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## Study on Catalytic Bio-Digestion of Sugarcane Bagasse Fiber.

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#### ABSTRACT

Bagasse like natural fiber reinforcement reduces the weight of the composite. However bagasse has residual sugar content which can be digested for biogas production through Anaerobic Digestion (AD). The sugarcane bagasse was delignified to remove lignin and hemicellulose. The bagasse was treated with 10% NaOH at 90 °C for 1.5 h and 10% PAA at 75 °C for 2 h. Production of biogas from treated and untreated bagasse was studied. The enzymatic digestibility of sugarcane bagasse was also greatly increased by alkali (NaOH)–peracetic acid (PAA) pretreatment. The temperature in the digester was maintained at 35-40 °C and pH between 6 and 7. Synthesis of iron oxide nanoparticle was done and used in the digester as catalyst. The gas chromatography (GC) test was done and the methane yields of the treated and untreated bagasse used in the bio digester were assessed. Scanning Electron Microscopy and Thermogravimetric anlysis of bagasse fiber before and after digestion is reported.

Keywords: Anaerobic Digestion; Bagasse; Peracetic acid; Gas chromatography.

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#### INTRODUCTION

Abundant quantity of agro fiber resources like sugarcane is available in many countries including India. Reported sugarcane annual production in India is 351,118,496 tonnes during the year 2013 [1]. Bagasse is the residue fiber remaining when sugar cane is pressed to extract the sugar. Traditionally, bagasse has been used as a fuel in the sugar factories as it has a good calorific value and, in smaller quantities, in paper mill. Nowadays a small quantity is also used in the particle board manufacturing [2].

Bagasse is composed of fibre and pith, the fibre is thick walled and relatively long. These fibres are mainly made of cellulose, hemicelluloses, lignin and pectin's, with a small quantity of extractives [3]. Compared to glass fibre and carbon fibres, natural fibres provide many advantages, such as, abundance and low cost, biodegradability, flexibility during the process and less resulting machine wear, minimal health hazards, and low density. Shortage of energy at rural areas needs the technologies of bio energy conversion [4]. Usage of waste materials for the conversion of energy has taken a significant step in sustainable development. The best method for producing biogas is by anaerobic digestion, which requires low investment and has good stability and is eco-friendly [5]. Anaerobic digestion process is simple and economical [6].

In the bioconversion of lignocelluloses biomass to fermentable sugars, the raw biomass should undergo pre-treatment to increase its enzymatic digestibility [7]. The specific purpose of pre-treatment is to reduce the sugar yields and to increase the bio-digestibility, also in the removal of lignin and hemicelluloses. It helps in disrupt the cellulose crystallinity and increase the porosity of the material [8, 9].

Therefore studying the properties of digested bagasse and comparing it with undigested one is important. The objective of this paper is to study the composition of biogas produced from treated and untreated bagasse and to study the structural characteristics of the bagasse fiber after chemical treatment with alkali-acid. The modification in the fibre structure was evaluated by scanning electron microscope (SEM) and Thermogravimetric Analysis (TGA).

#### **EXPERIMENTAL SECTION**

#### **Material description**

The bagasse obtained from nearby juice centre was dried and chopped into small pieces. About 6 kg of finely pieced bagasse was washed and dried under the sun. To create a digester a 50 L plastic drum was taken, a valve setting was done for the collection of the gas. The cap of the drum was kept air tight by using cotton threads and teflon linings. The digester has to air tight such that anaerobic bacteria grow inside the digester. Another digester of 30 L drum was used to digest 2 kg untreated bagasse. The pH of the slurry in the digester was initially at 4.5 once mixed with cow manure (source of anaerobic bacteria) with equal volume of water later reached to ~6.5.

#### **Pre-treatment with PAA**

Two stages of pre-treated process were carried out. 10 g of NaOH pallet was taken in a 100 mL flask, and then dissolved in 100 mL of water to form 10% of NaOH solution. For this solution raw bagasse of 5 g was added and heated in an oven at 90°C for 1.5 h. The sample was taken out from the oven washed and dried. The dried sample was again treated with PAA and heated in oven at 75°C for 2 h [4, 5]. Peracetic-acid was prepared by using hydrogen peroxide and acetic acid. 5% of acetic acid combined with 3% of hydrogen peroxide forms a peracetic acid solution.

