



## Statistical modelling and optimization of hydrolysis of urea to generate ammonia for flue gas conditioning

K. Mahalik<sup>a,b</sup>, J.N. Sahu<sup>a,c,\*</sup>, Anand V. Patwardhan<sup>d</sup>, B.C. Meikap<sup>a,e</sup>

<sup>a</sup> Department of Chemical Engineering, Indian Institute of Technology (IIT), Kharagpur, P.O. Kharagpur Technology, West Bengal 721302, India

<sup>b</sup> Department of Chemical Engineering, Gandhi Institute of Engineering and Technology, Gunupur, Orissa, India

<sup>c</sup> Department of Chemical Engineering, Faculty of Engineering, University of Malaya, Kuala Lumpur 50603, Malaysia

<sup>d</sup> Department of Chemical Engineering, Institute of Chemical Technology (ICT), Mumbai 400019, India

<sup>e</sup> School of Chemical Engineering, University of KwaZulu-Natal, Howard College Campus, King George V. Avenue, Durban 4041, South Africa

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### ABSTRACT

The present study is concerned with the technique of producing a relatively small quantity of ammonia which can be used safely in a coal-fired thermal power plant to improve the efficiency of electrostatic precipitator by removing the suspended particulate material mostly fly ash, from the flue gas. In this work hydrolysis of urea has been conducted in a batch reactor at atmospheric pressure to study the different reaction variables such as reaction temperature, initial concentration and stirring speed on the conversion by using design expert software. A  $2^3$  full factorial central composite design (CCD) has been employed and a quadratic model equation has been developed. The study reveals that conversion increases exponentially with an increase in temperature, stirring speed and feed concentration. However the stirring speed has the greatest effect on the conversion with concentration and temperature exerting least and moderate effect respectively. The values of equilibrium conversion obtained through the developed models are found to agree well with their corresponding experimental counterparts with a satisfactory correlation coefficient of 93%. The developed quadratic model was optimized using quadratic programming to maximize conversion of urea within experimental range studied. The optimum production condition has been found to be at the temperature of 130 °C, feed concentration of 4.16 mol/l and stirring speed of 400 rpm and the corresponding conversion, 63.242%.

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### 1. Introduction

Increasing population and industrial development demand sustainable electricity, which, in India, mainly depends on coal-based thermal power stations for the generation of electricity. However, the combustion of high-ash coal results in production of large quantity of ash, which essentially constitutes bottom and fly ash. Fly ash particles that are in the form of suspensions in the flue gas contribute to an increased suspended particulate matter (SPM) in the surrounding environment. As such, for safeguarding the environment, reduction in the emission levels of the SPM becomes essential. To achieve this, several pollution control devices such as cyclone separators, bag filters, and electrostatic precipitators (ESP) are being employed. Among the pollution control devices, ESP are most popularly used in thermal power stations to reduce

SPM levels [1–3]. This is mainly due to their: (i) higher efficiency of removal of particles (<0.01 μm in size), (ii) effectiveness over a wide range of operating temperatures and (iii) suitability for corrosive environmental conditions.

However, the efficiency of the ESP in India is adversely affected due to the large variations in operating conditions and the abrasive nature of high-ash domestic coal [3]. Stringent environmental regulation demands high removal efficiency of fly ash, even in older units. Additionally, many power companies have chosen to switch to low-sulfur coal as a means of meeting environmental restrictions on sulfur dioxide emissions. Uncertain fuel supplies have often forced the plants to use lower grades of coal. In many cases, these problems have resulted in the deterioration of precipitator efficiency resulting into particulate emissions exceeding the limits. It has also been demonstrated by earlier researchers [4–6] that the performance of ESP can be improved by: (i) changing the feed coal characteristics, (ii) increasing the collection plate area of the existing ESP, (iii) employing wet ESP to minimize re-entrainment, (iv) increasing or lowering the gas temperature and (v) addition of chemicals to modify the fly ash or the dielectric properties of ash in the ESP. However, most of these options are difficult to implement

\* Corresponding author at: Department of Chemical Engineering, Faculty of Engineering, University of Malaya, Kuala Lumpur 50603, Malaysia. Tel.: +60 379675295; fax: +60 379675319.

E-mail address: [jay\\_sahu@yahoo.co.in](mailto:jay_sahu@yahoo.co.in) (J.N. Sahu).

in a thermal power station, mainly, due to: (i) there are constraints associated with the feed coal (that is, cost associated with import, washing of coal and environmental issues associated with it, ash content, etc.), (ii) addition of more collection plate area in ESP requires more space and is highly expensive, (iii) installation and operating costs for employing wet ESP is too high apart from ash lump formation and its degradation as a construction material [6].

Under these circumstances, the flue gas conditioning (FGC) becomes inevitable [7–8]. FGC is a technique that involves addition of chemical additives to the flue gas in order to increase ash collection efficiency of ESP. Critical survey of literature reveals that FGC has several advantages such as: (i) less cost involved as compared to installation of additional ESP, (ii) lesser execution time requirement and (iii) more flexible and versatile to adopt, that is, even with variations in the operating parameters (such as coal characteristics, boiler load, ESP voltage and current change), SPM levels can be easily controlled to the desired level by simply adjusting the dosing amount of the FGC agents. These agents are quite helpful in improving the dielectric characteristics of fly ash particles, which results in enhanced ash collection efficiency of the ESP.

