferentes. Se encontró que la mezcla 1:1 v/v lodo anaerobio de planta de tratamiento de aguas residuales urbanas y estiércol de cerdo usada como inóculo, generó la mayor producción de metano acumulado, alcanzando 2740 mL de metano (0.343 m^3 CH_4/kg SV), usando una relación de 2 g SV de inóculo/g SV de sustrato, sin necesidad de neutralizar el pH del sistema.

Palabras clave: *Aguas residuales, Digestión anaerobia, Aceite de palma, Metano.*

RESUMO

O impacto ambiental gerado pelo uso de combustíveis fósseis, incentiva à sociedade a procurar novas fontes de energia renováveis, tais como o biodiesel. Na Colômbia, a matéria-prima mais utilizada para produzir biodiesel é o óleo de palma, devido a isso a sua produção tem aumentado drasticamente nos últimos anos, gerando efluentes com alta carga contaminante para o meio ambiente como consequência do processo. Dado que as características físico-químicas destes efluentes são propícias para a produção de biogás mediante digestão anaeróbia, este trabalho avalia a produção de metano a partir de água residual de uma empresa extratora de óleo de palma colombiana. Foram realizados experimentos de digestão anaeróbia em modo batch para avaliar a influência do pH e a relação inoculo/substrato utilizando dois inóculos diferentes. Encontrou-se que a mistura 1:1 v/v lodo anaerobio de tratamento de águas residuais urbanas e esterco de porco usada como inóculo, gerou a maior produção de metano acumulado, atingindo 2740 mL de metano (0,343 m^3 CH_4/kg SV), usando uma relação de 2 g SV de inóculo/g SV de substrato, sem necessidade de neutralizar o pH do sistema.

Palavras-chave: *Águas residuais, Digestão anaeróbia, Óleo de palma, Metano.*

1. INTRODUCTION

The environmental impact generated by the use of fossil fuels motivates the industry and society to search for new sources of renewable energy. Between them, biodiesel has demonstrated to be a biofuel with high potential to partially replace fossil fuels. In tropical and semitropical countries like Colombia, palm oil is used as the main raw material for biodiesel production.

The African palm culture in Colombia starts in 1945 with the *United Fruit Company*, in a banana cultivation zone in Magdalena state. This culture has extended until reaching 360000 hectare in 2010, making Colombia the first palm oil producer in Latinamerica, and 4th in the world. At present time, the territory designated for palm oil cultivation is distributed in 73 municipalities in 4 productive zones: North, Central, East and West (Lasso & Ramírez, 2011). Due to this, palm oil industrial sector has a unique opportunity to insert Colombia in a high position in the internal and external markets based on biofuels.

Several research projects are been carried out in the palm oil industry, conducted by *Federación Nacional de Cultivadores de Palma de Aceite (Fedepalma)*, the research association for this industrial sector. Between them, one of the most important is the Clean Development Mechanism Project (CDM) for methane capture, minimization of fossil fuels use and energy cogeneration. This project, approved in 2009 by the United Nations Organization, will have the capacity of generating each year 757067 green house gas emissions reduction certificates during the next 21 years. As raw material for cogeneration plant, several palm oil mill residues will be used. One of the objectives is to improve and adequate the oxidation ponds for Palm Oil Mil Effluent (POME) as anaerobic reactors for methane gas production, which can produce organic compost as well, which can be sold as organic fertilizer in the market.

The palm oil production process consist in sterilizing the fruits, separating them, macerating them, extracting the palm oil, clarifying it, and recovering the almonds from the resulting bagasse. As consequence, 0.7 tons of residual water per ton of processed fruit are generated (Tabatabaei *et al.*, 2010), generating environmental pollution problems due to the high organic load of the effluents. These effluents are mainly colloidal suspensions containing 95 - 96% H_2O, 0.6 - 0.7% oil, 4 - 5% of total solids from which 2 - 4% are suspended solids, pH 3.4 - 5.2 and high Chemical and Biological Oxygen Demand (COD and BOD) (Lam & Lee, 2011).

Based on this, the work presented here evaluates the potential production of methane using POME as substrate, with the objective of evaluating operational conditions for anaerobic digestion like pH and degradation time that maximize the methane production.

