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Numerical Evaluation and Analysis for Hydrogen Production Via Ethanol Steam Reforming

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Abstract

In the present study, two series of Ni/Ce/ZrO₂ catalysts were prepared. The first one is n% Ni/Ce_{0.74}Zr_{0.26}O₂ (n = 0, 2, 10 and 20 wt %). The second is 10%Ni / m (Ce/ZrO₂) (m = 0, 4, 6 and 8). Catalysts have been investigated for ethanol steam reforming (ESR) to produce hydrogen. The reaction was studied in an atmospheric flow system, the temperature range was 200-600 °C and water/ethanol (6, 8, 10 molar ratio). The effect of using H₂O₂ as an oxidant in auto-thermal reforming of ethanol has been also investigated (at 400 °C, and H₂O₂/ethanol ratio = 8) to get highest hydrogen selectivity with lower CO ratio. Numerical evaluation and analysis have been performed for the above obtained results. It has been observed that the ethanol conversion, hydrogen production and some of the various investigated relations are functions of more than one independent variable. So, the response surface methodology (RSM) has been employed to evaluate these relations.

Key Words: Numerical analysis, Response surface methodology, Ethanol steam reforming, Ni/Ce/ZrO₂ catalysts.

1. Introduction

In the future H_2 can become a major source of energy thus offering a potentially non-polluting, inexhaustible, and efficient and a cost attractive energy carrier. Hydrogen is mainly used as raw material for the chemical and refining industries. Moreover, in the near future, hydrogen will play an important role in the energy sector. In combination with fuel cells (Zhu *et al.* 2013) H_2 has been proposed as a major energy source which could contribute to the reduction of atmospheric pollution and greenhouse gases emissions, and reduction of global dependency on fossil fuels. So, the development of alternative methods for hydrogen production, especially from renewable sources, is attracted much attention (de Bruijn 2005; Armor 2005).

Among the several families of oxygenated fuels, alcohols are the better candidates. Methanol has attracted great interest because of its potential application in fuel cells (Wasmusa & Küver 1999; Murray *et al.* 1999); however, there are both health and environmental issues surrounding its use. Compared with methanol, ethanol is more attractive because it is less toxic, has high hydrogen content, has less environmental impact and is a renewable energy with several biomass sources (Cortright *et al.* 1999; Liguras *et al.* 2003). Reforming reactions for the production of hydrogen are the steam reforming of ethanol (SRE) (Josh *et al.* 2012), partial oxidation of ethanol (POE), oxidative steam reforming of ethanol (OSRE) and the ethanol decomposition (DE) reactions. In producing hydrogen from ethanol by steam reforming, the main catalytic reaction is shown in Eq. (1). Along with the hydrogen produced, non-renewable CO_2 is the other product.

$$C_2H_5OH + 3H_2O \rightarrow 6H_2 + 2CO_2 \tag{1}$$

$$CH_3CH_2OH \rightarrow CO + H_2 + CH_4$$
 (2)

In previous studies, several catalysts have been proposed to be further considered for practical applications in ethanol steam reforming. Nobel metal-based catalysts frequently exhibit better activity when compared to non-noble metal catalysts; however, these catalysts are very expensive (Fajardo *et al.* 2010). On the other hand, Ni-based catalysts have shown high activity and selectivity, moreover, they are economic (Haryanto *et al.* 2005; Vaidya *et al.* 2006; Leung *et al.* 2007).

Supports also play important roles in the SR of ethanol, as supports help in the dispersion of the metal catalyst and may enhance the metal catalyst activity via metal–support interactions. Supports may promote migration of OH groups toward the metal catalyst in the presence of water at high temperature, facilitating SR. In Ni-based catalysts, Ceria (CeO₂) is an interesting oxide with unique properties namely its ability to shift easily between reduced and oxidized states (Ce³⁺/Ce⁴⁺) and to accommodate variable levels of bulk and surface oxygen vacancies. These characteristics make it suitable for use as a support as well as catalyst in processes wherein reaction conditions fluctuate between oxidizing and reducing environments (Ebiad *et al.* 2012). Despite its widespread applications, the use of pure cerium dioxide is highly discouraged because it is poorly thermally stable as it undergoes sintering at high temperatures thereby losing its crucial oxygen storage and release characteristics (Zhu *et al.* 2013; de Bruijn 2005).

