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Research Article

RSM-based Optimization of Methane Yield From Palm Oil Mill Effluent

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Abstract: The palm oil industry generates huge amounts of Palm Oil Mill Effluent (POME), which poses environmental challenges due to its high organic content and toxicity. Anaerobic digestion (AD) is a potential method to treat POME and produce methane-rich biogas as an alternate source of renewable energy. However, the maximum methane yield requires a statistical approach to deal with the complex interactions of key operating parameters. This study employs Response Surface Methodology (RSM) for the optimization and modeling of methane yield from POME under the conditions of pH (7.0 - 7.2), temperature (35.8 - 42.5 °C) and recirculation ratio (0.76 - 3.06). Dual parameter optimization was conducted by analyzing interactions between pH, temperature and recirculation ratio in pairs, allowing better insight into combined effects on methane yield. The developed RSM model used to predict the optimum conditions for maximum methane yield while minimizing hydrogen sulfide concentration. Among these 80, 100 and 120 runs that were generated by Design Expert V.13 software, the developed RSM model with 80 runs demonstrated high predictive accuracy with a R^2 of 0.9294. The optimized conditions at 37.39 °C, pH 7.08 and recirculation ratio of 3.06 resulted in a methane yield of 0.2825 Nm^3 CH_4/kg COD removed and hydrogen sulfide concentration of 729.424 ppm, representing a 3.3% increase in methane yield and 33% reduction in hydrogen sulfide concentration to previous literature. These findings provide the reliability of RSM in enhancing biogas production efficiency, offering a valuable strategy for sustainable waste management and renewable energy generation in the palm oil sector.

Keywords: Response Surface Methodology (RSM); Palm Oil Mill Effluent (POME); Methane Yield; Hydrogen Sulfide Reduction; Anaerobic Digestion (AD).

1. Introduction

The palm oil industry is one of Malaysia's primary agricultural activities, contributing primarily to the country's economy. Even so, the process generates substantial waste, particularly Palm Oil Mill Effluent (POME). As stated on the Ministry of Plantation and Commodities official website, approximately 7.4 million tonnes of waste generated from palm oil mills in 2022, and this amount is expected to grow in the coming years [1]. POME gives significant environmental risks on soil and water qualities due to its high concentration of organic nitrogen, chemical oxygen demand (COD), biochemical oxygen demand (BOD) and oil and grease [2].

Anaerobic Digestion (AD) has emerged as an effective treatment method, capable of significantly reducing COD and BOD levels, by converting POME into methane, a renewable energy source [3]. POME treatment through anaerobic digestion in optimizing methane yield is essential as it converts a high-organic waste into a valuable renewable energy source, becoming an ideal feedstock for biogas production while reducing environmental risks such as water pollution and greenhouse gas emissions. Enhanced methane production offers economic advantages by lowering energy costs, enabling energy sales and supporting carbon footprint. Compared to other renewable fuels like hydrogen or biodiesel, methane from POME is more cost-effective, scalable, and compatible with existing infrastructure because it relies on waste rather than competing for agricultural resources. Overall, optimizing methane yield maximizes system efficiency, promotes sustainability, and strengthens energy security and environmental protection.

However, the efficiency of methane yield through AD is dependent on numerous operating conditions, which necessitated systematic optimization to maximize methane production and improve the overall performance of POME treatment. POME anaerobic digestion treatment has been studied using various modelling approaches, such as kinetic, mechanistic and empirical models. Kinetic models help understand the dynamic behavior of anaerobic digestion processes, but it might not account for all system complexities. Mechanistic models, on the other hand, offer a detailed biological, chemical, and physical insights. However, they require extensive data and computational resources. Hence, empirical models are selected to optimize methane yield through AD using Response Surface Methodology (RSM). RSM combines statistical modelling and experimental design to identify important factors and their interactions, enabling optimization while minimizing resource use and operational costs.

Although Gaussian and Surge models can achieve high R^2 values, they often struggle with high prediction errors in changing environments, especially when the data doesn't follow a normal distribution. This limitation can lead to inaccurate predictions, system instability and reduced efficiency in optimizing methane yield. In contrast, RSM is well-suited for dealing with these complex, non-linear relationships among variables and provides precise optimization of operational conditions. Building on these modelling foundations, recent studies have applied RSM to optimize various anaerobic digestion systems. Studies by Hoon et. al. [4], Abeng et. al. [5] and Olatunji & Madyira [6] demonstrated RSM's effectiveness in optimizing co-digestion, agricultural waste and biogas production from oxidative pretreated *Xyris Capensis*, showing that RSM is beneficial in enhancing the precision, efficiency, and applicability of methane yield modeling, making it a powerful tool in advancing renewable energy generation.

