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Pretreatment Impact on Biomethanation of Lignocellulosic Waste

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Abstract

Lignocelluloses are often a major or sometimes the sole components of different waste streams from various industries, forestry, agriculture and municipalities. Hydrolysis of these materials is the first step for either digestion to biogas (methane) or fermentation to ethanol. However, enzymatic hydrolysis of lignocelluloses with no pre-treatment is usually not so effective because of high stability of the materials to enzymatic or bacterial attacks. Pre-treatment helps to improve the process of hydrolysis. In this work different methods of pre-treatment were studied.

The present work illustrates the effect of acid, alkaline pre-treatment on different sizes of wheat straw and anaerobic digestion of treated biomass for the production of biogas in batch stirred tank bioreactor under particular parameters. The quality and quantity of biogas produced was analysed by gas chromatography and water displacement methods respectively. The Untreated wheat straw gave a biogas yield of 104 ml/g and methane content of 64%. Acid treated wheat straw gave biogas yield of 130, 140 and 134 ml/g and methane content of 68%, 72%, 75% for 1%, 2%, 5% acid concentration respectively. Similarly, for alkali treatment gave biogas yield of 124, 128, 126ml/g and methane content of 66%, 69%, 71% for 1%, 2%, 5% NaOH concentration respectively.

Keywords: Pre-treatment; Lignocelluloses; Biogas; Anaerobic digestion; Batch stirred tank bioreactor

Introduction

Biofuels are rapidly becoming a significant partner in our future energy needs. It can provide means to mitigate deleterious impacts of greenhouse gas emissions. Production of waste materials is an undeniable part of human society. For effective waste management utilization of waste is necessary. Anaerobic digestion is the process by which the waste treatment can be done easily and side by side it can be useful for the production of valuable products such as Biogas and organic manure [1]. There are different types of wastes such as industrial, forestry, agricultural and municipal waste which can be used as substrate for the Biogas production. Wheat straw is one of the most abundant agricultural wastes produced in the world [2]. However, due to its complex structure and high lignin content, its degradability and gas yields are low. The degradability can be improved by pre-treatment, making the material more accessible to microbial degradation thus increasing the biogas yield.

The composition of wheat straw is cellulose-30%, hemicellulose-50%, and lignin-15%. Lignin is responsible for the integrity, structural rigidity and resistance to swelling of lignocelluloses. Delignification process therefore can improve the rate and extent of enzymatic hydrolysis from the matrix polymers. The reason for improved hydrolysis by removal of lignin might be related to better surface accessibility to enzymes by increasing the population of pores after removal of lignin.

Li Sun et al. investigated the microbial response to straw as a feed stock for biogas production [3]. The addition of straw, pre-treatment of straw and operating temperature all affects the cellulose degrading community in biogas digesters, but there were no major differences in the digester performance and gas yield. Nekema et al. evaluated biogas production in batch and Up-flow anaerobic sludge bed (UASB) reactors from pilot-scale acid catalysed steam pre-treated and enzymatic hydrolysed wheat straw [4]. The results showed that the pre-treatment was efficient and, a sugar yield of 95% was obtained. The pre-treatment improved the methane yield compared to untreated straw. Chandra et al. studied experimental methane fermentation on untreated, NaOH and hydrothermal pretreated substrates of wheat straw. NaOH pre-treated substrate produced 87.5% higher biogas production and 111.6% higher methane production compared to the untreated wheat straw substrate [5].

Bondesson investigated wheat straw in two different process alternatives with simultaneous saccharification and fermentation (SSF) to ethanol and anaerobic digestion (AD) to biogas [6]. In her study three types of pre-treatment were done steam pre-treatment with water, acetic acid or phosphoric acid. The overall best yield was obtained when using phosphoric acid as impregnation material. The highest methane yield, 754 ml CH_4/g fed was achieved. Prasad Kaparaju investigated wheat straw hydro lysate for biogas production in continuous stirred tank reactor (CSTR) and (UASB) reactors [7]. In his study methane yields increased with increase in hydro lysate concentration. However, he concluded that, biogas process was affected by the reactor type and operating conditions. None of above work has illustrated the effect of acid and alkali treatment on a particular sieved size wheat straw particle.