In order to increase its thermal stability and ability to store and release oxygen during operations, other transition and non-transition metal ions could be introduced into the ceria cubic structure. According to the literature, introduction of zirconium into the ceria lattice greatly enhances the surface area, thermal stability and oxygen storage capacity, resulting in superior catalytic properties. Hence, ceria–zirconia solid solutions have been investigated with huge interest among other ceria-based mixed oxides (Zhang *et al.* 2014; Monte & Kaspar 2005). Roh *et al.* (2012) confirmed that Ni/Ce0.8Zr0.2O₂ shows a higher BET surface area than Ni/CeO₂ and better thermal resistance than Ni/ZrO₂ during the reduction process at 600 °C.

Srinivas *et al.* (2003) studied the influence of Ce/Zr ratio on the redox behavior of Ni in a series of NiO-CeO₂-ZrO₂ catalyst; they reported that the catalysts with Ce/Zr ratio of 1 are superior to the other compositions. The thermodynamic analysis of the water ethanol reaction shows that at high water concentration, SR of ethanol is favored. An increase in the amount of water also has the effect of increasing the extent of the WGS and methane SR reactions, which reduces the amount of the undesired products, CO and CH₄ (Fishtik *et al.* 2000).

In the hydrogen production via ethanol steam reforming it has been observed that the ethanol conversion, hydrogen production and some of the various investigated relations are functions of more than one independent variable. Therefore the Response Surface Methodology (RSM) has been employed to evaluate these relations.

2. Experimental

The Ce-ZrO₂ mixed oxide supports were prepared by co-precipitation with ammonia using an aqueous solution of cerium nitrate (Ce(NO₃)₂. $6H_2O$, 99% Fluka) and zirconium oxychloride (ZrOCl₂. $8H_2O$, 98% Aldrish). Details of the experimental section have been given elsewhere (Ebiad *et al.* 2012; Elsalamony *et al.* 2014).

3. Response surface methodology

RSM is a collection of mathematical and statistical techniques that are useful for the modeling and analysis of problems in which a response of interest is influenced by several variables and the objective is to optimize this response. Experimentation is made to determine the effect of the independent variables (factors) on the dependent variable say response of a process and a relation between them is usually illustrated through a regression model by using experimental data and optimization methods (Montgomery & Douglas 2005; Gendy et al. 2013; Deriase et al. 2012).

Because the form of the true response function f is unknown, we have to approximate it. Usually a low-order polynomial in some relatively small region of the independent variables space is appropriate. In many cases, either a first-order or a second order model is used.

For the case of two independent variables, the following second-order model would likely be useful as an approximation to the true response surface in a relatively small region.

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{12} X_1 X_2$$
(1)

This equation has been applied to investigate the effect of Nickel % loading (X_1) and temperature (X_2) over CeO₂-ZrO₂ catalysts on the production of hydrogen employing the data presented in (Ebiad *et al.* 2012). The equation takes the form:

$$Y = \beta_0 + \beta_1 * Ni + \beta_2 * T + \beta_{11} * (Ni)^2 + \beta_{22} * T^2 + \beta_{12} * Ni * T$$
(2)

Also it has been applied to assess the effect of varying the Ce/Zr (X_1) and water/ethanol (X_2) ratios on minimizing the bi- products (CO and carbon deposited) by auto thermal steam reforming of ethanol employing the data presented in (Elsalamony *et al.* 2013). The equation takes the form:

$$Y = \beta_0 + \beta_1 * (Ce/Zr) + \beta_2 * (W/E) + \beta_{11} * (Ce/Zr)^2 + \beta_{22} * (W/E)^2$$

