



Determination of water flow rate for the optimal biogas purification using water scrubbing technology at ambient temperature (23⁰C)

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ABSTRACT

Biogas has been proven to be a sustainable substitute for fossil fuel-based energy systems. It comprises mainly of CH₄, CO₂, H₂S, and water vapour. Biogas purification is crucial for making the gas suitable for various applications in the renewable energy sector. Water scrubbing has been widely used for enriching the methane percentage. Determining the optimal water flow rate in biogas scrubbing is crucial for enhancing methane content and reducing carbon dioxide and hydrogen sulfide impurities. Various studies have identified specific water flow rates that maximize efficiency in different scrubbing systems. This study focused on determining the water flow rate (WFR) required for the optimal biogas purification using water scrubbing technology. The study applied Response Surface Methodology (RSM), specifically Central Composite Design (CCD) in the design of the experiment (DoE) to optimize the water flow rate (WFR). WFR was varied at the rates of 3.5, 4.0, 4.5, 5.0, and 5.5 litres per hour (l/hr). The study used Design Expert software version 13 in the analysis of results. This study found that the highest methane enrichment of 91.45% was attained at the WFR of 5.0 litres per hour.

Keywords: Biogas Purification, Central Composite Design, Optimization, Response Surface Methodology, Water Flow Rate, Water Scrubbing

I. INTRODUCTION

Biogas is formed when bacteria decompose biological materials in the absence of oxygen through an anaerobic digestion process. According to Daniel-Gromke *et al.* (2018), biogas consists primarily of methane (CH₄) and carbon dioxide (CO₂), with small amounts of other gases such as Hydrogen Sulphide (H₂S) and trace amounts of water vapor. The exact composition varies depending on factors such as the feedstock used for biogas production and the efficiency of the digestion process (Bharathiraja *et al.*, 2018). The percentage composition of raw biogas ranges from 40-70% CH₄, 30-40% CO₂, 0 - 5000 ppm H₂S, and traces of water vapour (Al Mamun & Torii, 2015). Biogas burns very well when the methane content is more than 50%, and therefore, it can be used as a substitute for kerosene, charcoal, and firewood for cooking and lighting (Budzianowski, 2016). However, utilization of biogas as a renewable energy source has not yet attained its full potential due to the challenge of the inherent presence of impurities. These impurities make biogas inappropriate for vital commercial and domestic applications, such as transport fuel, requiring strict quality demands.

There are several techniques used to upgrade biogas, such as water scrubbing, cryogenic separation, pressure swing adsorption, membrane separation, chemical absorption, and physical absorption. (Andriani *et al.*, 2014). Water scrubbing is the widely used method in biogas purification due to water availability as the scrubbing material (Kapoor *et al.*, 2019). Water scrubbing is used to remove pollutants, contaminants, or unwanted gases from industrial exhaust gases, flue gases, or any other gas (Nock *et al.*, 2014). The process involves passing the biogas stream through a liquid, typically water or a water-based solution, to capture and dissolve the pollutants (CO₂ and H₂S). The water acts as a medium to absorb the pollutants, allowing for their removal from the gas stream (Tippayawong & Thanompongchart, 2010).

Vijay *et al.* (2006) developed a purification system to remove CO₂ from raw biogas of 40% CO₂ using a water scrubbing method. This system consisted of a single-stage compressor, a scrubbing column (150 mm diameter and 3,500 mm length), a water supply system, a gas supply system, a three-stage high-pressure compressor, and a storage cylinder. The study was conducted at various flow rates of pressurized water (at 1.0 MPa) and gas, ranging from 1.8 – 2.0 m³/hr and 1.0 – 3.0 m³/hr, respectively. This study concluded that the absorption of CO₂ in water depends on the flow rates of



gas and water; the optimum 98.62 % removal of CO₂ was achieved at gas and water flow rates of 1.5 m³ /hr and 1.8 m³ /hr, respectively.

Water scrubbing technology is mostly used for the removal of both CO₂ and H₂S in sewage-sludge-based biogas plants in France, Sweden, and the USA (Kapoor *et al.*, 2019). The studies show that after scrubbing using water as an absorbent material, 5-10% CO₂ is removed. Therefore, to enhance the biogas quality for a given scrubber, the process parameters such as WFR, GFR, Pressure, and RT should be optimized to meet the Indian Standard (IS)16087:2013 for biomethane.

