Journal of the Turkish Chemical Society Section: A Chemistry
Turkish Chemical Society

Biogas Production from Wheat Straw using Textile Industrial Wastewater by Co-digestion Process: Experimental and Kinetic Study

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Abstract: In the present experimental study, anaerobic co-digestion of wheat straw with textile industry wastewater (TIWW) was evaluated for biogas production. Anaerobic digestion systems were operated at ambient temperature (28-30 ºC) for 20 days. Five different ratios of wastewater with distilled water were added to wheat straw inoculated with cow dung operating in five digesters. Time-rate derivative models, including Gompertz's model and its related extensions, were used to represent yields. It has been found that the slurry containing wheat straw and cow dung digested with 75% diluted wastewater has the maximum production, while the slurry digested with only wastewater (not diluted with distilled water) has minimum production.

Keywords: Biogas, lignocellulosic biomass, bioenergy, wastewater, co-digestion

Submitted: October 16, 2021. :**Accepted:** March 31, 2022.

Cite this: Rani P, Pathak V, Bansal M. Biogas Production from Wheat Straw using Textile Industrial Wastewater by Co-digestion Process: Experimental and Kinetic study. JOTCSA. 2022;9(2):601–12.

DOI: https://doi.org/10.18596/jotcsa.1009483.

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INTRODUCTION

The continuous increase in the price of fossil fuels, greenhouse gas emissions and dependency on non-renewable energy has attracted the attention of researchers to explore sustainable and affordable energy resources. Rapid urbanization and industrialization have not only increased the energy demand but also released significant amounts of waste in various forms. Globally, the expected increase in energy demand is by 45-60% by 2030, and currently a major fraction of energy (85%) is accomplished from conventional energy sources (1, 2). The potential of biomass-based energy generation has to be recognized by various researchers who have found it as a reliable and affordable energy resource. A variety of biomass has been used for the production of biofuels depending upon the constituents or the type of bio-energy product depends upon the process applied for this conversion e.g. fermentation results into bio-ethanol or biodiesel while anaerobic digestion gives biogas production etc. among different bio-energy products, anaerobic digestion for biogas production is preferred over other chemical or biological methods due to its good output/input ratio. Anaerobic digestion (AD) is a biological process in which organic matter is decomposed by an assortment of microbes under oxygen-free conditions and produces biogas (about 50-75% CH_4 and 25-50% CO_2). Till date, a lot of work has been done for improvements in biogas production and is also in continuation. As in the process of anaerobic digestion, a huge amount of water is required for slurry formation, and thus water wastage is a drawback of anaerobic digestion. This can be improved by replacing distilled water with wastewater from various sources. As the living standards of people have been enhanced, this has resulted in the growth of industrialization. Hence, industrialization is increasing day by day, which releases more industrial wastewater (3). The type of wastewater depends upon the type of industry or the general used process. Industrial wastewater can be categorized as inorganic and organic wastewater. Inorganic wastewater mainly disposed of coal, steel, and metallic industries. Organic wastewater is mainly produced by the pharmaceutical, beverages, and textile industries (4).

The textile wastewater components are determined by the operations performed, type of the fabric, and the chemicals and dyes used. The color of the wastewater and the type of the dying material used play very important roles in the component of wastewater (5).The composition of industrial or textile waste varies with the enhanced variety of manufactured products and with the demand of consumers, so today wastewater has a large number of chemicals that need new methods for degradation and consumption in a sustainable manner (6). Most of the wastewater is discharged with no special treatment into ponds and rivers which leaves them highly polluted. This improper management of waste water from industries is the main cause of environmental hazards (7). Increased population demand for more textile products, as well as an increase in the number of textile industries and wastewater, are the main reason for water pollution worldwide. Specially the colored effluents of the textile wastewater breakdown into different chemical products, making the quality of water very harmful for aquatic life and causing eutrophication and perturbations in aquatic life (8). The use of textile wastewater for anaerobic treatment can help in controlling the water pollution (9). For the last few decades, wastewater has taken the attention of many scientists to be used for some other beneficial purposes like extraction of heavy metals, conversion into biofuels etc. Marques et al. (2001) (10) suggested that olive mill wastewater could be converted into 65-70% biogas using piggery effluent without any chemical modification or dilution with water.

