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Effect of Pretreatments on the Production of Biogas from Castor Waste by Anaerobic Digestion

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Abstract: Lignocellulosic biomass is a source of carbohydrates that can be used in the production of biogas. The aim of this study was to obtain biogas from biomass waste (leaves, stems and seed bagasse) of *Ricinus communis*, applying pretreatments such as temperature and humidity. We examined the effect of these pretreatments on the biomass, two enzymatic pretreatments (cellulase and cellobiohydrolase), two chemicals (NaOH and HCl) and two controls (dried castor straw and seed bagasse) on the methane content. The experiment was performed in two anaerobic digestion (AD) assays at a controlled temperature (37 °C) and at room temperature, with a hydraulic retention time (HRT) of 55 days. The results showed that the residues of the seed bagasse produced the highest biogas yields both at room temperature and at the controlled temperature since this material at 37 °C produced 460.63 mL gVS^{-1} under cellulase pretreatment; at room temperature, the highest level of production was found for the control (263.41 mL gVS^{-1}). The lowest yields at the controlled temperature and room temperature were obtained from residues of *Ricinus communis* treated with cellobiohydrolase and the seed bagasse treated with alkaline (15.15 mL gVS^{-1} and 78.51 mL gVS^{-1}, respectively). Meanwhile, the greatest amount of methane was produced by seed bagasse treated with cellobiohydrolase at a controlled temperature (92.2% CH_4) and the lowest content of CH_4 (15.5%) was obtained at a controlled temperature from castor straw under the control treatment.

Keywords: biomass; pretreatment method; castor waste; biogas; anaerobic digestion; methane

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1. Introduction

Dependence on fossil fuels for energy production and transportation, agriculture and rearing livestock and the industrial, commercial and residential sectors could increase the human-caused greenhouse gases (GHGs) involved in climate change. These gases include carbon dioxide, nitrous oxide, methane, ozone, chlorofluorocarbons and tetrachloride carbon, among others [1,2], with CO_2 being the main contributor to GHGs, increasing by 51% since 1990. However, the global warming potential of CH_4 is 21 times higher compared with the equivalent amount of CO_2 [3]. This is mainly produced by emissions from the agricultural sector and organic waste in landfills [4]. This has led to the development of sustainable alternatives for the use of CH_4 as a source of energy. One of them is obtaining

biogas (CH_4 and CO_2) from lignocellulosic biomass, which has a high yield of biogas, in addition to the production of fertilizers with low energy inputs [5], leading to proper management of the natural resources, ensuring healthy living conditions and developing strategies to boost the circularity of waste-management systems [6].

The raw lignocellulosic material of organic compounds used for chemical and energy generation processes contains mostly cellulose (32–45%), hemicellulose (19–25%) and lignin (14–26%) [7]. These hemicellulosic features can be found in plant species, an example being the castor oil plant *Ricinus communis*, which is a perennial shrub belonging to the Euphorbiaceae family that is native to Africa [8].

The main castor bean-producing countries in the world are India, Mozambique, China, Brazil, Myanmar and Ethiopia. World castor bean production during 2020 was 1.2 million t, with an average seed yield of around 700–1100 kg ha^{-1} [9]. The plant grows wild in many tropical and subtropical regions around the world. Castor seeds are a rich source of oil that can be extracted by grinding, boiling, pressing or with a solvent, among others [10]. In addition to medical applications, oil has long been used as an economical fuel for oil lamps. Due to its high proportion of ricinoleic acid, it is a valuable industrial raw material for lubricants, paints, cosmetic products, biodiesel and other uses [11].

The plants, particularly the seeds after extraction of the oil, contain traces of lectin ricin [12]. These residues in wet matter contain cellulose (38.4%), hemicellulose (22.4%) and lignin (20.2%), which are compounds that can be used for the production of biogas [7]. However, although these lignocellulosic materials have a considerable amount of carbohydrates, which can be converted into biogas, their recalcitrant structure is an obstacle to their direct conversion [13]. Therefore, a pretreatment process is a fundamental step that is needed to alter the structure of lignocellulosic materials. These include physical, chemical and/or biological methods, such as switching, extraction, acid treatment, alkaline pretreatment, oxidative pretreatment with peroxides and the use of fungi for the synthesis of enzymes, among others, to decrease the recalcitrance of the materials, that is, to reduce the crystallinity of the cellulose, increase the accessible surface area and remove the lignin and hemicellulose [14–16].

One of the most effective alkaline pretreatments is the use of sodium hydroxide, which can improve the production of biogas from lignocelluloses [17], while treatment with chloric acid has been carried out in oat straw, with the result that 85.5% of the hemicellulose content of oat straw was hydrolyzed, although hydrolyzation of the cellulose also occurred [18]. Relative to the increase in methane, the use of bagasse treated with sulfuric acid produced an increase of 18% compared with the untreated bagasse [19]. In addition, enzymes have been widely used for the hydrolyzation of organic matter to produce biogas. An example of this is that 13% more biogas was obtained from sugar beet pulp pretreated with enzymes compared with untreated pulp [20]. Similarly, another study carried out using the enzyme hydrolysate achieved the solubilization of 96.8% of the hemicellulose and 42.2% of the lignin in oat straw [18].

For the production of biogas after pretreatment, the substrate undergoes anaerobic digestion. Methane (CH_4) and other trace gases are produced in the absence of oxygen (O_2) in a four-stage process: hydrolysis, acidogenesis, acetogenesis and finally, methanogenesis [21,22]. The aim of this research was to analyze the potential to produce biogas from the residues (leaves and stems) of *Ricinus communis* due to the fact that in Mexico, more than 300 ha of this crop was cultivated in 2019 for oil extraction [23]. In addition to the perennial growth of the uncultivated plants, the crop leaves behind residues that can be used for biogas production due to their aforementioned characteristics. For example, one study explored the production of biogas from wastes, where they divided the waste of *Ricinus communis* into seed cakes, leaves and stems, achieving greater production from the seed cake than the other parts [24]. Other authors, such as Lingaiah and Rajasekaran, and Gollakota and Meher, evaluated the production of biogas from seed cake under varying C/N conditions and particle sizes [25,26]. However, our working group has

not found research where castor oil was used for the production of biogas, except for the aforementioned works.

Additionally, the effect of pretreatments, temperature and humidity on this biomass was explored, alongside the impact of these on the efficiency of the production of biogas. It is important to add that the process of digesting biomass requires less capital investment per unit of cost of production compared with other renewable energy sources, such as hydropower, solar power and wind power.

2. Materials and Methods

2.1. Sample Collection

The *Ricinus communis* residues included aerial parts (i.e., leaves and stems), called straw, and the seed bagasse resulting from the extraction of oil. For the collection of the straw, the plant material was obtained from plants located in Corregidora, Querétaro, México (20°31′40.1″ N, 100°25′42.7″ W). The plant material was used in a dry state (dehydrated in the shade at room temperature) and was named the dry aerial parts of *Ricinus communis* (APRc). The seed bagasse material of *Ricinus communis* (BSRc) was obtained from the residues of oil extraction using a tractor press (Zagaon Tech, Querétaro, México).