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(3)

$$+\beta_{12} * (Ce/Zr) * (W/E)$$

Moreover the use of hydrogen peroxide as an alternative commercial source of oxygen for the production of hydrogen has been compared to the use of steam over the various forms of Ce/Zr catalysts employing the data presented in (Elsalamony *et al.* 2013). The following equation has been applied:

$$Y = \beta_0 + \beta_1 * (Ce/Zr) + \beta_{11} * (Ce/Zr)^2$$
(4)

Ordinary Least Squares (OLS) method that minimizes the variance of the unbiased estimators of the coefficients is usually applied to estimate the coefficients of the equation (Draper & Smith 1998). We eliminate non-significant terms based on statistical parameter tests then rerun the model without them. The final model should contain only significant parameters (Box et al. 2005). To guarantee that the 'best fitting' equation fits the data well we assess the adequacy of the 'fitted' equation through the following indicators (Weisberg 2005; Hoffmann 2010).

 R^2 : is a measure of how well the regression line approximates the real data and should be at least 0.6 (60 %).

Calculated Regression F: Regression MS / Residual MS

The significance of the regression equations have been checked by comparing the value of the calculated F compared to the corresponding tabulated $F_{(k;n-k-1)}$ values with desired 0.05 significance level.

n: Number of experimental observations

k: Number of independent variables

Regression Significance F: It is considered that a significant relationship between the independent and dependent variables exists if this value $< \alpha = 0.05$.

The p- value for each regression coefficient: It is the percentage that tells how likely it is that the coefficient for that independent variable emerged by chance and *does not* describe a real relationship. The claim should be rejected if p < 0.05.

Confidence Limits are the 95% probability that the true value of the coefficient lies between the Lower and Upper 95% values.

4. Results and Discussion

The regression has been performed employing Microsoft Excel 2007 which determines the coefficients of the equation along with the statistical parameters which validate the results. Among these statistical parameters are R^2 , calculated *F*-ratio and Significance *F*. These values are presented in Table (1) for the various components investigated for equations 2&3. The significant parameters of the corresponding equation along with confidence interval and the *p*-value for the parameters are depicted in Tables (2-a&b). As for equation 4 the regression statistics along with the regression parameters are shown in tables (3-a, b&c). For Tables (3-a, b&c) the tabulated F-values are: Tab F(0.05,2,1) = 199.5, Tab F(0.05,1,2) = 18.5.

Regarding the *R* squared that indicate the goodness of fit between the experimental values and the corresponding equations, tables (1&3) presents the values for $R^2 > 0.6$ obtained for all the relations. This proves the adequacy of the corresponding equations for representing the experimental results. Also the tables reveal the higher values of the calculated *F*-ratio as compared to the corresponding tabulated ones which indicate the significance of the corresponding equation.

As for the significance *F* Table 2 shows that small values $< (\alpha = 0.05)$ have been obtained for all the relations. This indicates that there is a real relation between the independent variables and the corresponding dependent response variable for all the investigated cases. The low values of *p* depicted in Tables (2-a & b) and Tables (3-a, b & c) indicate that all the recorded coefficients are significant. This is also manifested in the small values of coefficients, limits in comparison with their corresponding ones which mean that they do not span the zero as a value for the parameter. It could be concluded that the various variables effect represented by the recorded corresponding parameters are statistically significant.

However for the relation H_2/CO , for the case of using H_2O , employing equation (4) we find that $R^2 = 0.964$ although the values of calculated F, significant F and P-values of the corresponding parameters are statistically insignificant, Tables (3-b&c). This relation needs further experimental work to be established.

4.1 Effect of % Nickel loading and temperature

From table (2-a) and Fig (1-a) it could be observed that ethanol conversion increased by increasing % Ni loading and temperature as indicated by the positive values of $\beta_1 \& \beta_2$ of eq. (2) reaching a maximum then start to decline slightly by further increase in % Ni and combined increase in %Ni and temperature as indicated by the small negative values of $\beta_{11} \& \beta_{12}$ respectively.