2. THEORETICAL FRAME

Anaerobic Digestion (AD) is one of the most efficient biological processes for treating both the organic fraction of residual solids, as well as the liquid effluents. In this process, organic compounds are solubilized and hydrolyzed by the microbial action to smaller compounds and Volatile Fatty Acids (VFA), which are then degraded into methane and carbon dioxide. The final products from AD are the recovery of energy (due to methane production) and biosolids with good physicochemical characteristics to be used as soil improver or organic fertilizer (Borzacconi, López & Viñas, 1995; Mata, Macé & Llabrés, 2000; Yu, Samani, Hanson & Smith, 2002).

The AD process is composed by four stages: hydrolysis, acidogenesis, acetogenesis and methanogenesis. Several environmental factors, such as pH, nutrients availability, temperature and redox potential affect the AD (Rittmann & McCarty, 2001). During AD process, the microbial consortium establishes a symbiotic equilibrium that generates the overall behaviour of the system, obtaining as consequence a certain conversion of organic carbon to methane (Pavlostathis & Giraldo, 1991). The modification of this equilibrium, by altering the stages sequence during separation, generates the possibility of establishing operational conditions that optimize all of them, allowing the improvement of the overall process (Mata *et al.*, 2000).

The application of the AD technology depends on the design, use and development of anaerobic reactors, and is determined by operational parameters that have direct influence on the metabolism of the microorganism, the kinetic of the reactions involved in the AD and the reactor design (Sharma, Unni & Singh, 1999). In order to improve the reaction velocity per unit of reactor volume, different reactor configurations have been tested, differing mainly in the immobilization and retention way for the microorganisms. The immobilization of biomass increases the system stability and the resistance to toxics action.

Depending on the fat, carbohydrate and protein content of the different substrates used for AD, the methane fraction contained in the biogas obtained varied between 50 to 75%. The composition of the biomass or substrate used determines the amount and final composition of the biogas produced. In general, fats and oils have a higher biogas yield (measured as m^3/ton VS), with an average methane content. Proteins have a comparative lower biogas yield, but with higher methane content. In the case of carbohydrates, they have an average yield and productivity of biogas, but with lower methane content. The concentration of solids is of importance for the biological process, for mixing effects and handling inside the reactor, as well as for the feeding system in the plant.

The different technologies and commercial systems for AD existent and emergent at industrial scale used in several countries like Japan, Switzerland, Spain, Austria, Germany, France, China, Italy and Belgium, present a biogas production between 0.10 to 0.15 m^3/kg of wet residue, while containing 50 to 70% methane in volume (Nichols, 2004).

3. MATERIALS AND METHODS

Physicochemical Characterization of Substrate and Inoculums

The wastewater (POME) used for the experiments was collected from a palm oil mill industry (San Alberto, Colombia).The physicochemical characterization of the substrate included pH, Total and Volatile Solids (TS, VS), Total and Volatile Suspended Solids (TSS, VSS), Volatile Fatty Acids (VFA), COD, BOD_5, Phenols, Fat and Oils content measurements. These analytics were carried out under *American Public Health Association* (APHA) standards (APHA, 1995).

Two different inoculums were used for the experiments. The first one (called PP inoculum) corresponded to a mixture composed by an anaerobic sludge coming from the municipal Wastewater Treatment Plant (WWTP) (Floridablanca, Colombia), and pig manure obtained from a pig processing plant (Los Santos, Colombia), in a proportion 1:1 (v/v) (Castillo, Cristancho & Arellano, 2006). The second one (called LP inoculum) corresponded to the anaerobic sludge coming from the oxidation ponds from the same palm oil mill industry that provided the substrate.

The physicochemical characterization of the inoculums included pH, TS and VS content, TSS and VSS content, VFA and alkalinity (Alk). These analytics were carried out under APHA standards (APHA, 1995).

