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Original Research Article

Optimization of Operating Conditions for CO Hydrogenation to Hydrocarbon *via* Response Surface Method

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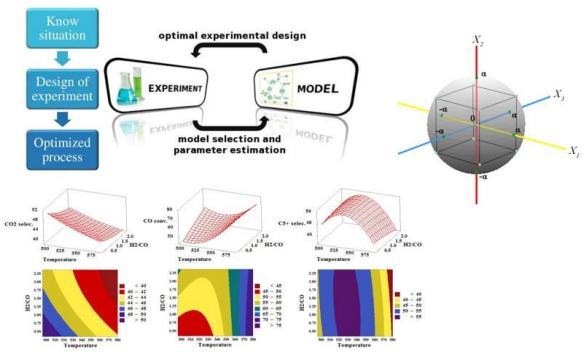
ABSTRACT

Clean hydrocarbon is an alternative source of other fuels like coal and natural gas. Based on the literature, the significance of hydrocarbon production via Fischer-Tropsch synthesis (FTS) process cause to develop a new mathematical algorithm response surface methodology (RSM)/ design of experiment (DOE). The influence of important factors, like pressure, temperature and feed ratio (H_2/CO) on the performance of the FTS are examined. The experiments are conducted in the range of: P = 1.9-3.75 bar, T = 523-563 K, and H_2/CO ratio = 0.85-1.85 at set space velocity (2000 h^{-1}). A second-order model is developed via RSM in terms of independent input variables to describe the CO conversion and selectivity of CO_2 and C_{5^+} as the responses. It is concluded that at low temperature and H_2/CO ratio, CO_2 selectivity increase significantly and C_{5^+} selectivity decreases appreciably when pressure increases. Moreover, at low pressure an increase in temperature, reduces CO conversion. According to contour plots and analysis of variance (ANOVA), it is illustrated that the maximum CO conversion was obtained at P = 3.21 bar, T =563 K and H_2/CO = 1.85 while for C_{5^+} the maximum is observed at P = 3.67 bar, T = 529.1 K, and H_2/CO = 0.91, and CO_2 selectivity is minimized at P = 1.93 bar, T = 563 K and H_2/CO = 1.85. The predicted conversion and selectivity are in good accordance with experimental results which is an indication of the accuracy of RSM methodology in designing and optimizing the FT process.

GRAPHICAL ABSTRACT



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Introduction

Owing to the decreasing fossil fuel resources and environmental challenges related to the increase of anthropogenic CO and CO_2 emissions, countless attention has been paid to investigate various methods of using these compounds as a feedstock for production of various chemicals and fuels [1-6]. FTS is a catalytic reaction process include a mixture of H_2 and CO converts into the hydrocarbon mixture compounds (CnH_{2n+2}, CnH_{2n}) [7-10]. The fundamental and practical significance of the FTS process draw considerable attention as an aromatic and sulfur free fuel. Therefore, many researches have been investigated this process from various perspectives include catalyst design, operating conditions and reactor configuration [11-13]. Moreover, numerous factors in FTS process are significant like pressure, temperature, feed ratio (H_2/CO), space velocity (GHSV), catalyst type and deactivation rate of catalyst [7, 14, 15].

The kinetic rate and operating conditions of the FTS reaction is significant for developing it to the industrial scale, being a prerequisite for the industrial process design, simulation and optimization [16]. As shown in Figure 1 CO rate consumption toward hydrocarbon production have been obtained in different pressures, molar feed ratio (H_2/CO) and temperatures. The low and high temperature FTS aims to produce heavy cuts like wax, diesel and lighter cuts like gasoline and diesel respectively as illustrated in Figure 1 (A). The reactors are considered based on the type of catalyst and the range of temperature in which these reactors run. Figure 1 (B) demonstrates that, In case of temperature rises toward producing the low boiling products, fluidized-bed reactor is the suitable reactor, and toward achieving high boiling products at reduced temperatures, the slurry phase and fixed-bed reactors are among the appropriate alternatives. Moreover, operating conditions and rate of reaction investigation can be reliable, in case of being enough the employed experimental data based on literature. As known, getting the abundant experiment reactor data is a costly and time-consuming procedure. Therefore, a mathematical model is best tools for estimating the experimental data that can be implemented in industrially scale [17-19].