Several advancements in biogas purification have been discussed, emphasizing on enhancing methane concentration and removing impurities, such as carbon dioxide, hydrogen sulfide, and water vapor (Angelidaki *et al.*, 2018; Das *et al.*, 2022; Sahota *et al.*, 2018; Salave & Desai, 2017; Sun *et al.*, 2015). Technologies such as membrane separation, pressure swing adsorption (PSA), and biological methods are being refined for enhanced efficiency and cost-effectiveness. Novel approaches include using low-cost materials for adsorption and exploring new catalysts for biogas conversion.

The previous work done by (Gantina *et al.*, 2020; Kapoor *et al.*, 2019; Nock *et al.*, 2014; Vijay *et al.*, 2006) indicates that most of the research previously undertaken focused mainly on water scrubbing as a biogas upgrading technology. These studies, however, concentrated mostly on purifying biogas using water scrubbing under high pressure, which consequently required high power to operate, limiting their application to commercial use. Few studies have focused on low-pressure water scrubbing and operated at room temperature, such as the work done by Mugagga *et al.*, (2022), who developed a system for optimizing and analyzing low-pressure water scrubbing. This study was conducted at a low pressure of 0.07 bar and ambient temperature (23 °C).

Several methods have been used for the optimization of process parameters of any given system (Zabava *et al.*, 2018). These techniques include: Box-Behnken, Taguchi, and Response Surface Methodology. RSM can be employed to optimize and analyze the effects of several independent factors on a process to obtain the maximum output (Karmoker *et al.*, 2019). Response surface methodology (RSM) is one of the most efficient and widely used mathematical and statistical tools for optimizing system performance. Hirkude and Padalkar (2014) used RSM to predict the optimal input parameters in an experiment to determine the effect of compression ratio on the performance of a CI engine fueled with biodiesel from waste fried oil.

The primary objective of this study, therefore, was to determine the optimal water flow rate required for the maximum biogas quality using water scrubbing technology at Ambient Temperature (23°C). This was achieved by varying the water flow rate in combination with other variables: gas flow rate, pressure, and gas retention time to establish the optimal methane yield. Response Surface Methodology, specifically Central Composite Design, was applied in the optimization of the water flow rate.

II. METHODOLOGY

2.1 Design of Experiment

A Design of Experiment (DoE) using a Central Composite Design (CCD) of the Response Surface Methodology (RSM) was used to develop a matrix of runs for conducting the experiment (Asadi & Zilouei, 2017). Upgraded biogas was collected for each run, and the composition was analyzed using a SKY2000-M4-WH multi-gas detector (Al Mamun & Torii, 2015). The CCD developed the matrix shown in Table 1. The experiments were conducted based on this narrative.

Table 1

Design of Experiment Matrix

Run	WFR (l/h)	GFR (l/min)	RT (sec)	P (bar)	CH ₄ (% Volume)	CO ₂ (% Volume)	H ₂ S (mg/m ³)
1	4.5	8.5	60	0.06			
2	4.5	8.5	60	0.06			
3	4.5	8.5	60	0.06			
4	3.5	8.5	60	0.06			
5	5.0	9.0	45	0.05			
6	4.5	8.5	60	0.06			
7	4.5	8.5	60	0.08			
8	4.5	8.5	30	0.06			
9	4.0	9.0	45	0.05			
10	4.0	8.0	45	0.05			
11	5.0	9.0	75	0.05			
12	4.5	7.5	60	0.06			

13	5.5	8.5	60	0.06			
14	5.0	8.0	45	0.07			
15	4.5	8.5	90	0.06			
16	5.0	8.0	75	0.07			
17	4.0	9.0	45	0.07			
18	4.5	8.5	60	0.04			
19	4.0	9.0	75	0.05			
20	4.0	8.0	75	0.07			
21	4.0	9.0	75	0.07			
22	4.5	8.5	60	0.06			
23	4.0	8.0	45	0.07			
24	4.5	9.5	60	0.06			
25	5.0	8.0	45	0.05			
26	4.5	8.5	60	0.06			
27	5.0	9.0	75	0.07			
28	5.0	9.0	45	0.07			
29	4.0	8.0	75	0.05			
30	5.0	8.0	75	0.05			