The different inoculums were also evaluated in order to determine the Specific Metanogenic Activity (SMA) which is the own capacity of the inoculums to produce methane (Díaz, Espitia & Molina, 2002). The experiments were carried out in 500 mL reactors. To each reactor, an inoculum volume equivalent to a concentration of 1.5 g/L VSS, and a mineral *balch* medium were added until reaching 300 mL operation volume. The experiments were carried out by triplicates during 15 days at 37±2°C incubation temperature. The biogas volume produced was quantified by alkaline displacement method using a Mariotte's bottle filled with *NaOH* 0.1 N solution for CO_2 retention (Díaz *et al.*, 2002). The SMA was expressed in g COD/g VSS* day.

pH Influence on Biogas Production

To quantify the pH influence in the AD process, the biogas production was evaluated using POME as substrate, at an Inoculum to Substrate Ratio (ISR) 0.5 g VS inoculum/g VS substrate at two differents pH: 4.8 and 7. The pH 4.8 corresponded to the original pH from POME and the pH 7 was reached by neutralization of the reactor content by adding *NaOH* 5 N every two days in anaerobic conditions. Two blank samples were prepared by adding the same inoculum quantity used in the experiment and completing the volume with distilled water. The experiments were carried out using 500 mL batch reactors with 300 mL operation volume by triplicate. Methane production was evaluated during 30 days at 37±2°C, measuring the volume of methane produced by the alkaline displacement method (Díaz *et al*., 2002). The final volume of methane produced by the substrate was determined by subtracting the methane produced by the blank sample. The methane accumulated yield $(Y_{p/s})$ during the retention time is expressed in m^3 CH_4/ kg VS of substrate.

Inoculum to Substrate Ratio (ISR) Influence on Biogas Production

To evaluate the ISR influence on biogas production three different ISR levels were evaluated: 1, 1.5 and 2 g VS inoculum/g VS substrate. 500 mL bioreactors with a 300 mL operation volume were used for the experiments. The inoculum and substrate concentrations were varied in order to maintain the operation volume constant. The blank samples were prepared by adding the amount of inoculum corresponding to each ISR experiment, and completing the volume by adding distilled water to the reactor. Both biorreactors (test and blank) were incubated at 37±2°C during 20 days. The experiments were carried out by triplicate. In order to obtain the final methane volume produced by the substrate, the methane volume produced by the blank was subtracted. Several operational parameters were measured during the experiments, like VFA, VS, COD content and pH by destructive sample every 4 days. The methane volume obtained in the experiments was measured by the alkaline displacement method (Díaz *et al*., 2002).

The maximal methane accumulated yield $(Y_{p/s})$ during the retention time is expressed in m^3 CH_4/ kg VS substrate, and it was calculated dividing the methane accumulated during the whole AD time at standard pressure and temperature conditions, by the amount of substrate added (g VS) (Raposo, Banks, Siegert & Borja, 2006). The accumulated methane productivity (Q_p) was also determined in mL CH_4/day.

4. RESULTS AND DISCUSSION

Physicochemical Characterization of the Substrate (POME)

Table 1 presents the physicochemical characterization of POME wastewater. As it can be observed, it presents a high solids content and COD that makes it an appropriate substrate for biogas production. This wastewater presents acid pH (4.8), and for this reason, it is convenient to evaluate if AD at this pH is possible, due to previous research that indicated optimal pH values between 6.8-7.2 (Díaz *et al*., 2002). The other parameter values are in the range reported by previous authors (Chan, Chong & Law, 2010; Mustapha, Ashhuby, Rashid & Azni 2003; Poh & Chong, 2009).

Table 1. Physicochemical characterization of POME substrate.

Parameter	Value
pH	4.8
TS (mg/L)	48000±492.5
VS (mg/L)	10100±92.5
TSS (mg/L)	34000±175
VSS (mg/L)	6200±25
VFA (mg/L)	6700±9.34
COD (mg/L)	72800±2.57
BOD_5 (mg/L)	24000±10%
Fats and oils (mg/L)	90±10%
Phenols (mg/L)	4±10%

Physicochemical Characterization of the Inoculums

Table 2 shows the physicochemical characterization of both inoculums used in the experiments. The inoculum composed by WWTP anaerobic sludge and pig manure (PP) (1:1 v/v) presents a pH higher than 7 giving to the reactor an increment in this value, which probably will favour the AD process. In contrast, sludge from palm oil mill oxidation pond (LP) has a more acidic pH and a semisolid appearance; therefore, the VFA and Alkaline content could not be measured.

PP sludge presents a higher content of Methanogenic Acetoclastic Bacteria, which are the main microorganisms capable of generating 2/3 of methane produced, being the main responsible of the anaerobic degradation of organic matter (Castro, 2012; Quintero, 2011).