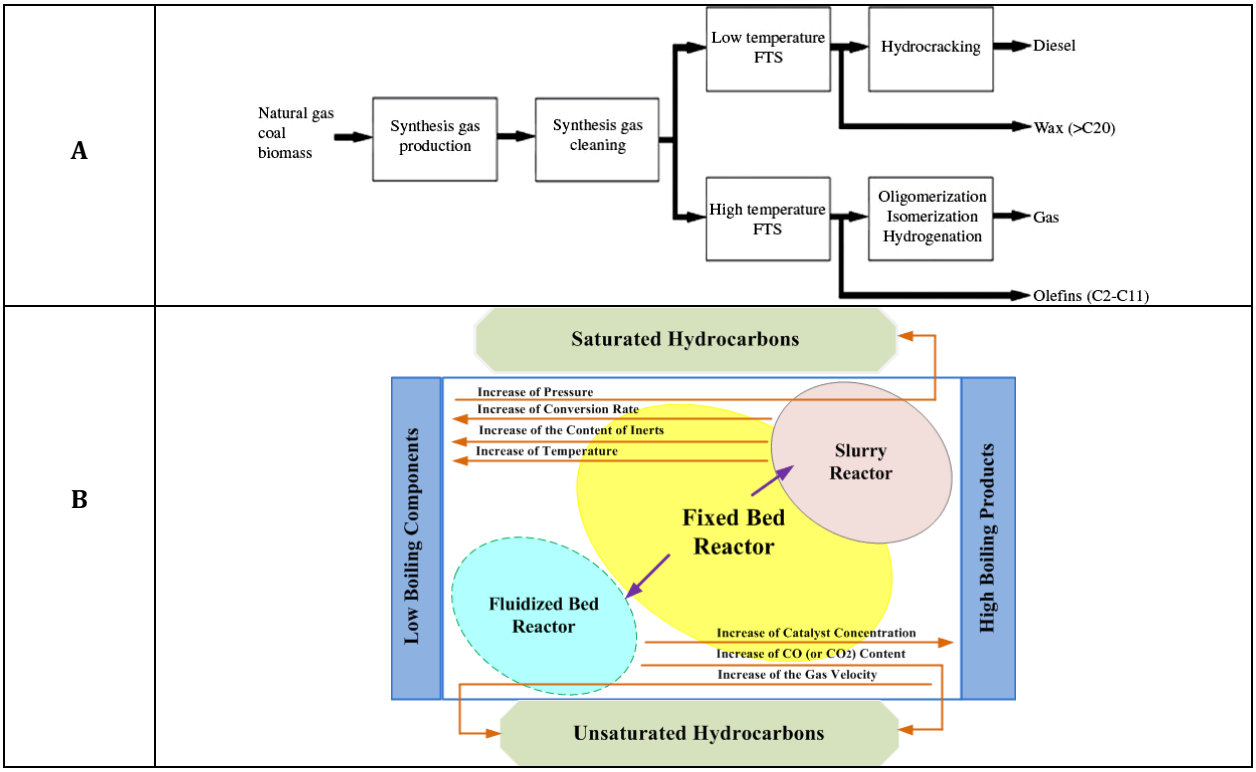

Figure 1: A) Overall process for the hydrocarbon production via the FTS, B) Impact of the process parameters on the product distribution in various types of reactors [20]

DOE is extensively used for various purposes such as optimization, design, and development of novel catalyst (include binder, promoter, base and active site), reactor configuration and operating conditions [21, 22]. DOE has several benefits such as decreasing cost and time. Generally, kinetic rate equations of FTS in term of nonlinear behavior and modeling based on statistical techniques have been studied in some researches.

The application of ANN in kinetic study and prediction of FTS products was investigated by Shiva et al. [23] and Sharma et al. [24], but application to kinetic study by RSM/ANN and optimization with hybrid of ANN/GA have not been addressed. However, the combination of RSM/ANN with ANN/GA may help us to evaluate the efficiency and optimize the conditions of reaction. This procedure could help us do better data fitting of kinetic rate equations and ultimately result in a successful optimization studies. The modeling of FTS reactor on various factor such as temperature, pressure and feed ration have been discussed.

As illustrated in Figure 2, Mansouri et al employed the Box-Behnken design (BBD) consisting of 15 experiments for developing model for reaction rate of FTS. The parameters in current research were changed such as the temperature, H_2 and CO partial pressure [25]. However, interaction of operating conditions and rate of feed ratio consumption via RSM based on the central composite design (CCD) and experimental design planning to create model for CO hydrogenation to hydrocarbon have not been reported. For the strategy of experimental design, several techniques illustrated in Figure 2 have been effectively exploited to the manufacturing: Central composite design (CCD), Fractional or full factorial design (FFD), Latin hypercube design (LHD) and Box-Behnken design (BBD). Among them, FFD analyzes all the factors at all p levels, and it is proficient of assessing interaction effects clearly by determining all feasible variables patterns. However, FFD enhances computational intensive in case of resolving second or higher order polynomial model. Hence, the FFD, including the famous orthogonal array assuming parameters independence is employed to the DOE. Likewise, CCD has as much data as the three-level FFD with fewer experiments. However, for the two-level experiment, CCD contains FFD, axial points and one center point, which would meaningfully improve the experiments data when large numbers of analyzed parameters are involved. In case of lack of subsequent experiments, BBD needs fewer designed points than CCD and is more appropriate for factors with non-linear elements [26].

