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Effect of Ultrasonic Pre-treatment on Cow dung Slurry with sludge water, food waste for Biogas Production in Anaerobic Digesters and result validation using Response Surface Methodology

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ABSTRACT

In this work Ultrasonic pre-treatment was performed on cow dung slurry in anaerobic conditions to observe the methane quality and hydraulic retention time (HRT). Response Surface Methodology (RSM) was used to determine the optimum ultrasonication time, temperature, and hydraulic retention time in this study on the ultrasonic pre-treatment of cow dung for the production of biogas. 13 experimental runs were developed in accordance with Central Composite Design with various set up conditions to observe the Responses, i.e., methane yield produced across 28 days after HRT. This was accomplished with the help of a software programme(Design Expert 12.0.1.0). In order to analyse the effects of the variables and their interactions to establish their optimal values, quadratic models for the responses were created, and a 3D response surface map was generated. The sonication time, temperature, and retention period following HRT were determined by numerical optimisation to be 35 minutes, 60°C, and 8 days, respectively.

Keywords: *Cow Dung; Sludge Water; Food Waste; Ultrasonic Treatment; Biogas; RSM; Methane, Anaerobic Digesters.*

1.0 Introduction

In the current scenario, where the globe struggles with issues like energy security, the consequences of utilising conventional fuels, environmental degradation, and global warming. Renewable and alternative fuel research is now in demand and growing in popularity. According to several studies, potential feedstock for the generation of biogas includes wastes from college canteens, industrial effluents, sludge water, agricultural wastes, cattle manures etc. (Nand et al., 1991a) (Aylin Alagöz et al., 2018a; Sawatdeenarunat et al., 2019; Vijayakumar et al., 2022; Viswanath et al., 1992a) . As of March 2021, Maharashtra had over 931 thousand biogas plants, making it the state with the most biogas plants in India. Second-place Karnataka has around 513 thousand plants. There were more than five million biogas plants in India (Fernández Lucía, 2023).

According to (Sagastume Gutiérrez et al., 2022) using biogas instead of firewood for cooking can cut the amount of greenhouse gas emissions by 11%. (R. C. Assunção et al., 2021) stated only 5% of the biogas generated globally gets converted to biomethane, fed into the petrol distribution system, or utilised as a fuel for vehicles. Worldwide food loss amounts to around 1.3 billion tonnes per year, or about one-third of all food produced. Because it is sustainable, converting food waste into energy has tremendous environmental implications (Habashy et al., 2021).

The wastes in different proportion with feed shows enhanced biogas production for an optimum proportion. (Stephen Bernard et al., 2020a) had shown in their experiment that optimum proportion of cowdung, vegetable waste and water was 1:1:2 respectively which yield 13.2% more methane. Another research on optimum proportion of cattle manure, food waste and sewage sludge were 70%, 20% and 10% respectively produced 31% more methane yield at 36 oC(mesophilic condition) while 67% more methane yield at 55oC(thermophilic condition) (Quiroga et al., 2014a). (Viswanath et al.,

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1992b) suggested that using fruit wastes and vegetable wastes to cow dung at two different HRT of 16 days and 24 days in which the yield gas formation was 74.5% and 59.03% respectively with methane content of 51 to 53% under mesophilic conditions. (Jyothilakshmi & Prakash, 2016) calculated the biogas from cow dung was 0.264 m3/kg volatile solid (VS) while from domestic waste it was 0.425 m3/kg volatile solid (VS). This experiment suggest that domestic wastes are potential feedstock for biogas production with 2 times efficient than cowdung slurry.

Biowastes are also a great feedstock for biogas production as well as solid waste management. In the context of solid waste management, biowastes are often divided into six categories: paper, glass, organic, plastic, metals, and others. An effective approach for turning biowaste into sustainable fuel for uses such as electricity production, heating, drying, and cooling is anaerobic digestion (AD). It is possible to use the biogas created by the anaerobic digestion of organic materials such as food scraps, cellulosic biomass, and animal manure as fuel for cooking, running motors, and producing power (Glivin et al., 2021).