As the nature of the synthetic dyes is toxic for each and every type of living beings so it should not be disposed to the water bodies directly. In the process of manufacturing the textile dyeing and printing more than 8000 chemicals are used in different processes. An average-sized textile mill having a production of about 8000 kg of fabric per day uses about 1.6 million liters of water (11). The World Bank estimated that around 20% of industrial water pollution is due to textile dyeing and finishing treatment given to fabric (12). Azo reactive dyes are very common for dying cotton fabric. These dyes cannot be treated using activated sludge treatment or other chemical treatment. The process of chemical coagulation and flocculation for adsorption is used to remove the color from the wastewater, but for the removal of the contaminants, an advanced oxidation process has been developed, but they are not cost effective due to their high consumption of energy so they cannot be applied in general. As a result, there is a need for a simple and effective process that can be used to make the textile wastewater nontoxic. This process must be cost-effective and environmentally safe. Anaerobic digestion is one of the biological treatments of textile wastewater that helps with contaminants' removal (11). Treatment of textile wastewater by using suitable microorganisms and media in up-flow anaerobic fixed bed reactor (UAFB) can remove COD and color up to 81.33% and 86.78%, respectively, at highest loading rate (13). The biogas production of various biomass using this wastewater can be improved as it provides good nutrients to the methane-producing micro-organisms. Incorporation of different biomass to generate biogas is called as "co-digestion". This helps optimize the biogas production (14). Different types of biomass can be considered as the best option as they have the large potential due to their abundance of carbohydrates, which can fulfill requirements for biogas generation and could satisfy fuel supply in the future (15, 16). The major problem in the process of conversion of lignocellulosic biomass into bio-fuels is the complex structure of the biomass that restricts the biological and chemical treatments needed to unfasten the poly-carbohydrates into mono-carbohydrates (17). Different countries have different biomass potentials depending upon temperature and other conditions (18). There is a vast variety of organic materials that can be used as a good substrate for biogas production, like agricultural waste, food and vegetable waste, sewage sludge, manure, and municipal waste, etc. (19).

Jijai et al., (20) suggested that enhanced bio-methane production from chicken manure when co-digested with Thai rice noodle wastewater in different ratios proves the effective role of co-digestion in boosting the biogas yield. The effect of phyto-degradation by *Pistia stratiotes* on wastewater of sugar-mill and its special use for biogas production was investigated and observed (21).

As the process of anaerobic digestion has been used for many years all over the world, it is not a new process. However, the important point is that it was processed without proper knowledge of the mechanism of the reaction in it, so it was not known earlier what the role of carbon content in the substrate was for this process to produce the biogas. Now as the chemistry behind the process of anaerobic digestion has become more clear, it helps understand the role of the structure of cellulose hemicelluloses and lignin in conversion into biomethane (22). To understand the process of anaerobic digestion of any substrate, the fundamental factors are as follows:

1. Providing proper contact between bacteria and carbon containing substrate,
2. Providing sufficient retention time for bacteria, and
3. Providing suitable uniform environment for depolymerization of complex carbohydrates (23). A lot of factors are there which affect the anaerobic digestion process like pH, temperature, retention time, and the nutrients present in slurry (24).

Agricultural wastes are made up of mainly carbohydrates and have a great potential to be converted into bioenergy and other value-added products. So, conversion to biogas is one of these products that gives many environmental benefits. The global production of wheat was 729 Tg in 2014, and it is one of the three most cultivated crops in the world. As the ratio of straw to grain is 1.5, the production of straw is more than one billion tons annually. Thus, wheat straw is a sustainable substrate due to its availability in abundant amount and at low cost (25). Wheat straw biomass is widely used for the production of biogas, hydrogen fuel, bioethanol, and other complementary products like briquette production (26).

In the present study, textile wastewater is used as a solvent for making the slurry from lignocellulosic biomass and inoculums. Lignocellulosic biomass gives better result when integrated with textile wastewater for the production of biogas. The process of biomethanation has been boosted up by the use of textile wastewater.

MATERIALS AND METHODS

Collection and Characterization of Wastewater

TWW was treated with hydrogen peroxide (H_2O_2) and sodium hypochlorite (NaOCl) before discharge to minimize its hazardous effects. To avoid further oxidation and degradation of wastewater constituents, the sample was stored in an air-tight plastic container at 4 °C without the addition of any chemicals. The physio-chemical characteristics of textile wastewater sample were determined in the laboratory as per Standard Methods for the Examination of Water and Wastewater, 21st Edition, American Public Health Association/American Water Works Association/Water Environment Federation, Washington DC, and are reported in Table 1. The characterization of each parameter has been carried out in triplicate.