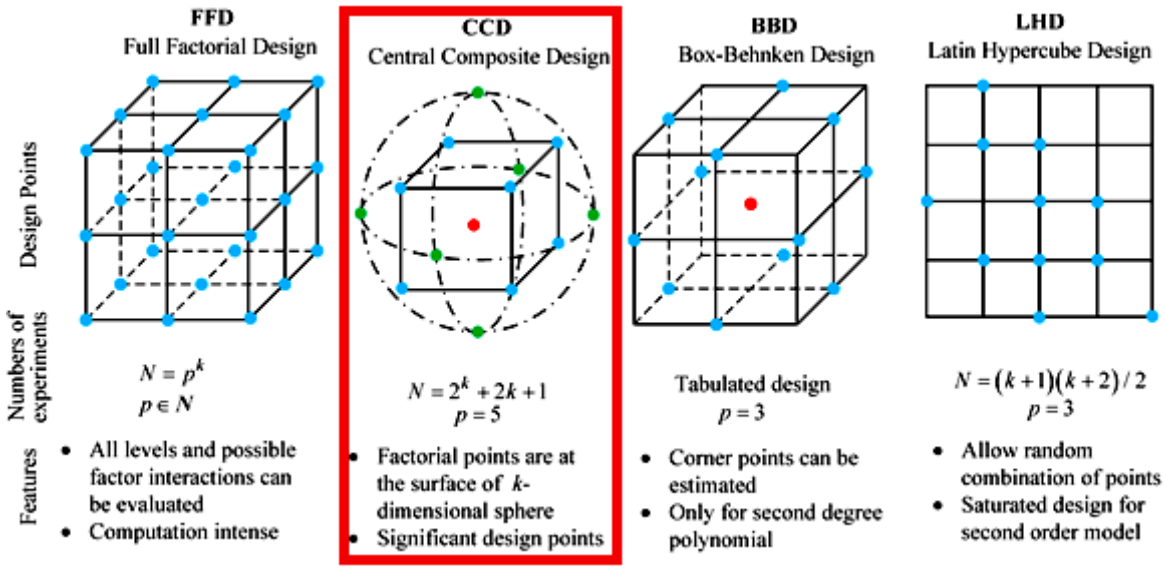

Figure 2: Schematic of all experimental strategies for RSM with special emphasis on scope of this research [26]

RSM is an impressive technique in current research in comparison with all other statistical methods. Due to, RSM is based on a set of statistical methods for DOE, developing the models, assessing the influences of parameters and try to find the optimum terms. Therefore, it can be concluded that RSM target is implementing the systematic modeling, optimizing and demonstrating the performances via statistical/ regression/graphical technique that lead to more understandable of the complex procedures. Recently, RSM has been employed to evaluate and optimize interactive effects of independent parameters in numerous biochemical and chemical processes such as, heterogeneous biodiesel production from waste cooking palm oil [22], Optimization of synthesis conditions of carbon nanotubes via ultrasonic-assisted floating catalyst deposition [21], steam reforming of methanol [27], Orthogonal Turning Process [28], water nanofluids pool boiling heat transfer coefficient at low heat fluxes [29]. Moreover, RSM and statistical methodologies have been exploited widely in several scientific fields such as reactor and catalyst design and also estimation of process operating conditions [30], a few researches for CO hydrogenation can be found based on statistical methodologies. Though, interaction of operating conditions and rate of feed ratio consumption using RSM based on the CCD and experimental design planning to develop model for FTS have not been reported.

In FTS, product quality can be affected by several factors like pressure, space velocity, temperature, feed ratio (H_2/CO), properties of catalyst, time-on-stream, selectivity, and reduction of catalyst. Considering these various factors' influence on FTS product distribution, optimization of these parameters to achieve the maximum process efficiency is a challenging task. RSM is a technique in order to develop, enhance and optimize empirical model building [22, 31]. The objective of DOE is to enhance a response that is affected through various independent input datas [32]. Modeling methods can be developed by several techniques such as, RSM, Taguchi or artificial neural network (ANN) [33], principal component analysis (PCA) [34].

Exploiting statistical techniques for FTS has been studied in many articles [35-37]. Generally, these studies focused on two or three independent variables [38-41]. However, in this research RSM has been employed in order to understand the interaction of effective parameters, like temperature, feed ratio (H_2/CO) and pressure on the performance of the FTS over Iron catalyst in packed-bed reactor. At first experimental section based on literature then experiment design, finally results and discussion include contour plots and analysis of variance (ANOVA).

Material and methods

Experimental section based on literature

According to the literature, sol-gel method is the efficient catalyst manufacturing method for synthesis 40%Fe/60%Ni/40 wt.% Al_2O_3 catalyst. The micro packed-bed reactor was constructed from stainless steel. The detailed explanation of the experimental set-up can be found in literature [42-45].

In a typical run, 1 gr of catalyst powder with Dp < 150 μm , to lower the internal mass diffusion resistance, was mixed with similar quartz wool (150 < Dpq < 250 μm) in order to obtain a uniform temperature in the packed-bed. Furthermore, the pattern of feed was assumed to be plug-flow, and the empirical reaction rate was defined according to Eq. (1):

$$\frac{W_{cat}}{F_{CO}} = \frac{X_{CO}}{-r_{CO}} \rightarrow -r_{CO} = \frac{X_{CO} F_{CO}}{W_{cat}} \qquad (1)$$

where, r_{CO} is rate of CO consumption, F_{CO} and W_{cat} are input CO flow rate and catalyst weight, respectively [46, 47].

Response surface methodology (RSM) and Design of Experiment (DOE)

As illustrated in Figure 3 merge of these algorithms can anticipate the interaction /dependency between the values of some

measurable outputs and those of a set experiments datas affect the outputs. Moreover, response value at different process conditions can be predicted. Finally the values of factors which create the optimum and best values of the response cab be found [48] .