Table 2. Physicochemical characterization of inoculums.

Parameter	PP	LP
pH	7.6	6.3
TS	65400±535 (mg/L)	29.2±0.57 (%w/w)
VS	28700±275 (mg/L)	23.5±0.43 (%w/w)
TSS	74000±300 (mg/L)	90600±68.4 (mg/L)
VSS	32200±950 (mg/L)	27000±89.7 (mg/L)
VFA	1200±16.5 (mg/L)	N.A
Alk	3400±25.6 (mg/L)	N.A

Evaluation of Specific Methanogenic Activity (SMA)

The parameters used in the calculation of SMA are presented in Figure 1 and Table 3. According to the results of SMA obtained, PP inoculum presented 0.113 g COD/g VSS*day, which demonstrates that it contains higher amount of methanogenic microorganisms when compared to LP. The values found are in the range reported by Field (1987), who measured different parameters for different inoculums types, reporting a SMA range between 0.02-0.2 g COD/g VSS*day.

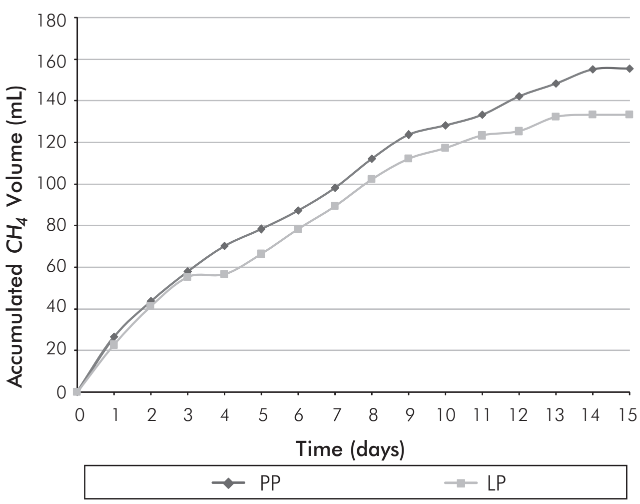


Figure 1. Kinetics of accumulated CH_4 production (mL) for each inoculum used. Initial solids concentration 1.5 g VSS/L pH= 7 T= 37±2°C.

Table 3. Evaluation of SMA for both inoculums.

Inoculum	SMA (g COD/g VSS*day)
PP	0.113
LP	0.086

pH Influence on Biogas Production

Figure 2 shows the kinetics of methane production for both different inoculums using POME as substrate. At pH 7 the accumulated methane volume was 778 mL for PP, and 786 mL for LP inoculum. On the other side, at pH 4.8 the accumulated methane volume was 647 mL for PP and 487 mL for LP inoculum. These results indicate that after neutralization at the beginning of the experimentation time, microorganisms adapted better to the medium, improving the starting up of the AD process, which consequently generates higher accumulated methane volume.

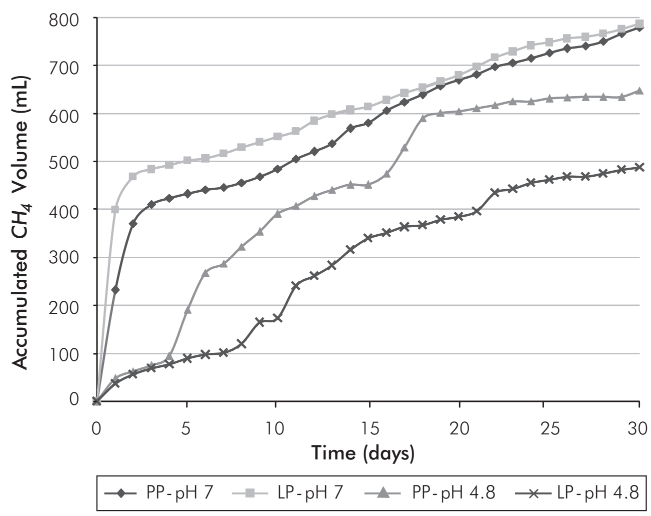


Figure 2. Kinetics of accumulated CH_4 production (mL) for both inoculums PP and LP at initial pH 4.8 and 7. ISR= 0.5 T= 37±2°C

Previous studies have demonstrated that AD at neutral pH increases the methane production because it favours the microorganism's growth, as well as avoids VFA accumulation which negatively affects the system stability (Poh & Chong, 2009; Chen, Cheng & Creamer, 2008). However, at the end of the AD process, for the experiments using PP inoculums,the difference in the final accumulated methane volume (at pH 4.8 and pH 7) is lower than the final accumulated methane volume obtained when using LP inoculum. That indicates that after the starting up, the AD process progresses at the same velocity in both cases. This can be attributed to the fact that PP inoculum has neutral pH (7.6), which contributes to the own microorganism's adaptability to the medium.