Table 1: Characteristic of the textile wastewater sample.

S.No.	Parameter	Wastewater sample
1	pH	8.6±0.14
2	COD	1390±3.5
3	BOD(5 days)	760±2.8
4	TDS	3400±8
5	TSS	168±1.8
6	Sulfides	6±0.16
7	Chlorides	78±0.24
8	Nitrates	4.5±0.12
9	Phosphates	5.8±0.11
10	Electrical conductivity	964±3.2
11	Oil & grease	12

*All the values except pH and EC are in mg/L.

Collection and Characterization of Wheat Straw and Cow Dung (inoculum)

Wheat (*Tritium aestivum*) straw for biogas production was collected from agricultural fields located in Faridabad (India). The straw was cut into pieces with a size smaller than 20 mm, prior to its application. Cow dung was used as an inoculum for the source of methanogens, which was collected from a local dairy farm located in the same region. The characterization of feed wheat straw and inoculum has been performed in the laboratory. The characteristics of wheat straw and cow dung are reported in Table 2. Wheat straw and inocula (cow dung) were analyzed for evaluation of total solids (TS), total-Kjeldahl nitrogen (TKN), pH, and volatile bolids (VS). TS was determined by drying in an oven at 100 °C for 48 h. VS content was determined by the mass of sample remaining after heating the dried milled sample at 550 °C for 4 h (27). The TS and VS of the substrate were determined before and after AD by 2540B method (27). The total carbon by Walkey&Black method (28), total nitrogen 4500-C (27), and chemical oxygen demand open reflux method (COD) by 5220B (27) of the substrate were analyzed. The pH value was detected by a digital pH meter (Cph-102).

Table 2: Compositional characterization of substrates.

Parameters	Cow Dung	Wheat straw
pH	6.2±0.12	7.1±0.14
TS%	32.4±0.11	76.4±1.22
VS%	21.2±0.01	86.23±1.35
TKN%	0.23±0.01	0.4±0.01
TOC%	4.75±0.21	8.82±0.22
COD(mgL^{-1})	80±1.2	200.0±4.3
C/N ratio	20.7±0.57	46.5±0.78
Celluloses%	19.6±0.16	39.5±0.56
Hemicelluloses%	21.8±0.24	26.8±0.34
Lignin%	1.9±0.14	6.6±0.12

*All the values except pH and C/N are in mg/L.

The wastewater was used at 25%, 50%, and 75% dilutions and at full concentration (100%).

Anaerobic Digestion Experiments

Anaerobic digestion experiments were performed in triplicate at room temperature (25-30 °C). Aspirator glass bottle (1 liter) was used as the bio-reactor. Wheat straw and cow dung were mixed together in 1:1 ratio and the slurry was prepared in proportion of 1:10 by using industrial wastewater diluted with distilled water. This wastewater is used as a top-up volume in collaboration with distilled water in various ratios to improve the biogas production from lignocellulosic biomass, i.e., wheat straw. Experimental setups have been prepared using wastewater and distilled water at 25%+75%, 50% +50%, 75%+25%, and at 100%+0%, respectively. A control set up was also tested for anaerobic digestion by loading wheat straw, cow dung, and distilled water in 1:10 for the determination of enhancement in biomethane potential of the substrate, which is important to evaluate the exact biomethane potential of wheat straw using industrial wastewater. Basic operating parameters of the digester set-ups are listed in Table 3 below. The slurry from all experimental set-ups was analyzed for physico-chemical characterization as soon as it was prepared and after completion of the experiment.

Table 3: Basic operating parameters of digester set up.

S. No.	Parameters	Particular detail
1	Hydraulic retention time (HRT)	20 days
2	Operating temperature	25-35 °C
3	C/N	20-30
4	Substrate concentration(TS)	76.40%
5	Substrate/inocula ratio	1:1

Liquid Digestate Characterization

After completion of experiments, the digested slurry was collected from the anaerobic digestion reactors and analyzed as per the methods discussed in the section titled “Collection and Characterization of Wheat Straw and Cow Dung (inoculum).

Kinetic Study of Biogas Production

The yield of AD process depends upon the rate of depolymerisation of substrate by the microbes. Thus, the kinetic study of the reaction has been observed using modified Gompertz model. In order to understand the influence of the addition of wastewater instead of simple water (in slurry preparation) on biogas production, a modified Gompertz model was employed to evaluate the cumulative biogas production. (29, 30)

$$Y(t)=P\exp\{-\exp mP\lambda-1+1\} \quad (1)$$

Where, Y (t) is the cumulative biogas yield (mL) with respect to time t (days), P is the maximum biogas production potential (mL), μ_m is maximum biogas production rate (mL/day), λ is lag phase (days). All parameters were estimated by using nonlinear curve fitting by using PAST 4.03 statistical software.