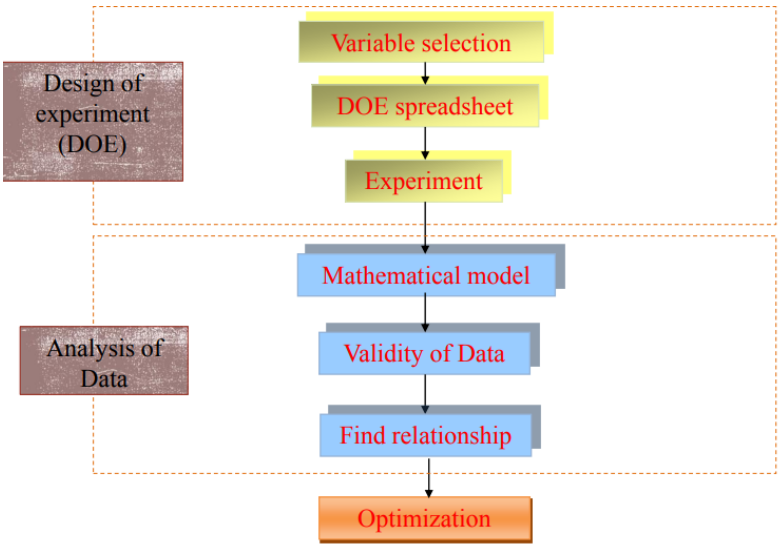

Figure 3: A new mathematical algorithm RSM/ DOE [48]

RSM is a mathematical/statistical method for optimization and modeling processes. The RSM can be employed in case of when several input variables affect quality or quantity characteristic of the response [49, 50]. In other word; the significant steps of RSM include: I) experiment design selection, II) coefficient estimation according to the mathematical model and response prediction, and III) model adequacy confirmation via variance analysis (ANOVA).

In fact, RSM is used as a principle tool for the enhancement of existing hydrocarbon designs, along with the design, formulation and development of new hydrocarbon [34].

In the RSM, coefficient estimation according to the mathematical model and response prediction, between inputs and output can be derived as follows Eq. (2):

$$y = f(x_1, x_2, x_3, \dots, x_n) \pm \varepsilon \qquad (1)$$

where y is the response, f is the function of response, x_1, x_2, x_3, …, x_n are the inputs and ε is the fitting error. A second order polyminal regression model can utilized to fit the data. The model of current study can be presented as follows Eq. (3):

$$f = a_0 + \sum_{i=1}^{n} a_i + \sum_{i=1}^{n} a_{ii} x_i^2 + \sum_{i<j}^{n} a_{ij} x_i x_j \pm \varepsilon \qquad (2)$$

where α_i and α_{ii} denote linear and quadratic effects of x_i and α_{ij} indicate the interaction between x_i and x_j, respectively. The RSM is a step-by-step procedure that contains the following seven steps:

1. Definition of input factors and the desired responses.
2. Central composite design (CCD) and experimental design planning.
3. Accomplishment of regression analysis for the polynomial model.
4. ANOVA calculating and finding the affected parameters.
5. Proposing a second-order polynomial model for response.
6. Optimization of the design parameters considering design constraints.
7. Validation and performing the experimental and design variables.

The ANOVA for regression importance is shown in Table 1.

Table 1: ANOVA based on the regression importance

Variation source	Sum of squares	Freedom degree	Mean square
Regression	$SS_R = \sum(\hat{Y}_i - \bar{Y}_i)^2$	$p-1$	$MS_R = SS_R/p-1$
Error	$SS_R = \sum(Y_i - \hat{Y}_i)^2$	$n-p$	$MS_E = SS_E/n-p$

Where n is the experiments run number and p shows the model parameters number.

The regression accuracy is determined through the Eq. (4) as follows:

$$R^2 = \frac{SS_R}{SS_R + SS_E} \quad (3)$$

The modification of R^2 is the adjusted R^2 that is adjusted for explanatory terms of model.

Adjusted R^2 is presented in Eq. (5) as follows:

$$R^2_{adj} = 1 - \frac{(1-R^2)(n-1)}{n-p} \quad (4)$$

The P-values and T-test were utilized to investigate the coefficients importance obtained via regression.

Result and Dissection

In current research, the influence of independent factors such as: pressure (x_1), temperature (x_2) and feed ratio H_2/CO (x_3) at set space velocity (GHSV=2000 h^{-1}) on four responses; %CO conversion and the selectivity of CO_2 and C_5^+ have been investigated. The prediction capability of experimental data based on literature compared with RSM responses is shown in Table 2.

Table 2: Operating conditions and experiment data as input data, and modeling output

