Biogas Production from Local Agricultural waste by using Laboratory Scale Digester

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Abstract

In rural areas, particularly in Jalgaon district, plenty cellulosic biomass in form of agriculture waste, are available in huge quantity. It has a bio-energy potential to fulfill the energy demand. Production of biogas using biomass is excellent solution in rural development technology to fulfill energy needs. Variety of regional lignocellulosic material is available whose biogas potential to be unexplored. Biogas production using lignocellulosic residues may improve energy availability, reduced the green house gases and pollution. Adaptation of small scale biogas digester could be the answer of solving these problems. The production of biogas mainly depends on available feedstock, carbon to nitrogen ratio and its other parameters. Bio energy potential of local agriculture waste or residues sample was studied by physical, chemical and thermal analysis. Chemical analysis of this agriculture waste showed less quantity of moisture, volatile solids and total solids, total nitrogen content while higher in and calorific values with carbohydrate content. On the other hand the chemical, physical, thermal treatment does improve neither the quality nor quantity of biogas by using agriculture residues as renewable source of energy. The present work is an attempt to study on the chemical analysis of agricultural waste with respect to its various parameters for biogas production to make an active feedstock. The paper also highlights the general presentation for design a small-scale biogas unit intended to be used for further analysis of screened materials for biogas production.

Keywords: Lignocellulosic, biogas digester, feedstock, nitrogen ratio, total solids, volatile solids, calorific values.

Introduction

Jalgaon is situated in the northern part of Maharashtra State in India. It has an area of 11,765 km$^2$ and a population of 3,682,690 (2001 census) of which 71% is rural. Majority of rural population depends on available agro-waste to fulfill energy demands either by use in domestic stove through burning it, in cattle feed and surplus waste is utilize to prepare compost. The designing of family or Lab-scale biogas plants is the prime important and necessary to work in the local rural setting. Not only designing but also maintenance of such plants should be minimal and that work smoothly for long time. Biogas production from local agro-waste by using Lab-scale digester minimises the use of agro-waste in burning for cooking food that result in controlling pollution by reduce emission of greenhouse gases. In Jalgaon district near about 60% of agro-waste is generated from crops like jawar, cotton, bajara, maize, grams and banana annually in post harvest treatment. This agro-waste has energy potential therefore necessary to utilise such agro-waste properly without creating pollutions. Adaptation of biogas technology by using lignocellulosic waste with cattle dung as source of microbes is eco-friendly technology. It is the prominent agriculture waste, renewable source and perennial crops with good calorific value, low ash content and cost effective.

The agro-waste contains large quantity of lignin, cellulose, hemicelluloses, nitrogen and carbon contents which is important source for biogas production. Various parameters such as total volatile solids, moisture, total solids, calorific values, and are among the main parameters affecting biogas production$^1$. Screening of suitable lignocellulosic substrate for biogas production is necessary to determine bio-energy potential of agro-waste. It includes determination of total solids (TS), volatile solids (VS), Calorific value, % total carbohydrates, moisture, lignin and cellulosic contents etc. In Farming practices, every year before sowing large amount of agriculture residues are burnt to avoid the problem of weeds and other plant pathological problems. On-form burning of agriculture residues creates the problems of environmental pollution, release of particles during burning leads to human and animal health problems. During burning of crop residues greenhouse gases are emitted like CO$_2$, CH$_4$, and nitrous oxide that causes global warming and major elements from plant like N, P, S and K are also loss that play a role in recycle of this element for next crop generation$^2$. In India, because of unavailability of sufficient crude oil and to fulfill the need of energy at domestic as well as at industrial sector, there is urgent need to find alternative, eco-friendly and safer source of energy. At the same time due to high transport costs end prices of fuel and fertilizer is expensive therefore necessary to find out cheaper sources of energy from natural resources. Use of biogas is excellent solutions in rural development technology to fulfill energy needs. Biogas is
metabolic product of anaerobic digestion, is mixture of methane
and carbon dioxide with minimum quantity of other gases like
hydrogen sulphide. The biogas using agro-waste and cattle
dung has a dual applications one is to manage waste and to
produce valuable energy from natural resources.

Related with the aspect present above, the next section of the
paper will present designing of lab scale anaerobic digester in
order to study the particularities of the anaerobic fermentation
process and for the process of degradation of different types of
agricultural waste material. It is necessary to increase the
participation of agriculture waste as renewable sources on the
whole energy production. In order to improve biogas production
quality and efficacy, the use of agricultural wastes
compositional data can be an effective tool to improve
efficiency and production of biogas. In this study designed a
small scale biogas plant for household production of biogas
which can be used as fuel for domestic purpose like for cooking,
boiling water and power generation etc. It is easy to handle and
there is no need of any expertise for its maintenance. Centre of
this system is agro-waste management and eco-friendly energy
utilization.