Therefore, this study applies Response Surface Methodology (RSM) to model and optimize the key parameters affecting methane yield during POME treatment, employing industrial-scale data for validation. It enables direct performance comparison with existing models from Chen et. al. [3] and Chong et. al. [7], lies in the enhancement of RSM using extended experimental designs while exploring how increasing the number of experimental runs impacts the statistical accuracy, predictive reliability, and robustness of the RSM models. This approach offers new insights into the effect of model complexity and data volume on optimization outcomes for methane yield and reduction of hydrogen sulfide concentration. Furthermore, this study introduces a dual-response modeling approach by concurrently analyzing both methane and hydrogen sulfide concentration. The extended model framework not only reinforces confidence in parameter interactions but also supports better-informed operational decisions for the AD process in treating POME, contributing a valuable perspective on the scalability and industrial relevance of RSM in optimizing full-scale biogas operations.

2. Materials and Methods

2.1. Data Collection

Data were obtained from a real industrial-scale biogas plant located in Pahang, Malaysia, operating with POME as the primary substrate. The study varied key operating parameters including pH (7.0 7.2), temperature (35.8 - 42.5°C), and recirculation ratio (0.76 - 3.06) while measuring the methane yield and hydrogen sulfide concentration.

2.2. Selection of Experimental Design and RSM Model Development

Design Expert v13 software was employed for modelling and optimizing methane yield while minimizing hydrogen sulfide concentration. The Box-Behnken Design (BBD) was selected due to its efficiency, requiring fewer runs and ability to accurately estimate main and interaction effects. BBD also avoids extreme operating points, ensuring safety and practicality in industrial conditions. Also, three sets of BBD-RSM modelling and optimization for 80, 100 and 120 runs were carried out to analyze the outcomes differences from the existing literature and the study's RSM models.

2.3. Pre-processing Data

Quadratic polynomial equations were developed for the 80, 100 and 120 runs. Analysis of Variance (ANOVA), R^2, adjusted R^2, and lack-of-fit tests were used to evaluate the statistical significance of each model term, as well as improvements in model fit, predictive accuracy and statistical reliability as the number of experimental runs increased.

2.4. Modelling and Optimization Using Response Surface Methodology (RSM)

The optimization began with validating the RSM model using the experimental data collected for predictive accuracy. 3D response surface plots were then utilized to evaluate the main effects and interactions of the independent variables. RSM was employed to identify operating conditions that simultaneously maximize methane yield and reduce H2S concentration. Rather than direct H2S removal, this approach focuses on process-level parameter adjustment to create conditions that suppress sulfide formation while favoring methanogenesis, thereby lowering H2S concentration. Subsequently, optimized parameter values were validated using additional experimental runs to confirm their practicality and effectiveness for industrial-scale applications. These outcomes served as the basis for further statistical evaluation and sensitivity analysis.

2.5. Performance Metrics

Developed RSM models were evaluated using R^2, Mean Absolute Error (MAE), Mean Squared Error (MSE), Root Mean Square Error (RMSE) and Mean Absolute Percentage Error (MAPE). The equations used for MAE, MSE, RMSE and MAPE are shown as listed below, respectively;

$$\frac{1}{x}\sum_{i=1}^{x}|A_i - P_i| \tag{1}$$

$$\frac{1}{x}\sum_{i=1}^{x}(A_i - P_i)^2 \tag{2}$$

$$\sqrt{\frac{1}{x}\sum_{i=1}^{x}(A_i - P_i)^2} \tag{3}$$

$$\frac{1}{x}\sum_{i=1}^{x}\frac{|A_i - P_i|}{A_i} \tag{4}$$

Where x is the number of observations, A_i is the actual value and P_i is the predicted value. The estimated range of methane yield after optimization should be around $\pm$ 1% of differences when compare it with actual data, and possibly be in range between 0.25 Nm3/kg COD removed until 0.35 Nm3/kg COD removed based on various literature reviews after done the optimization.

2.6. Performance Evaluation

A pareto chart was done to identify the most influential model terms affecting methane yield and hydrogen sulfide concentration by computing the percentage and cumulative contribution of each factor and interaction. Meanwhile, sensitivity analysis through pie-chart provided an estimate of the contribution of each input parameter to methane yield, based on the sum of squares (SS) for the model and linear terms obtained from ANOVA. The relative importance of each factor is calculated using the sum of squares to determine its contribution towards methane yield [3]. The Equation (5) used is as follows, where SS_f is the sum of squares of factor meanwhile SS_T is the sum of squares of all factors:

$$\frac{SS_f}{SS_T} \times 100\% \tag{5}$$

3. Results and Discussion

3.1. RSM Modelling and Optimization

The RSM model was developed to predict methane yield using the input variables; pH (A), temperature (B) and recirculation ratio (C). The relationship is expressed through the following second-order polynomial equation:

$$\text{methane yield}, y_1 = -249.03817 + 71.02848A - 0.097069B - 0.337371C + 0.034254AB + 0.096522AC - 0.007333BC - 5.12375A^2 - 0.001612B^2 - 0.013960C^2$$

The Analysis of Variance (ANOVA) test for all run sizes was illustrated at Table 1, Table 2 and Table 3 as shown below. It included the sum of squares, degree of freedom, mean square, F-values and P-values for each experimental run.

Table 1. ANOVA and significance test of each model term for 80 runs.

