Effect of Alkaline Pretreatment on Biogas Production from Corn (*Zea mays*) Crop Residues Biomass

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Abstract

Biogas represents a green energy source that has received major attention as an alternative for fossil fuel depletion. In this work, corn crop residues were used as feedstock for biogas production by anaerobic digestion. The effect of alkaline pretreatment was also evaluated. Proximate analysis was performed to inoculum samples in order to determine volatile and total solids content. Experiments were performed in duplicate varying both parameter particle size (0.5 mm, 1 mm, 2 mm, and *in natura*) and inoculum to substrate ratio (1:1, 2:1, and 3:1). The highest amount of biogas (392.75 mL) was obtained from corn stalk without pretreatment using 1 mm particle size and 1:1 inoculum to substrate (I:S) ratio. In addition, the highest yield (6.29x10^-2 m^3/kg SV substrate) was achieved for pretreated corn leaf biomass using *in natura* particle size and 3:1 I:S ratio, which confirms that alkaline pretreatment facilitates the hydrolysis stage during the anaerobic digestion process. These results suggested that alkaline pretreatment enhances biogas production from corn crop residual wastes.

**Keywords:** biogas, alkaline pretreatment, biomass, corn
1. Introduction

Nowadays, the environmental sustainability of processes is in the scientific community spotlight. Agricultural residues can provide large amounts of biomass as feedstock for biofuel and bioenergy production [5, 8]. Agricultural biomass residues are composed of cellulose, hemicellulose, and lignin and possess a high-energy content [3]. The use of this lignocellulosic biomass increases the economic value of residues and reduces the environmental impacts of its disposal [9]. Corn stover is a by-product generated by corn grain cultivation that has been widely studied for bioenergy production, and includes the husks, cobs, leaves, and stalks [13]. When transforming organic residues into methane anaerobic digestion plays a major role in converting the substrate [6]. Anaerobic digestion is a bioconversion process that can treat lignocellulosic biomass such as energy crops, crop residues, and agro-industrial by-products to produce biogas [12]. To improve this process, pretreatment step is required to weaken and break the bonds in the lignocellulosic complex and enhance microorganism accessibility to the carbohydrate polymers [7]. This work attempts to evaluate the effect of alkaline pretreatment on biogas production from corn crop residual biomass and establish suitable conditions of particle size and I:S ratio to achieving higher yields during this process.

2. Materials and Methods

The corn crop residual biomass was obtained in the rural area of the department of Bolivar and rumen liquid was provided by a local slaughter house. The substrates were prepared by separation of stalk and leaf in corn stover, then they were washed, exposed to solar radiation for drying and milled to 0.5 mm, 1 mm, 2 mm, and in natura.

Alkaline pretreatment and acid hydrolysis: In order to evaluate the effect of alkaline pretreatment on biogas production, substrates were kept in contact with 1% wt. NaOH solution (15 mL/g substrate) during 30 minutes at 121°C. Afterwards, they were washed and dried. In addition, acid hydrolysis is required to obtain simple sugar molecules from cellulose and hemicellulose. Hence, substrates with and without pretreatment were mixed with 1% vol. H₂SO₄ during 1 hour into an autoclave and samples were sent to anaerobic digestion.

Anaerobic digestion: The experiments were performed in duplicate according to operational conditions summarized in Table 1. Initially, a purge with nitrogen was carried out to ensure an anaerobic environment inside the reactor. The biogas produced was measured on a daily basis using the liquid displacement method. Different particle sizes (0.5 mm, 1 mm, and in natura) and I:S ratios of 1:1, 2:1, and 3:1 were evaluated in order to determine suitable conditions for biogas production. The inoculum was a mixture of pig mature and rumen liquid (80:20).
Table 1. Experimental conditions for anaerobic digestion

<table>
<thead>
<tr>
<th>Operational variable</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reactor volume</td>
<td>500 mL</td>
</tr>
<tr>
<td>Reaction volume</td>
<td>350 mL</td>
</tr>
<tr>
<td>Operation time</td>
<td>15 days</td>
</tr>
<tr>
<td>Temperature</td>
<td>Room temperature: 28°C±1°C</td>
</tr>
<tr>
<td>Stirring</td>
<td>Daily, manual</td>
</tr>
</tbody>
</table>

3. Results and Discussion

It has been reported that composition of lignocellulosic biomass such as corn stalk and leaf varies between 35-50%, 20-35%, 15-20%, and 15-25% for cellulose, hemicellulose, lignin, and ashes, respectively [10]. Inoculum composition also plays an important role for producing biogas. Table 2 summarizes the results for inoculum characterization. It was observed that pig manure has a higher total solids content than rumen liquid and substrates, which suggested the need for adding water to biodigester in order to reduce organic load and avoid inhibitory processes during biogas production [4].