Statistics

The anaerobic digestion experiments were performed in triplicate. The statistical calculations, modeling, optimization, and graphical work were performed using Microsoft Excel.

Economic Evaluation of Bioenergy Generation Process

An economic analysis of the experimental yields of the present study has been performed for the biomethane production process in this study. A comparison of different yields from energy recovery of bioenergy generation process and cost-effective combination was observed and analyzed. The evaluation is also compared to other wastewater-bioenergy conversions reported earlier by several researchers (31).

RESULTS AND DISCUSSION

Textile Wastewater Characteristics

The textile sector produces large amount of wastewater with lots of contamination and high pH and COD value. The pH of TWW samples is 8.6, which is alkaline in nature. COD and TOC of sample are appropriate after dilution for growth and activity of microbial population. The characterization of wastewater has been listed in Table 2. Oil and grease are low in value, but there is sufficiently high amount of other water pollutants, i.e., sulfides, chlorides, and phosphates. Total suspended solids and total dissolved solids are 168 and 1360 mg/L, respectively.

Feedstock Characteristics

Feedstocks are the most important parameters for designing and operating an anaerobic digester. The initial characteristics of the starting material have an effective role in initiation, process consistency, and bio-energy production during anaerobic digestion. Hence, the target of enhanced biogas production can be achieved. Initial pH of wheat straw was 7.1, while it was 6.2 for cow dung. Table 2 shows the characteristics of raw wheat straw and cow dung. Volatile solid content of wheat straw is very good i.e. 86.73% as compared to cow dung, i.e., 21.6%. The cow dung manure had already undergone the process of digestion, so it had a low value of volatile solids. C/N ratios of inocula used in the study were in between the optimal range of C/N ratio for anaerobic digestion, i.e., 20 to 30, while that of substrate was quite high. The co-digestion of lignocellulosic biomass with animal manure can adjust the C/N of slurry to an appropriate level.

Biogas Yield from Different Experimental Slurries

During the anaerobic digestion of all types of digester slurry, a significant change in all selected physico-chemical parameters was observed. The time course reduction in the selected slurry parameters has been given in Table 6. Among all the five digesting set ups of wheat straw, the highest percent reduction of pH, TS, TOC, VS, COD, TKN, and C/N ratio for 75% dilution of wastewater (SlurryA2) has been encountered at room temperature with maximum biogas production (588 mL). Table 4 shows that biogas production has been increased when the slurry contained distilled water and textile wastewater at 3:1 ratio. As the ratio of wastewater in the slurry increases, the activity of methanogens decreases, thus falling off the production of biogas. The daily and cumulative biogas production of all the digesters is shown in Figures 1 and 2, respectively.

Table 4: Design of the experimental set ups after preparation of experimental slurry for digestion of wheat straw with cow dung and yields obtained.

Set ups	Wheat straw: cow dung	Dilution of TWW in percentage	Distilled Water (mL)	Textile Wastewater (mL)	HRT	Biogas production (mL/kg VS)
A1	1:1	Control	600	0	20 Days	418
A2	1:1	75%	450	150	20 Days	588
A3	1:1	50%	300	300	20 Days	510
A4	1:1	25%	150	450	20 Days	456
A5	1:1	100%	0	600	20 Days	408

Daily biogas production of the anaerobic digesters is shown in Figure 1. It has been found that the slurry containing wheat straw and cow dung digested with 75% diluted wastewater has maximum production, while the slurry digested with only wastewater (not diluted with distilled water) has the minimum production.

