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Application of Central Composite Experimental Design for Optimization of Hydrogen Yield via Low Temperature Catalytic Bioethanol Steam Reforming

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Abstract

The integration of the response surface methodology (RSM) and ASPEN Plus (V8.8) was used to optimize the process parameters for bioethanol steam reforming over 10%Ni/MgO//H_2SO_4 modified metakaolin supported catalyst for hydrogen production. The response surface methodology designated with central composite design (CCD) was used, along with the thermodynamic analysis by Gibbs free energy minimization method. The optimum operating conditions that yielded the maximum hydrogen from the process optimization modelling using the best fitness function were obtained to be: steam-to-bioethanol molar ratio; 8 mol/mol, temperature; 400 ℃, and at atmospheric pressure. Both predicted and experimental data were in agreement, having coefficient of determination (R^2) of 0.9701 and adequate precision of 22.625. The validated results suggest (indicate) that the optimum parameters enable reliable operating conditions for steam reforming of bioethanol over the tested catalyst, yielding a bioethanol conversion of 99.5 % and hydrogen production of 3.72 mol/mol bioethanol.

Keywords: Bioethanol, Reforming, Optimization, Hydrogen, Yield

1.0 INTRODUCTION

In view of the exponential increase in global energy demand for essential services, coupled with the over dependence on fossil-derived energy sources, has intensified the need to harness renewable and environmentally benign energy resources. A renewable and sustainable energy resource, capable of meeting the ever growing energy demand and regulating the carbon cycle is receiving urgent attention at the moment. Out of several energy resources, an energy carrier such as hydrogen has been considered as viable option for clean economy, especially in the decarbonisation of energy sector and chemical-allied industries [1][2][3]. Due to enormous energy requirements for manufacturing and related industries, agricultural residues have been utilized as major substrates for bioethanol production. Bioethanol has the potential to be used as an alternative to carbon-fuels and enhance energy security, in view of its cleaning burning properties and its vast production renewable sources. [4]. Sunwoo *et al.* [5] reported that the global ethanol production through fermentation process is 97 %, while only 3 % is produced through the catalytic hydration of petroleum-derived ethylene. The prominent reforming mechanisms for producing hydrogen from bioethanol are steam reforming (ESR), partial oxidation (POX), and autothermal reforming (AR) [6]. However, steam reforming is chosen as promising mechanism for hydrogen production due to its cost effective, high efficiency (≈ 85 %), less task and substantial yield of hydrogen [7]. Notwithstanding, catalytic bioethanol steam reforming is prone to inactiveness (mostly by carbon deposition and sintering), due to side reactions and its endothermic nature [8]. At specific reaction conditions couple with efficient catalyst, undesired products formation from simultaneous side reactions with bioethanol steam reforming can be avoided [9][10]. To find the optimum operating conditions for hydrogen production, many experiments are needed. In addition, the reforming system and inherent reactions are quite complex, which possess a stern task in modelling and parameter identification [11]. Response surface methodology (RSM) is a widely used and effective technique for examining the effect of changing numerous factors on the output at a time. It is specifically applied in a situation where a number of input variables can influence a performance or response [12]. Furthermore, RSM can be a potential means to reduce the number, time and cost of experiment required for facts findings. Among the several methods of designing experiments, central composite design is preferentially selected because it yields reliable models with sizeable experiments for theoretical assessment of experimental space and better interactions across the prominent variables for optimum response. [13]. In this study, the relevance of independent variables on product distribution from low temperature bioethanol steam reforming over MgO doped/H_2SO_4 modified metakaolin supported-10 % Ni catalyst was investigated and optimal conditions were predicted and validated experimentally.

2.0 MATERIALS AND METHODS

2.1 Catalyst Preparation

Activated metakaolin was functionalized with 6M of tetraoxosulphate (VI) acid at 1:20 by weight. The modified metakaolin (Surface area = 471.7 m^2/g) was loaded with nickel content of 10 wt.% using an aqueous nickel (II) nitrate hexahydrate solution by wet impregnation method. The resultant was mixed with 1g of magnesium oxide, along with 50mL of ethanol and vigorously stirred at 250rpm for 1h. It was subsequently rinsed with warm distilled water to obtain neutral (pH of 7). The resultant slurry was dried under vacuum at 75 ℃ and subsequently calcined at 650 ℃ for 3 h (modified method of Olivares *et al.*[14]).