IC engines and natural gas networks can benefit greatly from biogas since it can be upgraded to biomethane. In the previous ten years, the installed capacity for producing biogas has more than quadrupled globally, and the development is spread out due to resources being readily available and developed national regulations. Around 90% of the world's biogas is produced in Europe, China, and North America combined.(Gupta et al., 2023) The world's most widely used clean fuel, biogas, really has its roots in India. It was first found in the middle of the eighteenth century, and it progressively became the technology of choice for addressing the energy shortage in rural hinterlands. Yet in the last ten years, it has expanded to play a bigger role from the standpoint of waste to energy (Mukherjee Abhijeet, n.d.).

The use of conventional energy sources, which are widely accessible and appear to be inexpensive, continues to pose a threat to biogas as a sustainable energy source.(Tumusiime et al., 2023) However, some studies demonstrate that pre-treatment techniques (Leca et al., 2023), the use of food wastes (Stephen Bernard et al., 2020b), sewage sludge (Xu et al., 2019), and the impact of ultrasonication (Aylin Alagöz et al., 2018b; Lizama et al., 2017; Zeynali et al., 2017; Zou et al., 2016) in the production of biogas have made the fuel an affordable and sustainable source for underdeveloped villages to use for cooking.

(Nand et al., 1991b) suggested anaerobic digestion [AD] of canteen/mess waste food with cow-dung in various proportions, the methane quality could be improved up to 50% and the hydraulic retention time could be shortened to 20 days as opposed to using cow-dung alone, which has methane quality of 41% at HRT of 30 days.

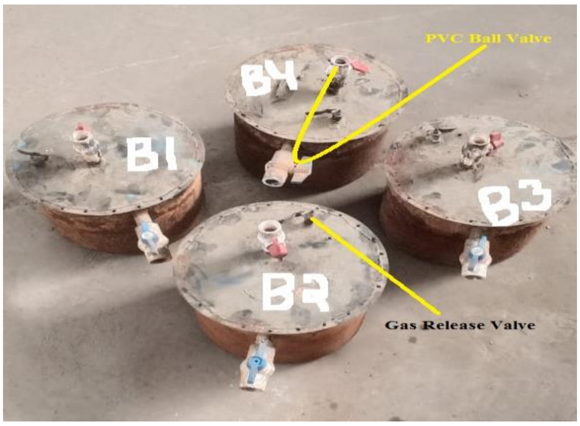
[ultra on manure digestate] found lab scale ultrasonication pre-treatment (USp) which increases the methane quality by 18% but require high specific energy which is negative energy balance between production of biogas and requirement of specific energy, but this can be cheaper pre-treatment at large scale. (Aylin Alagöz et al., 2018b) compared the pre-treatment with microwave and ultrasonic and found that microwave assisted pre-treatment required 9 times more specific energy than ultrasonic pre-treatment and enhanced only 10-15% biogas/methane yield. (Deepanraj et al., 2017) found the ultrasonic pre-treatment on food waste showed optimum yield of biogas also the VS removal rate would be maximum with USp.

2.0 Materials and Methods

2.1 Experimental setup

Experiment was conducted on batch type lab scale anaerobic digesters.

Figure 1: Anaerobic Digesters



Four anaerobic digesters (Fig.1) of 10 L volume each were used to keep cow dung slurry and feed in different proportion in absence of air. Each digester filled with 75% of full capacity i.e., 7.5 L. For attaining anaerobic condition there were two PVC ball valves at two separate locations are attached to each digester from where feed can be poured into the digesters. There is one gas release valve are attached to each digester so that biogas formed can be easily taken out into bags through a pipe (Fig.2) which can be connected to gas release valve at time of collection of biogas.