The present work studies pre-treatment of wheat straw with acid and alkali (1 vol%, 2 vol%, and 5 vol%) on mechanically grinded wheat straw (<75 µm, 75 µm, 150 µm, 212 µm 425 µm). It was shown that highest lignin content was extracted in 425 µm. The objective to use 212 µm particle size wheat straw was due to its abundant production during grinding. Hence, process was economical.

Material and Methods

Standards chemicals used (conc. sulphuric acid, sodium hydroxide

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and distilled water) were from Rankem. A Standard lignin used was from Merck. Standard biogas sample were from Centurion scientific pvt ltd.

Preparation of standard lignin solution

Standard lignin samples of 1 and 10 ppm were prepared by dissolving weighed quantity of pure lignin in appropriate volume of distilled water. The substrate wheat straw was collected from Dehradun.

Experimental method

Mechanical treatment of wheat straw: Raw wheat straw procured from Bidholi, Dehradun was first ground. The powdered sample was sieved. Five different sample sizes were collected: < 75 µm, 75 µm, 150 µm 212 µm, and 425 µm.

Estimation of acid soluble lignin content: 0.3 g of different particle size samples were taken mixed with 3 ml of 72% conc. H_2SO_4 [8] and kept in a water bath for 2 hrs for estimation of acid soluble lignin. The solution obtained was mixed with 84 ml water and autoclaved for 30 min at 121°C and 15 psi. Autoclaved solution was then filtered to separate residues and filtrate. Filtrate solutions were diluted to 100 ppm and the same were studied under UV spectrophotometer at 205 nm for lignin content. The process is shown in Figure 1.

Pre-treatment of wheat straw

Wheat straw pre-treatment was done by both acid and alkaline methods.

Sulphuric Acid pre-treatment of wheat straw: 50 g of mechanically treated wheat straw of 212 µm was treated with 1% H_2SO_4. The sample was mixed with 500 ml of 1% H_2SO_4 solution and kept in orbital shaker at 70°C, 150 rpm for 2 hrs. The solution was filtered and the residual biomass was washed twice with water and then dried. Similar treatment was done with 2% and 5% H_2SO_4 also. Figure 2 shows colour variation of the sample after treatment with 1%, 5% H_2SO_4. Colour difference indicates the removal of lignin in Figure 2. This pre-treated biomass is used for anaerobic digestion in reactor.

Alkali pre-treatment of wheat straw: Wheat straw was treated in presence of 500 ml of 1%, 2%, 5% NaOH in an orbital shaker at 150 rpm for 2 hours at 70°C. Reaction mixture was filtered, washed twice with water and then dried. The residual biomass was used for anaerobic digestion in batch reactor for biogas production.

Anaerobic digestion of pre-treated wheat straw: Wheat straw of size 212 µm after pre-treatment was digested in a bio-reactor. The pH of the slurry was maintained neutral. The cow dung was used as inoculums. The set-up is shown in Figure 3. Gas was collected in glass collecting port via gas nozzle of digester. The quantity of biogas produced was determined by the water displacement method. The reaction conditions maintained in bioreactor are given in Table 1.

Figure 1: Schematic representation of estimation of soluble lignin from wheat straw.

Figure 2: Acid (1%.5%) pre-treated wheat straw.

Figure 3: Batch type stirred tank reactor setup.

Reactor	Batch
Working volume	500 ml
Total volume	1 lt.
pH	6.8
Temperature	38°
HRT	4 days

Table 1: Reaction specification.