2.2 Experimental Design and Procedure

Three factors were chosen to determine their effects and interaction on two responses (ethanol conversion and hydrogen yield), using Design Expert software (12.0.3). The levels of such factors are shown in Table 1. Twenty experimental runs were performed to optimize the response based on the considered factors and levels. Model ESR for hydrogen production by Aspen plus was initially used as surrogate experiment to predict product distribution. The matrix for this design is presented in Table 2.

Table 1: Experimental factors and levels for ESR process

Independent Variables	**Coded**	**Low actual value**	**High actual value**	**References**
Temperature (℃)	A	350	450	[15] 16],
Pressure (atm)	B	0.5	1.5	[17]
Steam-to-ethanol ratio (mol/mol)	C	4	12	[18][19]

Table 2: Design matrix for the model

Std	**Run**	**Temperature (℃)**	**Ethanol/Steam Ratio (mol/mol)**	**Pressure (atm)**	**Ethanol Conversion (%)**	**Hydrogen Yield (%)**
20	1	400	8	1	100	51.4
17	2	400	8	1	100	51.4
18	3	400	8	1	100	51.4
3	4	350	12	0.5	100	54.45
12	5	400	14.7272	1	100	74.01
9	6	315.91	8	1	100	20.51
16	7	400	8	1	100	51.4
5	8	350	4	1.5	100	15
1	9	350	4	0.5	100	24.17
10	10	484.09	8	1	100	84.87
14	11	400	8	1.8409	100	40.74
4	12	450	12	0.5	100	86.7

11	13	400	1.2728	1	100	12.98
2	14	450	4	0.5	100	60.2
13	15	400	8	0.1591	100	85.81
19	16	400	8	1	100	51.4
7	17	350	12	1.5	100	36.18
6	18	450	4	1.5	100	41.02
15	19	400	8	1	100	51.4
8	20	450	12	1.5	100	79.97

2.3 Catalyst Performance Test

0.3 g of the catalyst sample was placed on glass wool at about 18cm off the top of the quartz tubular reactor. The respective flow rates of carrier gas (N_2) and reducing gas (10 % H_2) used for the test were 400 mL/min and 40 mL/min, at catalyst reduction temperature of 400 °C for 1h. The vaporized feed mixture (at 100 °C) was fed at 5mL/min by a constant flow pump at temperature of 400 °C, and pressure of 1 atm for 4h test reaction. The unreacted feed was trapped with chiller maintained at 2.5 °C, connected to the gas analyzer equipped with packed column (molecular sieve 5A) and TCD detector to analyse the resulting dry gases. The Equations 1-3 were used to evaluate the system's performance [16]:

$$\text{Ethanol conversion } (\mathrm{X_{ETOH}}) = \frac{CH_{4outlet} + CO_{2outlet} + CO_{outlet}}{2\times \mathrm{EtOH_{intlet}}} \times 100 \qquad (1)$$

$$\text{Hydrogen selectivity } (S_{H_2}) = \frac{F_j}{(\Sigma F_j)_{product}} \times 100 \qquad (2)$$

$$\text{Hydrogen yield } (Y_{H_2}) = (\mathrm{X_{EtOH}}) \times (S_{H_2})$$

(3)

wherer_i is the formation rate of product i, F_{EtOHin} represents ethanol inlet flow rate while $H_{2outlet}$, CO_{outlet}, $CO_{2outlet}$, $CH_{4outlet}$ represent moles of outlet gases from the reactor.

3.0 RESULTS AND DISCUSSION

3.1 ANOVA for Quadratic Response Surface Model for Hydrogen Yield

The significance of a factor and interaction of two factors within the model was checked using ANOVA [20]. A p-value is less than 0.05 and high F-value at 95 % confidence level, signify that the model is significant [21]. Table 3 is the ANOVA result of quadratic model for the hydrogen yield.