On the other side, when using LP as inoculum, it presents a higher difference in methane production at the two different pH used in the experiments. This is because LP inoculum is acidic (pH 6.3) in comparison to PP inoculum, which is not enough to neutralize the pH of the system, and consequently the AD system is unstable, producing lower amount of methane. Due to the difficulties in controlling the pH during the development of the AD process (in which the neutralization is necessary every two days) it was decided to continue working with both inoculums at the original pH of residual wastewater (pH 4.8), avoiding the continuous neutralization of the system inside the reactors.

ISR Influence on Biogas Production

The influence of ISR was evaluated on AD of POME in batch experiments. The different parameters affecting the AD process such as pH and VFA accumulation were measured. Due to this, they indicated acidification inside the reactor. The amount of inoculum and substrate to be added in each reactor were also evaluated.

Table 4 presents the accumulated methane volume and Q_p for both inoculums at the different ISR studied. The results showed that the higher methane production was obtained at ISR 2 using PP as inoculum, obtaining 2740 mL of accumulated methane volume. This demonstrates that at higher organic load of inoculum added to the reactor, the methane production increases, and consequently the volumetric productivity of methane per day.

From the data obtained,the percentage of VS removal was calculated, and it was related to the maximal accumulated methane volume obtained. As it can be expected for a typical AD process, while the VS concentration decreases, the accumulated methane volume production increases. This can be attributed to the operation time in which the organic matter degrades, which is used by the microorganisms for producing methane. As it can be observed, the % VS removal was in the range between 13.42 - 55.52% corresponding to LP at ISR 1 and PP at ISR 2, respectively. These data are concordant with the methane production results. The percentage of VS removal for PP at ISR 2 was higher than the reported by Forster, Pérez and Romero (2008), who studied the influence of solids in the AD process using urban solid residues as substrate, finding a value of 49.7% VS removal.

Figure 3 shows the kinetics of methane production at each ISR studied for both inoculums (PP and LP). The highest yield $(Y_{p/s})$ was obtained at ISR 2 using PP as inoculum, obtaining 0.343 m^3 CH_4/kg VS. This value was lower in comparison with that obtained with the same ISR but using fique pulp as substrate and a mixture between rumen fluid and pig manure as inoculum, reaching a maximal yield of 0.446 m^3 CH_4/kg VS (Quintero, 2011).

In the literature it was found that there is a linear relationship between methane yield and the ISR (Chen *et al.*, 2008; Chaiprapat & Laklam, 2011). Recent studies on the effect of ISR on AD of algae have demonstrated that methane yield decreased from 0.140 m^3 CH_4/kg VS to 0.094 m^3 CH_4/kg VS when the ISR was decreased from 2 to 0.5 (Dinsdale, Premier & Hawkes, 2000).

Table 4. Q_p, accumulated methane volume and % VS removal for the different ISR studied.

Methane Accumulated Volume (mL)			Q_p (mL CH_4/day)		% VS Removal	
ISR	PP	LP	PP	LP	PP	LP
1	1360±30	890±40	68±1.5	44.5±2	35.81	13.42
1.5	1922±28	947±17.5	96.1±1.4	47.4±0.88	51.82	31.70
2	2740±40	1411±9	137±2	70.5±0.45	55.52	44.60

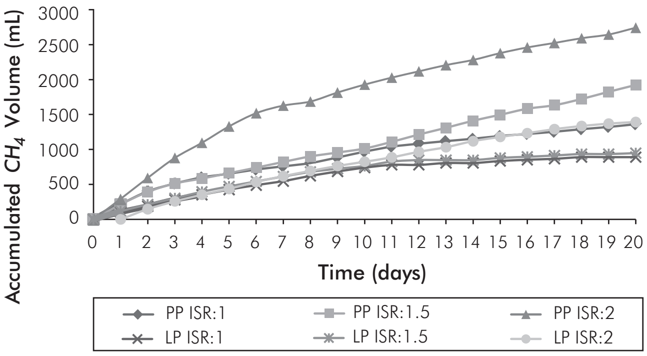


Figure 3. Kinetics of methane production at different ISR T=37±2°C Initial pH =4.8.