Figure 2: Biogas Collector Bag

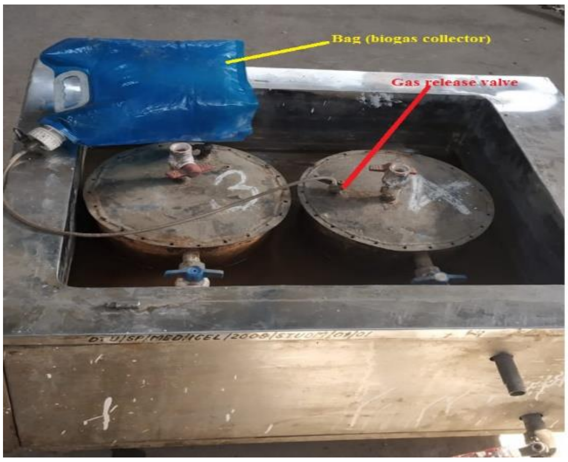
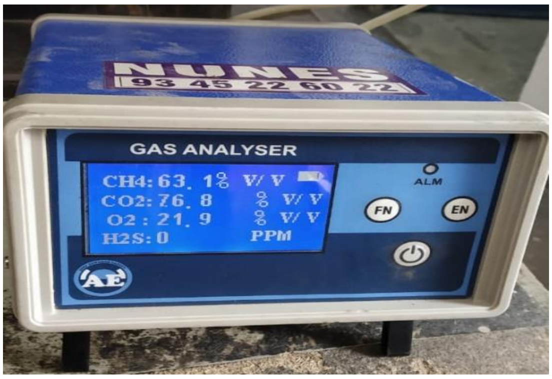


Figure 3: BioGas Analyzer



There is a biogas analyzer instrument(Fig.3) which can measure the content of methane (CH4), carbon-di-oxide (CO2), oxygen (O2) and hydrogen sulphides (H2S) present in the biogas obtained by anaerobic digestion. For pre-treatment of feedstock a ultrasonic cleaner machine (1.5kW,18kHz,230V) used with temperature control from 30 oC to 120 oC as shown in Fig.4.

Figure 4: Ultrasonic Cleaner

2.2 Feedstock used

In this experiment food wastes are taken from canteen and mess of Delhi Technological University. The collected food wastes were blended with kitchen blender. Cow dung was collected from nearby Shahabad dairy, New delhi. Sludge water was collected from plant of biogas inside campus of Delhi Technological University.

The pH of Food waste measured was 7.8 with the help of pH electrode (Deepanraj et al., 2017) (Stephen Bernard et al., 2020b). Digester capacity was of 10 L while 2.5 L was kept vacant for gas formation, total 7.5 L Cowdung slurry was made. Slurry was made with the help of sludge water, the proportion of cowdung : sludge water was taken as 1:1.5 (Stephen Bernard et al., 2020b) (Ounnar et al., 2012; Quiroga et al., 2014b) with additive of jaggery was 10gm per kg of cowdung . The total solid (TS) with OLR was 6 kg TS m-3day-1 (Viswanath et al., 1992c). Cow dung slurry was kept at HRT of 17 days at room temperature between 28oC and 32oC where methane yield was found 56% optimized from previous studies (Nand et al., 1991b).

3.0 Ultrasonic Pre-treatment

After HRT within 10 hours B1 and B2 was pre-treated with ultrasound for 35 minutes at 50oC and 60oC on regular basis for 7 days and it was found that methane yield was 58- 63% which was optimized from (Azman et al., 2020; Lizama et al., 2017; Quiroga et al., 2014b). The ultrasonic cleaner operates on 1.5kW power 18 kHz frequency (low ultrasound) with 230V supply. B3 and B4 was treated by coil inside it at 50oC and 60oC on regular basis for 7 days which yield methane was 43-45%.

It was seen that at thermophilic condition without USp, the methane yield was lower. After that the cowdung slurry was pre-treated with food waste where proportion of cowdung:food waste:sludge water was 1:1:1.5. Ultrasonication was given for 35 minutes for 10 days on regular interval on food waste treated cowdung slurry and it was observed that the methane yield was 63-67%.

The economic viability at the lab scale was better than the microwave assisted pre-treatment in terms of energy requirements for given energy produced (Deepanraj et al., 2017). However,the requirement of energy at lab scale biogas production was more which was negative energy (Azman et al., 2020).

