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A Response Surface Methodology Approach to Enhancing the Performance of CO2 Methanation Pilot Plant System

Nurul Noramelya Zulkefli[1,*], Wan Muhammad Faris Wan Ramli[1], Noraziah Muda[1], Ridzuwan Mohd Jais[1], Ahmad Lokman Omar[1], Muhammad Zul'izzuddin Muda[1], Mohd Asyraf Mohd Azmir Phang[1], Mohd Shalleh Ahmad Fauzi[1], Khairunisa Mazlan[2]

[1] Renewable Energy and Green Technology Unit, TNB Research Sdn. Bhd., 43000 Kajang, Selangor, Malaysia
[2] Faculty of Chemical and Energy Engineering, Universiti Teknologi Malaysia, 81310 Johor Bahru, Johor, Malaysia

ABSTRACT

Considerable attention has been devoted to the technological advancement of non-fossil fuel energy sources to mitigate carbon emissions and establish a sustainable energy infrastructure for future generations. One of the primary obstacles encountered in the generation of power from renewable energy sources pertains to the storage of the variable energy output. The integrated process has been developed to evaluate the feasibility of the carbon dioxide (CO_2) methanation reaction with the integration of renewable energy sources to power up hydrogen production. The Sabatier reaction principle was used to carry out CO_2 methanation in a pilot system of a two-stage catalytic packed bed methanation reactor. Hydrogen was added to get a conversion rate of up to 99%. The focus of this paper was to conduct a comprehensive examination of the response surface methodology (RSM) applied to a pilot system. The goal was to determine the optimal operating parameters for the carbon dioxide methanation process using a commercial catalyst known as nickel or alumina. The study looked at the reactions of CO_2 conversion and CH_4 selectivity, with two important factors being the temperature of the reactor column, which could be anywhere from 150 to 350 °C, and the flow rate of hydrogen, which could be anywhere from 7.9 to 9.9 L/min. The central composite design was selected as the methodology for constructing the optimal condition. Consequently, the numerical analysis conducted in this study was corroborated by experimental data, with an observed percentage error of around 0.5% under similar operating conditions.

Keywords:
Carbon dioxide; hydrogen; catalysis reaction; methanation; response surface methodology

1. Introduction

Climate change is a pressing issue that our planet is currently confronting. The current worldwide catastrophe is mostly caused by the escalating emissions of greenhouse gases, particularly carbon dioxide. The majority of emissions are worsened by the substantial impact of human activity, particularly the burning of fossil fuels for transportation or energy production, as well as industrial

* *Corresponding author.*
E-mail address: noramelya.zulkefli@tnb.com.my

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activities. Besides, the process of ethanol production also generates carbon dioxide emissions as a result of the carbon dioxide produced during fermentation. The waste gas produced during ethanol production has a high percentage of carbon dioxide [1]. The predominant technique under investigation to mitigate carbon dioxide emissions is carbon capture, storage, and utilisation (CCUS). This technology encompasses the processes of capturing, transporting, and storing CO_2 underground. Instead, the carbon dioxide (CO_2) could be used to make fuels and chemicals. For example, methane (CH_4) could be dry-reformed to make synthesis gas, or CO_2 could be hydrogenated to make methane, methanol, or higher alcohols [2].

To make use of the carbon dioxide released throughout the process and the hydrogen produced from renewable energy sources, the hydrogen and carbon dioxide are transformed into fuels with high-added value, such as methanol, dimethyl ether, and methane. The conversion of hydrogen and carbon dioxide into methane as the end product is intriguing because of its compatibility with the current infrastructure for storage and delivery. The methanation reaction, known as the Sabatier reaction, was initially investigated by Sabatier and Senderens at the start of the previous century [3]. The CO_2-methanation reaction is as follows:

$$CO_2 + 4H_2 = CH_4 + 2H_2O \qquad \Delta HR = -165.1 \text{ kJ/mol} \qquad (1)$$