2.2. Pretreatments

The residues were subjected to physical pretreatment by crushing in a universal mill (IKA-M20, Monterrey, México) and then sieved to a particle size of up to 500 µm to improve the methane yield of the lignocellulosic biomass. Subsequently, four different pretreatments were applied: two enzymatic (cellobiohydrolase and cellulase) [27], one alkaline (NaOH) [28] and one acidic (HCl) [18]. These were applied during the hydrolysis stage, in addition to leaving a control (a substrate without pretreatment). The times and temperatures of the enzymatic pretreatments were as shown in Table 1 for activating the enzymes without being too harsh for them. The alkaline treatment did not require heat as, when applied, it generates an exothermic reaction. However, the acid treatment must be maintained at a suitable temperature for the rupture of the lignin but without maintaining the substrate for a long time with that pH level, as it would partially degrade the sample [29]. Before being subjected to AD, the pH was neutralized using NaOH or HCl.

Table 1. Pretreatments applied to *Ricinus communis*.

Pretreatment	Time [h]	Temperature [°C]
Untreated (control)	0	Room temperature
Cellobiohydrolase enzyme extracted from *Hypocrea jecorina* (0.1% *w*/*v*)	18	60
Cellulase enzyme extracted from *Trichoderma longibrachiaum* (0.5% *w*/*w*)	18	60
NaOH (4% *w*/*w*)	24	Room temperature
HCl (4% *w*/*v*)	2	80

2.3. Scanning Electron Microscopy

The morphological changes were observed for all pretreatments imaged by scanning electron microscopy SEM using a JEOL microscope (model JSM-7401F, Mexico City, Mexico) with an accelerating voltage of 2 kV.

2.4. Physicochemical Analysis

The total solids (TS), volatile solids (VS) and fixed solids (FS) were determined according to the APHA's methods [30]. Chemical oxygen demand (COD) analyses were carried out via Boyles' photometric method for measuring the amount of oxidant consumed [31]. The quantification of sugars was performed by the sulfuric acid and phenol method, by

which the total sugars could be measured. In this method, 1 mL of each standard dilution, 1 mL of a 5% phenol solution and 5 mL of concentrated H_2SO_4 were added to HACH tubes for COD, in that order. The tubes were capped, homogenized and left to cool at room temperature, then placed in a water bath at 4 °C for 15 min. Only hexoses were measured by UV spectrophotometry at an absorbance of 490 nm [32].

2.5. Anaerobic Digestion Process

Digestion was conducted with 4% TS. The experiments were conducted in triplicate in 120 mL batch reactors. A head volume of 40 mL was left, and a volume of 80 mL was worked, of which 20% was inoculated with the inoculum of a biodigester that operates on horse manure. All bioreactors were hermetically sealed. The experiments were carried out at 37 °C and at room temperature. In addition, the biogas was measured daily via volumetric displacement [33] to obtain the hydraulic retention time (HRT) for 55 days.

2.6. Gas Chromatograph

The methane content was analyzed using a gas chromatograph (Thermo Scientific model TRACE1300, Mexico City, Mexico), equipped with a TCD (thermal conductivity detector) and a packed column (TracePLOT TG-BOND Sieve 5A 0.53 mm × 30 m). The temperatures of the furnace, injection port and detector were set to 70 °C, 100 °C and 120 °C, respectively. Helium was used as a carrier gas with a filtration rate of 20 mL min^{-1} [34].

3. Results

3.1. Impact of Pretreatments

For the evaluation of the effects of the pretreatments, the changes in the physical structures of the substrates were observed. The results of APRc are shown in Figure 1A–E and those of BSRc are in Figure 2A–E. The results showed little degradation in the structure for both controls (Figures 1A and 2A); however, at the end of the pretreatments (Figures 1B–E and 2B–E), the degradation of the morphological structures of the substrates was remarkable. Figure 1 shows that for almost all the pretreatments, the lignocellulosic material was fragmented, leading to the decomposition of the fibers, except in Figure 1D, which shows an arranged distribution of the fibers. However, in Figure 1D, one can also observe a group of small clusters that could be due to the application of the cellobiohydrolase enzyme. In the case of BSRc, Figure 2B shows an unusual structure, as microspheres can be observed throughout the area of the material, while the pretreatment with cellulase produced fissures distributed throughout the substrate, which contributed to adequate rupturing of the fibers. In Figure 2D,E, spongy structures can be observed, which lead to more destructive effects.

3.2. Physicochemical Characterization

The residues of the castor oil plant were characterized before (Table 2) and after (Table 3) application of the pretreatments. Table 2 presents the characteristics of the substrates on a dry matter basis, where the moisture and VS of the APRc were 122.2% and 1% higher than that of BSRc, which means a greater amount of water was needed to hydrate the bagasse before incubation. The values of TS and FS increased for BSRc by 1% and 1.6% compared with APRc.