Flue gas conditioning is required for low-sulfur coal containing less than 1.5% of sulfur when the gas humidity is lower than 15%. Conditioning agents cause adsorption of moisture on the particle surface, thus increasing its conductivity. Water, steam, ammonia (NH<sub>3</sub>), sulfur trioxide (SO<sub>3</sub>), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), sodium chloride (NaCl) and hydrogen chloride (HCl) are used as conditioning agents to increase dust conductivity [9–16]. More complex organic mixtures have also been proposed for gas conditioning, but NH<sub>3</sub> is still the most effective conditioning agent [14–16].

Ammonia has long been known to be useful in the treatment of flue/tail/stack gases from industrial furnaces, incinerators and the electric power generation industry and also for conditioning of the flue gas by which an improved collection and removal of particulate matter (fly ash) is obtained [16–28]. In the earlier days, aqueous or anhydrous ammonia was supplied as feedstock for selective catalytic reduction and selective non-catalytic reduction. Unfortunately, anhydrous ammonia as well as aqueous ammonia in strengths above 20%, pose to be a significant danger to the human health and are classified by Occupational Safety and Health Administration as hazardous chemicals. Their transportation, storage and handling triggers serious safety and environmental regulatory requirements for risk management plans, accident prevention programs, emergency response plans and release analysis. Aqueous ammonia solutions with low concentration render lower health and safety risks, but their usage results in a substantial increase in operating costs of selective catalytic reduction and selective non-catalytic reduction systems [29–30].

It has been determined that urea thermal hydrolysis is the preferred process for converting urea/water solution into a gaseous mixture containing ammonia, carbon dioxide and water vapor [31–37]. Therefore, in situ, generation of ammonia is essential for flue gas conditioning, especially in coal-fired thermal power plants. Urea is an ideal candidate to be substituted for ammonia, as the method of urea-to-ammonia conversion is a hydrolysis process. Urea is a nontoxic chemical compound, and for the purpose of satisfying small requirements (i.e. up to 50 kg/h), it presents essentially no danger to the environment, animals, plant life, and human beings. It is solid under ambient temperatures and pressures. Consequently, urea can be safely and inexpensively shipped in bulk and stored for long periods of time until it is converted into ammonia. Literature survey reveals that only few works have been reported for urea hydrolysis and most of them are patent protected [38–43]. However, none of the authors has disclosed the effect of reaction variable on the equilibrium conversion of hydrolysis of urea and their combined effect. Therefore, it was decided to study more thoroughly the phenomena of urea hydrolysis for the production of

ammonia in different application areas that requires the safe use of relatively small quantity of ammonia. In this work, an attempt has been made to develop a second-order non-linear model equation to show the effect of different reaction variables such as reaction temperature, initial feed concentration and stirring speed on the fractional conversion of hydrolysis of urea. In the present study hydrolysis of urea has been conducted at atmospheric pressure though the operating pressure plays an important role for this process. The reason being due to the fact that the rate of a heterogeneous reaction is function of temperature, composition of the phase and the pressure. However, the above operating variables are interdependent in that the pressure is determined given the temperature and composition of the phase according to the following expression:

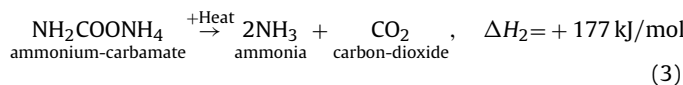
$$C = \frac{P}{RT} \quad (1)$$

where  $C$  is the concentration of the phase,  $P$  is the pressure,  $T$  is the temperature and  $R$  is the universal gas constant.

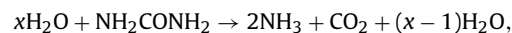
The conventional and classical methods of studying a process by maintaining the other factors involved at an unspecified constant level does not depict the combined effect of all the factors involved. The conventional technique for the optimization of a multivariable system usually defines one factor at a time. Such a technique needs to perform a lot of experiments and could not reveal the alternative effects between the components. This method is also time consuming and requires a number of experiments to determine optimum levels, which are unreliable. Recently, many statistical experimental design methods have been employed in chemical process optimization. Experimental design technique is a very useful tool for this purpose as it provides statistical models, which help in understanding the interactions among the parameters that have been optimized [44]. These methods involve mathematical models for designing chemical processes and analyzing the process results. Among them, response surface methodology (RSM) is one of the suitable methods utilized in many fields [44–47]. RSM is a collection of mathematical and statistical techniques useful for developing, improving and optimizing processes and can be used to evaluate the relative significance of several affecting factors even in the presence of complex interactions. The main objective of RSM is to determine the optimum operational conditions for the system or to determine a region that satisfies the operating specifications [47]. However, there is no information available in the literature regarding the optimization of hydrolysis of urea to produce ammonia. Therefore; it was decided to study more thoroughly the phenomenon of urea hydrolysis. The effects of operating parameters such as initial feed concentration, temperature and stirring speed were optimized using response surface methodology. The optimization of experimental conditions using RSM for hydrolysis of urea is not available in literature.