As for the Hydrogen production and the ratio H₂/(CO+CO₂) they also increased by increasing % Ni loading and temperature shown by the positive values of $\beta_1 \& \beta_2$ of eq. (2) reaching a maximum then start to decline slightly by further increase in % Ni as indicated by the small negative values of β_{11} , table (2-a) & Fig(1-a).

However as for the ratio H₂/CO₂ it increased by increasing % Ni loading and decreasing temperature as indicated by the positive values of β_1 and the negative value of $\beta_2 \& \beta_{12}$ of eq. (2), table (2-a) & Fig (1-a).

4.2 Effect of Ce/Zr and water/ethanol ratios

The ratios CO/CO₂ and CO/ (CO+CO₂) decreased by increase of both (Ce/Zr) and (W/E) ratios as indicated by the negative value of $\beta_1 \& \beta_2$ of eq. (3), table (2-b) & Fig (1-a).

The ratio (Ce/Zr) has undefined effect on the CO₂/CO ratio as indicated by the negative value of β_1 and the positive value of β_{11} of eq. 3, while there is a positive effect of the combined (Ce/Zr)* (W/E) as indicated by the parameter β_{12} , table (2-b). However the ratio CO₂/ (CO+CO₂) increased by increasing both (Ce/Zr) and (W/E) ratios as indicated by the positive values of $\beta_1 \& \beta_2$ of eq. (3), table (2-b) & Fig (1-b).

As for the ratios H₂/CO, H₂/CO₂ and H₂/(CO+CO₂) they have a parabolic relation with the ratio (Ce/Zr) as indicated by the negative value of β_1 and the positive value of β_{11} of eq. 3, table (2-b) & Fig (1-b).

Carbon deposition decreased by increase of both (Ce/Zr) and (W/E) ratios as indicated by the negative value of $\beta_1 & \beta_2$ of eq. (3) reaching a minimum then showed a slight increase by increasing (Ce/Zr) as indicated by the small positive value of β_{11} , table (2-b) & Fig (1-b).

4.3 Effect of hydrogen peroxide as compared to steam

The ratios CO/CO₂ and CO/(CO+CO₂) show a decreasing linear relationship with the ratio (Ce/Zr) while the ratio $CO_2/(CO+CO_2)$ shows an increasing linear relationship as pointed out by table (3-b) & Fig (1-c). The lower values of β_0 for the first two ratios and higher value of the last one reveal the oxidizing effect of H₂O₂ as compared to steam.

The ratio H₂/CO has a curvature relationship with the ratio (Ce/Zr) as indicated by table (3-c) & Fig. (1- c). H₂O₂ has a more pronounced effect as indicated by the higher value of β_0 .

	Eq.	la.		Regression		Residual	F Ratio		
Component	No.	\mathbf{R}^2	df	MS	df	MS	Calc. F	Tab. F	Significance F
Conversion, mole%	2	0.718	4	376.9	15	39.57	9.524	3.06	4.87E-04
H ₂ , mole%	2	0.710	3	3010.3	16	230.0	13.09	3.24	1.42E-04
H ₂ /CO ₂	2	0.649	4	251.4	15	36.25	6.94	3.06	2.28E-03
H ₂ /(CO+CO ₂)	2	0.665	3	4.309	16	0.4076	10.57	3.24	4.47E-04
H ₂ /CO	3	0.959	2	582.8	9	5.473	106.48	4.26	5.44E-07
H ₂ /CO ₂	3	0.613	2	0.4198	9	0.0588	7.143	4.26	1.39E-02
CO/CO ₂	3	0.980	2	8.118E-03	9	3.614E-05	224.7	4.26	2.084E-08
CO ₂ /CO	3	0.996	3	59.48	8	8.53E-02	697.3	4.07	5.17E-10
H ₂ /(CO+CO ₂)	3	0.616	2	0.3054	9	0.0424	7.204	4.26	1.35E-02
CO/(CO+CO ₂)	3	0.980	2	5.347E-03	9	2.464E-05	217	4.26	2.43E-08
CO ₂ /(CO+CO ₂)	3	0.980	2	5.35E-03	9	2.46E-05	217.0	4.26	2.43E-08
CD	3	0.981	3	1.524E-02	5	1.803E-04	84.54	5.41	1.051E-04