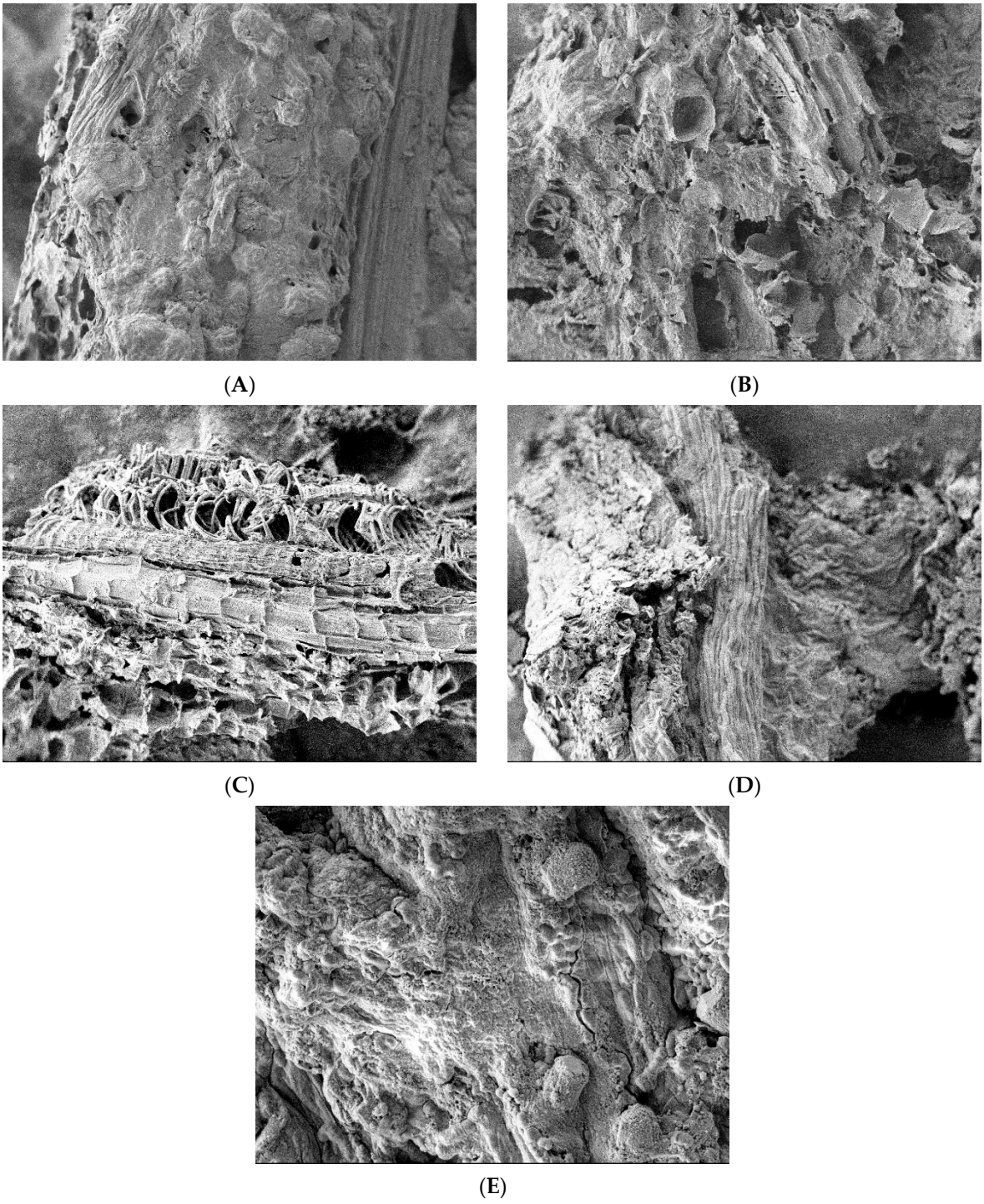

Figure 1. SEM images of APRc: (**A**) untreated; (**B**) treated with NaOH; (**C**) treated with cellulase enzyme; (**D**) treated cellobiohydrolase enzyme; (**E**) treated with HCl. (The figures (**A**–**D**) where with a resolution ×200 and 100 μm; figure (**E**) with a resolution ×5000 and 1 μm).

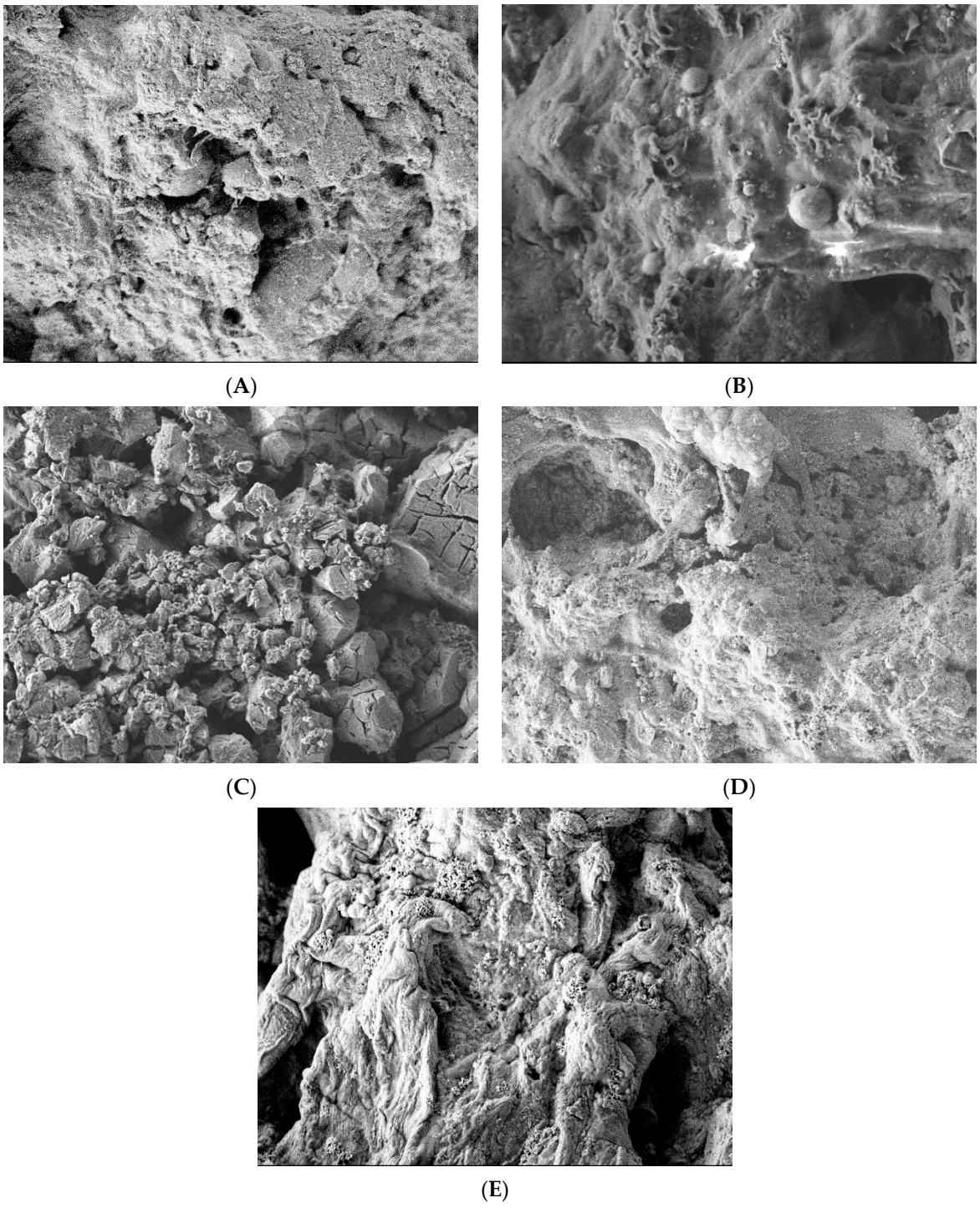

Figure 2. SEM images of BSRc: (**A**) untreated; (**B**) treated NaOH; (**C**) treated cellulase enzyme; (**D**) treated cellobiohydrolase enzyme; (**E**) treated with HCl. (Figure (**A**) it was made with a resolution of ×2000 and 10 µm; figure (**B**) with a resolution ×10,000 and 1 µm; the figures (**C**–**E**) were made with a resolution of ×5000 and 1 µm).

Table 2. Characterization of substrates on a dry basis.

Sample	Moisture [%]	TS [%]	VS [%]	FS [%]
APRc	9.9	90.2	74.4	15.9
BSRc	4.4	95.6	71.1	24.5

Table 3. Characterization and determination of TS, VS, sugars, and COD of castor residues with and without pretreatment.