Source	Sum of squares	Degree of Freedom	Mean square	F-value	p-value	Remark
Model	0.0491	9	0.0055	102.36	<0.0001	significant
A - pH	0.0033	1	0.0033	62.07	<0.0001	
B - Temperature	0.0032	1	0.0032	59.21	<0.0001	
C - Recirculation Ratio	0.0006	1	0.0006	11.23	0.0013	
pH X Temp	0.0005	1	0.0005	9.88	0.0025	
pH X RR	0.0005	1	0.0005	9.25	0.0033	
Temp X RR	0.0032	1	0.0032	59.88	<0.0001	
pH X pH	0.0137	1	0.0137	257.60	<0.0001	
Temp X Temp	0.0017	1	0.0017	32.10	<0.0001	
RR X RR	0.0018	1	0.0018	33.45	<0.0001	
R^2			0.9294			
Adjusted R^2			0.9203			

Table 2. ANOVA and significance test of each model term for 100 runs.

Source	Sum of squares	Degree of Freedom	Mean square	F-value	p-value	Remark
Model	0.0503	9	0.0056	134.91	<0.0001	significant
A - pH	0.0033	1	0.0033	79.81	<0.0001	
B - Temperature	0.0032	1	0.0032	76.12	<0.0001	
C - Recirculation Ratio	0.0006	1	0.0006	14.44	0.003	
pH X Temp	0.0005	1	0.0005	12.70	0.0006	
pH X RR	0.0005	1	0.0005	11.89	0.0009	
Temp X RR	0.0032	1	0.0032	76.99	<0.0001	
pH X pH	0.0138	1	0.0138	332.66	<0.0001	

Temp X Temp	0.0017	1	0.0017	41.46	<0.0001	
RR X RR	0.0018	1	0.0018	43.19	<0.0001	
R^2			0.9310			
Adjusted R^2			0.9241			

Table 3. ANOVA and significance test of each model term for 120 runs

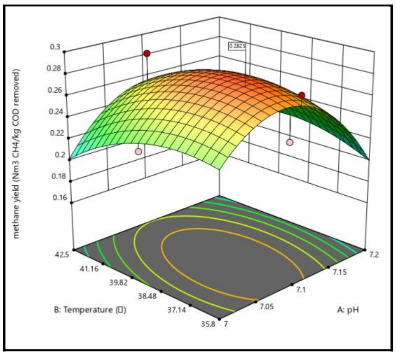
Source	Sum of squares	Degree of Freedom	Mean square	F-value	p-value	Remark
Model	0.0512	9	0.0057	167.59	<0.0001	significant
A - pH	0.0033	1	0.0033	97.54	<0.0001	
B - Temperature	0.0032	1	0.0032	93.04	<0.0001	
C - Recirculation Ratio	0.0006	1	0.0006	17.65	0.0013	
pH X Temp	0.0005	1	0.0005	15.53	0.0025	
pH X RR	0.0005	1	0.0005	14.53	0.0033	
Temp X RR	0.0032	1	0.0032	94.10	<0.0001	
pH X pH	0.0138	1	0.0138	407.71	<0.0001	
Temp X Temp	0.0017	1	0.0017	50.81	<0.0001	
RR X RR	0.0018	1	0.0018	52.94	<0.0001	
R^2			0.9320			
Adjusted R^2			0.9265			

Referring Table 1, Table 2 and Table 3, the data consistently show that the quadratic RSM model is statistically significant ($p < 0.0001$). For all run sizes, the quadratic pH term is the strongest contributor with the highest F-values, which are 257.60, 332.66 and 407.71 respectively. The interaction between temperature and recirculation ratio also remains significant across all models. Increasing the run size improves model fit, with R^2 increasing from 0.9294 to 0.9310 to 0.9320.

3.2. 3D Surface Plots

All 3D surface plots across the 80, 100, and 120 runs were shown as Figure 1, Figure 3 and Figure 5 for methane yield, meanwhile Figure 2, Figure 4 and Figure 6 for hydrogen sulfide reduction, showing the optimized responses and the interaction effects among all those parameters (pH, temperature, RR).

The optimized parameters were pH of 7.08, temperature of 37.39 °C, and recirculation ratio of 3.06, resulting in methane yield of 0.2825 Nm^3 CH_4/kg COD removed. For hydrogen sulfide concentration, the calculated values were 729.424 ppm, 729.529 ppm, and 729.548 ppm for 80, 100, and 120 runs respectively, showing a slight decrease with increased experimental runs. Certain dual parametric optimizations such as pH with temperature and temperature with recirculation ratio showed significant effects on methane yield. These interactions emphasize the importance of considering the combined parameters effects, as they play a crucial role in enhancing methane yield while simultaneously reduce hydrogen sulfide concentration.