Operation conditions			Experimental data based on literature			Predicted data by RSM		
P (x_1)	T(x_2)	H_2/CO (x_3)	%CO conv.	C_5^+ selec.	CO_2 selec.	%CO conv.	C_5^+ selec.	CO_2 selec.
3.25	533	0.70	41.70	51.42	48.12	40.13	49.25	49.67
2.35	553	0.75	61.92	53.31	41.73	63.11	52.20	42.85
3.35	563	0.75	52.37	47.31	45.42	51.11	49.33	47.02
2.95	563	0.85	55.40	44.86	43.36	53.23	42.76	44.70
2.35	563	0.85	69.96	47.28	41.03	68.20	49.11	42.67
2.35	533	0.85	54.40	53.41	43.73	55.23	51.55	42.56
1.95	543	0.85	77.03	56.02	42.57	78.78	54.76	43.66
3.75	543	0.85	46.16	59.16	47.43	48.12	60.26	46.20
1.95	563	0.85	84.39	47.10	40.76	82.78	45.78	38.90
1.95	553	0.85	81.66	53.08	41.22	78.94	51.16	42.76
1.90	523	0.85	74.04	52.21	45.12	75.31	51.33	44.18
1.95	523	1.24	69.22	50.77	44.39	72.24	52.07	45.54
2.25	523	1.38	56.49	50.94	44.61	54.38	52.30	43.23
2.70	523	1.70	51.75	52.66	44.63	52.94	50.13	43.20
3.45	533	0.85	43.32	56.52	48.21	44.56	58.12	47.12
1.95	523	0.85	72.04	52.25	45.16	70.07	54.37	46.41
2.85	543	1.10	51.40	54.05	43.56	52.75	55.93	42.18
2.85	523	1.10	49.82	50.75	46.51	48.46	49.05	45.22
2.70	563	1.10	56.63	46.27	41.20	57.71	44.43	42.44
3.25	543	1.24	51.40	58.77	44.85	52.76	57.13	43.50
2.95	533	1.24	51.27	51.95	44.40	52.32	53.28	45.34
3.10	553	1.24	51.87	54.60	42.65	50.23	53.20	44.21
1.95	533	1.40	68.47	51.11	42.36	69.75	53.12	43.38
2.75	543	1.45	52.24	55.39	42.14	51.11	54.43	41.10
1.95	553	1.45	77.82	51.73	40.08	76.05	53.44	41.12
1.95	563	1.45	80.51	45.96	39.96	82.29	43.23	38.04
2.35	533	1.65	58.47	51.01	42.49	59.13	52.55	43.70
1.95	543	1.70	72.49	53.27	40.98	69.31	54.77	42.13
2.75	553	1.80	54.94	56.30	40.60	53.58	55.20	38.11
1.95	553	1.85	78.54	49.62	39.70	77.13	48.32	41.25

To examine the goodness of model fit, the normal probability plot of residuals was employed. In the case of residuals with normal distribution, commonly all points of the plot form are fit over a straight line. Figure 4 illustrates that three responses residual plots are distributed normally.

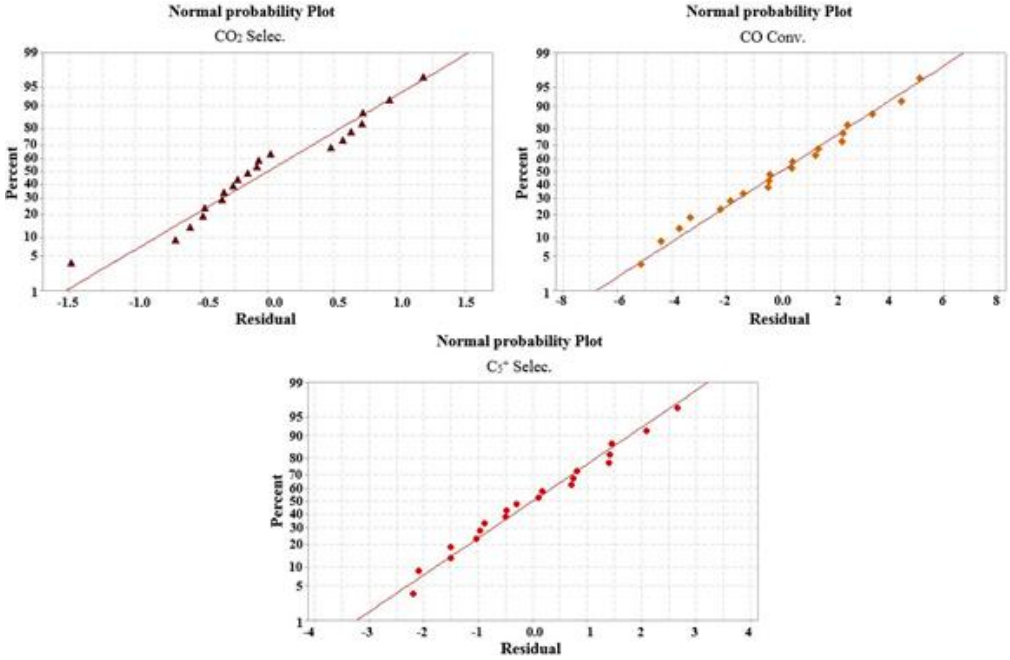

Figure 4: Normal probability plots of three responses

For the evaluation of the model parameters, ANOVA was employed and some of statistical terms such as R^2, R^2_{adj} for all responses were derived, that depicted in Table 3. The accuracy of the model is examined via lack-of-fit tests. When data has replication, the pure error lack of fit test is automatically performed by RSM. The null hypothesis would be met if P-value is the probability of achieving a consequence at least as extreme as the one in the sample data assuming. In case of the P-value to be less than 0.05, the parameters effect on the output response would be impressive. Moreover, coefficient of SE would be defined based on the estimated standard deviation of the coefficient estimate [42] (see Table 3).