Table 1. Regression Statistics and Analysis of Variance

Table 2-a.	Estimated	Regression	Parameters	for Equation	(2)
		-0		1	

		Component								
Regr	ression Parameters	Conversion	H_{2}	H ₂ /CO ₂	$\frac{H_2}{(CO+CO_2)}$					
	Coeff.	59.97	-19.82	28.97	-0.7197					
β ₀	± C.L.	13.07	23.57	26.96	0.9921					
	P-value	6.69E-08	9.37E-02	3.69E-02	1.44E-01					
	Coeff.	3.549	6.066	1.846	0.2304					
β ₁	± C.L.	1.864	3.648	1.093	0.1536					
	P-value	1.03E-03	2.81E-03	2.63E-03	5.81E-03					
	Coeff.	0.0690	0.1075	-0.1614	4.247E-03					
β ₂	± C.L.	0.0302	0.0508	0.1402	2.14E-03					
_	P-value	2.04E-04	3.78E-04	2.68E-02	6.68E-04					
	Coeff.	-3.63E-03		-3.55E-03						
β ₁₂	± C.L.	2.69E-03		2.58E-03						
	P-value	1.16E-02		1.02E-02						
	Coeff.	-0.0787	-0.2294		-9.039E-03					
β ₁₁	± C.L.	0.0734	0.1760		7.41E-03					
	P-value	3.74E-02	1.39E-02		1.99E-02					
	Coeff.			2.08E-04						
β ₂₂	± C.L.			1.71E-04						
	P-value			2.09E-02						

						Component			
	gression ameters	H ₂ /CO	H ₂ /CO ₂	CO/CO ₂	CO _{2/} CO	CO/(CO+CO ₂)	$CO_2/(CO+CO_2)$	H ₂ /(CO+CO ₂)	CD
	Coeff.	17.363	2.717	1.873E- 01	6.336	1.610E-01	0.8390	2.347	1.0473
β ₀	± C.L.	3.042	0.3152	2.052E- 02	0.3871	1.694E-02	0.0169	0.2677	0.2155
	P-value	4.11E-07	1.13E-08	6.85E-09	2.66E-10	4.79E-09	1.83E-15	9.78E-09	5.834E-5
	Coeff.	-3.029	-0.2997	-1.228E- 2	-0.6732	-9.966E-03	9.97E-03	-0.2506	-0.1312
β1	± C.L.	1.740	0.1803	1.327E- 03	0.2834	1.096E-03	1.10E-03	0.1532	0.0736
	P-value	3.42E-03	4.49E-03	6.05E-09	5.90E-04	7.08E-09	7.08E-09	4.92E-03	5.925E-3
	Coeff.			-3.495E- 3		-2.872E-03	2.87E-03		-0.0290
β ₂	± C.L.			2.404E- 03		1.985E-03	1.99E-03		7.045E-3
	P-value			9.39E-03		9.64E-03	9.64E-03		1.303E-4
	Coeff.				0.0849				
β ₁₂	± C.L.				0.0221				
	P-value				2.09E-05				
	Coeff.	0.7552	0.0343		0.1576			0.0317	8.333E-3
β ₁₁	± C.L.	0.215	0.0223		0.0274			0.0190	6.101E-3
	P-value	2.37E-05	6.98E-03		9.98E-07			4.32E-03	1.708E-2

Table 3-a. Regression Statistics, Analysis of Variance and

Estimated Regression Parameters for Equation (4)