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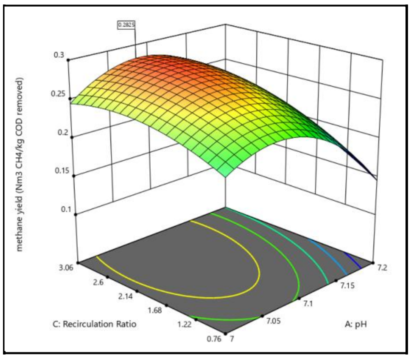

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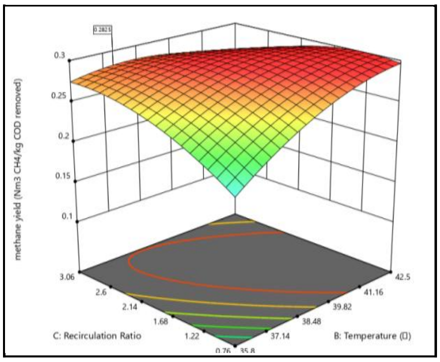
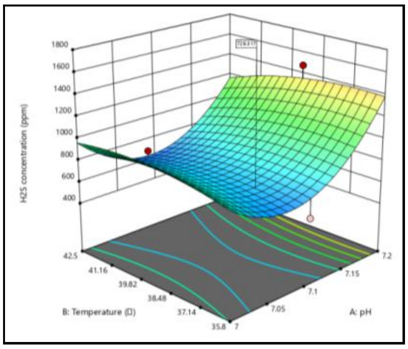


(**c**)

Figure 1: 3D surface plots for methane yield of 80 runs (a) pH x temperature; (b) pH x recirculation ratio; (c) temperature x recirculation ratio.



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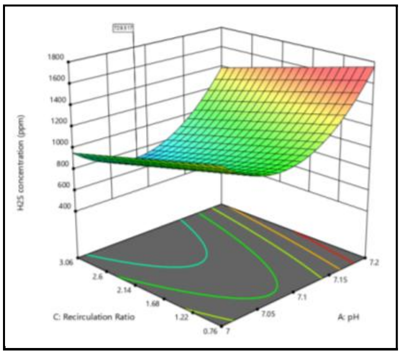

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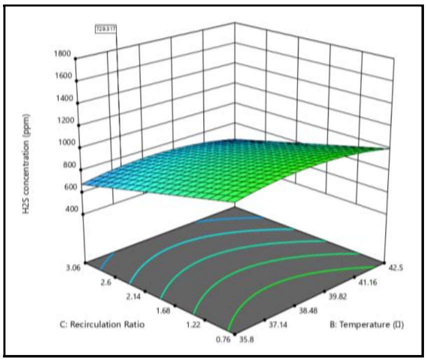

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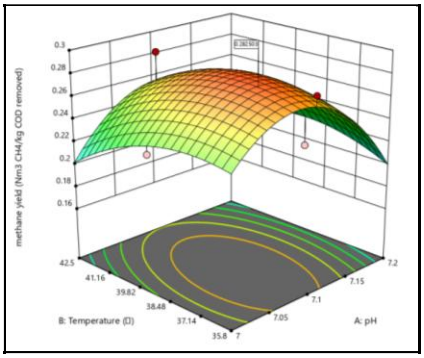
Figure 2: 3D surface plots for hydrogen sulfide reduction of 80 runs (a) pH x temperature; (b) pH x recirculation ratio; (c) temperature x recirculation ratio.



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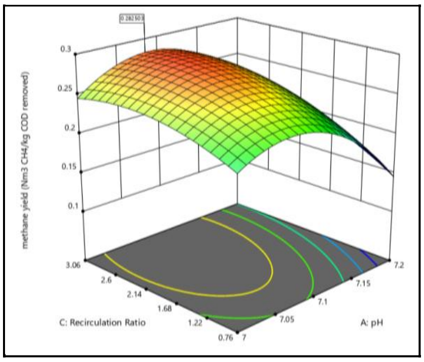

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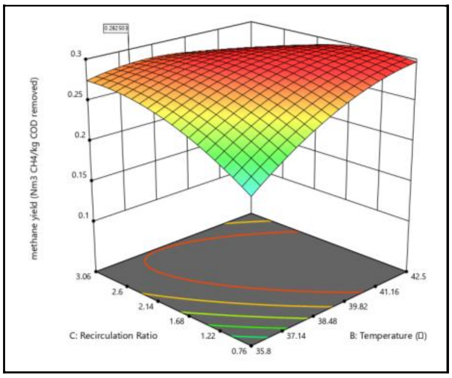

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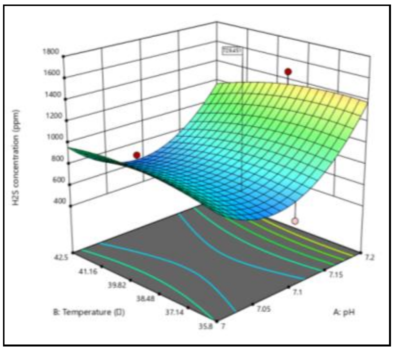
Figure 3: 3D surface plots for methane yield of 100 runs (a) pH x temperature; (b) pH x recirculation ratio; (c) temperature x recirculation ratio.