Table 3: Regression coefficient and ANOVA of the RSM modeling for all responses

	%CO conversion			CO_2 selectivity			C_5^+ selectivity		
Variables	SE coeff.	T-value	P-value	SE coeff.	T-value	P-value	SE coeff.	T-value	P-value
Constant	1.63	33.27	0.000	0.368	116.35	0.000	0.777	71.96	0.000
P (x_1)	1.08	-11.66	0.000	0.244	6.39	0.000	0.515	2.49	0.032
T(x_2)	1.08	5.93	0.000	0.244	-8.92	**0.000**	0.515	-6.37	0.000
H_2/CO(x_3)	1.08	1.68	0.125	0.244	-5.43	0.000	0.515	-0.48	0.638
x_1*x_1	1.05	5.13	0.000	0.238	2.80	0.015	0.502	-1.21	0.255
x_2*x_2	1.05	3.45	0.006	0.238	1.02	0.016	0.502	-8.78	0.000
x_3*x_3	1.05	-0.11	0.917	0.238	1.15	-	0.502	-040	0.700
x_1*x_2	1.41	-1.55	0.153	0.319	-0.16	-	0.673	0.91	0.384
x_1*x_3	1.41	1.97	0.078	0.319	-2.60	-	0.673	2.36	0.040
x_2*x_3	1.41	-1.24	0.242	0.319	0.68	-	0.673	-0.80	0.443
Lack-of-fit			0.570			0.807			0.518
	R^2	98.92%		R^2	97.33%		R^2	95.93%	
	R^2(adj)	95.96%		R^2(adj)	95.22%		R^2(adj)	92.57%	

All plots (surface and contour) of yields (% CO conv., CO_2 selec. C_5^+ selec.) in comparison to temperature, pressure are shown in Figure 5.

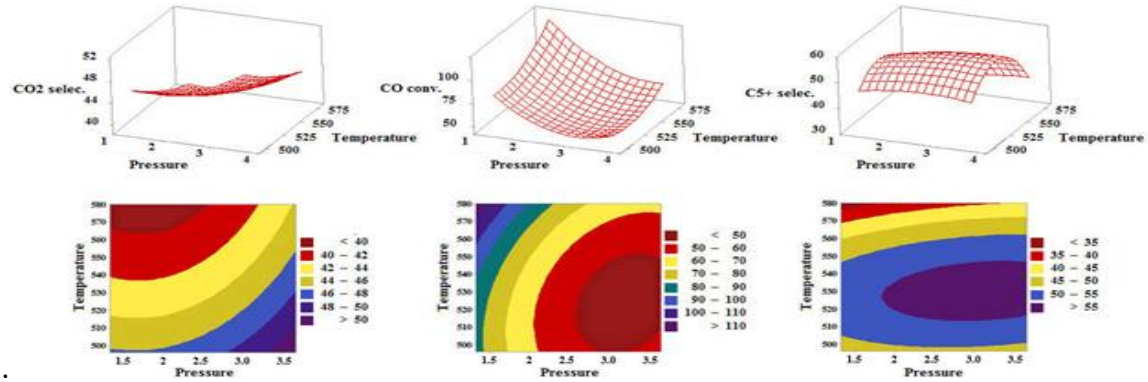

Figure 5: Surface and contour plots of yields vs temperature, pressure (H2/CO=1.35 hold value)

All plots (surface and contour) of yields (%CO conv., CO_2 selec., C_5^+ selec.) in comparison to H_2/CO, pressure are shown in Figure 6.

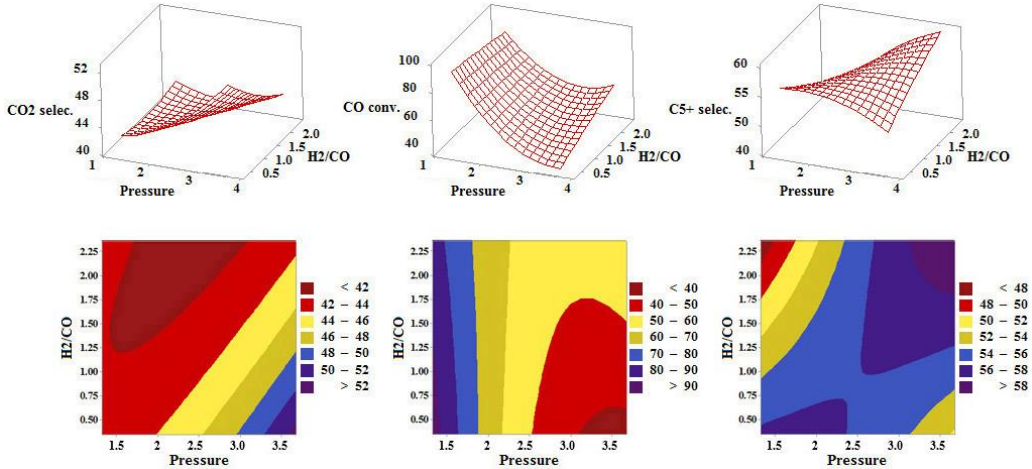

Figure 6: Surface and contour plots of yields vs H_2/CO, pressure (T=538 hold value)

All plots (surface and contour) of yields (%CO conv., CO_2 selec., C_5^+ selec.) in comparison to H_2/CO, temperature are illustrated in Figure 7.