2.2 Experimental Setup

Optimization of biogas purification was designed to upgrade the raw biogas by removing incombustible gases from biogas mixtures, specifically CO_2 and H_2S , using water scrubbing technology (Jiang *et al.*, 2020). The study was conducted using a plastic water scrubber of height 0.5m and diameter 0.1m. The scrubber comprised three sections: the bottom section for the gas inlet, the middle section for the scrubbing process, and the top section for the water inlet and gas outlet.

The experimental setup comprised three sections: Raw biogas collection and sampling, optimization scrubber, and collection unit. The first section involved raw gas sampling, analysis of raw gas composition, and a gas flow rate control meter. The second section involved the biogas purification process through the scrubber. Water was sprayed from the top of the scrubber while biogas was fed from the bottom of the scrubber upwards. This ensured the maximum gas-water contact and hence purification of CO_2 and H_2S dissolved in water, and was discharged as wastewater through the wastewater outlet pipe at the base of the scrubber. The methane-enriched biogas moved upwards due to its reduced density to the last stage. Finally, the third section involved the collection and sampling of the upgraded gas for composition analysis using the SKY2000-M4-WH multi-gas detector.

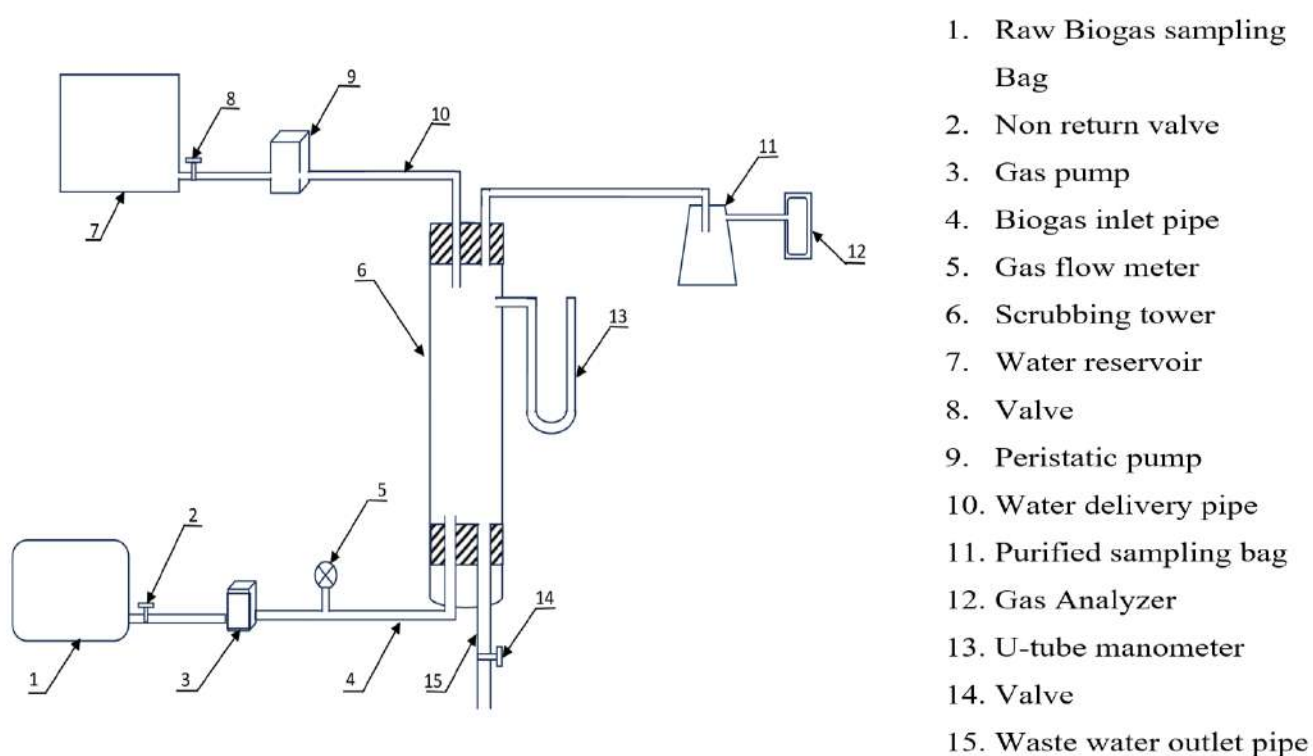


Figure 1
Experimental Setup

2.3 Experimental Procedure

The study involved varying the water flow rate at 3.5, 4.0, 4.5, 5.0, and 5.5 litres per hour (l/hr) in the scrubbing tower. A Design of Experiment (DoE) using a Central Composite Design (CCD) of the Response Surface Methodology (RSM) (Asadi & Zilouei, 2017) was used to develop a matrix for runs of experimentation. Upgraded biogas was collected for each run, and the composition analyzed using a SKY2000-M4-WH multi-gas detector (Al Mamun & Torii, 2015).

The raw biogas was first tested for percentage composition by volume of CH₄, CO₂, and H₂S. Then, the peristaltic pump was turned on, and the water flow was opened to test for any leakages. The gas pump was switched on, and gas leakages were checked. The downward flow of water and upward flow of the gas allowed time for the gas to interact and absorption