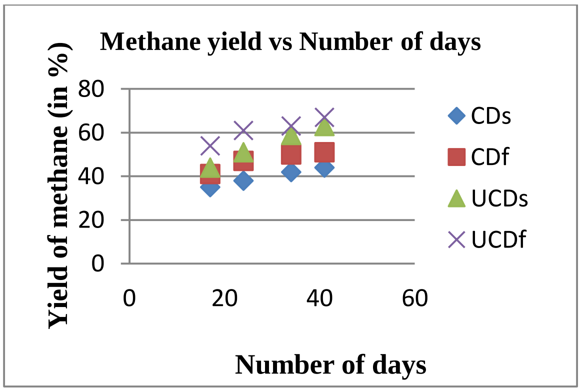
4.0 Model Validation

The Central Composite Design (CCD) forecast was tested and confirmed when the ideal circumstances were attained. Utilising the expected values, this was done experimentally, and the proportion of methane output produced was recorded as "responses". To demonstrate the model's viability, a comparison between experimental and anticipated values was done.

5.0 Results and Discussions

Figure 5 indicates the methane quality in biogas as number of days increases after HRT of 17 days.

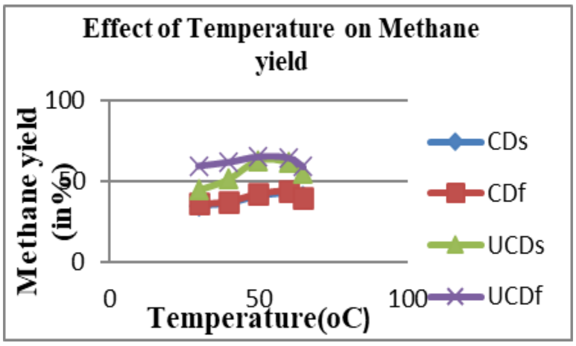
Figure 5: Percentage Methane Yield vs Number of Days after HRT



Digesters B1 and B2 was pre-treated with ultrasonic cleaner at 50oC and 60oC respectively for 35 minutes for 10 days and found the result in increasing of methane yield of 63% and 65% as compared to B3 and B4 which was maintained at 50oC and 60oC respectively which yield methane of 44% and 44.7% at lower pace as compared to energy supplied and cost associated with it.

Feed of food waste and sludge water in proportion of cow dung:sludge water:food waste is 1:1.5:1 shows the maximum yield of methane in biogas produced. From fig.5 and fig.6 it was clear that food waste leads to maximum methane yield of 69% with ultrasonic pre-treatment at thermophilic condition.

Figure 6: Temperature Variation on Methane Yield with or without Treated Substrate



The pH value of CDs observed initially was 7.8 and finally it was 6.5 this pH was acidic not suitable for anaerobic digestion. At the end of 50th day CDs slurry shows degradation of methane yield in biogas produced.

6.0 Result Validation through RSM

The percentage of methane yield produced from UCDs under different sonication time, temperatures and the digestion time after HRT conditions set by Central composite design are presented in Table 2.The percentage of methane was obtained across 28 days after HRT and responses are recorded for 17,20,23,26,28 days are displayed. The highest methane yield percentage was found 65.63% in run #7 for 8 days after HRT while lowest was observed 24.27% in run #13 for 28 days after HRT. Different responses were caused by variations in the values of the manipulable variables (factors).

Table 1: Ranges of Ultrasonication Time and Temperature Input for Design Expert®

Factor	Name	Units	Type	SubType	Minimum	Maximum	Coded Low	Coded High	Mean	Std. Dev.
A	sonication time	minutes	Numeric	Continuous	10.00	60.00	-1 ↔ 10.00	+1 ↔ 60.00	35.00	17.68
B	temperature	degree celcius	Numeric	Continuous	20.00	100.00	-1 ↔ 20.00	+1 ↔ 100.00	60.00	28.28

This made stating the necessary link between the variables and answers, and as a result, model equation 1 was created.

6.1 Factors optimization

The effect of ultrasonication time and temperature on methane yield percentage is displayed on 3D plot as shown in Fig.6. The display shown a higher interaction between ultrasonication temperature and methane yield percentage, and a marginal interaction on ultrasonication time and methane yield percentage.