#### $CH_3COOH + H_2O_{\clubsuit}CH_3COOH + H_2O$

The pre-treatment was conducted to two other raw biomass i.e. rice husk and saw dust along with bagasse to check the increase in digestibility of sugarcane bagasse compared to the other two. With same chemical composition all the three substances were taken in a 50 ml glass beaker and 5g of each sample was taken and first it was treated with 10% NaOH solution and kept in an oven for heating at  $90^{\circ}$ C for 1.5 h. After



this process the contents were washed thoroughly with DI water and dried such that there was 25% moisture content in the substance. SEM images of the treated bagasse and digested bagasse was also obtained. Similar treatment procedure was followed for remaining 4kg bagasse to be put in a 50 L digester.

#### Treatment with iron oxide nanoparticles

Synthesis of iron-oxide nanoparticle was done using polyol process [10]. Iron oxide nanoparticle reduces the retention time and digestion temperature [6]. The synthesis was done by reduction of ferric chloride using ethylene glycol. Here 2 g of ferric chloride is reduced by 40 mL ethylene glycol and 0.1 ml 34% HCl. The solution is heated and stirred until the solution become brown. The nanoparticles are formed in the solution. Precipitate is repeatedly washed with ethanol to remove unused FeCl<sub>3</sub>. The separation of nanoparticles from the solution is done by centrifugation. Approximately 0.2 g nanopartcle is used per kg of dry bagasse in the digester.

#### Gas chromatograph (GC)

Gas chromatography (GC) is a common type of chromatography used in analytical chemistry for separating and analyzing compounds that can be vaporized without decomposition. Typical uses of GC include testing the purity of a particular substance, or separating the different components of a mixture (the relative amounts of such components can also be determined). In some situations, GC may help in identifying a compound. In preparative chromatography, GC can be used to prepare pure compounds from a mixture. In gas chromatography, the mobile phase (or "moving phase") is a carrier gas, usually an inert gas such as helium or nitrogen. The stationary phase is a microscopic layer of liquid or polymer on an inert solid support, inside a piece of glass or metal tubing called a column (a homage to the fractionating column used in distillation). The instrument used to perform gas chromatography is called a gas chromatograph (or "aerograph", "gas separator"). The gaseous compounds being analyzed interact with the walls of the column, which is coated with a stationary phase. This causes each compound to elute at a different time, known as the retention time of the compound. The comparison of retention times is what gives GC its analytical usefulness.

The GC was a single column GC with helium as a carrier gas. A TCD detector was used in the column for the detection of the gas. The temperature was set to 200°C. The pressure was at 83.6 kPa and the velocity of the flow was 25.4 cm/sec having a split ratio of 10 (name of the column: Stabilwax). The column length of the column was 30 m and inner diameter of 0.25 mm.

#### **RESULTS AND DISCUSSIONS**

#### Gas chromatograph test (GC)

After 60 days of digestion gas sample were collected in four 0.5 L rubber balloons from each digester and sent for analysis. Gas chromatograph was done using TCD detector and using helium as a carrier gas. Both the graph detected methane and the concentration in each sample is as shown in the graphs (fig. 1, 2). The graph (fig 1) shows the composition of gas sample obtained from the digester that contained untreated bagasse.

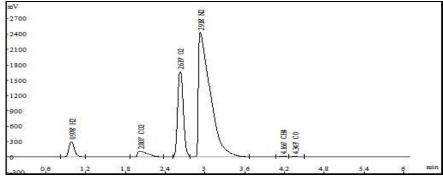


Fig. 1 GC test of biogas obtained from a digester containing untreated bagasse.



Rt Time	Area	Height	Concentration	Name	
0.978	294245	300	0.4380	H <sub>2</sub>	
2.007	115169	256	44.32	CO <sub>2</sub>	
2.619	1668358	1796	15.35	02	
2.918	2425337	2400	69.43	N2	
4.166	23728	105	0.1057 CH <sub>4</sub>		
4.343	16232	110	0.1144 CO		

Table 1: Properties of the peaks obtained from the graph.

From the graph (fig 2) and the table shown (table 2) it is clear the concentration of air that mainly contains nitrogen and oxygen is more compared to methane. This can be due to the contamination of the sample gas with air during the filling or during the testing process. This can be avoided by proper designing of the gas collection process. From the two graphs we can clearly see that the treated bagasse helped in better production of gas from the digester. This graph shows more amount of nitrogen and oxygen is due to contamination of air during filling of the gas or while testing the sample.

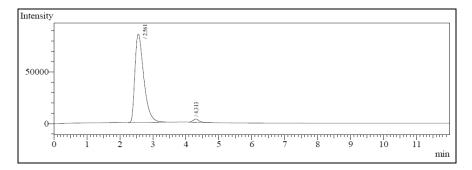


Fig 2 GC test of biogas obtained from a digester containing treated bagasse.

Table 2 properties of the peaks obtained from the graph.