Gas analysis: Bio gas was analysed by GC (Nucon 5700). GC conditions are given below:

Injection volume-100 µl

Mobile phase-Argon

Column Make-Stainless Steel

Column ID-(HEYSEP.Q)

Run time-10 minute

Results and Discussion

Delignification of wheat straw

The absorbance of lignin standard was determined at 1 ppm and 10 ppm as 0.791 and 3.261.

Estimation of acid soluble lignin content

The observations obtained from acid pre-treatment of wheat straw are summarized in Table 2 and Figure 4.

Absorbance is increased as the particle size of the wheat straw is decreased. It was found that mechanical treatment had good impact on delignification. Particle (<75 µm) yield more lignin because particle has large surface contact with acid 212 µm yield 10.7% lignin.

Acid pre-treatment of wheat straw

Above observation indicate that the acid soluble lignin removal was found more in 5% H_2SO_4 reaction condition as shown in Table 4. But

Particle size	Absorbance of soluble lignin (1 ppm)	Absorbance of soluble lignin (10 ppm)	%lignin (1 ppm)	%lignin (10 ppm)
425 nm	1.363	1.643	0.8	10.5
212 nm	1.370	1.671	0.9	10.7
150 nm	1.376	1.732	1.0	11.1
75	1.379	1.759	1.1	11.3
<75	1.387	1.836	1.2	11.8

Table 2: Effect of particle size on lignin removal.

% H_2SO_4	Absorbance of 100 ppm solution	Lignin removal (g/lit)
1	3.205	2.836
2	3.312	2.930
5	3.506	3.102

Table 3: Acid treatment.

%NaOH	Absorbance 100 ppm solution	Lignin removal (g/lit)
1	2.945	2.606
2	3.102	2.745
5	3.314	2.932

Table 4: Alkali treatment.

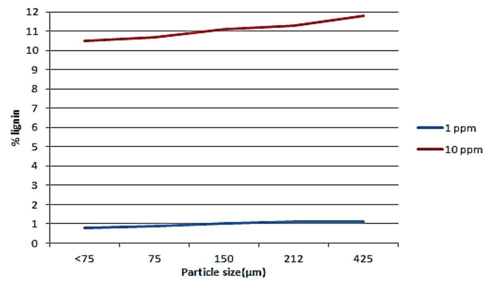
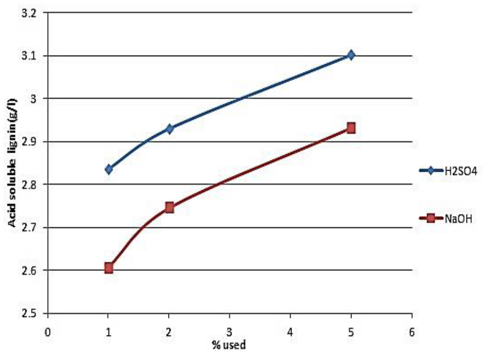


Figure 4: Comparative graphical representation of lignin removal.



Above figure indicates that acid treatment has more impact on lignin removal than alkali.

Figure 5: Comparative effect of alkali, acid treatments on lignin removal (g/lit).

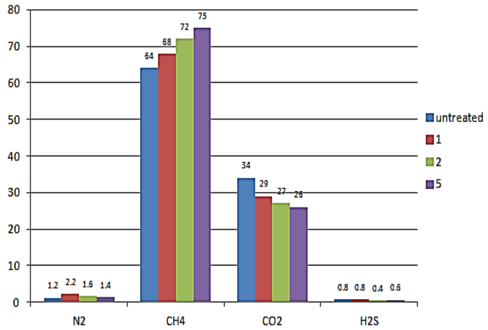


Figure 6: Comparative chromatogram of biogas after (1%, 2%, 5%) acid treatment.