## 2. Reaction pathway

The basic chemistry employed in the process is the reverse of that employed in industrial production of urea from ammonia and carbon dioxide and employs two reaction steps as follows [32,48–49]:



The first reaction in which urea hydrolyzes to form ammonium carbamate is mildly exothermic, while the second reaction, in which ammonia and carbon dioxide are produced, is strongly endothermic, with the result that the reaction to release ammonia and carbon dioxide requires heat and quickly stops when the supply of heat is withdrawn.



$$\Delta H_{\text{over}} = +161.5 \text{ kJ/mol} \quad (4)$$

The overall reaction is endothermic and the first reaction, i.e. urea to ammonium carbamate reaction is a slow reaction and the second reaction is very fast and undergoes completion.

### 3. Experimental set-up and technique

Experimental arrangement used for optimization of production of ammonia from urea in semi-batch reactor is same as that has been used in our earlier experimental study on reaction kinetic [37]. First urea solution of different concentration (1.6, 2.67, 2.64, 3.33, 4.16, 5.64 and 6.66 mol/l) was prepared. In each case, the volume of the solution was taken as 500 ml. Then the urea feed solution of 1.67 mol/l was fed into the reactor. Adjusting the stirrer speed at 100 rpm heat was supplied from the bottom of the reactor at a controlled rate by means of electricity. As the temperature increases initially evaporation of water will take place. The decomposition of urea takes place slowly at around 110 °C. The temperature was kept constant at 110 °C in order to maintain isothermal condition. As the reaction starts, the product which is a gaseous mixture of ammonia, carbon dioxide and water vapor comes out through the condenser. In the condenser, where cold water was circulated, gaseous product mixture gets condensed and was collected into a beaker. Then the residue of the reactor was taken out and its volume was measured after it becomes cold. An additional amount of water was added to the residue to make the volume equals to the initial volume of the solution and its refractive index was determined by refractometer. The concentration of the reactor content (unreacted urea) was determined from the calibration chart obtained previously by Mahalik et al. [37]. Same procedure was repeated for other concentrations (2.673, 5.646 and 4.16 mol/l) temperature (110, 130, 140 and 150 °C) and stirring speed (221, 400, 578 and 700 rpm). Then by comparing the initial concentration and final concentration the conversion was found out. The details of design of experimental matrix have been presented in Table 3 where as Table 2 represents the ranges of variables.

### 4. Response surface modelling

Factorial design is a statistical method that uses quantitative data from appropriate experiments to determine the regression model equation and optimum operating condition. A standard RSM design called central composite design (CCD) has been applied in this work to study the variables for the conversion of ammonia from urea. The central composite design has been widely used for fitting a second-order model. By using this method, modelling is possible and it requires only a minimum number of experiments. The CCD consists of  $2^n$  factorial runs with  $2n$  axial runs and ' $n_c$ ' center runs (six replicates). These designs consist of a  $2^n$  factorial or fraction (coded to the usual  $\pm 1$  notation) augmented by  $2n$  axial points ( $\pm\alpha, 0, 0, \dots, 0$ ), ( $0, \pm\alpha, 0, \dots, 0$ ), ..., ( $0, 0, \dots, \alpha \pm$ ), and  $n_c$  center points ( $0, 0, 0, \dots, 0$ ) [50]. Each variable is investigated at two levels. In this case, main effects and interactions may be estimated by fractional factorial designs running only a minimum number of experiments. Individual second-order effects cannot be estimated separately by  $2^n$  factorial designs. Therefore, the central composite

**Table 1**

Actual value and corresponding coded value of the variables.

Code	Actual level of variable
$-\alpha$	$A_{\min}$
$-1$	$[(A_{\max} + A_{\min})/2] - [(A_{\max} - A_{\min})/2\beta]$
$0$	$(A_{\max} + A_{\min})/2$
$+1$	$[(A_{\max} + A_{\min})/2] + [(A_{\max} - A_{\min})/2\beta]$
$+\alpha$	$A_{\max}$

Where  $A_{\max}$  and  $A_{\min}$  are maximum and minimum values of  $A$ , respectively;  $\beta$  is  $2^{n/4}$ .

design was employed in this study. The responses and the corresponding parameters are modelled and optimized using analysis of variance (ANOVA) to estimate the statistical parameters by means of response surface methods. Basically, this optimization process involves three major steps, which are; performing the statistically designed experiments, estimating the coefficients in a mathematical model and predicting the response and checking the adequacy of the model.