Table 3: ANOVA result for the hydrogen yield via ESR

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	9597.06	9	1066.34	36.00	<0.0001	Significant
A-Temperature	4443.08	1	4443.08	150.02	<0.0001	
B-Steam-to-ethanol ratio	3529.52	1	3529.52	119.17	<0.0001	
C-Pressure	1211.79	1	1211.79	40.92	<0.0001	
AB	24.47	1	24.47	0.8261	0.3848	
AC	0.2926	1	0.2926	0.0099	0.9228	
BC	1.40	1	1.40	0.0474	0.8321	
A^2	1.14	1	1.14	0.0386	0.8482	
B^2	179.83	1	179.83	6.07	0.0334	
C^2	167.36	1	167.36	5.65	0.0388	
Residual	296.17	10	29.62			
Lack of Fit	296.17	5	59.23			
Pure Error	0.0000	5	0.0000			
Cor Total	9893.23	19				

$R^2 = 0.9701$, Adjusted $R^2 = 0.9431$

3.2 Mathematical Model Equation for Hydrogen Yield

The impact of every factor, two factors and their interaction on response was verified with the aid of ANOVA [22]. Equation 4 represents the real regression expression explaining the correlation between the input variables and the response (based on coded factors). Intercept of the equation is 51.51, which helps to predict the precise results of the hydrogen yield.

$$\text{Hydrogen yield (\%)} = 51.51 + 18.04A + 16.08B - 9.42C + 1.75.AB + 0.1913AC + 0.4187BC - 0.2815A^2 - 3.53B^2 + 3.41C^2 \quad (4)$$

The closeness of the predicted data to the actual experimental data with respect to the model variables can be determined by the P-value [23]. If p-values are greater than 0.100, the model term is insignificant; if less than 0.05, the model term is significant [24]. Based on the Table 2, the quadratic model presented in this work is significant because the p-value is less than 0.05 [25]. Hence, A, B, C, B^2 and $C^2$are significant model terms, while AB, AC, BC and A^2 are insignificant model terms. At relatively moderate pressure, it is evident that steam/ethanol molar ratio and temperature are very significant in the production of hydrogen via catalytic bioethanol steam reforming. Based on the ANOVA result, the model is suitable to be used for hydrogen production via low temperature bioethanol steam reforming.

3.3 Adequacy Test of the Models

The consistency of the developed model for hydrogen yield was confirmed using R^2-values, adjusted R^2, C.V. and adequate precision (as shown in Table 4).

Table 4: Consistency check of hydrogen yield response surface for full quadratic model

Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	Adequate Precision	Coefficient of variance
Quadratic	5.44	0.9701	0.9431	0.7635	22.6254	9.9%

The good prediction of a model can be ascertained with R^2 value greater than 0.9, which indicate the significance effect on the response. Similarly, Adj R^2 helps to realign the number of terms which are insignificant in a regression model, while predicted R^2 prevents over fitting of a regression mode [23]. R^2 value of the developed regression model was determined to be 0.9701, which implies good fit that can explain 97.01% of the variability. The predicted and adjusted R^2 were determined to be 0.7635 and 0.9431, respectively. It is in reasonable agreement, because 0.2 is the maximum allowable difference [13][26]. The adequacy precision of the model is determined to be 22.6254, which is greater than the desirable value of 4 [27]. Coefficient of variation (CV %) is used to indicate the credibility of result and it should be less than 10 % [28]. The CV of 9.9 % obtained in this study, implies that the model is significant.

3.4 Parity between the Predicted Values and the Experimental Values/Normal Plot

The normal plot is to indicate whether residuals follow a normal distribution, such that points follow straight line and no response transformation required [21][27], as shown in this study (Figure 1). The regression model that is been developed can be validated with the parity chart and the accuracy of the correlation can as well be determined with it. When the predicted and observed values of the responses are scattered and close to the 95% prediction limits with 45^o, it implies that the developed model has almost perfect fit. Figure 2 shows the parity plot for the hydrogen yield.