This reaction is strongly exothermic and is controlled by chemical equilibrium [4]. Because of speed limits, the CO_2 methanation process is mostly done at low temperatures (250–400°C) with catalysts based on nickel. Because of thermodynamic equilibrium, undesirable competing reactions such as CO methanation and the reverse water-gas shift reaction can occur at high temperatures, as follows:

$$CO + 3H_2 = CH_4 + H_2O \qquad \Delta HR = -206.3 \text{ kJ/mol} \qquad (2)$$

$$CO_2 + H_2 = CO + H_2O \qquad \Delta HR = 41.2 \text{ kJ/mol} \qquad (3)$$

Therefore, one of the main issues in reactor scale-up is to remove heat generated by this exothermic reaction and maintain the process at a relatively low temperature without generating hot spots or quenching the reaction [5]. Moreover, the operation must be performed at an appropriate temperature range to prevent unwanted competing reactions [6]. Initially, the method was employed to eliminate small amounts of carbon oxides from the gas used in the production of ammonia [7]. Recently, there has been a resurgence of interest in CO_2 methanation due to its application in power-to-gas technology and biogas upgrading. Power-to-gas technology involves the conversion of excess renewable energy into hydrogen, which is then combined with CO_2 from power plants, industrial processes, or biological sources. This chemical reaction results in the production of methane, which can be conveniently stored and delivered using the existing natural gas infrastructure.

There are two methods available: biological methanation and thermochemical methanation [8]. Biological methanation, conducted using an anaerobic digester, operates at a lower temperature in comparison to thermochemical methanation. However, a drawback of this technique is the extended reaction duration due to hydrogen's limited ability to dissolve in the liquid phase, as stated by Hervy *et al.,* [9] Another alternative is thermochemical methanation, which employs a catalyst to function at a controlled temperature ranging from 300 to 450 °C. A Ni-based catalyst is commonly used as a commercial catalyst for methanation. Ahmad *et al.,* [10] did research that showed the Ni-Ce/eg-C_3N_4 catalyst could change 83% of CO_2 into methane and 99% of CO_2 into methane at 297 °C. To study

how CO_2 methanation works, Yin *et al.,* [11] used Ni-based catalysts made from hydrotalcite and replaced them with Fe (Ni3-Fe0.5-calc).

The conversion of CO_2 to methane using a Ni3-Fe0.5-calc catalyst resulted in a 78% conversion rate at a temperature of 200 °C. The operational parameters, such as temperature, pressure, the ratio of hydrogen to carbon dioxide, and the composition of the feed, affect how much methane is produced by the thermochemical methanation process. Ren *et al.,* [12] investigated the impact of contaminants, specifically N_2, steam, and O_2, on CO_2 methanation. Their findings demonstrated that the presence of trace amounts of O_2 in the feed improved the CO_2 methanation process. This was attributed to the production of additional *OH groups, which effectively promoted the conversion of intermediates into methane.

However, there are still unresolved issues with the reaction's implementation. The product yield is influenced by the process parameters and catalyst. A lot of work has gone into looking into different aspects of CO_2 methanation, such as the catalytic aspect, process design, and the problems that come with putting it into practice [8,13]. The current catalysts under development often exhibit good selectivity towards CH4; however, enhancing the conversions at lower temperatures remains a significant priority. The rate at which CO_2 is converted and the preference for methane can also be changed by things like temperature, pressure, gas hourly space velocity (GHSV), and the initial concentration of CH_4 [2].

In the past, researchers have primarily used the OFAT (one-factor-at-a-time) optimisation strategy to investigate nickel/alumina-based catalysts for the methanation reaction. The OFAT methodology is a complex and labour-intensive method for assessing numerous process parameters to obtain a comprehensive understanding of the methanation process. Assessing the relative significance of various components in a complicated process is challenging with this technique, especially when evaluating how two or more aspects interact with each other. The Response Surface Methodology (RSM) is a widely recognised mathematical and statistical technique that aims to minimise the time and effort needed to carry out experiments, create optimisation models, assess the impact of parameters, and identify the optimal values of independent parameters [14]. The optimal customized approach is a multivariate response surface methodology employed in experimental design [15]. To optimise operational parameters, a statistical method is required to assess the relationship between elements influencing the process and the process output. The face-centred composite design (FCCD) is typically employed to establish the connection between one or more response variables and a group of quantitative experimental factors. It is used to identify the factor settings that maximise the response. It was also utilised to accommodate a quadratic surface. This strategy not only helps optimise the individual and interaction impacts of operating parameters [17]. This method examines a set of experimental parameters, which can be either quantitative or qualitative, in connection to one or more responses. This study aims to investigate how response surface approaches can improve methane output.