$$Y = f(A_1, A_2, A_3, A_4, \dots, A_n) \quad (5)$$

where  $Y$  is the response of the system and  $A_i$  is the variables of action called factors. The goal is to optimize the response variable ( $Y$ ). It is assumed that the independent variables are continuous and controllable by experiments with negligible errors. It is required to find a suitable approximation for the true functional relationship between independent variables and the response surface [47]. The experimental sequence was randomized in order to minimize the effects of the uncontrolled factors. The response was used to develop an empirical model that correlated the response (%conversion) to the variable (concentration, temperature and stirring speed) using a second degree quadratic equation as given by Eq. (6):

$$Y = a_0 + \sum_{i=1}^n a_i A_i + \sum_{i=1}^n a_{ii} A_i^2 + \sum_{i=1}^n \sum_{j>1}^n a_{ij} A_i A_j \quad (6)$$

where  $Y$  is the predicted response,  $a_0$  the constant coefficient,  $a_i$  the linear coefficients,  $a_{ij}$  the interaction coefficients,  $a_{ii}$  the quadratic coefficients and  $A_i, A_j$  are the coded values of the variables. The codes are calculated as function of the range of interest of each factor as shown in Table 1 [51]. In developing the regression equation, the test variables were coded according to the equation:

$$x_i = \frac{(X_i - X_i^*)}{\Delta X_i} \quad (7)$$

where  $x_i$  is the coded value (dimensionless) of the  $i$ th independent variable,  $X_i$  is the uncoded value of the  $i$ th independent variable,  $X_i^*$  is the uncoded value of the  $i$ th independent variable at the center point and  $\Delta X_i$  is the step change value [52]. The number of tests required for the CCD includes the standard  $2^n$  factorial with its origin at the center,  $2n$  points fixed axially at a distance, say  $\alpha$ , from the center to generate the quadratic terms, and replicate tests at the center; where  $n$  is the number of dependent variables [53]. The axial points are chosen in such a manner that they allow rotatability, which ensures that the variance of the model prediction is constant at all points equidistant from the design center [54]. Replicates of the test at the center are very important as they provide an independent estimate of the experimental error. For three variables, the recommended number of tests at the center is six [55]. Hence, the total number of tests ( $N_t$ ) required for the three independent variables are:

$$N_t = 2^n + 2n + n_c = 2^3 + (2 \times 3) + 6 = 20 \quad (8)$$

**Table 2**  
Level of independent variables.

Variables	Symbol	$-\alpha$	$-1$	$0$	$+1$	$+\alpha$
Temperature ( $^{\circ}\text{C}$ )	$A_1$	110	118	130	141	150
Feed Concentration (mol/l)	$A_2$	1.66	2.67	4.16	5.64	6.66
Stirring speed (rpm)	$A_3$	100	221	400	578	700

**Table 3**  
Experimental design matrix and response.

Run	Coded level variable			Actual level variable			Conversion $Y$ (%)
	$A_1$	$A_2$	$A_3$	$A_1$ ( $^{\circ}\text{C}$ )	$A_2$ (mol/l)	$A_3$ (rpm)	
1	-1	-1	-1	118	2.673	221	20.581
2	+1	-1	-1	141	2.673	221	24.726
3	-1	+1	-1	118	5.646	221	43.469
4	+1	+1	-1	141	5.646	221	52.223
5	-1	-1	+1	118	2.673	578	53.714
6	+1	-1	+1	141	2.673	578	64.531
7	-1	+1	+1	118	5.646	578	38.5
8	+1	+1	+1	141	5.646	578	66.6
9	$-\alpha$	0	0	110	4.16	400	53.835
10	$+\alpha$	0	0	150	4.16	400	50.938
11	0	$-\alpha$	0	130	1.66	400	25.388
12	0	$+\alpha$	0	130	6.66	400	53.28
13	0	0	$-\alpha$	130	4.16	100	15.905
14	0	0	$+\alpha$	130	4.16	700	54.08
15	0	0	0	130	4.16	400	63.623
16	0	0	0	130	4.16	400	63.623
17	0	0	0	130	4.16	400	63.6
18	0	0	0	130	4.16	400	63.05
19	0	0	0	130	4.16	400	63.376
20	0	0	0	130	4.16	400	63.068

## 5. Results and discussion

### 5.1. Development of model

The statistical software package “Design Expert” has been used for regression analysis of experimental data and to draw response surface plot. ANOVA has been used to estimate the statistical parameters. The complete experimental range and level of variables are given in the Table 2 and Table 3 which shows the design of experiments together with the experimental results. As suggested by the software, the quadratic model has been selected which was not aliased. The final empirical model in terms of coded factor for conversion of urea ( $Y$ ) is shown in Eq. (9):