of CO₂ and H₂S. Methane, being insoluble in water, moved to the gas outlet, whereas CO₂ and H₂S, which are soluble in water, were removed through the wastewater outlet. The upgraded biogas was then passed through a SKY2000-M4-WH multi-gas detector to determine its composition. The optimization was achieved by varying a set of 30 runs of WFRs at 4.0, 4.50, and 5.0 l/h, respectively. The upgraded biogas was assessed for CH₄, CO₂, and H₂S composition using a SKY2000-M4-WH multi-gas detector.

2.4 Gas Analysis

The gas composition was analyzed offline by the SKY2000-M4-WH multi-gas detector as shown in the figure 2 below. The SKY2000-M4-WH multi-gas detector was fitted directly to the gas delivery pipe from the scrubber to record the gas composition.



Figure 2

The SKY2000-M4-WH Multi-Gas Detector

III. FINDINGS & DISCUSSION

3.1 Findings

Response Surface Methodology, particularly CCD, was chosen for the analysis of the results since it allows multiple variables to be examined at a time, unlike the conventional statistical methods (Veza *et al.*, 2023). The experimental results that were collected are illustrated in Table 2, shown below;

**Table 2***Results of the Experiment.*

Run	WFR (l/h)	GFR (l/min)	RT (sec)	P (bar)	CH ₄ (% Volume)	CO ₂ (% Volume)	H ₂ S (mg/m ³)
1	4.5	8.5	60	0.06	78.06	17.32	11
2	4.5	8.5	60	0.06	78.23	17.40	11
3	4.5	8.5	60	0.06	77.82	17.26	11
4	3.5	8.5	60	0.06	76.54	17.82	12
5	5.0	9.0	45	0.05	72.94	19.24	15
6	4.5	8.5	60	0.06	81.07	17.16	11
7	4.5	8.5	60	0.08	78.12	17.04	11
8	4.5	8.5	30	0.06	74.38	18.03	14
9	4.0	9.0	45	0.05	78.02	16.28	10
10	4.0	8.0	45	0.05	81.36	15.02	10
11	5.0	9.0	75	0.05	86.36	12.05	9
12	4.5	8.5	60	0.06	80.66	15.76	10
13	5.5	8.5	60	0.06	83.65	16.22	10
14	5.0	8.0	45	0.07	86.14	14.73	10
15	4.5	8.5	90	0.06	78.02	17.29	11
16	5.0	8.0	75	0.07	91.45	6.91	0
17	4.0	9.0	45	0.07	82.85	14.60	8
18	4.5	8.5	60	0.04	71.68	21.52	18
19	4.0	9.0	75	0.05	82.69	17.22	11
20	4.0	8.0	75	0.07	89.82	10.12	7
21	4.0	9.0	75	0.07	86.20	13.68	9
22	4.5	8.5	60	0.06	79.01	17.35	11
23	4.0	8.0	45	0.07	84.63	15.84	10
24	4.5	9.5	60	0.06	78.47	17.24	11
25	5.0	8.0	45	0.05	83.67	16.25	10
26	4.5	8.5	60	0.06	84.52	15.41	10
27	5.0	9.0	75	0.07	88.63	10.95	8
28	5.0	9.0	45	0.07	83.84	15.94	17
29	4.0	8.0	75	0.05	85.28	14.58	10
30	5.0	8.0	75	0.05	87.12	11.82	9