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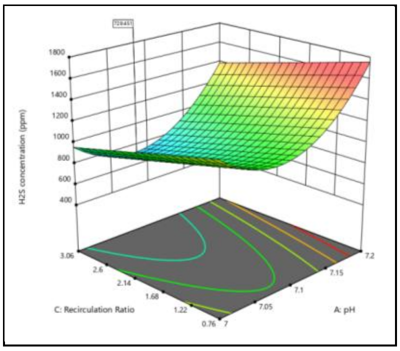

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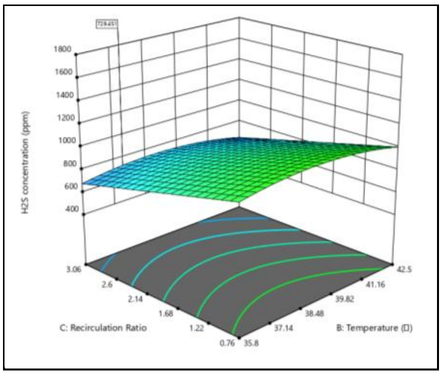

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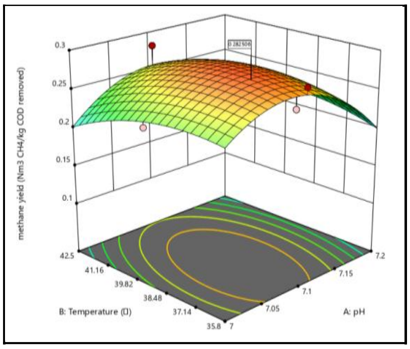
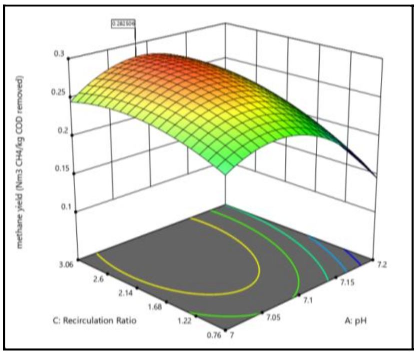
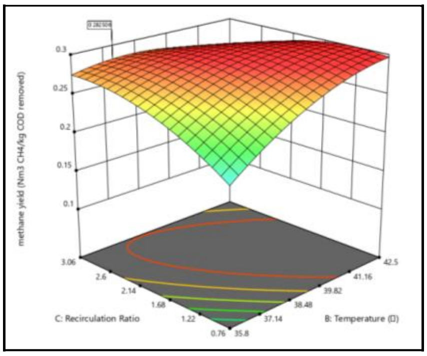
Figure 4: 3D surface plots for hydrogen sulfide reduction of 100 runs (a) pH x temperature; (b) pH x recirculation ratio; (c) temperature x recirculation ratio.



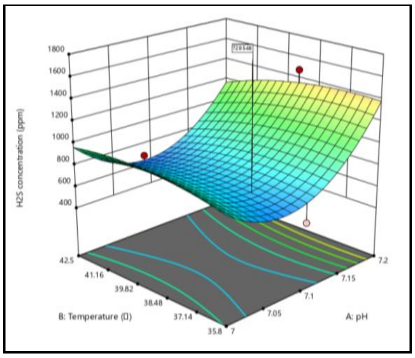


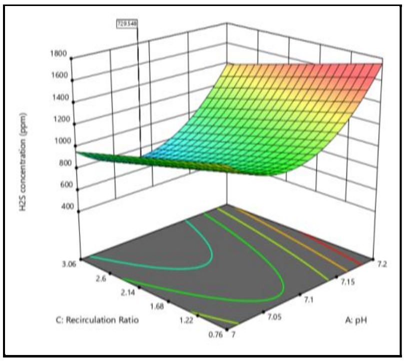
(a) (b)



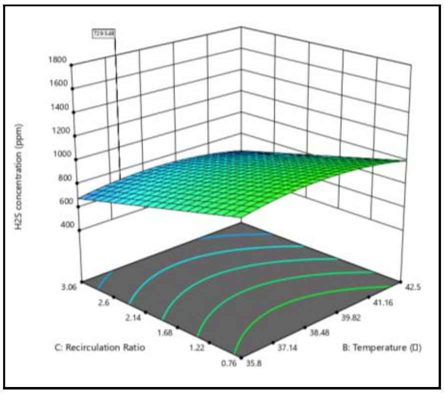
(c)

Figure 5: 3D surface plots for methane yield of 120 runs (a) pH x temperature; (b) pH x recirculation ratio; (c) temperature x recirculation ratio.





(a) (b)



(c)

Figure 6: 3D surface plots for hydrogen sulfide reduction of 120 runs (a) pH x temperature; (b) pH x recirculation ratio; (c) temperature x recirculation ratio.

3.3. Performance Analysis

3.3.1. Statistical Metrics

The performance of the RSM models was evaluated using the statistical metrics, which were R^2, MAE, MSE, RMSE and MAPE, that was constructed at Table 4.