Moreover, this study investigated the influence of temperature and H_2 feed flow rate on a methanation system at a pilot scale. The study examined the performance of four response variables: the conversion of CO_2 in Reactor 1, the selectivity of CH_4 in Reactor 1, the conversion of CO_2 in Reactor 2, and the selectivity of CH_4 in Reactor 2.

2. Materials and Methods

2.1 Methanation

This investigation utilised commercial carbon dioxide (CO_2) gas with feed flow rate were set at 2.2 L/min for each run. Then, the hydrogen was generated using water electrolysis using sustainable

energy, specifically solar power, to dissociate the H_2 and O_2 molecules. Subsequently, the H_2 is stored in the tank prior to its utilisation in the methanation process. in this study, the feed flow rate is mimic to gas ratio of CO_2:H_2 which is the H_2 flowrate were varied. The H_2 flow rate were varied between 7.92, 8.91 and 9.90 which is referring to gas ratio CO_2:H_2 as 1:3.6, 1:4.05, 1:4.5, respectively. The catalyst employed in this investigation consists of commercially available Nickel/alumina. The constituent gases were analysed using an Agilent gas chromatography (GC) instrument. All this material was utilised for the methanation process through the research of response surface methods.

2.2 Experimental Design and Statistic Analysis

The experiments in this study were conducted utilising the central composite design with the assistance of Design-Expert software (version 11.0, Stat-Ease, Inc., Minneapolis, MN, USA). Two distinct variables, specifically H2 flow rate (L/min) and temperature (°C), were utilised. The measured response variables were Reactor 1's CO_2 conversion rate, Reactor 1's CH_4 selectivity, Reactor 2's CO_2 conversion rate, and Reactor 2's CH_4 selectivity. The studies were conducted in a randomized order to mitigate the influence of extraneous variables on the response. The design consists of 13 runs: 4 factorial points, 4 axial points, and 5 replicates' centre points. The amounts of each factor for the primary composite design are specified in Table 1 and were determined based on prior experimental investigations.

Table 1
Levels of input experimental variables

Factors	−1	0	+1
H_2 flowrate, L/min	7.92	8.91	9.90
Temperature, °C	150	250	350

An analysis of variance (ANOVA) was conducted using the Design-Expert program (version 11.0, Stat-Ease, Inc., Minneapolis, MN, USA) to analyse the CO2 conversion (%) and CH_4 selectivity (%) data for reactors 1 and 2. However, this article will focus on analysing the data from Reactor 2, as it is considered to be the primary reactor for the methanation process. The experimental data were calibrated using a quadratic equation:

$$Y = \beta_0 + \Sigma\beta_i X_i + \Sigma\beta_{ii} X_i^2 + \Sigma\beta_{ij} X_i X_j \quad (4)$$

The response variable, Y, is determined by the independent variables, X_i and X_j. The intercept is denoted as B_0, the linear coefficient as B_i, the quadratic coefficient as B_{ii}, and the interaction coefficient as B_{ij}. The independent variables were assigned codes A and B. The polynomial equation was expressed as:

$$Y = \beta_0 + \beta_1 A + \beta_2 A + \beta_3 A + \beta_{11} A^2 + \beta_{22} B^2 + \beta_{12} AB \quad (5)$$

The variable Y reflects the anticipated outcome, either CO_2 conversion or CH_4 selectivity. A corresponds to the H_2 flowrate and B corresponds to the temperature (°C). The selection or rejection of the model terms was determined by the probability value (P) at a confidence level of 95%. The polynomial equation model's fit quality was assessed using the coefficient of determination (R^2), adjusted R^2, and "adequate precision". Three three-dimensional (3D) response surfaces were created, each accompanied by its corresponding contour plot. These surfaces were developed to

analyse the impact of the two-factor levels. The study examined the concurrent influence of two factors on the response variable using 3D graphs.

3. Results and discussion

3.1 Model Analysis and Regression Equation

To investigate the impact of process parameters on the catalytic efficiency of a commercially available nickel/alumina catalyst, a statistical method was utilised to design the experiments utilising face-centred composite design (FCCD). This FCCD is a three-level practical in an RSM study by considering the experimental design in which the axial points are focused on the cubic surface rather than the sphere, and the α value is equal to 1 [18]. Hence, the flowrate of hydrogen and operating temperature have been optimised with the FCCD, and the operational correlation between independent factors and the response has been developed.