The pH is one of the most important parameters in the methane production process, because microorganisms are pH-dependent in order to correctly develop its metabolic activity (Chaiprapat & Laklam, 2011). It can be observed -in Figure 4- the pH evolution at different ISR in the experiments using both inoculums. Lower pH values during experiments were observed for the reactors inoculated with LP, caused by the lower initial pH of this inoculum. Higher pH values were observed in those reactors inoculated with PP.

During the whole degradation time, the experiments using PP inoculum at ISR 2 showed appropriate pH values, demonstrating that at neutral conditions the methane production increases, being coincident with the optimal range of pH 6.8-7.2 reported by previous studies (Lam & Lee, 2011; de La Rubia, Raposo, Rincón & Borja, 2009).

In Figure 5, the behaviour of VFA/Alk ratio during the experiments can be observed. In the case that the reactors were inoculated with LP at ISR 1 and 1.5, they presented acidification due to the fact that VFA concentration was in a range between 8000 - 8500 mg/L, showing a decrease in methane production. According to the literature, VFA concentration higher than 8000 mg/L causes reactor acidification and inhibits the methane production (Quintero, 2011).

pH stability during experiments using PP at ISR 1.5 and 2 can be attributed to the buffer capacity of the inoculum itself (Poh & Chong, 2009), represented by the Alk values between 10000 - 15000 mg $CaCO_3$/L for

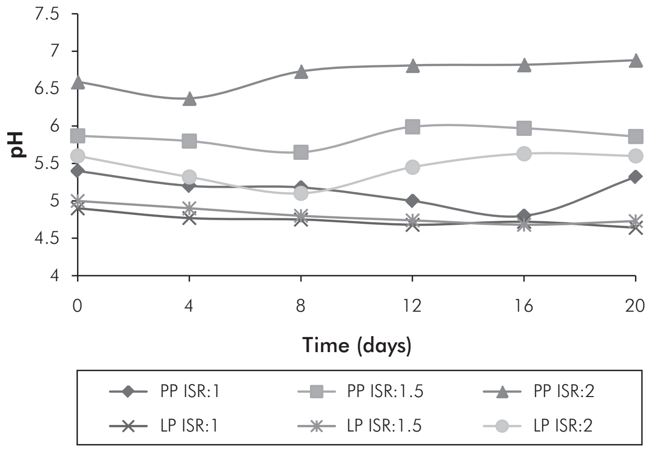


Figure 4. Evolution of pH at the different ISR studied using LP and PP inoculums. T= 37±2°C, Initial pH= 4.8.

PP inoculum at ISR 2. Apparently, the media and/or the microorganisms that are present in this inoculum exert a buffer effect over the bioprocess (Dinsdale *et al.*, 2000), as was previously evidenced by the pH influence evaluation.

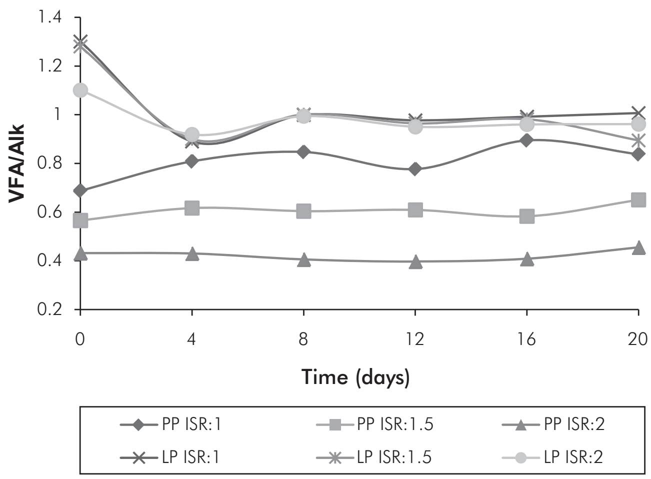


Figure 5. Evolution of VFA/Alk ratio at T= 37±2°C Initial pH= 4.8 for both inoculums.