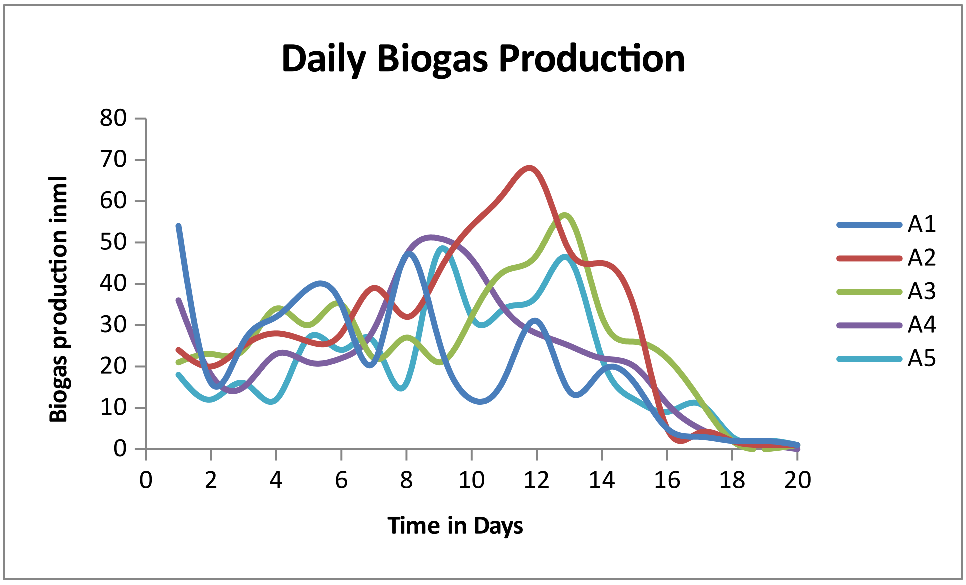


Figure 1: Daily biogas production from different slurries.

Daily and cumulative production showed that 75% dilution of wastewater provides a suitable environment for the bacterial colostra to grow and procure depolymerization of complex carbohydrates into simple ones and then to methane.

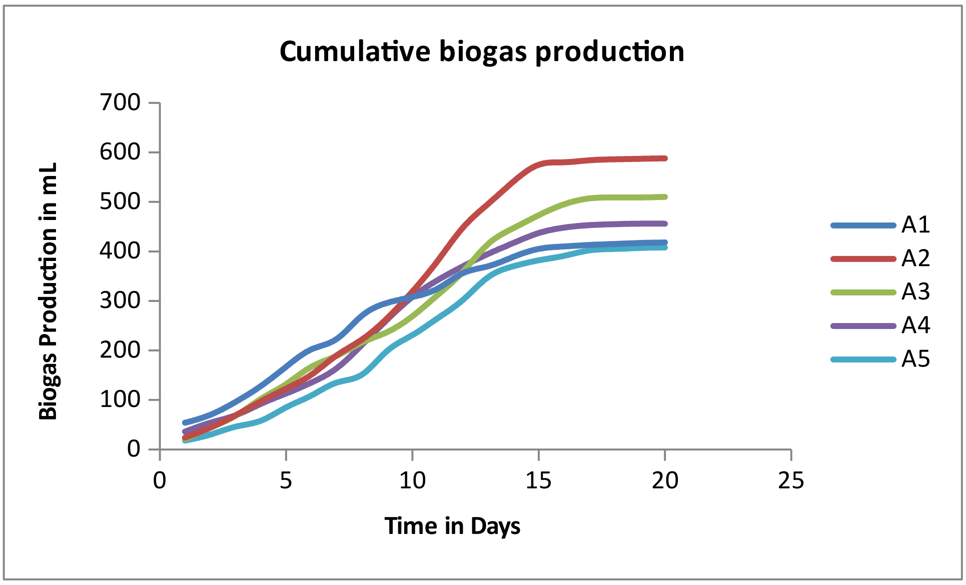


Figure 2: Cumulative biogas production of different slurries.

Effect of Wastewater Composition on Biogas Generation

The ions present in the slurry are crucial parameters as they determine the granulation and stability of the reactor (32).Thus, methanogens perform according to the environment they are provided with. Methanogens need some nutrients that help the working capability of the microbes for methane production (33). The experimental work performed shows that higher concentration of wastewater lowers the biogas production due to hindrance in methanogens' activity. Also, as the concentration of wastewater remains at appropriate level, it provides proper nutrients to microbes, thus enhances the production of biogas. This can be properly understood by the change in CODs before and after slurry of each sample. Chemical oxygen demand (COD) of any biomass or slurry is calculation of the oxygen equivalent of the organic matter content. Change in COD is amount of oxygen removed by changing organic compounds to CH_4, a significant amount of CO_2, H_2, and negligible amounts of other gases like H_2S (34). These changes in CODs are shown in Fig 4. The decrease in CODs of all 5 slurry samples are 56.23%, 26.60%, 31.36%,72.49% and 33.74% respectively. This shows that the maximum decrease in CODs occurs in the digester having a wastewater concentration of 25%, i.e., sample A2. This can easily come to the fact that accurate ions in limited amount can enhance the potential and activity of methanogens for better performance. Similarly, Manjula and Mahanta (21) showed the effect of the change in CODs in boosting biogas production.