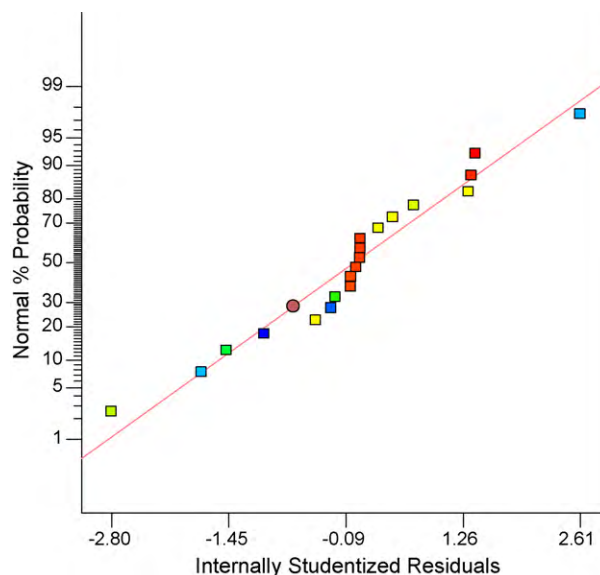
$$Y = 63.24 + 3.44A_1 + 6.16A_2 + 10.73A_3 + 2.74A_1A_2 + 3.25A_1A_3 - 7.94A_2A_3 - 0.92A_1^2 - 7.54A_2^2 - 9.07A_3^2 \quad (9)$$

Eq. (7) has been used to visualize the effect of experimental factor on conversion percentage. The model  $F$  value of 16.85 implies that the model is significant. Further Values of “Prob  $> F$ ” less than 0.0500 indicate that the model terms are significant. In this case,  $A_1$ ,  $A_2$ ,  $A_3$ ,  $A_2A_3$ ,  $A_2^2$  and  $A_3^2$  are significant model terms. The “Lack of fit  $F$ -value” of 840.72 implies that the Lack of Fit is significant. There is only a 0.01% chance that a “Lack of fit  $F$ -value” this large could occur due to noise. Adequacy Precision measures the signal to noise ratio and it compares the range of the predicted values at the design points to the average prediction error. A ratio greater than 4 is desirable. The ratio of 11.847 in our study, indicates an adequate signal. Hence this model can be used to navigate the design space.

One of the most important parts of the data analysis is to check the adequacy of the developed model. This is done by examining the residual plots which have been shown in the Figs. 1 and 2. It is observed from the Fig. 1 that there was neither response transformation nor any apparent problem with normality. Fig. 2 shows the internally studentised residual vs predicted percentage conversion

which represents a random scatter plot indicating that the variance of original observation is constant for all values of the response. This is also an indication that there was no need for transformation of response variables. However, this plot would exhibit a funnel shaped pattern if the variance of the response depended on the mean level of response [56].

The actual and the predicted percentage conversions have been shown in Fig. 3 and it is observed that the values of  $R^2$  and  $R_{adj}$  have been found to be 93% and 82%. The fair correlation coefficient 82% might be due to the insignificant terms in Table 4 as well as the non-linear influence of the investigated variables on the response.



**Fig. 1.** The studentized residuals and normal percentage probability plot of conversion of urea for production of ammonia.

**Table 4**  
Analysis of variance (ANOVA) for response surface quadratic model for generation of ammonia from urea.

Source	Sum of squares	Degrees of freedom	Mean square	F value	Prob > F	Remark
Model	4757.98	9	528.66	16.85	<0.0001	significant
A <sub>1</sub>	161.37	1	161.37	5.14	0.0467	significant
A <sub>2</sub>	518.47	1	518.47	16.53	0.0023	significant
A <sub>3</sub>	1572.51	1	1572.51	50.13	<0.0001	significant
A <sub>1</sub> A <sub>2</sub>	59.91	1	59.91	1.91	0.1971	
A <sub>1</sub> A <sub>3</sub>	84.62	1	84.62	2.70	0.1315	
A <sub>2</sub> A <sub>3</sub>	504.52	1	504.00	5216.08	0.0025	significant
A <sub>1</sub> <sup>2</sup>	123.04	1	123.04	3.92	0.0758	
A <sub>2</sub> <sup>2</sup>	818.61	1	818.612	6.10	0.0005	significant
A <sub>3</sub> <sup>2</sup>	1185.97	1	1185.97	37.81	0.0001	Significant
Residual	13.67	10	31.37			
Lack of fit	313.30	5	62.66	840.72	<0.0001	Significant
Pure error	0.37	5	0.075			
Correlation total	5071.65	19				
R <sup>2</sup>						93%
R <sub>adj</sub> <sup>2</sup>						82%
Adequacy precession						11.847

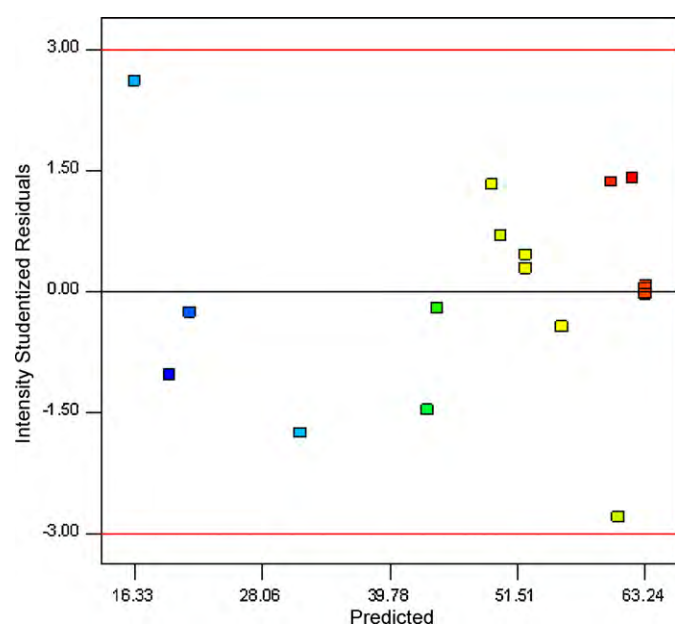


Fig. 2. The predicted conversion of urea and studentized residuals plot.