Table 2
Central composite experimental design for the RSM optimization

		Factor 1	Factor 2	Response 1	Response 2	Response 3	Response 4
Std	Run	A:H_2 flowrate	B:Temperature	R1 CO_2 Conversion	R1 CH_4 Selectivity	R2 CO_2 Conversion	R2 CH_4 Selectivity
		LPM	°C	%	%	%	%
9	1	8.91	250	36.3	61.5	51.6	73.1
4	2	9.9	350	85.5	95.9	97.4	99.3
11	3	8.91	250	72	71.3	72.8	87
3	4	7.92	350	91.4	97.6	98	99.4
12	5	8.91	250	32	62.8	47.4	77.8
8	6	8.91	350	77.8	91.7	93.8	97.8
7	7	8.91	150	4.2	6	5	8.1
1	8	7.92	150	15.4	3.2	16.8	10
13	9	8.91	250	42	72.9	68.3	85.7
6	10	9.9	250	58	60.3	67.3	72.5
5	11	7.92	250	46.9	73.8	74.6	89.2
2	12	9.9	150	7.2	4	15.1	5
10	13	8.91	250	52.7	74.5	72.8	87.5

The FCCD was implemented to construct a predictive model for the interaction between process factors and the response variable. The process variables selected for the methanation process were the H_2 flowrate (A) and temperature (B). To optimise the process parameters linked to methane production, a series of experimental runs, as shown in Table 2, was conducted. Table 3 displays the recommended linear model that has been utilised for ANOVA analysis.

Table 3
Summary of model selection in RSM for CO_2 conversion

Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	PRESS	
Linear	11.37	0.8700	0.8440	0.8256	1735.81	Suggested
2FI	11.98	0.8702	0.8269	0.7938	2051.67	
Quadratic	12.78	0.8850	0.8029	0.7279	2708.09	
Cubic	14.35	0.8965	0.7516	0.7556	2432.24	Aliased

The RSM analysis generated a predictive model, represented by Eq. (6), for the response of CO_2 conversion in Reactor 2. The CO_2 conversion in Reactor 2 is determined by the total reaction that occurs in Reactor 2 (R2).

$$\text{R2 } CO_2 \text{ conversion} = +60.07 - 1.60A + 42.05B \quad (6)$$

3.2 ANOVA Analysis for CO_2 Conversion in Reactor 2

The FCCD model was evaluated by statistical testing by putting the experimental CO_2 conversion data into ANOVA analysis, as shown in Table 4. The estimated probability, often known as the p-value, is an important parameter that determines the relevance of each component in the model. The ANOVA findings in Table 4 indicate that the model was statistically significant due to its low p-value of 0.0001 and high F-value of 45.79. Furthermore, the chosen process parameters were deemed significant due to the observed low p-value.

Table 4
Analysis of variance (ANOVA) for central composite design for CO_2 conversion

Source	Sum of squares	df	Mean square	F-value	p-value	
Model	10624.58	2	5312.29	45.79	< 0.0001	significant
A-H2 flowrate	15.36	1	15.36	0.1324	0.7235	
B-Temperature	10609.21	1	10609.21	91.45	< 0.0001	
Residual	1160.15	10	116.02			
Lack of Fit	567.54	6	94.59	0.6385	0.7035	not significant
Pure Error	592.61	4	148.15			
Cor Total	11784.73	12				

Temperature (B) was identified as the most influential parameter in CO_2 conversion at Reactor 2, based on its high F-value (91.45) and low p-value (<0.0001). The Lack of Fit F-value of 0.64 indicates that the Lack of Fit is not statistically significant compared to the pure error. The probability of a Lack of Fit F-value of this magnitude occurring solely due to noise is 70.35 %. A lack of major lack of fit is beneficial for the model's fit.

The coefficient of determination (R^2) value is a crucial measure for assessing the quality of a model which shown as in Table 5. Researchers have established that good statistical models of best fit should have an R^2 value between 0.75 and 1 [19,20]; hence, the R^2 value achieved, which is 0.90, implies that 90 % of the data variability is accounted for by the regression model, demonstrating its fitness and appropriateness.

Table 5
Model summary statistics response of CO_2 conversion

R^2	0.90
Adjusted R^2	0.88
Predicted R^2	0.85
A_{deq} Precision	16.87

The predicted R^2 value of 0.85 shows a decent level of agreement with the adjusted R^2 value of 0.88, indicating that the discrepancy between the two values is less than 0.02, which is similar to the pattern in a study by Sunarti and Ahmad [17]. A_{deq} Precision measures the ratio of the signal strength to the background noise level. A ratio over 4 is preferable. According to the ratio of 16.87, the signal is considered sufficient. This paradigm is applicable for navigating the design space.