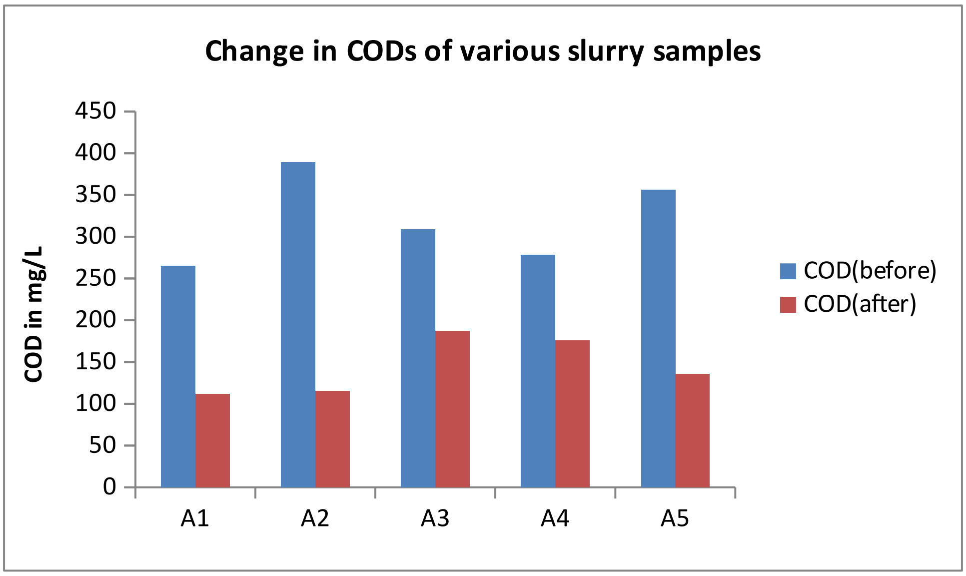


Figure 3: Change in CODs of various slurries samples.

Statistical Analysis of the Results of Experiments

Data were analyzed for means and variances, and statistical significance was determined using non-linear regression using Excel with a threshold p-value of 0.05. The analysis of samples shows that coefficients and other values are best suited for A2 sample, satisfying the experimental work.

Table 5: Statistical analysis of slurry samples.

Sample	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
A1	68.2	15.80438906	4.315257	0.000417	34.9962108	101.40379
A2	-30.03157895	21.03385729	-1.42777	0.170477	-74.222073	14.158915
A3	-9.105263158	13.21098212	-0.68922	0.499474	-36.860507	18.64998
A4	-22.8	11.0269566	-2.06766	0.053357	-45.966776	0.3667761
A5	-19.25789474	13.43097183	-1.43384	0.168761	-47.475319	8.95953

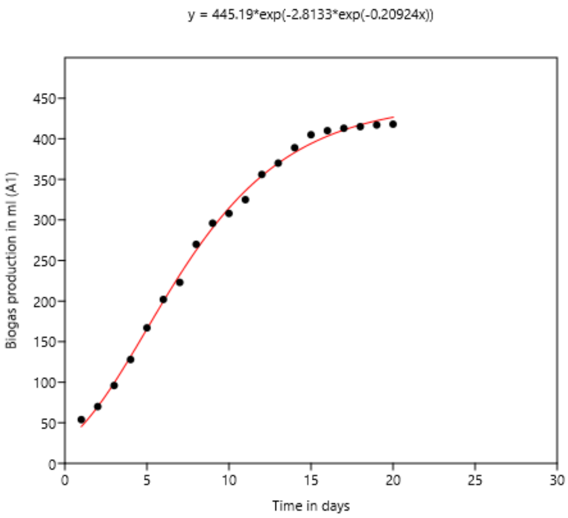
ANOVA showed a significant difference in biogas yield from A2 slurry as compared to A1 slurry ($p < 0.05$). This increase in biogas production from A2 slurry might have happened because textile wastewater in appropriate ratio with distilled water has good nutritive value for methanogenic microorganisms. Thus, it would have provided an additional microbial workforce to degrade and convert the available polysaccharides into biogas. CD could have also alleviated the potential toxicity of textile wastewater due to its complex composition containing chelating agents and the ability to enhance the degradation of recalcitrant compounds. Moreover, the C/N ratio of the A2 mixture was also in a particular range, i.e, 21–25.