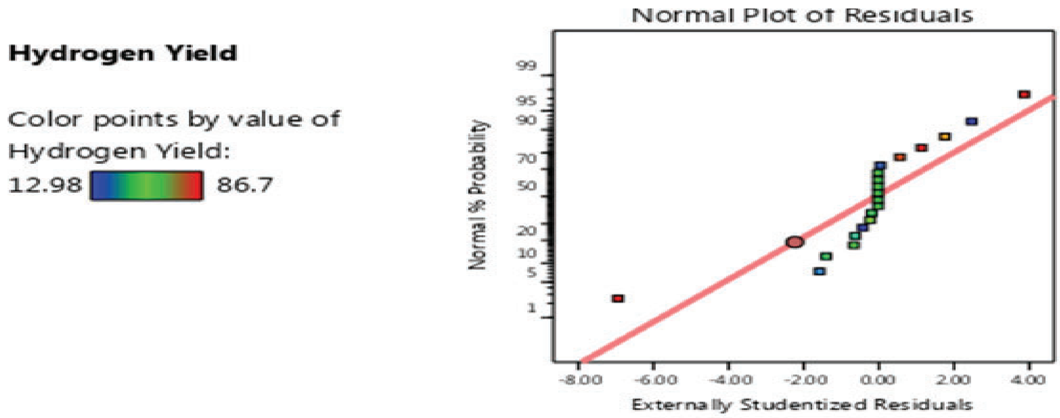

Figure 1: Normal probability plot of hydrogen yield response surface quadratic model

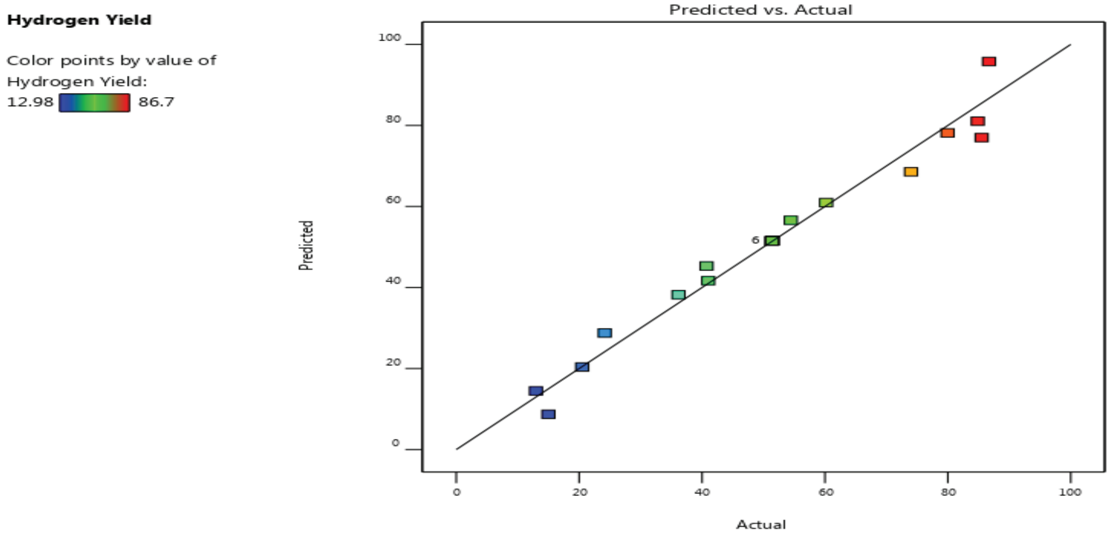

Figure 2: Predicted and experimental parity plot of the model for hydrogen yield

3.5 3D and 2-D Contour Plots of Independent Variables with Hydrogen Yield

The impacts of input parameters on the studied responses are shown using 3D surface plots [23]. The impacts of chosen input factors and their interactions on the hydrogen yield from ESR are presented as surface response and contour plots (see Figure 3a-c).