3.1.1 Methane Maximization

From Table 2, run 16, in which the experiment was set at a water flow rate (WFR) of 5.0 litres per hour, gave the highest purification percentage of 91.45%. In the control experiment, the WFR was maintained at 4.5 litres per hour. It gave a purification of 80.66%. This showed an improvement of 10.79% compared to run 16. Srichat *et al.* (2017) used chemical scrubbing and achieved 89.3% CH₄. Similarly, Akila *et al.* (2017) used Pressure swing Adsorption and attained 93.90% Methane. The purification Percentage achieved (91.45%) closely compares with the findings of the above studies.

3.1.2 Carbon Dioxide Minimization

The control experiment (run 12) reduced the CO₂ content to 15.76%, while run 16 gave the highest minimization of 6.91%. This showed an improved reduction of 8.85%. Vijay *et al.* (2006) used water scrubbing and achieved an optimum 98.62 % removal of CO₂. However, despite high performance, this system was operated at a high pressure of 1.0 MPa, which consequently meant high energy was required and a high cost of purification. Work done by Aphichat Srichat *et al.* (2017), Pugalendhi and Boopathi (2017), and Vrbova and Ciahotny (2017) concentrated on maximizing the CH₄ content - minimal attention was given to the CO₂ content. Andriani *et al.* (2014) recommended that water scrubbing is a better option for removing carbon dioxide when water is available in the required quantity.

3.1.3 Hydrogen Sulfide Minimization

Table 2 shows that run 16 gave the highest minimization of Hydrogen Sulphide (H₂S) of 0% while the control experiment minimized to 10%. According to Jiang *et al.* (2020), Hydrogen sulfide (H₂S) is highly soluble in water and establishes an ionization equilibrium once it is dissolved in water. Dissociation of the H₂S molecule forms a hydrosulphide ion (HS⁻), which then dissociates to form the sulphide ion (S⁻²) through the ionization of water. This explains the high efficiency of removing H₂S from biogas when using water scrubbing technology.



3.1.4 Prediction Models

The ANOVA was used to develop linear regression models used for predicting the methane content, carbon dioxide, and hydrogen removal efficiency. The models show how water flow rate relates to methane enhancement as well as carbon dioxide and hydrogen sulfide minimization.

The prediction model (equation) in terms of actual factors was given as:

$$y = 1.96x_1 - 2.69x_2 + 0.14x_3 + 204.16x_4 + 74 \quad (1)$$

where;

- y = percentage of methane (%)
- x_1 = water flow rate (litres/hour)
- x_2 = gas flow rate (litres/minute)
- x_3 = retention time (seconds)
- x_4 = pressure (bar)

The linear prediction model equation (1) above shows that the water flow rate exhibits direct proportionality to the methane yield. This implies that the methane yield increases with an increase in the water flow rate.

The prediction model for CO₂ in terms of actual factors is given by equation (2).

$$y = -1.056x_1 + 1.47x_2 - 0.08x_3 - 119.37x_4 + 20.34 \quad (2)$$

where;

- y = percentage of CO₂ (%)
- x_1 = water flow rate (litres/hour)
- x_2 = gas flow rate (litres/minute)
- x_3 = retention time (seconds)
- x_4 = pressure (bar)

From the above linear prediction model equation (2), the water flow rate was indirectly proportional to the methane yield. This implies that the methane yield decreases with an increase in the water flow rate.