The values of biogas produced by different set-ups are compared with the predicted values obtained from the statistical analysis of all the experimental yields. This also helps to estimate the result that A2 gives the minimum difference between predicted and experimental yield.

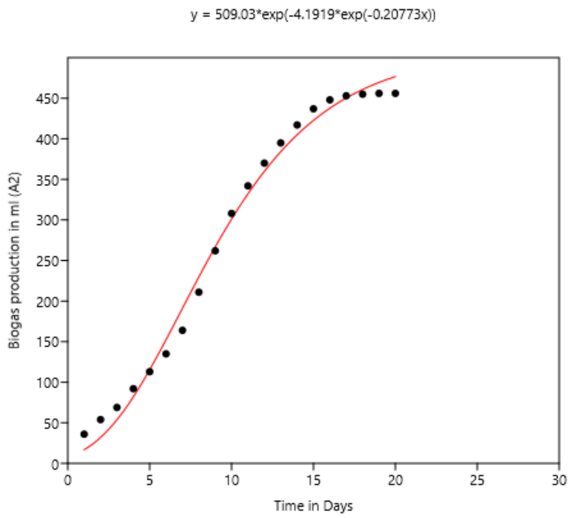
Kinetic Study of Biogas Production Using Modified Gompertz' Model

It is well known that the action of microbes on substrate for depolymerization of carbohydrates or complex constituents differs in response to the conditions of reactors such as temperature, pH, and nutrient availability. Microbial growth was also analyzed at changing concentrations of wastewater using kinetic study of reacting and found that microbial action is good in 25% and 50% of wastewater in slurry, not much in 75% and was decreased in 100% concentration. Kinetic study of anaerobic digestion revealed that a low concentration of textile wastewater helps in microbial growth and, hence, enhanced biogas production. Also, 25% is the optimum concentration for increasing the yield of anaerobic digestion by enhancing depolymerization of lignocellulosic biomass, while 100% concentration lowers the production as compared to the control.

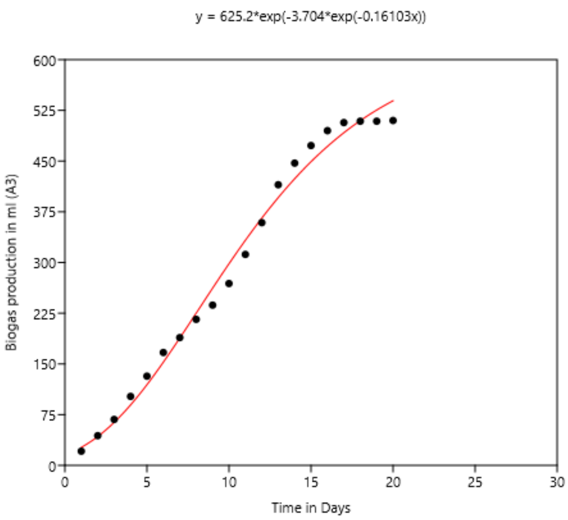
Biogas daily cumulative production data of each test group was fitted best to the modified Gompertz' model. The kinetic parameters of biogas production are shown in Figure 4. This shows that the correlation coefficient for the Gompertz's model was found to be 0.99 for all test groups, which shows a good fit of the model with experimental data.



(a)



(b)



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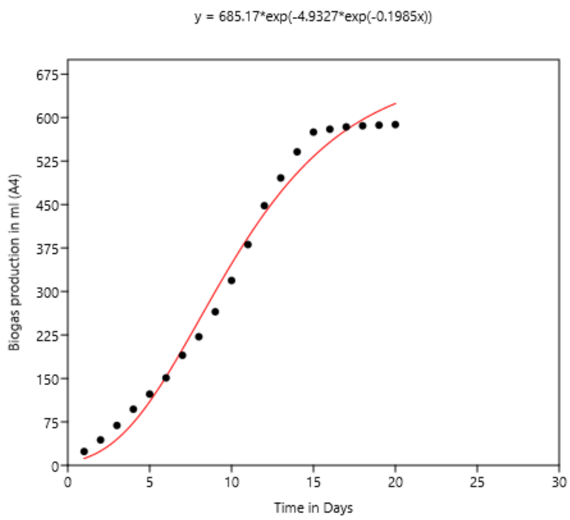