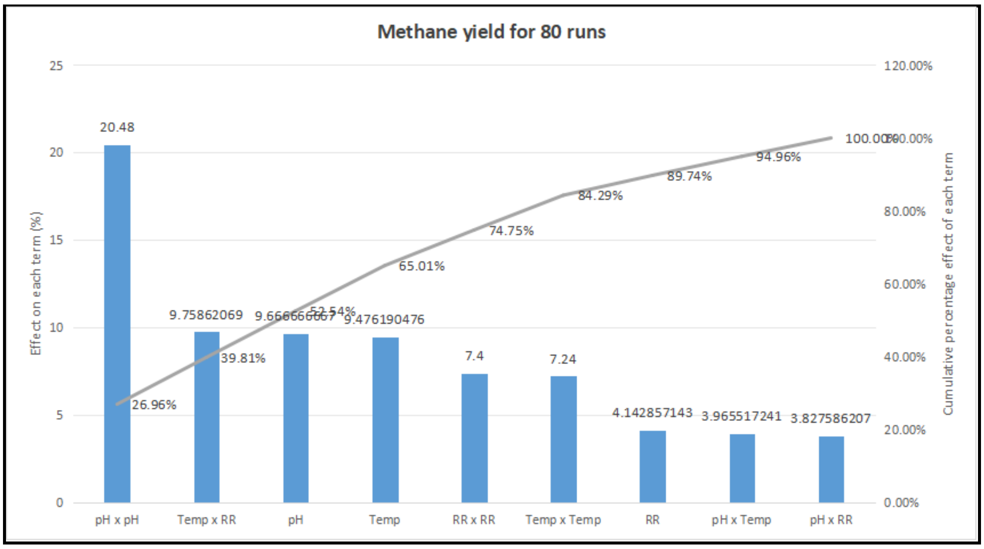
Table 4. Performance metrics for BBD-RSM models

	80	100	120
R^2	0.9294	0.9310	0.9320
MAE	0.00222	0.00178	0.00148
MSE	0.00005	0.00004	0.00003
RMSE	0.00683	0.00611	0.00558
MAPE (%)	0.01000	0.00800	0.00666

Overall, the 80 runs model provides strong predictive ability with acceptable accuracy and low error values while 120 runs give the smallest error values and largest correlation coefficients, providing highly accurate predictions in optimizing methane yield.

3.3.2. Pareto Chart

Pareto chart was done to identify the most important model terms that contributed to methane yield and hydrogen sulfide concentration, which were shown in Figure 7, Figure 8 and Figure 9.



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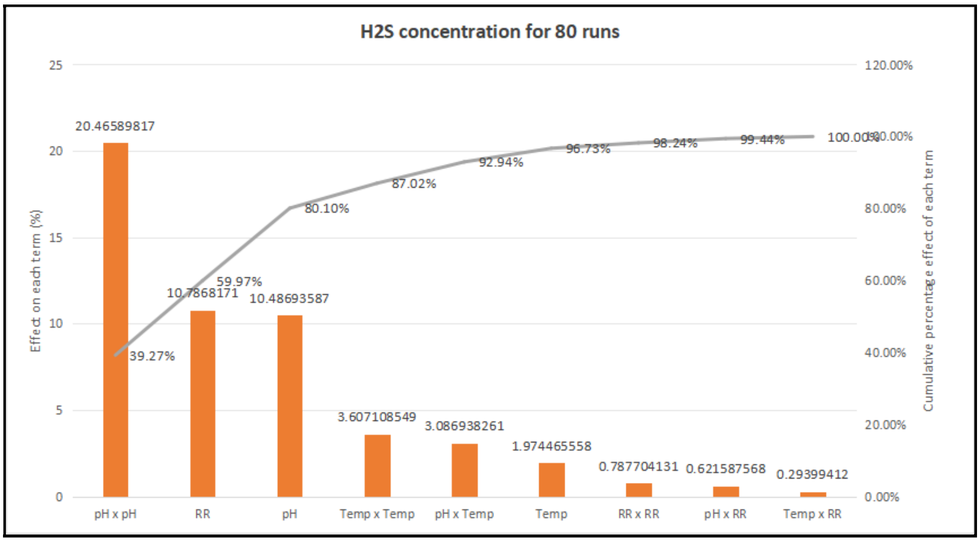

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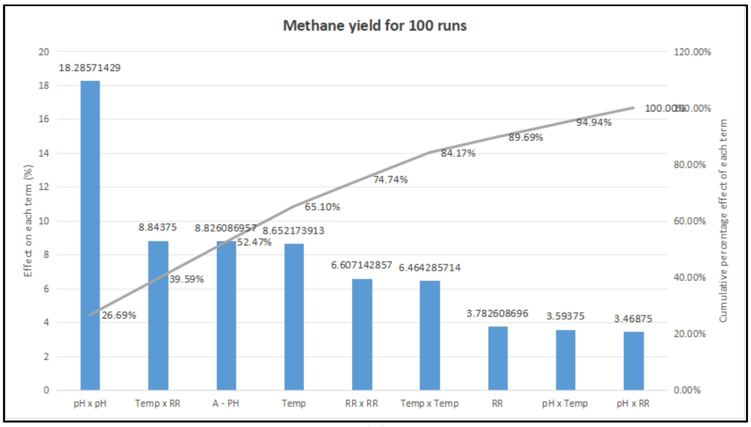
Figure 7: Pareto chart for 80 runs (a) methane yield; (b) hydrogen sulfide reduction.