Rt Time	Area	Height	Concentration	Name
2.561	1593460	85515.7	96.48503	Air
4.313	37411.7	3115.4	3.51497	Methane Gas

#### Scanning electron microscope (SEM)

Bagasse fiber can also be used as natural fiber reinforcement in composites [3]. Therefore it is interesting to know whether digested bagasse fiber retains the similar structure or not. This SEM image shown in Fig. 3 is the image of a digested bagasse which is treated with sodium hydroxide and peracetic acid. In this image we can clearly see the porous in the medium. This shows the digestibility of the material has an increased compared with raw bagasse [12]. We can see how the fibers have separated and bumps in the image show the removal of lignocelluloses content in the material.

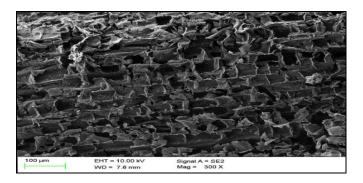


Fig 3. SEM image of a bagasse kept in a digester.



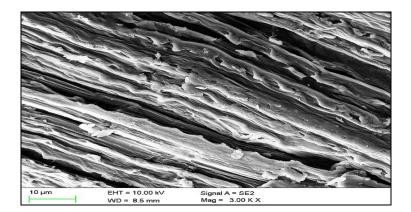


Fig 4 SEM image of a bagasse which is treated with PAA.

In this SEM image (Fig 4), the decomposed material is characterized. Clearly the long fibers are broken compared with undigested bagasse. Therefore digested bagasse may not be suitable as natural fiber for reinforcement.

#### Thermogravimetric analysis testing (TGA)

The thermogrvimetric analysis (TGA) of synthesized samples is performed to study the stability of the samples. The corresponding Derivative thermograms (DTG) are also shown. In the TGA graph at the starting of the temperature the material starts drying [14]. The first deformation of the curve signifies thermal decomposition with the formation of gaseous reaction products (Fig 5 (a)). Another deformation at the end of the graph indicates decomposition of the material around 400°C. Many gravimetric effects have nothing to do with chemical reaction or melting process. The most frequently observed are drying steps. These usually occur at the beginning of the temperature program. This graph also shows an escape in moisture content between 100°C to 120°C. The treated bagasse remains stable till 230°C whereas digested bagasse decomposes at 200°C. However this temperature is higher than other natural fibers like wood [15].

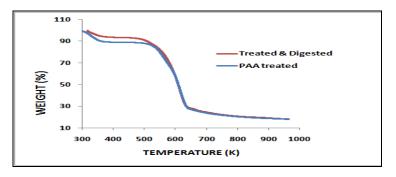


Fig. 5(a) TGA graph of PAA treated and digested bagasse;

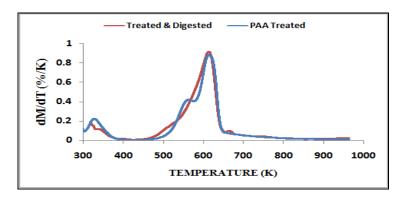


Fig 5(b) DTA graph of PAA treated and digested bagasse.

8(1)



The material under study and an inert reference are made to undergo identical thermal cycles. Any temperature difference between sample and reference is recorded. The differential temperature is then plotted against time or against temperature (DTA curve or thermogram) [14]. The peak in the graph shows the temperature at which the material melts or oxidizes (Fig 3 (b)). Fine -grained powder should be used to achieve greater contact area and better equilibrium conditions. The time at any temperature must be sufficiently long in order to permit completeness of reactions [14]. If an exothermal event takes place, then the temperature of the sample will exceed that of the reference and a maximum will be observed on the curve. This graph shows a peak formation at a temperature of 340°C. This sharp increases in the peak shows that the material is undergone an exothermic reaction. Hence it can be proved that this bagasse contains substantial amount of energy. Therefore even the digested bagasse is high on energy content hence can be dried and combusted suitably.

#### CONCLUSION

Sugarcane bagasse is an agro natural fiber and used as reinforcement in composite. But the bagasse can be digested to produce biogas. The bio-digestion by anaerobic process is found to be suitable method to produce the energy from bagasse. The pre-treatment process should be chosen to accelerate digestion process. The iron oxide nanoparticles improve biogas production. When using fibrous material it should be treated properly to remove the lignocellulose material. The temperature and pH also should be maintained to get a better yield of biogas. The SEM image of the treated bagasse and digested bagasse was taken. This shows the digested bagasse decomposes completely. The TGA analysis of the bagasse was done to check the thermal stability of the fibers. Even the digested bagasse has comparable thermal stability with undigested one.

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