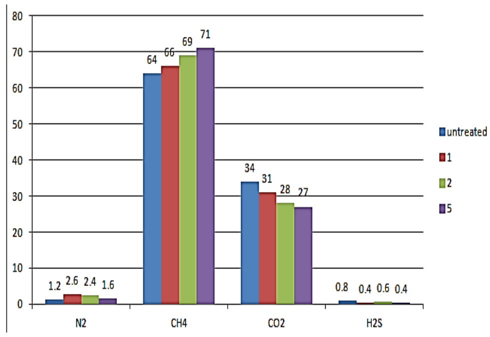


Figure 7: Comparative chromatogram of biogas obtained after (1%, 2%, 5%) alkaline treatment.

2% H_2SO_4 was feasible to use for acid hydrolysis and moreover lignin removal was found nearby to 5% H_2SO_4 (Table 3).

Alkaline pre-treatment of wheat straw

The observations obtained from alkaline treatment of wheat straw are summarized in Table 4.

Results of alkaline pre-treatment show that 2% NaOH stage is beneficial for the removal of lignin, but more lignin was extracted out in the presence of 5% NaOH as shown in Table 4 and Figure 5.

Biogas production from acid and alkaline pre-treated wheat straw

A standard Biogas sample was run on GC. The composition was (CH_4-62%), (CO_2-36%). This is used to calibrate the GC.

The chromatogram indicates percentage of methane content increases from 64% to 75% with increase in the concentration of sulphuric acid from 1% to 5%. As it already seen in Figure 4 that 5% acid has good impact on lignin removal. Acid treatment also disrupts crystalline structure of cellulose. Hence accessibility of microorganism to biomass surface increases the 2% acid pre-treatment give better biogas methane yield comparable 5% sulphuric acid. Hence preferred because it is found economical than 5% acid pre-treated as require less alkaline solution to maintain pH of pre-treated slurry (Figures 6 and 7).

Above chromatograph indicates percentage methane content was found more after 5% alkali treated wheat straw as compared to 2% NaOH. 5% alkali pre-treatment extracts more soluble lignin as compare to other.

The Figures 8 and 9 indicates biogas yield/g was found more after 2% acid pre-treated and wheat straw while the rest of treatment also

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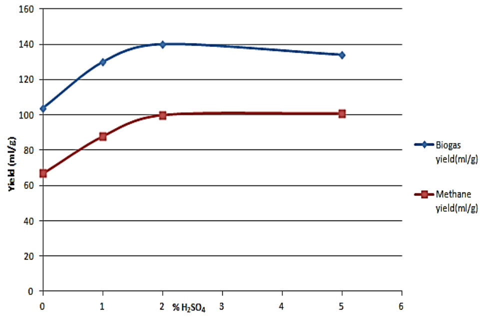

Figure 8: Yield of biogas, methane/gm from acid pre-treated wheat straw.

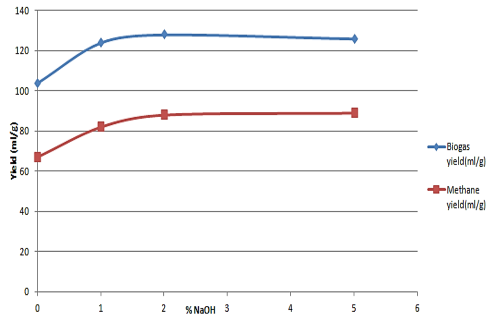

Figure 9: Yield of biogas, methane/g from alkali pre-treated wheat straw.

yield good volume of biogas per gm of biomass. Methane yield was found more after 2% acid, 5% alkaline pre-treated wheat straw

Conclusions

Different pre-treatment methods for delignification of wheat straw were tried. 5% conc. H_2SO_4 yields more acid soluble lignin as compared to 2% conc. H_2SO_4, but 2% yield more biogas. Alkaline pre-treatment is also good method for delignification of wheat straw. However, the methane content of biogas obtained from acid treated biomass was 10% more as compared alkaline treated biomass. Acid treated wheat straw can be further upgrade to pilot plant to yield biogas which can be used for power generation to solve growing energy demand. Acid pre-treatment provide pathway for utilization of wheat straw for energy production.

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