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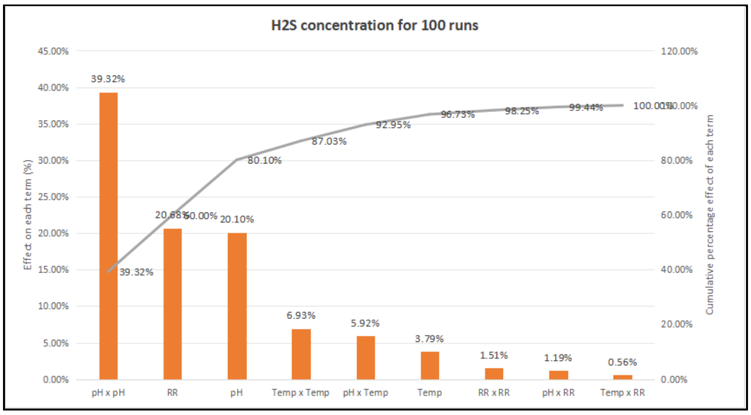

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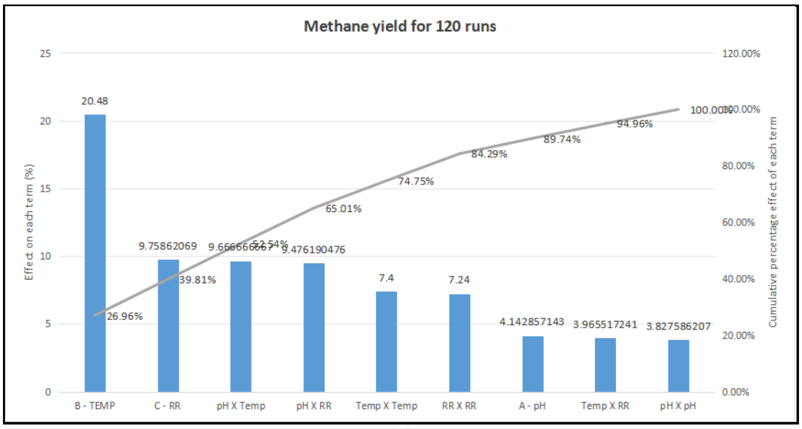
Figure 8: Pareto chart for 100 runs (a) methane yield; (b) hydrogen sulfide reduction.



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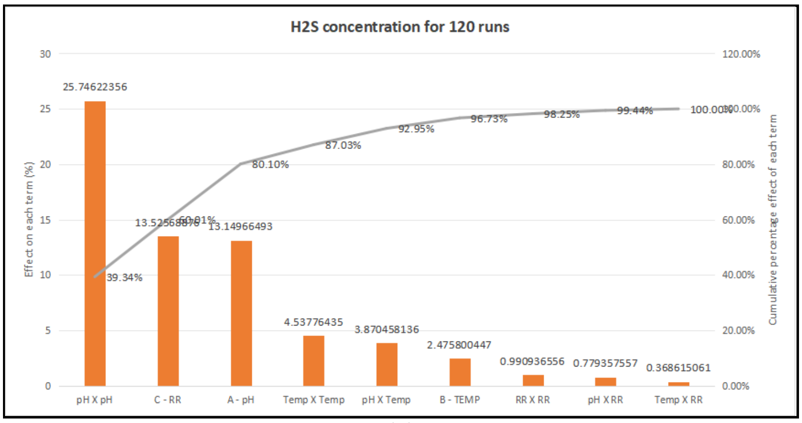

(**b**)

Figure 9: Pareto chart for 120 runs (a) methane yield; (b) hydrogen sulfide reduction.

Across all run sizes (80, 100, and 120), the quadratic pH term (pH × pH) consistently showed the highest influence on both methane yield and hydrogen sulfide reduction. For methane yield, pH × pH contributed 18.29% to 20.48%, followed by the interaction between temperature and recirculation ratio (Temp × RR) at 8.83% to 9.76%. Other linear and interaction terms such as RR (linear), pH × Temp, and pH × RR had much smaller effects, generally below 5%. For hydrogen sulfide reduction, pH × pH remained the dominant factor, contributing 20.47% to 39.32%. The linear terms of recirculation ratio and pH also contributed significantly at 10.49% to 20.68%. In contrast, quadratic RR, pH × RR, and Temp × RR consistently showed minimal effects below 2%, indicating very limited influence on hydrogen sulfide variability

3.3.3. Sensitivity Analysis

Figure 10 shows the relative contributions of temperature, pH and recirculation ratio to methane yield, that was illustrated through pie chart as shown below;