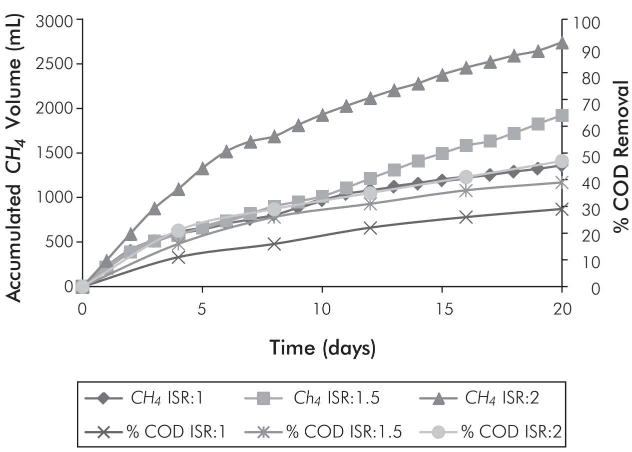
For an optimal operation of the AD process, the range for the VFA/Alk ratio should be between 0.1 - 0.4. These values are adequate for this type of systems in order to avoid acidification or inhibition during the AD process (Zeng, Yuan, Shi & Qiu, 2010; de La Rubia *et al.*, 2009). The VFA/Alk ratio presented a value near to 0.4 for PP at ISR 2, being the lowest of this process.

When the values of VFA/Alk ratio are lower than 0.4, the AD is favoured, while at values higher than 0.8 the process fails, decreasing the methane production. This was evidenced by the lower methane production for those reactors inoculated with LP, because the VFA/Alk ratio was between 0.8 - 1.4.

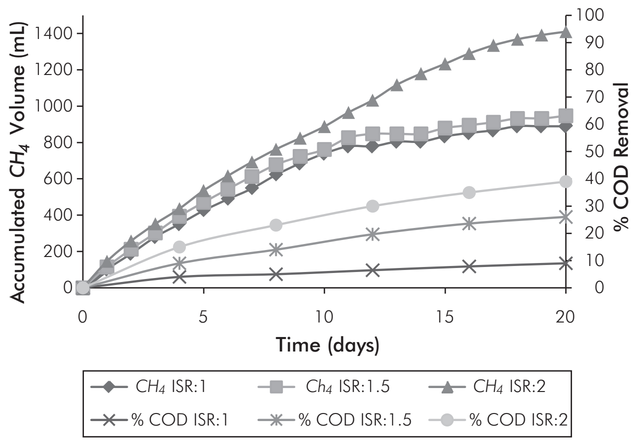
Figure 6 shows the evolution of the accumulated COD (%) removal during the experimentation time, varying between 9% for LP at ISR 1 and 47% for PP at ISR 2. These values are in the range between 27-83% COD removal reported by (Chan *et al*., 2010). It can be observed that while the accumulated methane volume increased, COD (%) removal also increased, showing a direct relationship between these two factors.

While ISR increases, methane production and COD and VS removal increase. This fact can be explained because at higher ISR, a higher concentration of microorganisms capable of degrading the organic matter emerge, being favoured also by the higher pH value (near 7) present in the system during the experimentation time.

Finally, for the anaerobic degradation process of POME it was found that the higher methane production was reached using a mixture 1:1 v/v WWTP anaerobic sludge: pig manure (PP) as inoculums at a ratio 2 g VS inoculum per gram of VS substrate. At these conditions, it was possible to obtain 2740 mL of accumulated methane volume (0.343 m^3 CH_4/kg VS), without any pH neutralization step.



(a)



(b)

Figure 6. COD (%) removal in comparison with accumulated CH_4 volume. T=37±2°C Initial pH 4.8 a) PP b) LP.

5. CONCLUSIONS

- This research have demonstrated that methane production by AD using palm oil mill effluent as substrate, and other residues as inoculums, is viable, making possible to solve pollution problems and inadequate soil uses in the environment. This process can also lead to implement a source of renewable energy available to the same industrial sector.

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