6		Regression Statistics					β ₀			β ₁		
Comp. Feed	R ²	Reg.	Res.	F Calc	Sign F	Coef.	CL	P value	Coef.	CL	P value	
60/60	H_2O	0.995	6.49E-03	1.72E-05	377.2	2.64E-03	0.1678	0.0162	5.06E-04	-0.0136	0.0030	2.64E-03
CO/CO ₂	H_2O_2	0.978	2.99E-03	3.32E-05	90.06	1.09E-02	0.1098	0.0226	2.27E-03	-9.24E-03	4.19E-3	1.09E-02
со	H ₂ O	0.994	4.21E-03	1.25E-05	335.8	2.96E-03	0.1444	0.0139	4.98E-04	-1.10E-02	2.57E-3	2.96E-03
$(CO + CO_2)$	$\mathrm{H}_{2}\mathrm{O}_{2}$	0.982	2.23E-03	2.10E-05	106.2	9.28E-03	9.92E-02	1.79E-02	1.76E-03	-7.98E-03	3.33E-3	9.28E-03
CO2	H_2O	0.994	4.21E-03	1.25E-05	335.8	2.96E-03	0.8556	0.0139	1.42E-05	0.0110	0.0026	2.96E-03
$(CO + CO_2)$	H_2O_2	0.982	2.23E-03	2.10E-05	106.2	9.28E-03	0.9008	0.0179	2.14E-05	7.98E-03	3.33E-3	9.28E-03

Table 3-b. Regression Statistics and Analysis of Variance

H ₂ O					H ₂ O ₂				
\mathbf{R}^2	Reg.	Res.	F Calc	Sign F	R2	Reg.	Res.	F Calc	Sign F
0.964	185.4	13.67	13.56	0.189	1.000	166.6	0.0113	14734.1	5.83E-03

of the ratio H_2/CO for Equation (4)

Table 3-c. Estimated Regression Parameters of the ratio H₂/CO for Equation (4)

Regression		H ₂ O		H ₂ O ₂				
Parameters	Coeff.	± C.L.	P-value	Coeff.	± C.L.	P-value		
β ₀	18.03	46.76	0.1282	20.48	1.34	3.29E-03		
β1	0.7126	3.312	0.2233	0.5321	0.0953	8.97E-03		
β ₂	-2.727	26.76	0.4187	-1.296	0.7696	2.97E-02		

4.4 Graphical representation of the response surface

In general, the response surface can be visualized graphically. The graph is helpful to see the shape of a response surface; hills, valleys, and ridge lines. Hence, the function $f(X_1, X_2)$ can be plotted versus the levels of X_1 and X_2 . In this graph, each value of X_1 and X_2 generates a corresponding Y-value. This three-dimensional graph shows the response surface from the side and it is called a response surface plot.

Equation (2) has been applied to represent the response surface of the various components as a function of the input variables of %Nickel loading (X_l) and temperature (X_2) . Also, Equation (3) has been applied to represent the response surface of the various components as a function of the input variables of Ce/Zr (X_l) and water / ethanol (X_2) ratios.

The various developed equation models along with the calculated parameters depicted in Tables (2-a&b) have been employed, (utilizing Matlab 7), to display the Response Surface plots for ethanol conversion and formation of Hydrogen and the various components as shown in Figure 2.Together with the corresponding experimental values.







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Figure 1-c. Response surface of various components

5. Conclusion

The response surface methodology (RSM) was a good tool to study the effect of the operating variables on hydrogen production via ethanol steam reforming employing the prepared Ni /Ce_{1-x} Zr_xO_2 catalysts. The developed least squares regression models showed good prediction for the experimental results with a correlation coefficient of more than 0.6 for all the presented cases. The various variables effect represented by the recorded corresponding parameters of the applied equations are statistically significant. The response surface illustrated in the three dimensions depicted the response of the investigated relations to the variation of the studied parameters.

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