Prediction model for hydrogen sulphide: The prediction model in terms of actual variables is given by equation (3)

$$y = -27.2x_1 - 26.7x_2 + 1.15x_3 - 645.83x_4 + 4.75x_1x_2 - 0.2x_1x_3 - 12.5x_1x_4 + 0.008x_2x_3 + 112.5x_2x_4 - 6.25x_3x_4 + 168.52 \quad (3)$$

where;

- y = percentage of H₂S (mg/m³)
- x_1 = water flow rate (litres/hour)
- x_2 = gas flow rate (litres/minute)
- x_3 = retention time (seconds)
- x_4 = pressure (bar)

In the linear prediction model equation (3), the water flow rate exhibits indirect proportionality to the methane yield. Similarly, the water flow rate in combination with the gas flow rate has a direct proportionality to the methane yield, while the intersection of water, gas retention time, and pressure has an indirect proportionality to the methane yield.

3.1.5 Analysis of Variance (ANOVA)

ANOVA for the linear model of methane yield, Other Statistics: Adjusted R² = 0.2978, Predicted R² = 0.0936, Adequate precision = 7.741, C.V. % = 5.04, Std. Dev. = 4.12, and Mean = 81.71

Table 3

ANOVA for the Linear Model of Methane Yield

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	276.61	4	69.15	4.08	0.0112
A-WFR	23.05	1	23.05	1.36	0.2548
B-GFR	43.52	1	43.52	2.56	0.1218
C-RT	110.00	1	110.00	6.48	0.0174
D-P	100.04	1	100.04	5.90	0.0227
Residual	424.24	25	16.97		
Lack of Fit	390.31	20	19.52	2.88	0.1223
Pure Error	33.93	5	6.79		
Cor Total	700.85	29			

From Table 3 above, a large F-value of 4.08 and a small P-value of 0.0112 implied a more significant influence on the corresponding response variable (Quanhong and Caili, 2005). R² represents the coefficient of determination of the linear regression model. It measures the variability of the actual response that can be defined by the corresponding



independent factors (Mukhopadhyay *et al.*, 2013a). The predicted R^2 of 0.0936 indicated reasonable agreement with the Adjusted R^2 of 0.2978. The threshold is that the disparity between the adjusted R^2 and the predicted R^2 should be about 0.2 (P. Subha and Jayaraj, 2019). The difference here was 0.2042. This showed a strong correlation between the observed and the predicted values. ANOVA for the linear model of carbon dioxide yield

Table 4

ANOVA for the Linear Model of CO₂ Yield

Source	Sum of squares	df	Mean square	F-value	p-value
Model	96.65	4	24.16	4.04	0.0116
A-WFR	6.67	1	6.67	1.11	0.3012
B-GFR	12.98	1	12.98	2.17	0.1532
C-RT	42.80	1	42.80	7.15	0.0130
D-P	34.20	1	34.20	5.72	0.0246
Residual	149.56	25	5.98		
Pure Error	3.00	5	0.6009		
Cor Total	246.21	29			

The Analysis of Variance (ANOVA) was employed to assess the statistical significance of the linear model (Nanduri *et al.*, 2008). From table 4 above, the sequential p-value of 0.0116 is significant since $p < 0.05$. A model F-value of 4.04 was established. This implies that there was a chance of less than 1.16% that a value this much could be caused by noise. Consequently, the linear model was selected and applied in this study. ANOVA for the linear model of hydrogen sulphide.

Table 5

ANOVA for the Linear Model of H₂S

Source	Sum of squares	df	Mean square	F-value	p-value
Model	183.38	10	18.34	3.62	0.0076
A-WFR	0.0417	1	0.0417	0.0082	0.9286
B-GFR	22.04	1	22.04	4.36	0.0506
C-RT	45.38	1	45.38	8.97	0.0074
D-P	35.04	1	35.04	6.93	0.0164
AB	22.56	1	22.56	4.46	0.0482
AC	39.06	1	39.06	7.72	0.0120
AD	0.0625	1	0.0625	0.0124	0.9127
BC	0.0625	1	0.0625	0.0124	0.9127
BD	5.06	1	5.06	1.00	0.3297
CD	14.06	1	14.06	2.78	0.1119
Residual	96.12	19	5.06		
Pure Error	0.8333	5	0.1667		
Cor Total	279.50	29			

The ANOVA tables 3, 4, and 5 have p-values of 0.0112, 0.0116, and 0.0076, respectively, implying that the linear models for methane, carbon dioxide, and hydrogen sulfide yields were significant since their p-values were less than 0.05 for the chosen confidence interval of 95% (Quanhong & Caili, 2005). From Table 5, the ANOVA for H₂S yield, the water flow rate is a significant factor when combined with other factors, particularly gas flow rate and gas retention time.