Table 2. Proximate analysis of inoculum

<table>
<thead>
<tr>
<th>Components</th>
<th>Pig manure&lt;sup&gt;a&lt;/sup&gt; (%)</th>
<th>Rumen liquid&lt;sup&gt;b&lt;/sup&gt; (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total solids</td>
<td>60.76 ± 1.38</td>
<td>0.97 ± 0.02</td>
</tr>
<tr>
<td>Volatile solids</td>
<td>53.85 ± 1.26</td>
<td>0.84 ± 0.03</td>
</tr>
</tbody>
</table>

<sup>a</sup>pH 6; <sup>b</sup>pH 7

3.1. Biogas production without alkaline pretreatment: Figures 1-3 shows the accumulated production of biogas using corn leaf, stalk and stalk/leaf biomasses without alkaline pretreatment. It was found that 1:1 I:S ratio exhibited the highest biogas production for all experiments using the corn crop residual biomasses without pretreatment. The effect of particle size was mainly observed in corn stalk/leaf biomass. For corn stalk/leaf and corn leaf biomasses, the maximum amount of biogas (342.75 mL and 387.5 mL, respectively) was obtained using 0.5 mm particle size. However, the highest biogas production from corn stalk biomass (392.75 mL) was achieved using 1 mm particle size. The accumulated biogas production is not enough to determine the suitability of lignocellulosic biomass as feedstock. The effectiveness of pretreatment on lignocellulosic biomass is generally assessed by measuring the methane yield during its subsequent anaerobic digestion [3]. Hence, biogas production yield was calculated in order to consider the biodegradability of substrate.
Figure 1. Biogas production from corn stalk/leaf biomass using different I:S ratio and particle size.

Figure 2. Biogas production from corn stalk biomass using different I:S ratio and particle size.

Figure 3. Biogas production from corn leaf biomass using different I:S ratio and particle size.
Figures 4-6 shows biogas production yield when corn crop residual biomass (stalk/leaf, stalk, and leaf) is used. This potential of methane production indicates the volume of biogas produced per unit of mass of volatile solids [2]. It was observed that the lowest biogas production yield was obtained for 1:1 I:S ratio despite the high amount of biogas produced in comparison to other I:S ratios, which is attributed to the acidification of raw material due to accumulation of acids in hydrolysis stage causing unfavourable conditions for anaerobic digestion process [4]. The highest biogas production yield was achieved with 3:1 I:S ratio for all substrates used. The reduction of particle size enhances biogas production, however, methane production yield is not necessarily increased [1]. It explains that both corn stalk and corn leaf exhibited the highest biogas production yield using *in natura* particle size (Figure 5 and 6), while for corn stalk/leaf, it was achieved using 0.5 mm particle size (Figure 4). The low biogas production yield obtained for 1:1 I:S ratio is due to inappropriate proportion between inoculum and substrate. It has been reported values for inoculum to substrate ratio above 2 for optimum performance during biogas production [11].

Figure 4. Biogas production yield from corn stalk/leaf biomass using different inoculum: substrate ratio and particle size

Figure 5. Biogas production yield from corn stalk biomass using different inoculum: substrate ratio and particle size
3.2. Biogas production with alkaline pretreatment: Anaerobic digestion was performed using pretreated biomass with NaOH under suitable particle size and I:S substrate ratio based on biogas production yield. This pretreatment is applied to facilitate the hydrolysis stage during the anaerobic digestion process [13]. Figures 7 and 8 show biogas production and its yield from both pretreated biomass and without alkaline pretreatment (control). It was observed that 1:1 I:S ratio exhibited the lowest biogas production yield, which can be attributed to the presence of high amount of acids produced in acidogenic stage in comparison to digestion of biomass without pretreatment. Hence, the accumulation of these acids causes acidification of the medium and low values of pH reduces the activity of microorganisms for acetogenic and methanogenic stages reducing the biogas production [11].

Figure 6. Biogas production yield from corn leaf biomass using different inoculum: substrate ratio and particle size

Figure 7. Accumulated biogas production using suitable values for particle size and inoculum to substrate ratio
As is shown in Figure 7, the highest amount of accumulated biogas (392.75 mL) was obtained for corn stalk biomass without pretreatment using 1 mm particle size and 1:1 I:S ratio. However, the highest yield (6.29x10^-2 m^3/kg SV substrate) was achieved for pretreated corn leaf biomass using in natura particle size and 3:1 I:S ratio.

![Figure 8. Biogas production yield using suitable values for particle size and inoculum to substrate ratio](image)

**4. Conclusions**

This work evaluated the effect of alkaline pretreatment on biogas production from corn crop residual biomass. The experiments performed to biomass without pretreatment indicated that highest biogas production was achieved using 1:1 I:S ratio. Regarding particle size parameter, it was identified as suitable values 0.5 mm particle size for corn stalk/leaf and corn leaf biomasses with biogas production of 342.75 mL and 387.5 mL, respectively. For corn stalk, 1 mm particle size reported 392.75 mL of biogas. The biogas production yield suggested that 3:1 I:S ratio is suitable for carrying out anaerobic digestion due to this potential of methane production considers biodegradability of substrate. The corn crop residual biomass pretreated with NaOH exhibited the highest yield 6.29x10^-2 m^3/kg SV substrate for corn leaf substrate using in natura particle size and 3:1 I:S ratio. These results suggested that lignocellulosic biomass from corn crop residues is suitable for producing biogas and that its yield is enhanced by the use of alkaline pretreatment.

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**References**


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