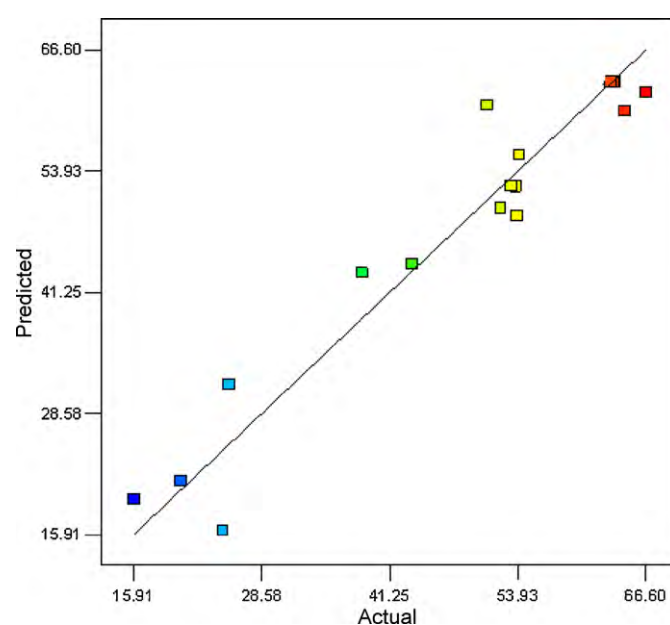


Fig. 3. Comparison of actual and predicted value of conversion of hydrolysis of urea.

### 5.2. Combined effect of stirring speed, temperature and feed concentration on conversion of urea

To study the individual and interaction effect of three factors such as temperature, concentration and stirring speed on conversion of ammonia from urea, the response surface methodology was used and three dimensional surface plots were obtained which has been shown in Figs. 4–9. A careful observation of ANOVA results reveals that stirring speed initial concentration and temperature have significant effect on the conversion. However stirring speed imposes the greatest effect while temperature imposes the least. On the other hand, quadratic effect of stirring speed and interaction effect of stirring speed and concentration has also significant effect on the conversion. Furthermore the quadratic effect of temperature and interaction effect of temperature and concentration impose least effect.

The combined effect of stirring speed and concentration on conversion at a constant temperature of 120 °C is shown in Fig. 4. The conversion is a function of both the initial urea concentration and the stirring speed. The latter parameter affects to a greater extent the conversion that increases exponentially up to a maximum value of 55%. This is due to the fact that as stirring speed increases, the

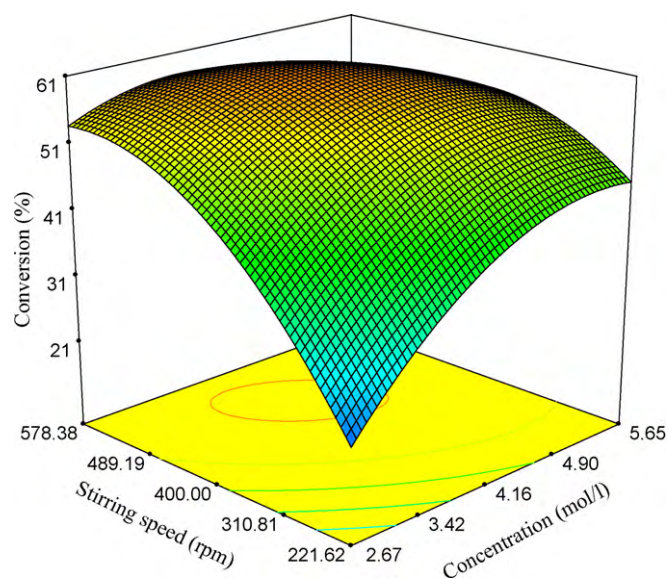
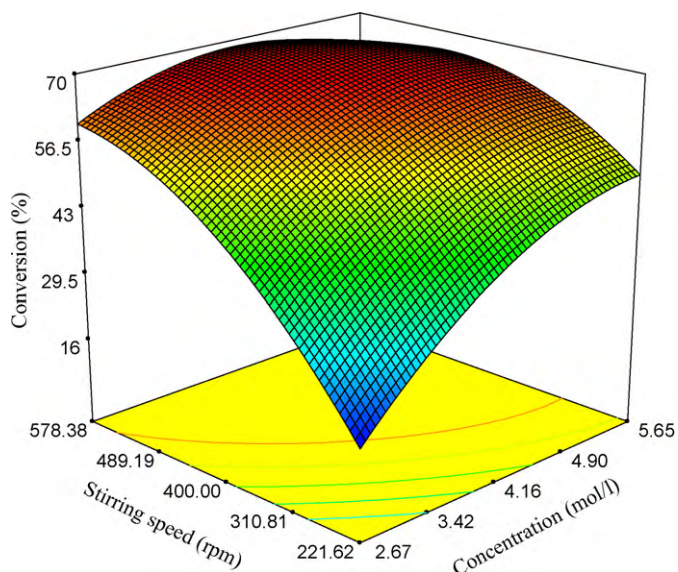