Material and Methods

Design of laboratory scale biogas digester unit4-5: The
materials required construction for biogas unit are as follows:-
A plastic tank of 100 litres capacity, PVC pipe (diameter- 2.5
inch), MTA-3(2.5 inch), FTA-3 (2.5 inch), Elbow (2.5 inch), T-
Pipe(1.25 inch), Check nuts, tank nipple, Cap (2.5 inch),
Reducer (2.5inch ×1inch), Garden pipe, plastic transparent jars-
2, valves (1.25 inch), PVC cement, necessary equipments etc
show in fig no.1

Figure-1
Necessary equipments

The main parts of Laboratory scale biogas production unit
includes, Batch bio digester, Inlet pipe, Outlet pipe, Overflow
slurry collection unit, Gas collection unit

Batch Bio-digester: A plastic tank of 100 litres capacity is used
for the construction of the bio-digester. It consists of a lid
containing a inlet pipe and gas line. At the bottom of digester
there is outlet pipe connected to digester. At a height of 41 cm
from bottom there is slurry outlet5.

Inlet pipe: PVC pipe of 65 cm height is used as the inlet. It is
inserted in the tank through lid which opens at bottom of tank
such that it will dip in the waste matter in tank. At the top of this
pipe there is a wide open end to add inoculums, waste food, cow
dung easily.

Outlet pipe: A PVC pipe of 18 cm is used as outlet located at
the bottom side of bio-digester. It is used for removal of all
content at the time of biogas plant washing.

Overflow slurry collection unit: Overflowed slurry produced
in the bio-digester is collected by using a slurry outlet at a
height just above the working volume of bio-digester. Slurry
produced when increased above the working volume is flowed
outside the bio-digester through the slurry outlet and collected
in a airtight bottle which can be used again as inoculums for the
digester. The slurry collection bottle is properly airtight which
will prevent formation of odour.

Gas collection unit: Biogas produced in the bio-digester will
flow through the gas line which consists of three valves for
proper collection of biogas. It is collected in a transparent
plastic jar by downward displacement of water.

Methodology

For Bio-digester: Plastic tank of 100 litres capacity was taken.
Two circles of diameter 2.5 inch were marked on tank, one at
bottom and another at height of 41 cm from bottom. Two holes
diameter of 2.5 inch were cut at those two marks with the help
of hot metal ring. MTA and FTA of same diameter were
attached in the holes and attach 2.5 inch PVC pipes on them.
Bottom PVC pipe is closed with end cap which is batch washing
outlet. Second PVC pipe at upper side was attached to reducer
and connected to slurry container. A hole of diameter 2.5 inches
was cut on the lid and same size MTA and FTA were attached
to it. A pipe of same diameter and length 65 cm was inserted in
that such that it just touches the bottom of tank and was fixed in
lid which acts as inlet. Another hole of diameter 1.25 inch was
cut in the lid and same size tank nipple was attached in lid and
to the gas valve which act as gas outlet which is further
connected to gas reservoir.

For Gas Reservoir: For making reservoir of gas, air tight jar
was taken and garden pipe was fitted in that and filled with
water. This jar was placed in plastic tray containing water.

For Slurry Collection: Reducer of upper PVC pipe was
attached to garden pipe and inserted in the tank for collection of
overflowed slurry. Finally, all the attachments were sealed with
the help of PVC cement and M-seal to maintain anaerobic conditions and to prevent leakage of produced gas.

**Analysis of bio-energy potential of regional agro-waste:**

**Sample Collection and Preparations:** Lignocellulosic substrate for biogas production, five crops from Jalgaon region was identified and samples were collected. Selection of samples on the basis of their availability, annual production per acres, residues generated per quintal, strategies used for disposal of agro-waste. Sample chosen for analysis of bio-energy potential and substrates for biogas production were soybean straw, wheat stalk, ground nut shells, black gram straw and red gram straw. Selected samples were dried at room temperature then subjected to oven treatment at 105°C for 4 hrs, fine powder prepared by passing through 250 mm sieve and stored in air tight polythene bags for further analysis.