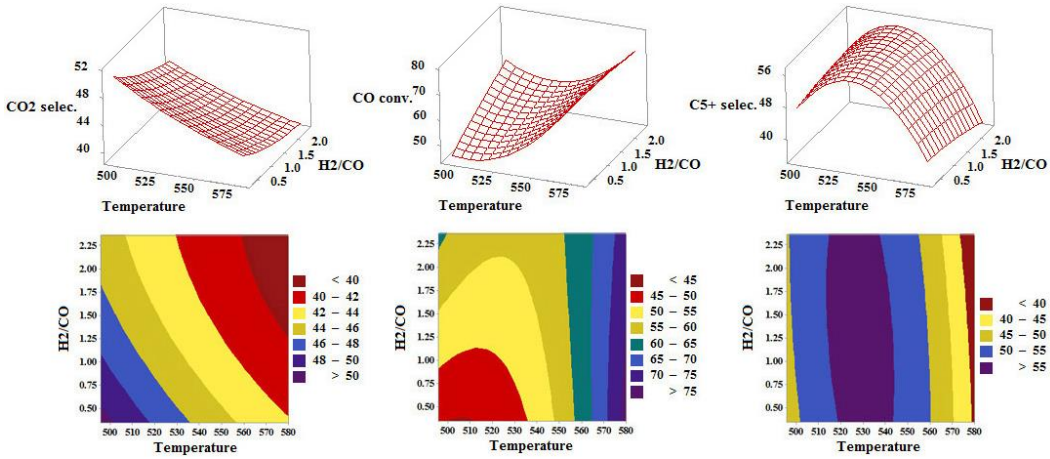

Figure 7: Surface and contour plots of yields vs H_2/CO, temperature (P=1.5 bar hold value)

Based on ANOVA in Table 3 the full quadratic equations interaction the responses with the independent factors, such as: pressure (x_1), temperature (x_2) and feed ratio (H_2/CO) (x_3) are depicted as follows:

$$\%CO\ conversion = 7.1x_1 - 5.63x_2 + 56.8x_3 + 11x_1^2 + 0.0058x_2^2 - 0.31x_3^2 - 0.1244x_1x_2 + 6.60x_1x_3 - 0.117x_2x_3 + 1468 \quad (6)$$

$$CO_2\ selectivity = 2.35x_1 - 0.519x_2 - 9.1x_3 + 1.360x_1^2 + 0.00038x_2^2 + 0.756x_3^2 - 0.0029x_1x_2 - 1.976x_1x_3 + 0.0145x_2x_3 + 210 \quad (7)$$

$$C_5^+\ selectivity = -18.4x_1 + 7.45x_2 + 14.7x_3 - 1.24x_1^2 + 0.007x_2^2 - 0.55x_3^2 + 0.035x_1x_2 + 3.78x_1x_3 - 0.0358x_2x_3 - 1910 \quad (8)$$

In the optimization part, the target was set to maximum CO conversion which was found at P= 3.21 bar, T=563 K and H_2/CO=1.85; also it was achieved that the minimum CO_2 selectivity could be obtained in P=1.93 bar, T=563 K and H_2/CO=1.85. The maximum of C_5^+ selectivity was achieved at P = 3.67 bar, T = 529.1 K, and H_2/CO = 0.91. Figure 8 shows the optimization plots of all quadratic equations.

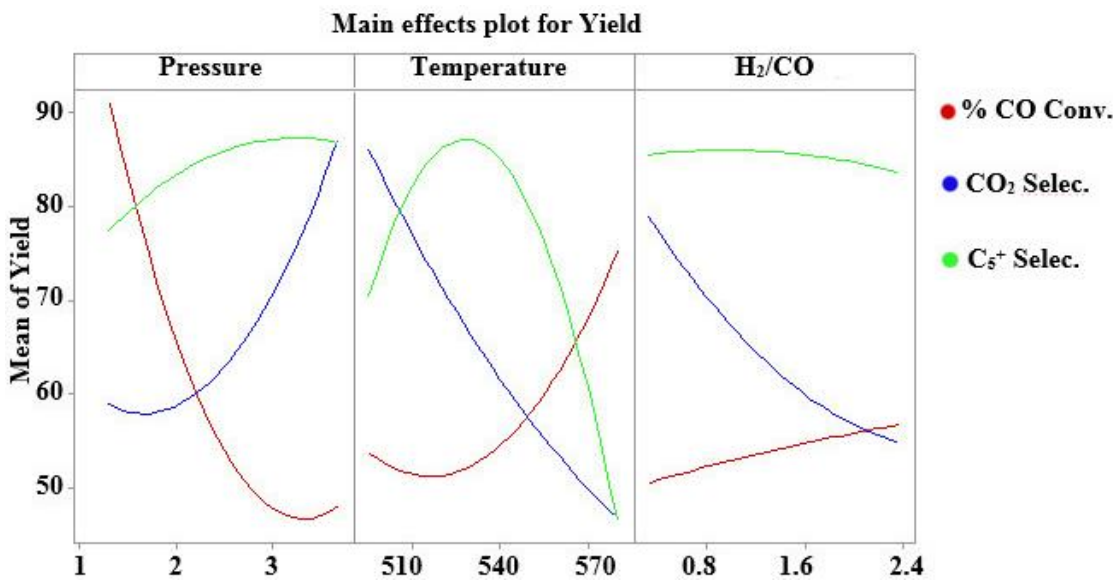

Figure 8: All quadratic models optimization

To evaluate the capability and performance of the RSM model, Figure 9 shows the comparative error plot for the RSM model with the experimental data for two choices responses (%CO conversion and CO_2 selectivity). It was concluded that RSM model has logical competence and worthy efficiency to anticipate, the experimental data based on literature.