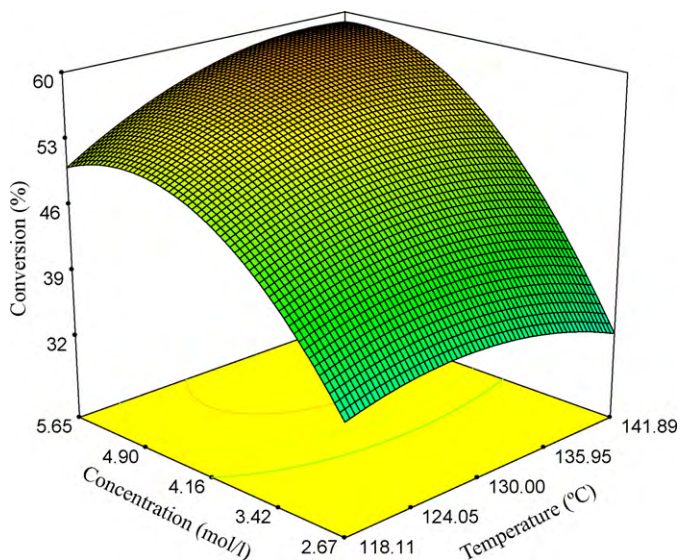
Fig. 4. Combined effect of stirring speed and initial concentration on conversion at temperature 120 °C.



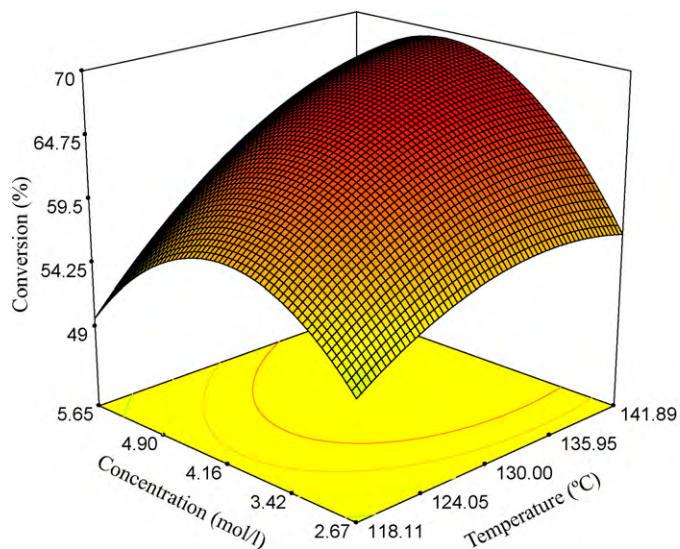
**Fig. 5.** Combined effect of stirring speed and initial concentration on conversion at temperature 141 °C.

rate of mass transfer also increases, which in turn increases the formation of ammonia and carbon dioxide thereby decreasing the unreacted urea in the reactor. Furthermore, the direct function of initial concentration with conversion may be due to the presence of more amount of urea in the feed. Due to this more amounts of ammonia and carbon dioxide is generated. Similarly, Fig. 5 represents the corresponding effect but at a different temperature of 141 °C.

Fig. 6 shows three dimensional response surfaces of the combined effect of temperature as well as feed concentration at a constant stirring speed of 300 rpm. With an increase in temperature and concentration, the conversion increases exponentially and reaches a maximum of 59% at a stirring speed of 300 rpm. The direct function of conversion on temperature may be due to the endothermic nature of the urea hydrolysis reaction. And the reason for conversion being the direct function of concentration may be attributed to the fact that more concentrated urea feed solution possesses less excess water (more urea) than that of less concen-



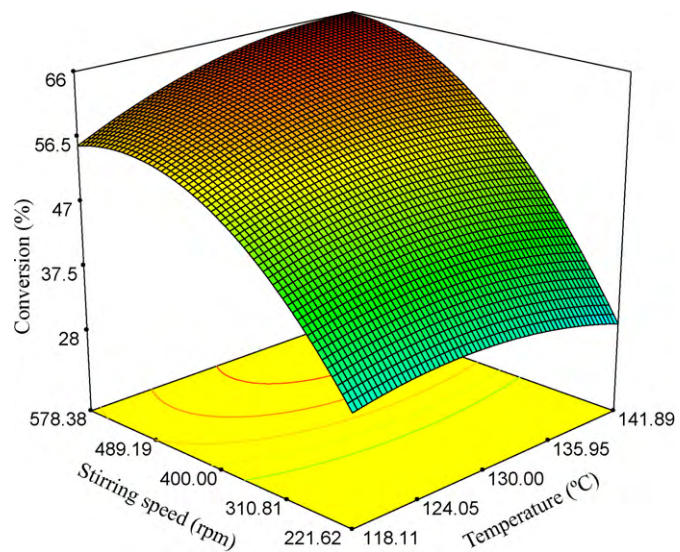
**Fig. 6.** Combined effect of temperature and initial concentration of urea on conversion at stirring speed of 300 rpm.