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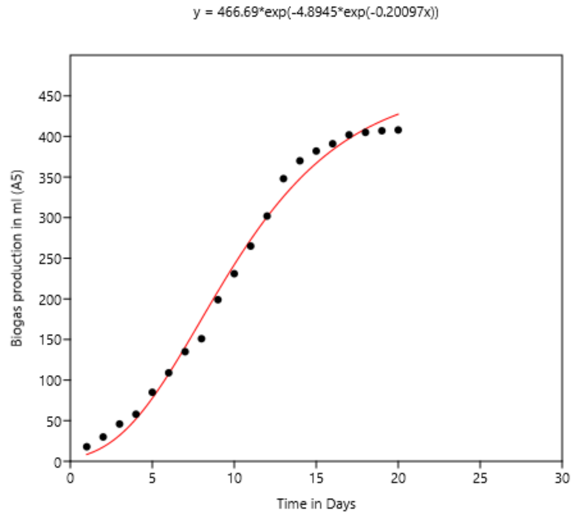

(e)

Figure 4: Kinetics study and predicted biogas production from the slurry.

Economic Analysis of Bioenergy Production Profit and Energy Recovery Capacity

The economic analysis for energy recovery and cost effectiveness has been carried out for biomethane production from textile wastewater. Energy recovery from textile wastewater was measured in terms of the biomethane production that can be generated per unit of substrate utilization and the energy value of the produced energy. The assumption is based on the experimental study, performed by Park et al. (35). Biogas production from slurry A2 is maximum, i.e., 588 mL/kg of VS, which estimates about 60% of methane i.e. 353 mL/kg of VS, as suggested by other researchers (31).

Methane production from slurry containing 25% textile wastewater =353 mL/kg of VS = 0.35 L/kg of VS

COD of slurry A2 after biomethane production = 115 mg/L

Economic Profit of Methane produced (35) =0.35 L /kg of VS * 0.57 $/m^3 = 0.2 $/m^3

Pollution Reduction in Textile Wastewater

Textile wastewater has pollutants that can cause a bad effect on aquatic life. Thus, reduction in these pollutants must be made before reviving to water bodies. Anaerobic digestion helps in the reduction of these pollutants. Analysis of pre and post-characterizations of textile wastewater in this study has shown that the chemical moieties that may cause water pollution can enhance the biological activities of microbes.

Table 6: Analysis of pre- and post-characterizations of textile wastewater.

Conc. (%)	25			50			75			100		
Parameters	Initial	Final	R*(%)	Initial	Final	R*(%)	Initial	Final	R*(%)	Initial	Final	R*(%)
Chloride	62	38	38.7	65	50	23	78	56	28.2	86	59	31.39
Nitrate	2.4	0.8	66.7	3.6	2	44.4	4	2.4	40	4.4	2.1	52.27
Phosphate	4.2	2	52.4	4.8	2.6	45.8	5.2	3.1	40.3	5.6	4.3	23.2
Sulfate	4.3	1.2	72	5.6	2.3	41	5.8	3.4	41.4	6	4.5	25
COD	1540	854	44.5	1765	1240	29.17	1778	1380	22.38	1840	1540	16.3

Table 6 has shown that the value of chlorides in wastewater reduced by 38.7% after anaerobic digestion in a 25% concentration of slurry. The results given in table 6 specifically show that pollutants are effectively reduced in all slurry samples, with the highest level in 25% of the slurry sample.

CONCLUSION

In the present study, the optimization and kinetic modelling of biogas production potential of wheat straw biomass co-digested with cow dung using industrial wastewater was investigated. It has been concluded that with the help of textile wastewater, biogas production can be boosted up. Biogas production by using an appropriate amount of wastewater enhances the depolymerization of polysaccharides by providing proper nutrients to microbes. It has been suggested that an appropriate amount of textile wastewater helps in microbial growth and enhances biogas production. The result in enhanced biodegradability was observed as change in CODs and TOCs before and after of each slurry samples. The economic analysis for energy recovery and cost effectiveness provides a smart approach to use textile wastewater in bioenergy production. A pollution reduction study of the textile wastewater has shown that the use of textile wastewater in biomethane generation helps in the control of water pollution.

CONFLICT OF INTEREST

On behalf of all authors, the corresponding author states that there is no conflict of interest.

ETHICAL STATEMENT

This article does not contain any studies with human participants or animals performed by any of the authors.

ACKNOWLEDGMENTS

Manav Rachna University for financial support and providing all necessary materials and instruments for conducting research.

AUTHOR CONTRIBUTIONS

PR, MB and VVP did the experimental work, experiment design and manuscript editing.

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