Table 2. Methane yield % for various setup circumstances

Run	Factor 1 A:sonication time minutes	Factor 2 B:temperature degree celcius	Response 1 Methane yield %
1	10	20	29
2	35	60	55
3	60	100	25.6
4	35	60	59.8
5	60	60	45.3
6	10	60	50.1
7	35	60	63
8	35	60	59.8
9	35	60	56.4
10	10	100	27
11	35	20	44.6
12	60	20	53.7
13	35	100	20.2

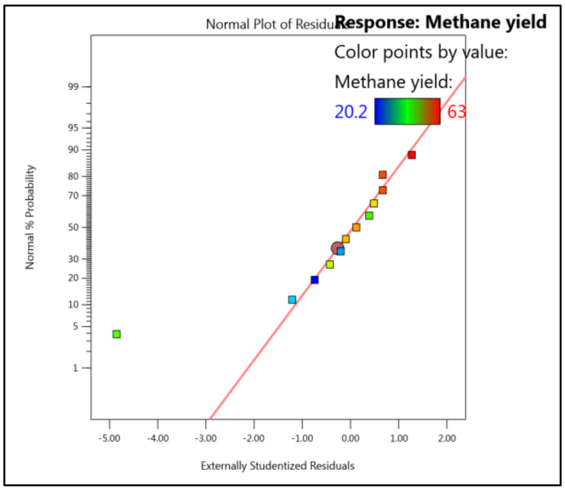
The yield of methane grew as the temperature of the ultrasonication process rose, but it eventually started to fall as the process parameters departed from their ideal range. The sonication duration did have an impact on gas generation, however it had a little impact in comparison to the ultrasonication temperature.

The Central Composite Design forecasted the ideal circumstances for the highest methane output using numerical and point prediction optimisations. According to the forecast, the ideal parameters for the highest output methane yield were sonication duration of 35 minutes, ultrasonication temperature of 60°C, and anerobic digesting period of 8 days following HRT. The greatest methane output that could be expected under these circumstances was 63%. As a result, the simplified quadratic model for the methane yield produced from cow dung, which was determined for ultrasonication duration (A) and ultrasonication temperature (B), is presented as follows in 1:

$$\text{Methane yield} = 1.10762 + 0.514833 * A + 1.67218 * B - 0.006525*A*B - 0.013924*B^2 \quad (1)$$

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor.

Figure7 (a): Plot of Residuals vs Normal Probability



This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space. The normal probability plot of residuals and the expected vs. actual methane output is shown in Figure 7(a) and (b). Plots demonstrate that the testing results are typical; as a result, the model is successful in estimating methane yield output.

Fig 7 (b): Plot of Actual and Predicted Values

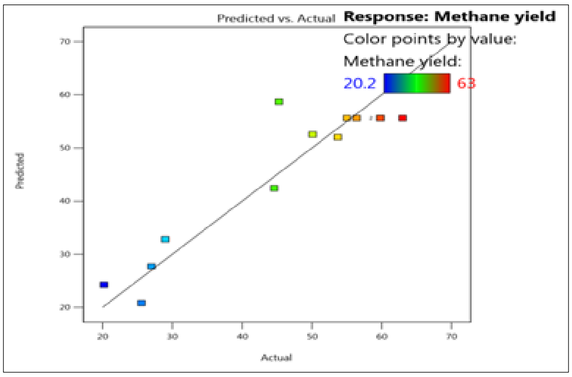
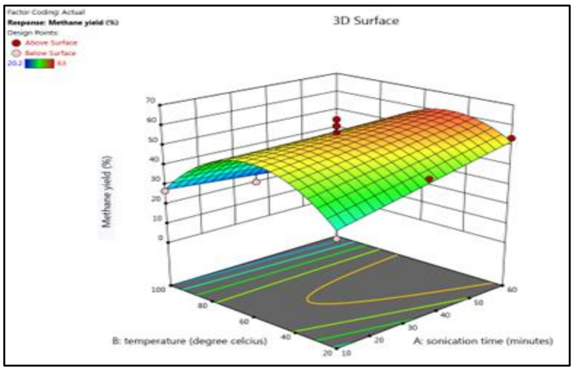


Fig 8: 3D Surface Showing Interaction between Sonication Time, Ultrasonication Temperature and Methane Yield %



The regression equation's 3D response surface map (Fig. 8) revealed modest connections between the variables. The findings demonstrated that equation 1's model was correct and satisfactory, and that the response model was sufficient for representing the anticipated optimisations. Such interactions were noted in prior research (Ghaleb et al., 2020; Ibrahim et al., 2021) that used RSM for prediction in ultrasonicated biogas production and RSM for methane production optimisation.