Sample	TS [g L^{-1}]	VS [g L^{-1}]	Total Hexoses [mg L^{-1}]	COD [mg L^{-1}]
APRc Untreated	31	26	514.4	749.4
APRc NaOH	44	37	451.1	1607.3
APRc Cellulase	92	87	815.6	1661.7
APRc Cellobiohydrolase	280	270	246.8	4355.3
APRc HCl	138	101	898.5	4816
BSRc Untreated	32	31	238.2	918.6
BSRc NaOH	166	163	106.3	1801
BSRc Cellulase	24	22	528.3	2800.2
BSRc Cellobiohydrolase	48	46	379.2	1376.7
BSRc HCl	68	36	618.2	1720.4

Table 3 presents the physicochemical characterization of the samples after the pretreatment had been carried out, in addition to the control. The results show that the pretreated APRc had an increased TS content relative to the control; specifically, the treatment with cellobiohydrolase showed the greatest increase in the concentration of TS, followed by the treatments with HCl, cellulase and NaOH, which increased TS content by 803.2%, 345.2%, 196.8% and 42%, respectively. There was also an increase in the concentration of VS compared with the control in the same order as that of TS, with increases of 938.5%, 288.5%, 234.6% and 42.3% for cellobiohydrolase, HCl, cellulase and NaOH, respectively.

For the TS of BSRc, this order changed, since for this substrate, the greatest increase was found for the samples pretreated with NaOH, followed by HCl and cellobiohydrolase compared with the control, with increases of 418.8%, 112.5% and 50%, respectively. However, the treatment with cellulase decreased TS by 25%. For the VS of this substrate, NaOH was the pretreatment that produced the highest concentration relative to the control, followed by cellobiohydrolase and HCl, with increases of 425.8%, 48.4% and 16.1%, respectively. Similar to the results for TS, the treatment with cellulase decreased TS by 29%.

Regarding the chemical analysis of the APRc, the pretreatments with the cellobiohydrolase enzyme and NaOH showed a decrease in the concentration of carbohydrates of 52% and 12.3%, respectively, relative to the control; however, the pretreatments with HCl and cellulase showed decreases of 74.7% and 58.5%, respectively. In terms of the COD of this substrate, the concentrations were changed by each of the pretreatments relative to the control. The highest concentration was found for HCl, followed by cellobiohydrolase, cellulase and NaOH, which produced increases of 542.7%, 481.2%, 121.7% and 114.5%, respectively. For the BSRc, the concentration of hexoses decreased by 55.4% with NaOH; however, pretreatments with HCl, cellulase and cellobiohydrolase increased the concentration of hexoses by 159.5%, 121.8% and 59.2%, respectively. The same trend was seen for the COD concentrations because the treatment that increased its concentration to the greatest extent was cellulase, followed by treatment with alkaline, acid and, finally, cellobiohydrolase, with increases of 204.9%, 96%, 87.3% and 49.9%, respectively, relative to the control.

3.3. Effect of Pretreatments on the Generation of Biogas

3.3.1. Biogas from Aerial Parts of *Ricinus communis*

For the production of biogas from APRc with different pretreatments both at room temperature (Figure 3) and at a controlled temperature (Figure 4), a hydraulic retention time (HRT) of 55 days is preferred. If we compare both figures, it can be observed that the temperature was an important factor in the yield of biogas, because for each pretreatment of the same substrate, yield increased with temperature. The reactors exposed to room temperature generated 166.6 mL·gVS^{-1}, while at a controlled temperature, the yield was 227 mL·gVS^{-1}; with NaOH, the yield increased from 249.9 mL·gVS^{-1} to 353.5 mL·gVS^{-1}, while for the treatment with cellulase enzyme, the increase was more than double, rising from 51.9 mL·gVS^{-1} to 114.1 mL·gVS^{-1}. On the other hand, for the pretreatment with

cellobiohydrolase, at room temperature, 15.2 mL·gVS^{-1} was obtained, but at 37 °C, the yield reached 108.5 mL·gVS^{-1}; this was the treatment with the highest percentage increase. The HCl treatment also showed an improvement with temperature, increasing from 66.1 mL·gVS^{-1} to 111.5 mL·gVS^{-1}. From these data, it can be observed that the alkaline treatment, at both temperatures, produced the highest yield of biogas compared with the enzymatic pretreatments.

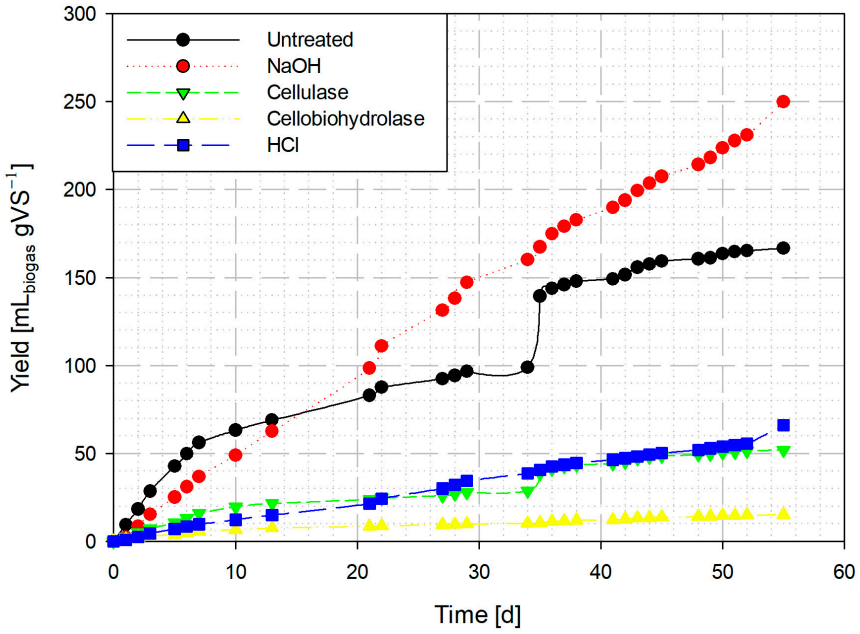

Figure 3. Biogas production yield in pretreated biomass of APRc at room temperature.

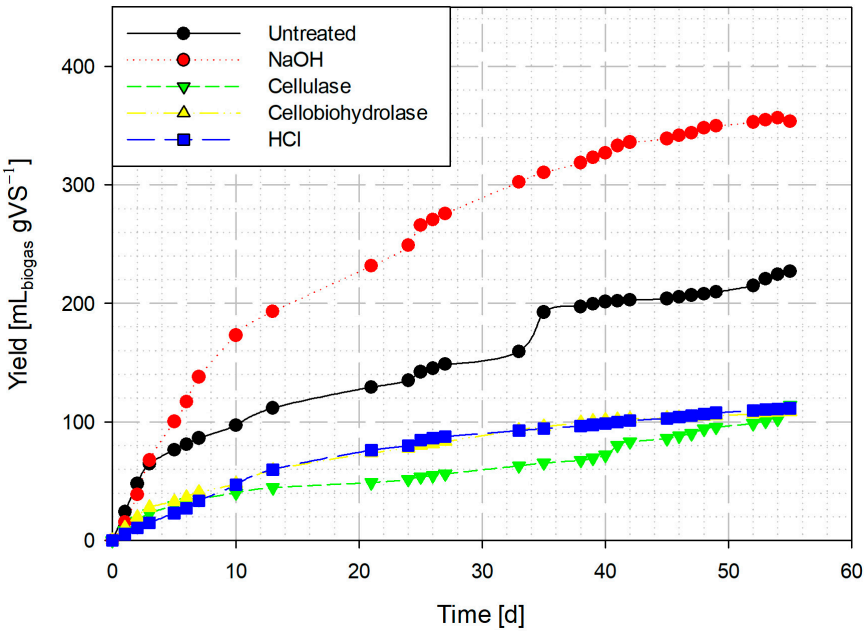

Figure 4. Biogas production yield in pretreated biomass of APRc at 37 °C.