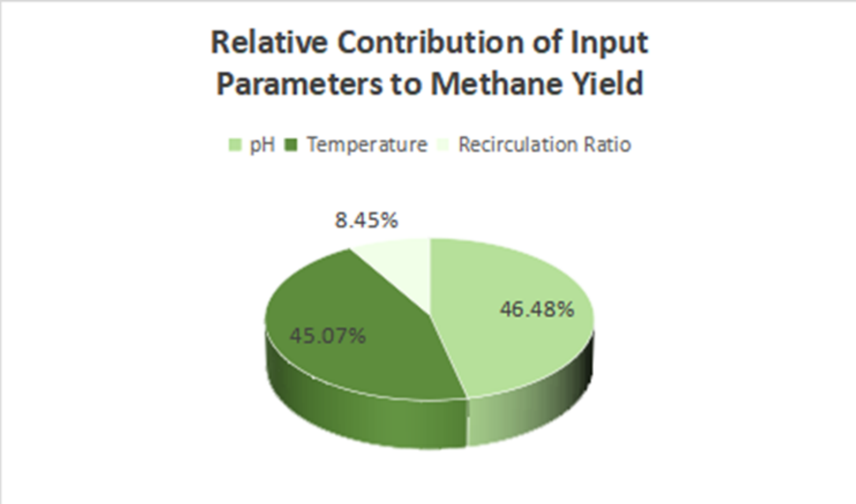

Figure 10: Relative contribution of input parameters to methane yield.

pH emerged as the most influential factor with 46.48%, followed closely by temperature at 45.07%, while recirculation ratio made least contribution at 8.45%. Due to high sensitivity and directly regulate microbial activity, even small changes in pH and temperature can substantially affect methane yield. This emphasizes the necessity for accurate control of these parameters during the anaerobic digestion to maintain stable microbial performance and maximize biogas production.

3.3.4. Data Comparison between Experimental and Article Data

Table 5 compares the literature outcomes for optimum conditions by Chen et. al., [3] against the optimized conditions and predicted responses obtained in this study.

Table 5. Comparison results between experimental and article data for three sets of experimental runs using BBD-RSM by Design Expert Software.

Source	**Article**	**Experimental**		
Number of experimental runs	80	80	100	120
pH	7.36	7.08	7.08	7.08
Temperature (°C)	42.0	37.39	37.39	37.39
Recirculation Ratio	2.5	3.06	3.06	3.06
Methane Yield (Nm³ CH_4/kg COD removed)	0.2733	0.2825	0.2825	0.2825
H_2S **concentration (ppm)**	1086.4	729.424	729.529	729.548
Desirability	0.799	0.868	0.868	0.868

All datasets provided highly similar optimized solutions, demonstrating the reliability and consistency of the RSM-BBD models. The referenced article reported optimal conditions at pH 7.36, 42.0 °C, and a recirculation ratio of 2.5, yielding 0.2733 Nm^3 CH_4/kg COD removed with 1086.4 ppm of hydrogen sulfide concentration. In contrast, this study consistently converged on pH 7.08, temperature at 37.39 °C, and a recirculation ratio of 3.06 across all run sizes, predicting a higher methane yield of 0.2825 Nm^3 CH_4/kg COD removed (3.3% improvement), and a substantially lower hydrogen sulfide concentration of 729.4 to 729.6 ppm (33% reduction).

These improvements are attributed to a better-regulated microbial environment as pH, temperature and recirculation ratio directly influence methanogenic activity and system stability. Operating at pH 7.08 supports methanogenesis and reduces hydrogen sulfide formation, the mesophilic temperature of 37.39°C enhances stability and energy efficiency compared to thermophilic conditions, and the higher recirculation ratio improves microbial retention, substrate contact and digestion efficiency while diluting localized hydrogen sulfide.

The desirability index further supports these findings where the referenced article reported 0.799, whereas all models developed in this study achieved 0.868, highlighting the effectiveness of RSM in identifying the optimal range of operating conditions that maximize methane yield and minimize hydrogen sulfide concentration. Additional runs (100 and 120 runs) merely provide further confidence and statistical reinforcement to support that 80 runs already significant and become a strong model, from both referenced article and this study.

3.3.5. Limitation and Recommendations

The optimization was based solely on a controlled experimental set of conditions and narrow parameter ranges, which can limit generalizability to other digestate systems or feed mixture compositions. Furthermore, the developed model is specific to the plant data used from those existing literature and may not be applicable to other plants with different conditions. Also, the interaction effects of other possibly influential variables, such as hydraulic retention time (HRT), organic loading rate (OLR), and microbial population dynamics, were not examined.

To strengthen the findings and real application of the study, the inclusion of other parameters (HRT, OLR and variability of feedstock) can enhance the robustness and applicability of the model further. Integration of advanced techniques, such as microbial community analysis or hybrid model techniques (such as combination of RSM and machine learning) could further increase prediction accuracy and system adaptability. Lastly, incorporation of economic and environmental impact analysis would improve a more holistic optimization framework for POME biogas production.

4. Conclusions

This study verifies the effectiveness of Response Surface Methodology (RSM) in modeling and optimizing key parameters in the anaerobic digestion of POME to maximize methane yield and reducing hydrogen sulfide concentration. Using 80 experimental runs and refined model parameters, the optimized conditions at 37.39 °C, pH 7.08 and recirculation ratio of 3.06 produced a methane yield at 0.2825 Nm^3 CH_4/kg COD removed and hydrogen sulfide concentration of 729.424 ppm. These values represent a 3.3% increase in methane yield and 33% reduction in hydrogen sulfide concentration compared to previous literature. Overall, the findings reinforce the consistency, reliability and robustness of RSM as a process optimization modeling technique in biogas production processes.

Declaration of Competing Interest: The authors declare no conflict of interest.

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