The prediction's validation indicated a 63% methane output. As 4% was the recorded percentage error, this result was quite near to the expected figure of 65.63%, confirming the sufficiency and validity of the predicted models. The percentage error should not be more than 30 % (Chun et al., 2015). In order to confirm the prediction and modelling abilities of RSM, Analysis of Variance (ANOVA) (Table 3) for the response surface model fit was performed. The ability was assessed using the coefficient of determination (R2), enough precision, and lack of fit values for key model parameters. The R2, Adjusted R2, Predicted R2, and Adequate precision values were 0.8732, 0.8097, 0.5453, and 9.3999 respectively, indicating that the model was highly significant according to the ANOVA, which had a low P-value of 0.0012 and a high F-value of 13.77.

The regression model's F-value demonstrated its importance, which is consistent with the findings of (Montingelli et al., 2016). It has been recommended that the value should be about 0.80 for the excellent fit of a model when using the R2 to assess the model's goodness of fit (Pei et al., n.d.). The model's high R2 values attest to its consistency with the experimental data (Giwa et al., 2013). Model terms are significant when their p values are lower than 0.05. Model terms are not significant if the p value is higher than 0.10. The values suggested that the study's model was sufficient for the design of space navigation, according to (Stat-Ease Handbook for Experimenters, n.d.)

The adjusted R2 of 0.8097 was quite consistent with the predicted R2 of 0.5453, and the square of the correlation coefficient (R2) value was near to 1. The model was inferred to be significant by the model F-value in the F-test. Noise has a very little possibility (0.12%) of causing an F-value this significant. The model source's Sum of Squares was 2325.93. Each regression source's degrees of freedom (DF) was correspondingly one, giving the model source a total DF of 4. The sum of squares divided by the matching DF yielded the model's mean squares, which came to 581.48. The model in this case has a Lack of Fit F-value of 298.06 and an Adequate Precision of 9.3999. According to the Lack of Fit, the relative inaccuracy was considerable.

There is a potential that this will happen as a result of the noise.

Table 3: Variance Analysis

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	2325.93	4	581.48	13.77	0.0012	Significant
A-sonication time	57.04	1	57.04	1.35	0.2787	
B-temperature	495.04	1	495.04	11.72	0.0090	
AB	170.30	1	170.30	4.03	0.0795	
B^2	1603.54	1	1603.54	37.96	0.0003	
Residual	337.90	8	42.24			
Lack of Fit	298.06	4	74.52	7.48	0.0384	Significant
Pure Error	39.84	4	9.96			
Cor Total	2663.83	12				

7.0 Conclusion

According to the study's findings, cow dung anaerobic digestion produced more methane when ultrasonic pre-treatment was applied. in ideal ultra-sonication period, Temperature and hydraulic retention time were shown to be crucial for maximising methane output. For the highest percentage of methane output from cow dung, numerical optimisation identified an ultrasonication period of 35 minutes, temperature of 60oC, and retention time of 8 days following HRT. These criteria are stated in a model equation that was developed. Ultrasonic pre-treatment have an issue with its energy consumption as compared to biogas production at the lab scale. Further research on alternative energy source can be done for reducing energy demand for ultrasonication from conventional fuel.

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Nomenclature

HRT: Hydraulic Retention Time
TS: Total Solid
CDs: Cow dung slurry
VS: Volatile Solid
FW: Food Wastes
AD: Anaerobic Digestion
Usp: Ultrasonic pre-treatment
OLR: Organic Loading Rate
Usp+CDs+FW = UCDf
RSM: Response Surface Methodology