3.3.2. Biogas from Bagasse Seed of *Ricinus communis*

The yield of BSRc at room temperature and a controlled temperature is shown in Figures 5 and 6, respectively. In Figure 5, a maximum yield of 263.4 mL·gV^{-1} was observed for the control, followed by 206.6 mL·gVS^{-1} for the treatment with HCl, 202.5 mL·gVS^{-1} for cellulase, 196.7 mL·gVS^{-1} with cellobiohydrolase and, finally, a minimum of 42.1 mL·gVS^{-1} with the NaOH treatment. However, the yields were increased by controlling the temperature, and the pretreatments produced an increase over the untreated bagasse. In Figure 6, this effect can be observed, because the pretreatment that achieved the highest yield was the enzymatic cellulase (460.6 mL·gVS^{-1}), followed by the acid treatment (373.6 mL·gVS^{-1}), the control (369.2 mL·gVS^{-1}), the cellobiohydrolase enzyme treatment (100.4 mL·gVS^{-1}) and, finally, the alkaline treatment (78.5 mL·gVS^{-1}).

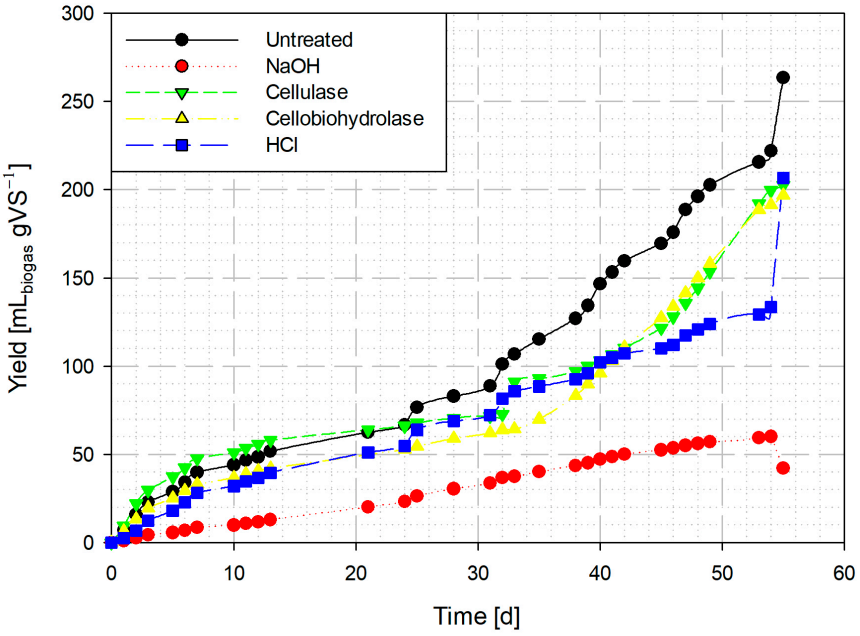

Figure 5. Biogas production yield in pretreated biomass of BSRc at room temperature.

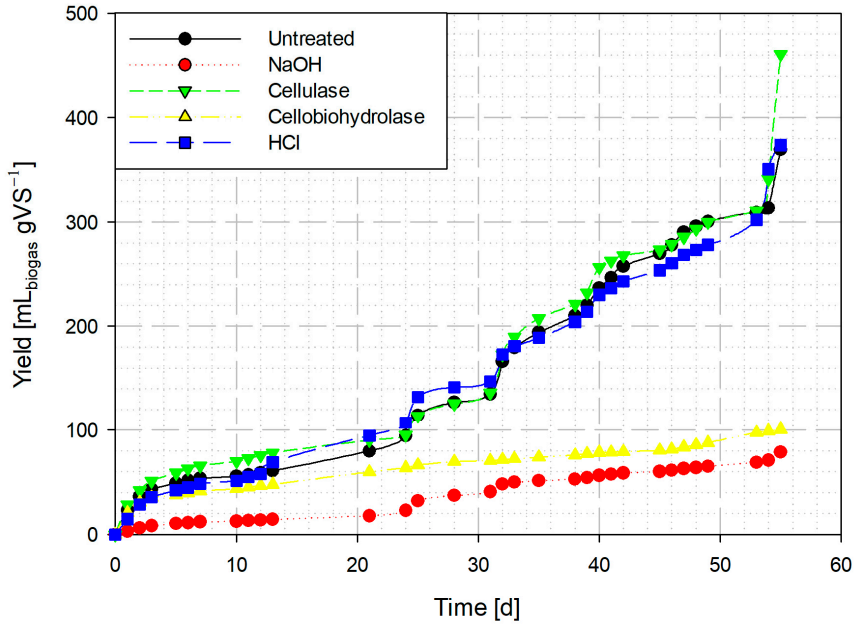

Figure 6. Biogas production yield in pretreated biomass of BSRc at 37 °C.

3.4. Methane Content

The amount of methane obtained from biogas from the controls and the pretreatments at two different temperatures was observed (Table 4). The results showed an increase in the methane content when pretreatments were administered compared with the controls at both temperatures. At room temperature, the APRc increased the yield by 251.6%, 65.8%, 37.4% and 8.4% with the HCl, NaOH, cellulase and cellobiohydrolase treatments, respectively, compared with the control. At the same temperature, the BSRc produced higher increases of 48.6%, 29.9%, 16.8% and 10.3% after treatment with cellobiohydrolase, HCl, cellulase and NaOH, respectively. At 37 °C, the greatest increase in the methane content was for the APRc: 262.8%, 190.8%, 190.3% and 50.7% for NaOH, cellulase, cellobiohydrolase and HCl, respectively, compared with the control. For the BSRc substrate, the methane concentration increased by 81.1%, 67.2%, 27.9% and 19.7% through treatment with cellobiohydrolase, NaOH, cellulase and HCl, respectively.

Table 4. Methane content of substrates with and without pretreatment at room temperature and at 37 °C.