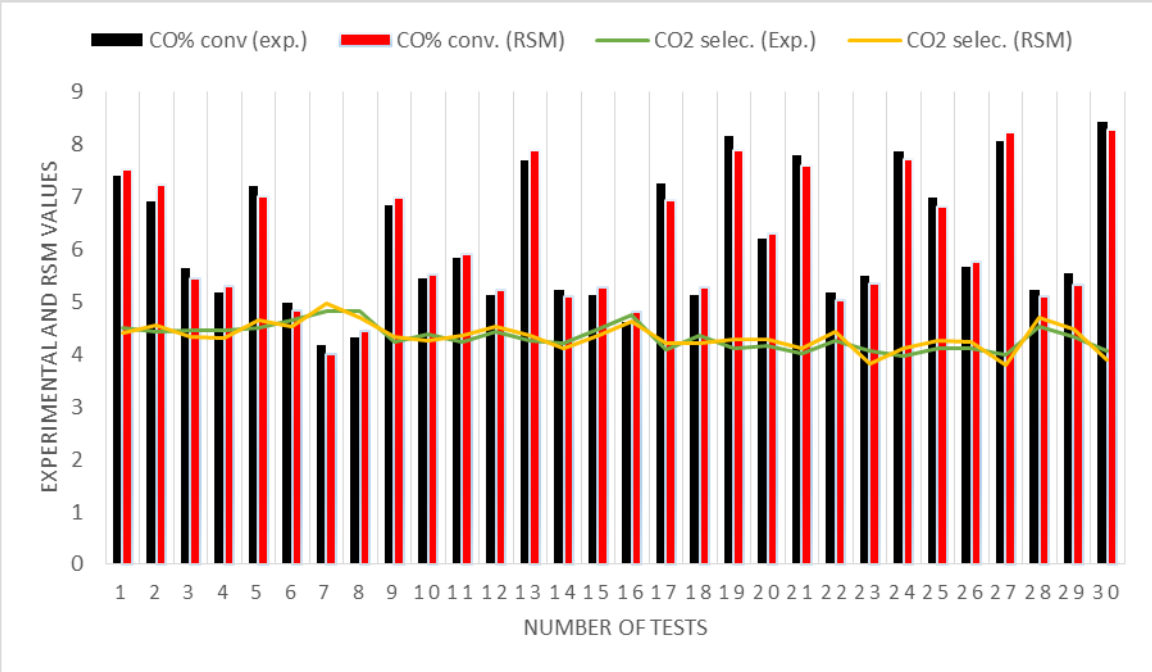

Figure 9: Comparing the experimental data based on literature and predicted RSM values for two responses

Conclusion

The influences of independent parameters (temperature, pressure and feed ratio (H_2/CO)) on CO conversion and the selectivity of CO_2 and C_{5^+} were studied in the presence of bi-functional Fe/Ni catalyst supported on alumina in a packed-bed reactor using DOE/RSM. Generally, it was observed that, by increase in pressure, CO conversion and C_{5+} selectivity were decreased. The selectivity of CO_2 increased when pressure was decreased. Also, increase in temperature caused a rise in CO conversion.

Results revealed that, the CO_2 selectivity grew significantly at low temperature and H_2/CO ratio, when temperature and H_2/CO ratio decreased. Increase in pressure caused an increase in CO_2 selectivity appreciably. As observed, C_{5^+} selectivity fell appreciably when pressure rose. At low temperature, C_{5^+} selectivity was increased, but at high temperature, this behavior turned upside down. Furthermore, C_{5^+} selectivity grew when H_2/CO ratio increased.

RSM is a proficient technique for demonstration of effective input factors and responses. In this study, the upper limit of conversion of CO (%) was obtained at P = 3.21 bar, T =563 K and H_2/CO = 1.85; for C_{5^+} selectivity was at P = 3.67 bar, T = 529.1 K, and H_2/CO = 0.91, and the minimum selectivity of CO_2 was P = 1.93 bar, T = 563 K and H_2/CO = 1.85. It was concluded that RSM models were proficient tools for demonstrating the behavior of the effective parameters on responses where they are complex functions of processing parameters.

Nomenclature

a_{ii}	quadratic effect of x_i
X_{CO}	CO conversion percentage
$-r_{CO}$	CO consumption rate $(^{mol}/_{(kgcat.s)})$
f	function of response
$x_1, x_2, x_3, \ldots, x_n$	RSM inputs variables
ε	fitting error
n	number of experiments
a_{ij}	interaction between x_i and x_j
F_{CO}	input flow rate of CO $(^{mol}/_{s})$
SS_E	Error sum of squares
SS_R	residual sum of squares
p	the number of model parameters
W_{cat}	the weight of catalyst (kg)

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Conflict of Interest
We have no conflicts of interest to disclose.

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