3.3 ANOVA Analysis for CH_4 Selectivity in Reactor 2

The RSM analysis produced a prognostic model, denoted as Eq. (7), for the CH_4 selectivity response in Reactor 2. The CH_4 selectivity in Reactor 2 is dictated by the overall reaction taking place in Reactor 2. While Table 6 shows the suggested model for this study were quadratic model.

$$R2\ CH_4\ selectivity = +81.97 - 3.63A + 45.57B + 1.23AB - 0.4793A^2 - 28.38B^2 \quad (7)$$

Table 6
Summary of model selection in RSM for CH_4 selectivity

Source	Sequential p-value	Lack of Fit p-value	Adjusted R^2	Predicted R^2	
Linear	0.00	0.02	0.78	0.64	
2FI	0.90	0.01	0.75	0.29	
Quadratic	0.00	0.66	0.97	0.94	Suggested
Cubic	0.42	0.82	0.97	0.97	Aliased

The FCCD model's statistical validity was checked by using ANOVA to look at the experimental data on CH_4 selectivity, which can be seen in Table 7. The estimated probability, commonly referred to as the p-value, is a crucial parameter that determines the significance of each component in the model. The ANOVA results presented in Table 7 demonstrate that the model achieved statistical significance, as evidenced by its low p-value of 0.0001 and high F-value of 89.98. Moreover, the selected process parameters were considered significant because of the observed low p-value. Based on its high F-value (369.24) and low p-value (<0.0001), the parameter that had the biggest influence on CH_4 selectivity at Reactor 2 was temperature (B). The Lack of Fit F-value of 0.57 suggests that the Lack of Fit is not statistically significant when compared to the pure error. The likelihood of observing a Lack of Fit F-value of this magnitude entirely because of random variation is 66.45%. The absence of a significant lack of fit is advantageous for the model's fit.

Table 7
Analysis of variance (ANOVA) for central composite design for CH_4 selectivity

Source	Sum of squares	df	Mean square	F-value	p-value	
Model	15179.38	5	3035.88	89.98	< 0.0001	significant
A-H2 flowrate	79.21	1	79.21	2.35	0.17	
B-Temperature	12457.93	1	12457.93	369.24	< 0.0001	
AB	6.00	1	6.00	0.18	0.69	
A^2	0.63	1	0.63	0.019	0.89	
B^2	2224.40	1	2224.40	65.93	< 0.0001	
Residual	236.18	7	33.74			
Lack of Fit	70.63	3	23.54	0.5689	0.66	not significant
Pure Error	165.55	4	41.39			
Cor Total	15415.55	12				

Table 8 shows the predicted R^2 value of 0.98 is reasonably consistent with the adjusted R^2 value of 0.97, indicating a discrepancy of less than 0.2. A_{deq} Precision quantifies the ratio of signal to noise. A ratio over 4 is preferable. A ratio of 24.94 indicates a satisfactory signal. This paradigm is applicable for navigating the design space.

Table 8
Model summary statistics response of CH_4 selectivity

R^2	0.98
Adjusted R^2	0.97
Predicted R^2	0.94
A_{deq} Precision	24.94

3.4 Interactive Effects of Process Parameters and Optimization

Process optimization was performed using the FCCD interface of the RSM. The optimisation results were acquired by examination of the surface response plots of interactive effects between the process variable and CO_2 conversion. Figure 1 displays the 3D response surface plot, which shows practically a linear relationship with the CO_2 conversion.