Sample at Room Temperature	CH_4 [%]	CO_2 * [%]	Sample at 37 °C	CH_4 [%]	CO_2 * [%]
APRc Untreated	15.5 ± 2.9^{D}	84.5 ± 2.9^{A}	APRc Untreated	20.7 ± 12.6^{D}	79.3 ± 12.6^{A}
APRc NaOH	25.7 ± 6.1^{CD}	74.3 ± 6.1^{AB}	APRc NaOH	75.1 ± 14.7^{AB}	24.9 ± 14.7^{CD}
APRc Cellulase	21.3 ± 7.1^{D}	78.7 ± 7.1^{A}	APRc Cellulase	60.2 ± 12^{ABCD}	39.8 ± 12^{ABCD}
APRc Cellobiohydrolase	16.8 ± 2.3^{D}	83.2 ± 2.3^{A}	APRc Cellobiohydrolase	60.1 ± 7.6^{ABCD}	39.9 ± 7.6^{ABCD}
APRc HCl	54.5 ± 7.7^{BC}	45.5 ± 7.7^{BC}	APRc HCl	31.2 ± 16.8^{CD}	68.8 ± 16.8^{AB}
BSRc Untreated	60.3 ± 15.8^{AB}	39.7 ± 15.8^{CD}	BSRc Untreated	50.9 ± 8.5^{BCD}	49.1 ± 8.5^{ABC}
BSRc NaOH	66.5 ± 6.7^{AB}	33.5 ± 6.7^{CD}	BSRc NaOH	85.1 ± 17.2^{AB}	14.9 ± 17.2^{CD}
BSRc Cellulase	70.4 ± 15.9^{AB}	29.6 ± 15.9^{CD}	BSRc Cellulase	65.1 ± 11.4^{ABC}	34.9 ± 11.4^{BCD}
BSRc Cellobiohydrolase	89.6 ± 13.5^{AB}	10.4 ± 13.5^{D}	BSRc Cellobiohydrolase	92.2 ± 12.9^{A}	7.8 ± 12.9^{D}
BSRc HCl	78.3 ± 19.2^{AB}	21.7 ± 19.2^{CD}	BSRc HCl	60.9 ± 20.3^{ABCD}	39.1 ± 20.3^{ABCD}

* Data were the difference between 100 minus CH_4 concentration, and include traces gases; Different letters indicate statistical difference between columns Tukey test, $\alpha = 0.05$.

4. Discussion

The change in the morphological structures of substrates with and without pretreatment is important to observe the degradation of lignocellulosic material to know the effect caused by pretreatments on the matter [35]. In this sense, when comparing the APRc with other raw materials but with the same pretreatments, it can find that after applying heat treatment combined with cellulase to wheat straw it was observed that the severity of the pretreatment conditions made the compact straw structure more porous than the control, making the substrate more accessible to microorganisms in the next step of digestion [36].

On the other hand, when thermal and NaOH pretreatments were applied to plant residues of *Eruca sativa* (Brassicaceae), the pretreatment at 0 °C produced deep pitting between the substrate compartments, while the pretreatment at 100 °C showed empty lumens, which represented that the layers of substrate cover were destroyed after pretreatment thus increasing the porosity of the residues with respect to the untreated [35]; these results with alkaline pretreatment were not performed for the APRc of this research with different temperatures. However, it was also possible to observe the destruction of the fibers that cover the plant as well as the bites that the pretreatment caused. Regarding the pretreatment with HCl, a study focused on wheat straw revealed that wheat straw fibers soaked for 2 h in deionized water were complete and orderly; however, after 2 h of acid pretreatment, the fiber structure became disordered and even defragmented, so this study points out that the pretreatment was able to break the hydrogen and covalent bonds between the units lignocellulose [37]. In the case of APRc with the same pretreatment, the cracks were observed and a porous surface that the acid caused was noticed. These results agree with the description that pretreatments help accelerate the destruction of plant compounds as mentioned above for their own research.

As for the bagasse substrate, Sánchez-Cantú et al. [38], who carried out pretreatment with an enzymatic cocktail extracted from the fungus *Pleurotus djamor* to obtain bioethanol from the bagasse of the seed of *Ricinus communis*, found that the sample presented two different microstructures: the first, most of the sample, was identified as the contribution of the seed coat of bagasse and the second microstructure presented a spherical shape that corresponded to the lipid and protein bodies in this research, for the BSRc with enzyme cellulase cracks caused by this pretreatment is shown, which indicates the operation of the same; however, when applying the pretreatment with cellobiohydrolase, the concentrations of oil that still contains the substrate can be observed at first sight, indicating the presence of protein as suggested by the aforementioned authors. Similarly, Bateni et al. [39] used NaOH at 0 and 100 °C on the bagasse of the seed of *Eruca sativa*, where they found that the untreated bagasse did not significantly affect the porosity of the substrate; however, the low-temperature pretreatment resulted in some pores dispersed in the structure while the high-temperature treatment led to an agglomerated structure in the pretreated bagasse, which was mainly due to the gelatinization of starch at 100 °C and its expulsion to the surface forming a coating. Therefore, the alkaline pretreatment of the bagasse of the *Eruca sativa* seed was unsuccessful in increasing porosity, while for BSRc, with alkaline pretreatment the SEM image showed that in the structure of the substrate, they left oil residues; this can be observed in the formation of microspheres found in this oil content was due to the fact that the extraction was carried out by hydraulic pressure and at 95 °C.

On the other hand, Monlau et al. performed the acid treatment at 170 °C on the bagasse of the sunflower seed, thus determining that the pretreatment was effective in eliminating hemicelluloses, in addition to the structural modification of the bagasse during the pretreatment [13]. Then, the presence of oil in pretreatments may be an additional nutrient source for methanogenic bacteria in AD [40].

Regarding physical characterization, in 2018, Kaur et al. reported 11.1% moisture for previously dehydrated *Ricinus communis* residues (leaves and stems) [7]. These same authors reported 88.9 and 74.3% of TS and VS, respectively, which were like those obtained in this study, in addition to 9.2% of SF; on the other hand, Kalogiannis et al. obtained 4.4% of SF in dehydrated aerial parts of [41] *Ricinus communis*, this value was 1.7 times lower, respectively, than that reported in this study.

For seed bagasse, one research observed 5.6% moisture, while another reported 10% [42]; the first value is close to 4.4% reported in this paper, the difference may be due to the method of extraction of the oil since for this research it was performed by mechanical extraction, while the authors performed by Soxhlet. Data of the bagasse of oil seeds belonging to the Euphorbiaceae family have also been carried out and are similar to those determined in this research, an example is the 7.5% moisture obtained on the bagasse of the seed of *Jatropha curcas* [43] or 8.3% achieved with the same substrate [44], most of these studies are only done on the bagasse of the seed (and not on the aerial parts of the plant) because it is the raw material for the extraction of the oil. In relation to TS, FS and VS for BSRc, 92.7% and 5.6% were achieved, respectively; but no VS determined [42], values decreased by 3 and 77% compared to those shown in Table 2. VS determination has also been reported for *Jatropha curcas* seed bagasse of 17.3 and 72.7% [43,45]. The authors do not report the origin of bagasse; therefore, it is difficult to determine the difference and similarity to the first and second values reported in this study.