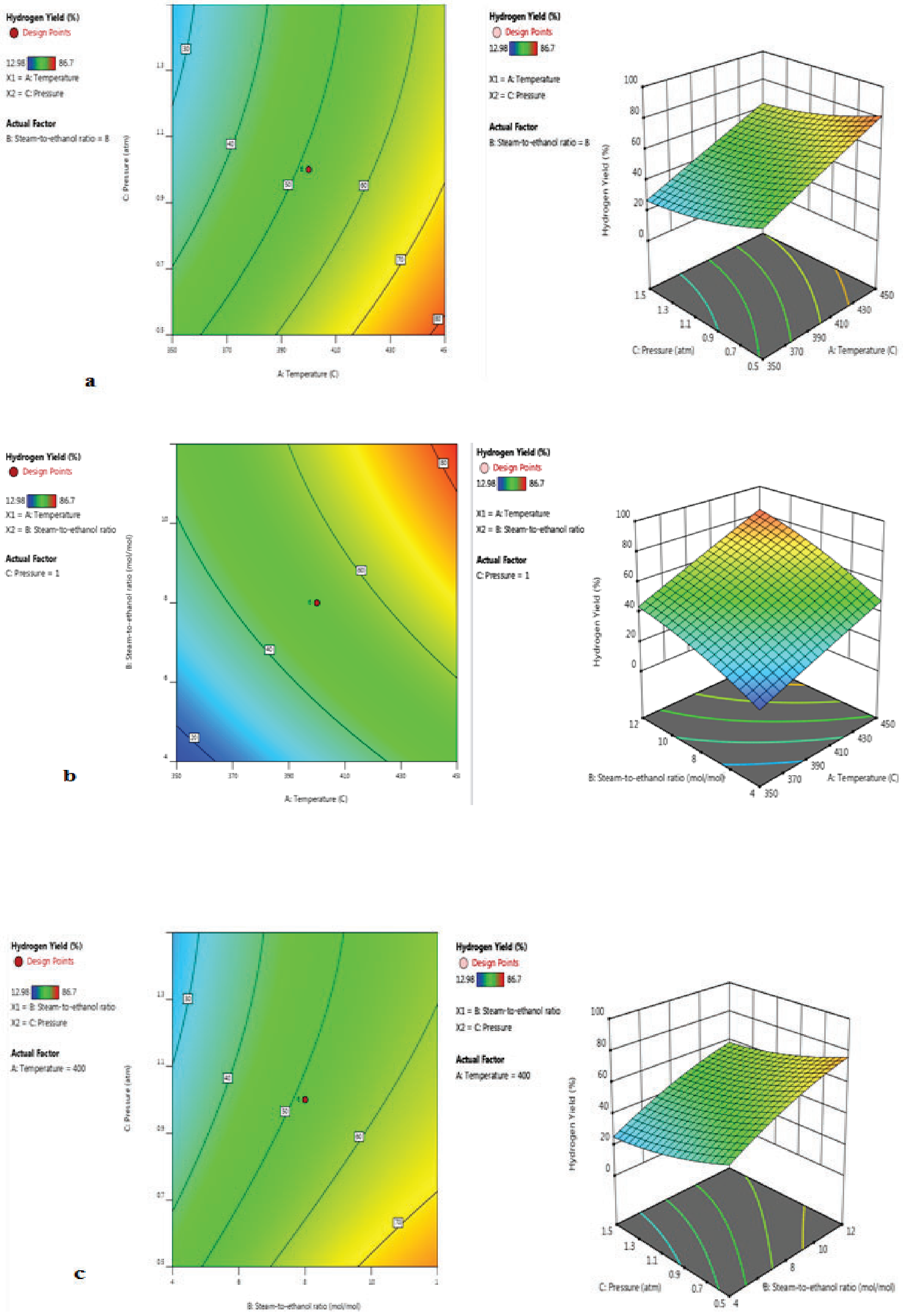

Figure 3: Representation of the 3D response surface and 2D contour plots for describing the interaction of different variables on hydrogen yield: (a) the interaction between pressure and temperature at steam-ethanol molar ratio of 8 mol/mol, (b) the interaction between steam-ethanol

molar ratio and temperature at pressure of 1 atm, and (c) the interaction between pressure and steam-ethanol molar ratio at temperature of 400 °C

As shown in the Figure 3a, hydrogen yield increases with increase in temeperature and slight increase in pressure at constant steam/ethanol molar ratio of 8 mol/mol. At pressure of 1atm and temperature of 400 °C, the highest hydrogen yield was obtained. In view of the endothermic nature of the ESR reaction, the hydrogen yield from ethanol conversion increases as temperature increases [29][30]. Figure 3b shows that the interaction of temperature and steam/ethanol molar ratio is significant on hydrogen yield. The highest hydrogen yield was obtained to be 51.51 %. at steam/ethanol molar ratio of 8mol/mol and temperature of 400 °C. Hydrogen yield from ESR reaction is enhanced when both factors increase [30][31]. As shown in Figure 3c, hydrogen yield increases with increase in steam/ethanol molar ratio and slight increase in presssure. The highest hydrogen yield obtained at steam/ethanol molar ratio of 8mol/mol and pressure of 1 atm was 51.51 %. It has been proven using thermodynamic equilibrium calculation to show that at higher pressures, the resultant products of ESR are low hydrogen yield and high methane production. Furthermore, pressutized operations culminate into catalyst inactiveness, as a result of carbon deposition. CO conversion via WGSR and prevention of catalyst deactivation by coke formation via ethylene decomposition can be achieved with the use of high steam/ethanol molar ratio[32][31][30]. The above results showed that hydrogen yield is affected more by the interaction of steam/ethanol ratio and temperature.