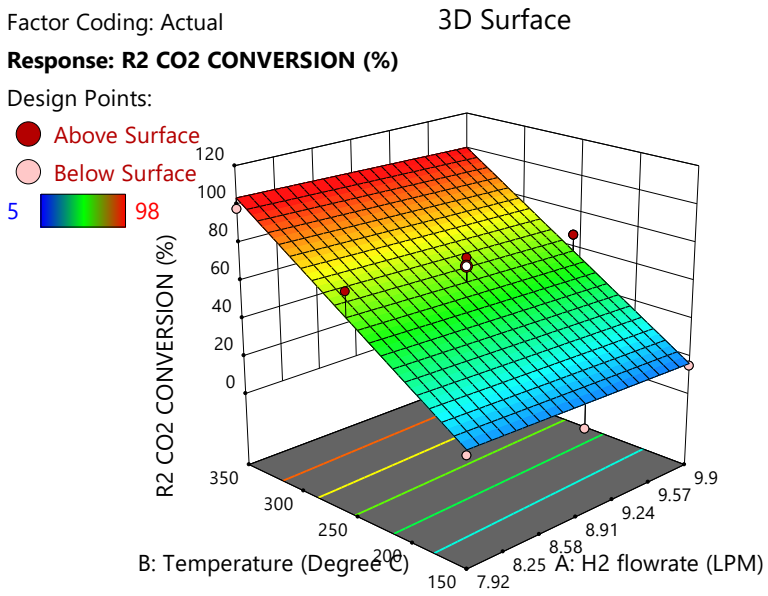

Fig. 1. Surface plot of interaction between the significant factors (A) H_2 flowrate and (B) temperature for CO_2 conversion

The graph in Figure 1 illustrates the relationship between the surface responses for CO_2 conversion in Reactor 2 (Y) and two variables: the H_2 flow rate (A) and the operating temperature (B). The graph shows that the conversion of CO_2 increases as the reaction temperature rises, reaching its maximum value at the optimal temperature. The endothermic nature of the reaction is attributed to this [21]. Similarly, raising the flow rate of H_2 leads to an increase in CO_2 conversion up to a particular threshold, provided that the applied temperature is similarly increased. However, at a constant temperature, increasing the flow rate of H_2 only leads to a minor variation in CO_2 conversion. The result obtained suggests that the presence of H_2 in the feed acts as a potent oxidising agent, leading to a favourable increase in CO_2 conversion [22,23].

The figure labelled Figure 2 displays a surface plot illustrating the combined influence of H_2 flowrate and operating temperature on CH_4 selectivity in Reactor 2. Concerning CH_4 selectivity, the plot reveals a semi-spherical relationship between the operating temperature and H_2 flowrate. According to the trend shown in Figure 2, the selectivity of CH_4 decreases gradually as the flowrate of H_2 steadily increases. This demonstrates that the flow rate of H_2 has an impact on activity, as evidenced by a consistent decrease in CH_4 selectivity with an increasing H_2 flow rate. According to

previous research, this aligns with the idea that a higher gas hourly space velocity (GHSV), like the flow rate of H_2 in this study, means that H_2 molecules interact with active sites more quickly [22,24].

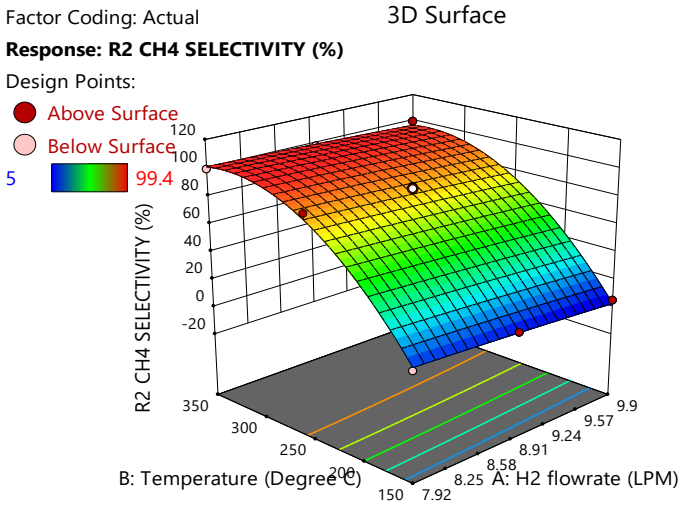

Fig. 2. Surface plot of interaction between the significant factors (A) H_2 flowrate and (B) temperature for CH_4 selectivity

Figure 3 depicts the desirability plot of the optimisation for the objective function. Desirability is a quantitative measure that ranges from zero (0) at the lower limit to one (1) at the upper limit, as stated in reference [25]. The peak desirability function was found within the numerical optimisation. The desirability function is dependent on the proximity of the lower and upper bounds to the actual optimal conditions. According to this study, the desirability value is 0.968, indicating the highest level of desirability performance. Hence, the optimal condition observed in this study might be employed to establish another experiment for additional validation.