The concentrations of TS and VS after the application of the pretreatments shown in Table 3, can be compared with the concentrations of TS and VS for pretreatments with sulfuric acid 5% (*v*/*v*) and alkaline 3N with NaOH, to the pulp of sugar beet, where they observed decreases in concentrations in relation to untreated beet, finding decreases of 25% and 22.7% of ST and VS for alkaline treatment, while for beet treated with the H_2SO_4 they obtained decreases of 22% and 14.8% of TS and VS, respectively [46]. Therefore, in comparison with this research, it is corroborated that it is advisable to dehydrate the aerial parts of *Ricinus communis* before pretreating them, due to the increase in TS and VS concentrations.

Another study applied a mixture of cellulases obtained from a microalgae community to the production of biogas, and found that the concentration of VS increased by 5324.7% for pretreated algae with enzymes with respect to the control [34], the increase in VS concentrations performing pretreatment with enzymes for APRc was also reflected; however, these increases were not so noticeable, because in the substrate in which the greatest increase was had was in the pretreated APRc with cellobiohydrolase with 938%. For the bagasse of the seed with the same pretreatment presented lower VS content per liter, the reason for the minority effect of the pretreatment on the bagasse could be the degradation of hemicellulose, which mostly contains xylan and does not let the pretreatment act on the substrate [24].

For the chemical analyses performed on the samples, shown in Table 3, chemical pretreatments (alkaline and acid) performed on rice straw to obtain biogas can be compared, observing that the conversion of carbohydrates with 2% NaOH (*w*/*w*) increased by 55%, while for treatment with 1% HCl (*w*/*v*), the improvement was 30% greater. This same study evaluated the effects of these pretreatments on COD, finding similarities, since the alkaline treatment obtained 14,800 mg L^{-1}, while the acid treatment did 9300 mg L^{-1} [47]. These improvements in carbohydrate concentrations were higher than those obtained in this research compared with aerial parts with the same pretreatments; on the other hand, the comparison of COD with the APRc in this study showed higher concentrations, since the increase with NaOH was 821% and the HCl was from 93%. The increase in COD concentrations is a parameter indicating the total chemically oxidizable material in the sample, and therefore, the energy content of raw material; in addition, the acid solution indicates that the cellulose degrades faster with the pretreatments [48,49]. Another study conducted on rice straw under hydrothermal pretreatment (cellulase at 190 °C) improved its carbohydrate concentration by almost 200% more than the control [36]. For the APRc with the same pretreatment, an increase was observed; however, it was not as noticeable as that of the aforementioned work, since it was only 59% higher than the control.

Waste cake has been used to obtain an enzyme cocktail, which was applied to food waste from a cafeteria to obtain ethanol. In this publication, the concentration of carbohydrates increased by 535% with the application of pretreatment [48,50]; on the other hand, enzyme pretreatment (amylase, glucoamylase and protease) to the same substrate for biogas production was also evaluated by Zhang et al. [51] they obtained an increase in COD of 1.5% with pretreatment respective to the control. These values, both carbohydrate and COD, are close to the increases obtained for APRc and BSRc pretreated with cellobiohydrolase. Chemical pretreatments have also been carried out on spent grains from the brewing industry for bioethanol production, where the concentration of carbohydrates was evaluated in alkaline and acid treatments, finding a decrease of 46% for the substrate treated with 1% NaOH and an increase of 26% for the treated with 1% HCl [52]. In this sense, the BSRc with the same pretreatments had similarities for both treatments, since there was a decrease in the concentration with NaOH of 55%; however, the acid treatment obtained an increase of 159%.

As for the yield of biogas produced, the application of *Ricinus communis* waste for obtaining biogas is scarce; however, several pretreatments have been carried out on raw materials with lignocellulosic content, a substrate used is the oilseed husk with which research has been carried out in the production of biogas. Venkateshkumar et al. [52] evaluated this substrate with acid and alkaline pretreatment, using 1, 2, 3% of HCl and 4, 6, 8% of NaOH; the substrate was dried and subsequently ground leaving a particle size of between 0.5 and 0.7 mm. The experiment was performed in 500 mL reactors inoculated with cow dung maintaining a temperature of 35 ± 2 °C and an HRT of 45 d. The results obtained demonstrated the positive effect of pretreatments on cotton husks, because their negative control (substrate without pretreatment) obtained a yield of 33 mL gVS^{-1}, while in the positive control (only cow dung), they obtained 193 mL gVS^{-1}, reaching higher yields with pretreated substrate compared to the negative control. However, different pretreatment with HCl yielded lower yields than positive control; the highest production of

biogas was obtained by the treatment with a concentration of 1%, and still the production decreased by 40%; otherwise, the pretreatments with NaOH managed to increase the yield, except for the concentration to 8%, which decreased 40% compared to the positive control, achieving a higher yield concentration of 6% and increasing production by almost 123%.

If comparing the results obtained from chemical pretreatments with the APRc in this work, it can observe the same trend in terms of performance, since in our case, it also decreases when applying pretreatment with HCl by 51%, decreased more than when applied to the husk of the cottonseed. On the other hand, it increases with NaOH by almost 56%. Although a higher yield was achieved with this substrate, the same increase was not obtained as with the cottonseed husk; however, the concentrations applied are different. These chemical pretreatments applied to lignocellulose plants, indicate the effectiveness of alkaline pretreatment, but also give us an approach to knowing the conditions of acid pretreatment, since it becomes abrasive in lignocellulosic material and, therefore, loses effectiveness [29]. Additionally, Bateni et al. [24] achieved a methane yield of 186.4 mL gVS^{-1} from *Ricinus communis* seed bagasse with 8% alkaline pretreatment (*w*/*v*)l this treatment decreased methane yield by 26% compared to the control. A negative effect was also obtained in the BSRc since at room temperature it decreased by 78% and at controlled temperatures the decrease was 84%, which shows that alkaline pretreatment is not a good candidate for this substrate, and instead it was probably due to the oil that remained in the bagasse structure. This can be observed in Figure 2B and could induce the saponification reaction instead of performing its purpose as a pretreatment; consequently, the effectiveness of the pretreatment was reduced and the pretreated substrates produced less performance during anaerobic digestion [24,53].

On the other hand, enzymatic pretreatments have also been applied to lignocellulosic substrates; an example is the evaluation of the production of biogas in *Secale cereale* (Gramineae), *Brassica napus* straw (Brassicaceae) and *Vicia faba* straw (Fabacea), which were pretreated with a preparation liquid of cellulase and cellobiase extracted from *Trichoderma longibrachiatum* and *Aspergillus niger*, these pretreated substrates were inoculated with 20 mL in flasks of 100 mL with an HRT 67 d. The controls were also evaluated (without pretreatments), which yielded a biogas production of 360, 420 and 440 mL gVS^{-1} for *Secale cereale*, *Brassica napus* and *Vicia faba*, respectively, this enzymatic pretreatment, according to the authors, it only had a significative effect on *Secale cereale*, while for *Brassica napus* and *Vicia faba*, biogas productivity and performance were similar to the controls [54]. The effect caused by the enzymes for the APRc of this study was negative, since at controlled temperature, the use of cellulase decreased production by almost 50%, while for the enzyme cellobiohydrolase, the decrease was greater than 52% for the BSRc at 37 °C, and the cellobiohydrolase affected the yield by 72%, while in the same substrate with cellulase, the highest biogas yield was obtained, increasing by 24% respect to the control.