3.6 Optimization Solution from the Model of Hydrogen Yield

The optimum conditions for the attainment of high hydrogen yield were determined using the desirability function. Desirability values ranges from 0 to 1, and the recommended values adequate to explain variability are between 0.75 and 1[33]. The optimization solution from the model of hydrogen yield is as shown in Table 5. Based on the optimum operating conditions, ethanol conversion and hydrogen yield were predicted to be 100 % and 51.51 %, respectively. The ramp of the predicted optimization solution is shown in Figure 4.

Table 5: Optimized solution from the model of hydrogen yield

Independent Variables and Responses with Desirability					
Temperature (℃)	**Steam-to-Ethanol Ratio (mol/mol)**	**Pressure (atm)**	**Ethanol Conversion (%)**	**Hydrogen Yield (%)**	**Desirability**
400	8	1	100	51.5129	1.000

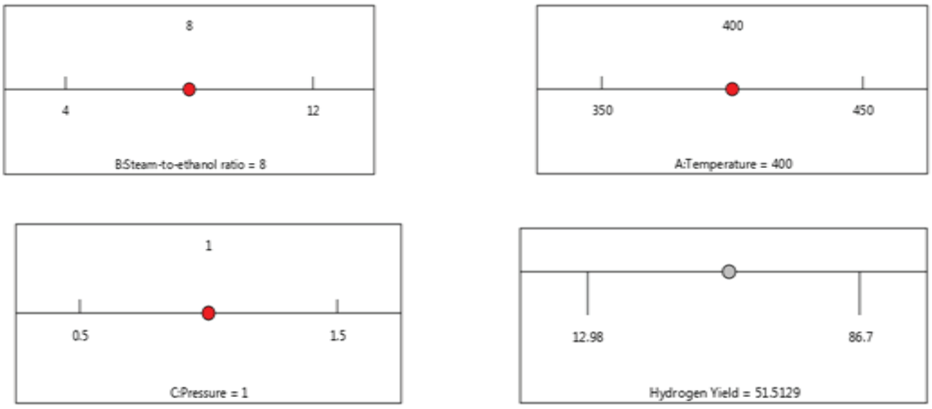

Figure 4: Numerical optimization of process variable for hydrogen yield at desirability of 1.0

3.7 Comparison of Optimum Condition Obtained from RSM between Predicted and Experimental Values of Hydrogen Yield

Hydrogen yield showed that the statistical model is valid since the +5 % allowable error based on 95 % confidence level is exceeded due to the effectiveness of the catalyst, and negligible difference between observed and predicted ethanol conversion values were obtained, as shown in Table 6.

Table 6: RSM results comparison between predicted and experimental values

Responses	**Observed Value (%)**	**Predicted Value (%)**	**Residual (%)**
Ethanol Conversion (%)	99.5	100	-0.5
Hydrogen yield (%)	62	51.51	+ 10.49

4.0 CONCLUSIONS

Optimization of input parameters for maximizing hydrogen yield from catalytic bioethanol steam reforming reaction (ESR) using central composite design-based response surface methodology has been carried out. A two-level, three-factor experimental design was performed to evaluate the combined effects of temperature (350-450 °C), steam/ethanol molar ratio (4-12 mol/mol) and pressure (0.5-1.5 atm) on hydrogen yield. Second order quadratic regression model was developed based on the limits of the examined variables to relate the synergy among them. Having adequate precision of 22.625 and correlation coefficient of (R^2) of 0.9701, it implies that the regression model has good predictive performance. The results of optimization showed that minimization of carbon monoxide and methane and maximization of hydrogen yield could be realized at steam-to-ethanol molar ratio of 8 mol/mol, temperature of 400°C and at atmospheric pressure. The hydrogen yield was predicted to be 51.5 % and validated value was 62 %, which showed that there is good agreement between the predicted and actual values and the model accurately predict the responses.

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COMPETING INTERESTS

The authors declare that they have no competing interests, financial or nonfinancial, that could be perceived as influencing the content or conclusions of this paper.

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