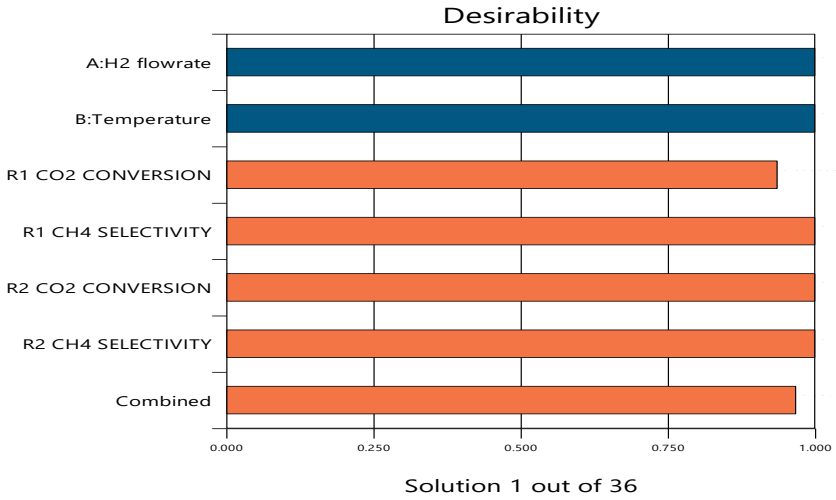

Fig. 3. Bar chart for desirability analysis

The RSM Analysis (Figure 4) yielded the optimal conditions of 102.29 % CO_2 conversion and 99.4 % CH_4 selectivity at Reactor 2, with H_2 flowrate and temperature set at 8.8 L/min and 350 °C, respectively.

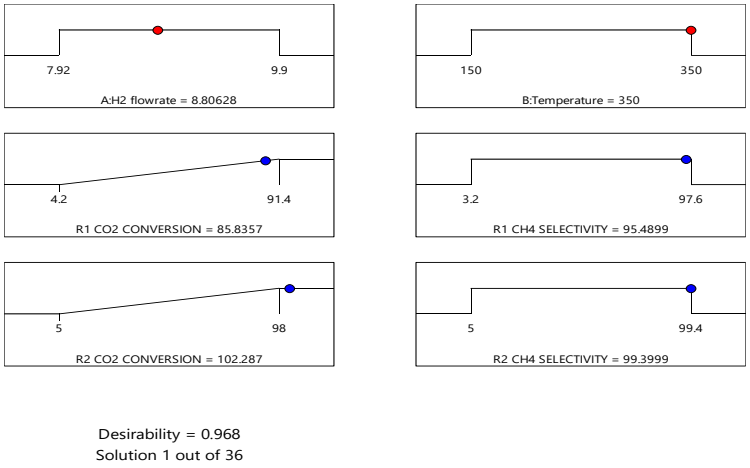

Fig. 4. Ramp plot for desirability analysis

Additionally, a complementary experiment was conducted to verify the accuracy of the regression model using the projected values that yielded the best results. Generally, to check the validity of the suggested model for maximum CO_2 conversion and CH_4 selectivity, one needs to conduct experiments for the optimum conditions. Three experiments were conducted for H_2 flowrate at 8.8 L/min and temperature at 350 °C. The average CO_2 conversion and CH_4 selectivity are 100 % for both were obtained from the validation experiments; that is very close to the predicted value with a discrepancy of 2.29 % and 0.6 % between the observed and expected values. Therefore, the suggested model for optimum CO_2 conversion and CH_4 selectivity process is accepted.

4. Conclusions

Based on the results shown above, the commercial nickel/alumina catalyst was better at changing CO_2 into CH_4 when it was heated up and the H_2 flow rate was set to its ideal level. The flow rate of H_2 is a key factor in determining the best contact time between H_2 and the catalyst. This allows CO_2 to be converted to CH_4 more selectively. In addition, the primary factor that significantly affects CO_2 conversion and CH_4 selectivity is the elevated working temperature of 350 °C, and the gas ratio is compatible with the Sabatier coefficient, which is 1 CO_2: 4 H_2, or else the H_2 feed flow rate at 8.9 L/min. But more research needs to be done to find out how to improve the CO_2 conversion at lower temperatures using different catalysts. This could lead to more useful and cost-effective results for the methanation process. In conclusion, our research shows that a higher temperature is the best setting for common commercial catalysts in the methanation process, which leads to more CO_2 being converted.

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