3.1.6 Response Surface Plots

The two-dimensional contours and three-dimensional surface plots relating water flow rate and gas flow rate for the optimal responses are highlighted as in figures 3.1a, 3.1b, 3.2a, 3.2b, 3.3a, and 3.3b below. The plots help visualize the relationship between the input variables and the responses. From the figures 3, 4, 5, 6, 7, and 8 which show the contour and surface plots reveal that the optimal methane content of 91.45%, and the optimal CO₂ reduction of 6.91% were achieved at the water flow rate of 5.0 litres per hour.

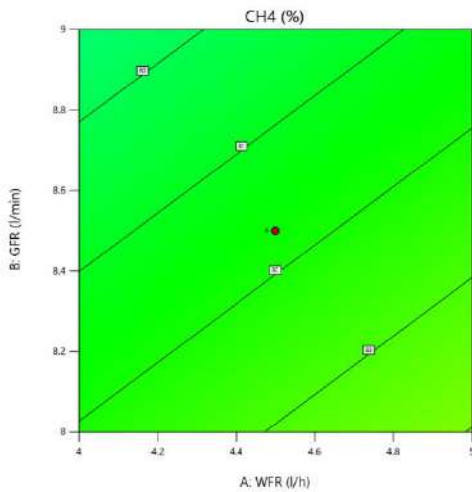


Figure 3
2D contour plot for WFR and GFR for CH₄

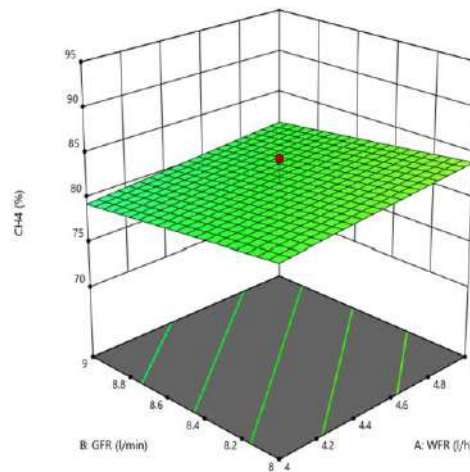


Figure 4
3D surface plot for WFR and GFR for CH₄

The 2D contour and 3D surface plots for interaction of WFR and GFR for CH₄ shown in figures 3 and 4 respectively, reveal that the optimal methane content was attained at a water flow rate of 5.0 Litres per hour.