Regarding biogas quality, Almeida et al. [55] used *Aloe vera* and *Opuntia robusta* species with high cellulosic content for methane production, finding contents of 23.96% and 10.19% CH_4 for *Opuntia robusta* and *Aloe vera*. If we compare those results with the untreated APRc and BSRc in our research, we found that the CH_4 for the APRc are among the ranges obtained for *Opuntia robusta* and *Aloe vera*, since both temperatures were lower than the CH_4 found in *Opuntia robusta* but higher than *Aloe vera*; in contrast, for BSRc, the contents were higher than the highest value found (24%), since the methane content more than doubled in both temperatures. These results of the use of biomass without pretreatments to obtain methane show us the high concentration of lignin and the need to pretreat waste with lignocellulosic characteristics since the application of pretreatments allows it to reduce the possible negative effects on the production of biogas [56,57].

The pretreatments have been sought to lignocellulosic material to raise the methane content in the production of biogas, an example is the application of chemical pretreatments to parts of spruce pine (leaves, branches, bark, wood, fruits and their mixtures), these substrates were dried and crushed at room temperature to later use pretreatments with phosphoric acid, ethanol and sulfuric acid. Once the pretreatment was applied, the

substrates were divided into solid and liquid parts, obtaining 75% methane content for the solid substrate of leaves pretreated with H_2SO_4 and 65% CH_4 for the liquid fraction. The lowest CH_4 content was 30% for the same substrate but with concentrated H_3PO_4, and they also found the highest methane content of 67 to 82% for solid mixtures using H_2SO_4 [58]; although the APRs of this experiment were not pretreated with H_2SO_4, it can be compared with HCl, where compared to the higher methane content for the solid part of the leaves with H_2SO_4, the CH_4 for the pretreated APRc with HCl at room temperature the content decreased by 27.3%; however, at a controlled temperature, there was a decrease of more than 58%. Despite this decrease, the result is similar to the methane obtained from the substrate of the liquid part of leaves pretreated with H_3PO_4; as for the BSRc at controlled temperature, it is inside the parameters found in mixtures treated with H_2SO_4, while BSRc at room temperature was 9% lower than the lowest concentration of CH_4 found in mixtures of different parts of spruce pine.

Another chemical pretreatment used is the application of NaOH, where CH_4 concentrations of 54.9% have been found for food waste with this pretreatment [59], and 79.5% CH_4 applying 8% NaOH pretreatment to *Miscanthus sacchariflorus* [60], where compared to the application of the same pretreatment, APRc at room temperature decreased by 53.2% compared to food waste. However, at a controlled temperature, an increase of 36.8% was obtained; in the case of *Miscanthus sacchariflorus*, the APRc at 37 °C and at room temperature was decreased by 5.5 and 67.7% of CH_4. On the other hand, the application of alkaline pretreatment to BSRc, comparing it with food waste, shows an increase in methane concentration is observed for both temperatures, since at room temperature it increased 21.1%, while at 37 °C the increase was 55%. Although if we compare the BSRc with *Miscanthus sacchariflorus*, at room temperature it decreased by 16.3%, but at controlled temperature, an increase of 7% was obtained; these results indicate the positive effects of the application of alkaline pretreatment to different substrates, because in terms of this research, it was possible to increase the quality of biogas in almost all cases at controlled temperature.

To increase methane concentration and lignin degradation, enzyme pretreatments have been used. Hashemi et al. [61] used birch wood (*Betula pubescens*) as a substrate on which they first performed steam explosion pretreatment, and then applied pretreatment with four commercial enzymes of MetGen: Metzyme FORCI 017, MetZyme FORCI 0215, MetZyme FORCI 018 and MetZyme FORCI 032, finding methane concentrations of 60, 61, 61 and 60%, respectively for each enzyme. Compared to the pretreated APRc with enzyme cellulase and cellobiohydrolase at controlled temperature, methane concentrations are within the range obtained for the four enzymes used in birch; however, the pretreated APRc with cellulase and cellobiohydrolase at room temperature decreased more than double and more than triple, respectively, in comparison. However, the use of enzyme cellulase in BSRc had an increase of 6 and 15.4% of CH_4 at 37 °C and at controlled temperature compared to that obtained from *Betula pubescens*, but where the greatest difference was found was in the methane obtained from BSRc pretreated with cellobiohydrolase at room temperature and controlled, increasing 46.9 and 51.1%. Another study by Petersson et al. [54], where they used a liquid preparation of cellulose and cellobiase applied to *Secale cereale*, *Brassica napus*, and *Vicia faba*, found the theoretical yield of methane based on the assumption that volatile solids were stoichiometrically converted to CO_2 and CH_4; they reported concentrations of 96, 85 and 75% methane for *Secale cereale*, *Brassica napus* and *Vicia faba*, respectively. Although good concentrations of CH_4 were found for the APRc and BSRc, the maximum concentration obtained from *Secale cereale* was not reached; however, the BSRc is very close to 37 °C, which is only below 3.8% CH_4. It should be noted that the comparison is being made with a theoretical concentration.

5. Conclusions and Future Perspectives

Ricinus communis residues are substrates with potential for biogas production, with BSRc showing the highest biogas yields, followed by APRc, achieving higher yields at 37 °C than at room temperature in both cases.

The pretreatment that showed the highest methane content at a controlled temperature and at room temperature was BSRc treated with cellobiohydrolase enzyme, which achieved 92.2% and 89.6%, respectively. Although this enzymatic pretreatment showed an improvement in the production of methane, it is difficult to obtain the enzyme. It would, therefore be convenient to evaluate the fungus *Hypocrea jecorina* as an enzymatic source for the production of biogas to avoid high costs.

However, it is important to emphasize that this research is expected to establish a method for obtaining biogas from the waste generated by the cultivation of *Ricinus communis* in Mexico, with the aim of collaborating in the global strategies to reduce dependence on fossil fuels and to reduce GHGs, as established by the Kyoto Protocol and Agenda 21.

In addition, the production of biogas from organic waste is a way to assist communities with scarce resources that do not have gas utilities for cooking food, which promotes the use of firewood that causes the felling of trees and increases the likelihood of degenerative diseases associated with the combustion of plant residues. For future work, the results obtained in the laboratory by this experiment can be extrapolated to develop a functional biodigester that could be beneficial for low-income communities.

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Abbreviations

AD	Anaerobic digestion
APRc	Aerial parts of *Ricinus communis*
BSRc	Bagasse seed of *Ricinus communis*
C/N	Carbon/Nitrogen ratio
COD	Chemical oxygen demand
FS	Fixed solid
GHGs	Greenhouse gases
HTR	Hydraulic retention time
SEM	Scanning electron microscopy
TS	Total solid
VS	Volatile solid

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