**Estimation of the total solids of agro-waste:** Total solids were determined by subtracting moisture from hundred percent. Loss of weight when ignited at 105°C was measure of total organic content of agro-wastes.

\[
\%TS = \frac{\text{Weight of petridish with sample} - \text{Weight of petridish}}{\text{Weight of sample}} \times 100
\]

**Estimation of the moisture content** of agro waste: Moisture content of agro-waste was measured by loss of weight after drying at temperature 105°C for 4 hr. Moisture content usually is given to priority because too much moisture leads to anaerobic condition, toxic substance, mal odours and slow decomposition. Moisture content using the following equation,

\[
\%MC = \frac{\text{Weight of sample} - \text{Weight of sample after drying}}{\text{Weight of sample}} \times 100
\]

**Estimation of the volatile solids agro waste:** The amount of residues left after the ignition of oven dried sample at 575°C. To determine volatile solid and Ash analysis, oven dried moisture free sample were weighed as 1g each in moisture free and pre weighed crucibles and place at the muffle furnaces at 575±5°C for minimum 4hrs. The volatile solids % and ash% were calculated as following formulae,

\[
\% \text{ Ash} = \frac{\text{Weight of crucible sample} - \text{Weight empty crucible}}{\text{Weight of crucible}} \times 100
\]

\[
\% \text{ VS} = 100 - \% \text{ Ash}
\]

**Estimation of calorific value of agro waste:** Calorific value measured by using Toshniwal oxygenic bomb calorimeter. Oven dried 1g samples were used to making special small tablet by using hand operated press. Then tablet subjected to burn in the oxygen enriched atmosphere of the bomb of calorimeter were estimated in terms of cal/g. The calorific value indicates that the burning abilities and actual energy available in agro-waste presented in per unit mass – MJ/kg.

**Determine the total carbohydrate of agro biomass by phenol sulphuric acid method:** Prepared sample subjected to total carbohydrate analysis as, in hot acidic medium glucose is dehydrated to hydroxyl methyl furfural. This forms a green colour product with phenol and has absorption maximum at 490nm. Weigh 100 mg of the sample into a boiling tube. Hydrolyze by keeping it in a boiling water bath for 3hrs with 5ml of 2.5 N HCl and cool to room temperature. Neutralize it with solid sodium carbonate until the effervescence ceases. Make up the volume to 100 ml and centrifuge. Collect the supernatant and take 0.5 and 1ml aliquots for analysis. Pipette out 0.2, 0.4, 0.6, 0.8 and 1ml of the working standard into a series of test tube. Pipette out 0.1 and 0.2 ml of sample solution into two separate test tubes Make up the volume in each tube to 1ml with water. Set a blank with 1ml of water. Add 1ml of phenol solution to each tube. Add 5ml of 96% sulphuric acid to each tube and shake. After 10 min shakes the content and place in water bath at 25-30°C for 20 min. Measure colour at 490nm and calculate amount of % total carbohydrate present in sample solution by using formula,

\[
\% \text{ total carbohydrate} = \frac{A - A_0}{0.4} \times 100 \text{ mg of glucose}
\]

**Estimation of nitrogen of agriculture waste:** Total Kjeldahl’s nitrogen method is used to analyze nitrogen contained in organic molecules and ammonium but not nitrate. Agricultural waste contains nitrogenous compounds and are usually proteins which are converted to ammonium by anaerobic digestion. The sample were digested with conc. H₂SO₄ in the presence of catalyst under which condition the nitrogenous compounds are converted to ammonium sulphate by steam distillation in the presence of strong alkali, ammonia is liberated which can be estimated by suitable means on the average most protein have 16% nitrogen in the composition. In the other words, 1mg nitrogen equals 6.25 mg protein. Thus by finding out the amount of ammonia formed from a known amount of sample, one can calculate the amount of protein present.

Ammonia gas captured by boric acid and to form ammonium borate complex. As the colour of the receiving solution changes from bluish green to pink when the ammonia collected, and the equation is given by,

\[
\% \text{ Nitrogen} = \frac{(\text{Standard acid (ml) - Blank (ml)}) \times N \text{ of acid} \times 1.007}{\text{weight of sample in gm}}
\]

Reagents: 2% boric acid solution made by dissolving 10gm of boric acid in 480 ml of distilled water. 20 ml of 0.1% bromocresol green added to this solution (in 95% ethanol) and 4 ml of 0.1% methyl red solution. Standard HCl solution 0.01 N, Sodium Hydroxide solution 40% w/v. Conc. Sulphuric acid, Catalyst Selenium dioxide powder, Standard 1% ammonium sulphate solution.
Processing the sample: A weighted quantity of the sample (about 0.5ml solution) containing approximately 1mg of sample is taken in Pyrex digestion tube. Add 1ml of conc. H₂SO₄ carefully and pinch of (about 20mg) catalyst. Place the tube in digestion rack or sand bath at low flame so that the solution just boils. Continue the heating till the solution become colourless or clear. If necessary a few drop of H₂O₂ may be added at the final stage to clear digest. After cool make up the known volume.