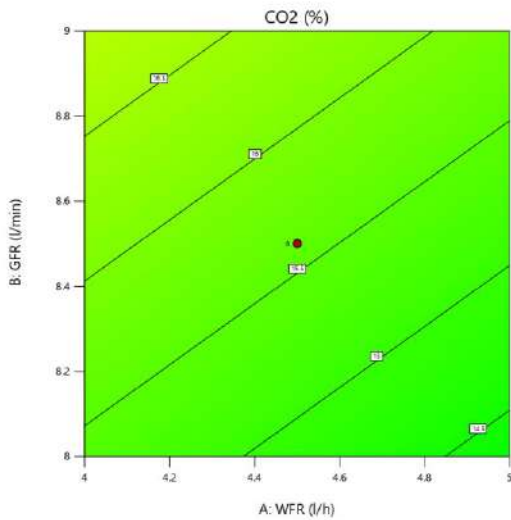


Figure 5
2D contour plot for WFR and GFR for CO₂

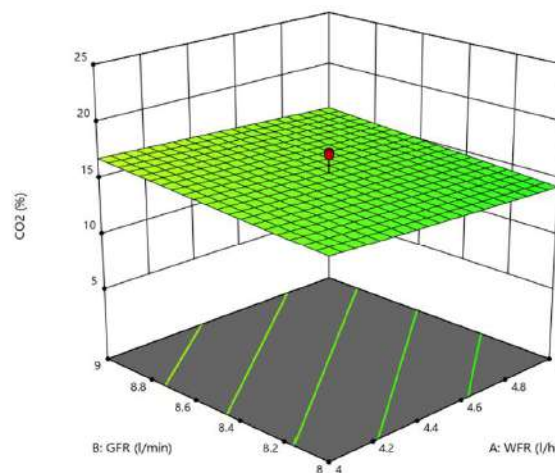


Figure 6
3D surface plot for WFR and GFR for CO₂

The 2D contour and 3D surface plot for interaction of WFR and GFR for CO₂ shown in figures 5 and 6 respectively, shows that the maximum CO₂ removal of up to 6.91% was attained at a water flow rate of 5.0 Litres per hour.

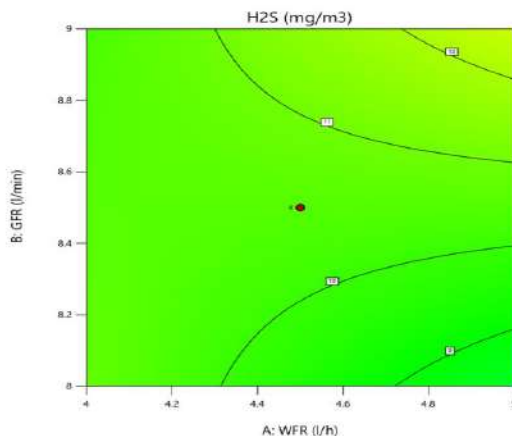


Figure 7
2D contour plot for WFR and GFR for H₂S

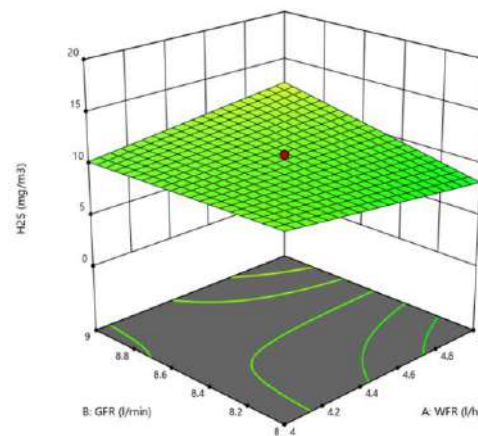


Figure 8
3D surface plot for WFR and GFR for H₂S



The 2D contour and 3D surface plot for interaction of WFR and GFR for H₂S shown in figures 6 and 7, respectively, indicate that the 100% removal of H₂S was attained at a water flow rate of 5.0 Litres per hour.

IV. CONCLUSION & RECOMMENDATIONS

4.1 Conclusion

In this study, the water flow rate required in the optimization of biogas purification by water scrubbing was investigated, and its performance was analyzed using RSM and CCD techniques. The system's optimal performance was investigated through an experimental matrix of thirty runs. Four control factors (Water Flow rate, gas flow rate, scrubber pressure, and gas retention time) were varied to attain a combination that resulted in the optimal performance of the system. The optimal performance of the system was attained at a water flow rate (WFR) of 5 litres per hour. Compared with the control experiment, methane was purified from 80.66% to 91.45% - an improvement of 10.79%, carbon dioxide was reduced from 15.76% to 6.91% - a reduction of 8.85%, and hydrogen sulphide went down from 10 mg/m³ to 0 mg/m³ - 100% removal.

From the results, methane concentration increases with an increase in water flow rate. This, therefore, implies that optimization of water flow rate is vital in achieving the maximum methane content. This study, contrary to high-pressure water scrubbing technology, is cost-saving as no energy or power is needed, making it suitable for use at both domestic and commercial levels. Further research is to be undertaken to establish the impact of the purity of water on the optimal process.

4.2 Recommendations

Optimization of water scrubbing has proven to yield a high methane content; further research should be conducted to establish the effect of water purity on the quality of enriched biogas.

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