Standardization: Introduce known amount of standard ammonium sulphate solution (total volume 10ml) into flask C through the funnel. Then add 10 of 40%NaOH through the funnel. Take 10 ml boric acid solution in flask at the condenser delivery end in such a way that the tip is beneath the surface of liquid. Now heat the flask A filled with water to produced steam this steam is passed through the content of the flask C and the ammonia form there by the alkaline treatment of ammonium sulphate is carried along with it through the condenser outlet. Ammonia is fixed by boric acid as ammonium borate. Continue passing steam for 10min then remove the receiver flask after rinsing out the tip of condenser. Stop heating of flask A, on cooling this will create a back suction so that the content in flask C will be sucked into trap B. Add about 10ml of water through the funnel quickly, so that it also be sucked into flask B, while rising flask C the apparatus is now ready for the next round of operation. The content in the receiver flask are now titrated against 0.01N HCl, till the colour changes from bluish green to pink. Note the volume of acid used and calculate the strength of ammonia in the distillate and repeat with different concentration of ammonium sulphate. A blank titration without any ammonium sulphate will be useful.

Calculation: On the basis of stoichiometric relationships involved in the titration, 1ml of 0.01N HCl is equivalent to 140µg of nitrogen as present in ammonia. Thus from the volume of the standard HCl used for titration the amount of nitrogen in sample can be calculated. The calculations for % nitrogen or % protein must take into account which type of receiving solution was used and any dilution factors used during the distillation process. If the sample weight is in milligrams, the molecular weight of nitrogen should be changed to 1400.67.

Figure-2
Prepared samples of Agro-wastes
Results and Discussion

A laboratory scale biogas production unit is designed. All parts are assembling as per production of biogas at laboratory scale. Bio energy potential of agro waste sample was studied by physical, chemical and thermal analysis. The moisture content of selected local agriculture residues like soybean straw (12.2%), black gram stalk (14.8%) and wheat stalk (15%) was found to be higher as compared to ground nut shells (8.9%), red gram straw (11.9%) shown to figure 3. Analysis of moisture in solid substrates is co-related with growth of microorganism. As microbes can absorbs only dissolved solids, so moisture content in residues provide a medium for metabolic and physiological processes. Volatile solids of wheat stalk (73.1%), soybean Straw (71.2) and black grams stalk (70.1) was also much higher than that of red gram straw (67.4%) and ground nut shell (68.2%). The total solids of wheat stalk (25.0%), Soybean straw (23.2%) and Ground nut shells (28.1%) were found to be more. % Ash content of wheat stalk (18.8%), ground nut shell (16%) were found to be higher when compared with the soybean straw (12.50%), black grams stalk (11%). Calorific values of Soybean straw (4170.211Kcal/Kg), Ground nut shells (4200.635Kcal/Kg), Black Gram stalk (3938.265 Kcal/Kg) were found to be high as shown in figure 4; which serve as potential of biogas production. The production of biogas depend on the choice of feedstock with its C:N ratio. The carbohydrates present in feedstock provide energy to producer organism and supports its growth. % total carbohydrate was estimated by phenol sulphuric method. It was found that black gram stalk (30.1%), wheat stalk (26.4%), soybean straw (21%) as higher carbohydrate content while as groundnut shells (11%) red gram straw (13%) shown in figure-6. Nitrogen content was also most important parameters for good anaerobic digestion. Additionally, nitrogen also serves essentially as a constituent of protoplasm. % Nitrogen content estimated by TKN method was found to be 1.8% in red gram straw, 1.9% in groundnut shells as higher nitrogen content when compared with soybean straw 0.35%, wheat stalk 1.2% and black grams stalk 1.1%. Figure-5.
Figure-4
Estimation of calorific values

Figure-5
Estimation of % Nitrogen
Figure-6
Estimation of % total carbohydrate

Figure-7
Micro – Assembly of Kjeldahl’s Apparatus for Nitrogen estimation
Figure-8
Laboratory scale biogas production unit

Figure-9
Standard graph of total carbohydrate by phenol sulphuric acid method

\[ y = 0.003x \]
\[ R^2 = 0.999 \]
Conclusion

Out of five agriculture wastes samples, wheat stalk, soybean straw and black grams stalk has bio-energy potential and found to be suitable feedstock for biogas production at laboratory scale. Also by using the chemical, physical, thermal pre-treatment to this feedstock does improve the quality of their biomass as an efficient feedstock for bio-energy production. Volatile solids estimated was helps to compare % amount of biomass fraction actually available for energy production and calorific values estimated was useful to screen biomass on the basis of their energy efficiency of burning abilities. Nitrogen content important for the C: N ratio that controls the pH value of the slurry. Total carbohydrate of the black gram stalk, red gram straw, wheat stalk, were found to be high as compared to soybean straw, groundnut shells so it act major sources glucose and carbon. On the other hand availability of these agriculture wastes is a major concern in bio-energy production Lab-scale. There is need to run one batch of biogas production by using selective agriculture waste as feedstock and laboratory scale biogas production unit.

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