**Fig. 7.** Combined effect of temperature and initial concentration of urea on conversion at stirring speed of 500 rpm.

trated solution. Therefore, more amounts of ammonia and carbon dioxide are produced leaving less unreacted urea in the reactor. As less excess water is present in more concentrated feed solution, less amount of heat energy is required to achieve a desired level of production of ammonia. Therefore, more is the concentration of feed solution; more is the saving in energy. Similarly, it is observed from Fig. 7 that the maximum conversion of 65% is found out when temperature and feed concentration vary from 118 to 141 °C and 2.67 to 5.65 mol/l, respectively, at a stirring speed of 500 rpm.

The combined effect of temperature and stirring speed at a feed concentration of 3.33 and 5 mol/l has been shown in Figs. 8 and 9 respectively. It is obvious from Fig. 8 that with an increase in stirring speed and temperature, the conversion increases. A maximum conversion of 55% was obtained when the stirring speed was increased from 221 to 278 rpm through a temperature variation from 118 to 141 °C. Higher conversions due to higher stirring speed may be due to the fact that stirring enhances the rate of heat and mass transfer thereby decreasing the mass transfer resistance. On the other hand, from Fig. 9, which shows the above effect but at a feed concentra-



**Fig. 8.** Combined effect of temperature and stirring speed on conversion at initial concentration of 3.33 mol/l.

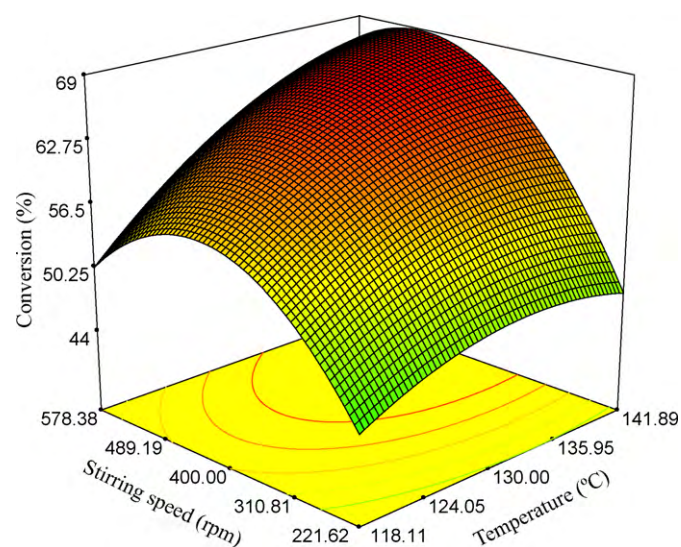
**Table 5**  
Optimum condition and model validation.

Temperature, $A_1$ ( $^{\circ}\text{C}$ )	Feed concentration, $A_2$ (mol/l)	Stirring speed, $A_3$ (rpm)	Conversion, $Y$ (%)	
			Predicted	Experimental
130	4.16	400	63.242	63.39

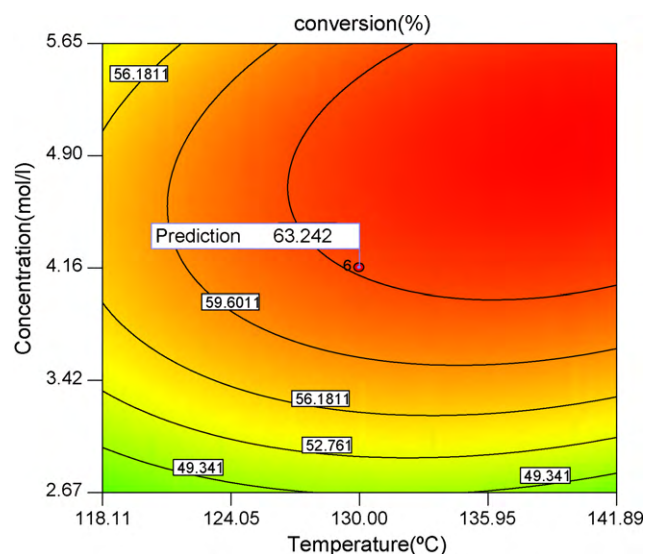
tion of 5 mol/l, it has been observed that a maximum conversion of 66.6% is determined when the stirring speed and temperature vary from 221 to 578 rpm and 118 to 141  $^{\circ}\text{C}$  respectively.

### 5.3. Optimization by response surface modelling

The determination of the optimum reaction variables for maximizing the production of ammonia from urea was one of the vital parts of this experimental study. The developed quadratic model equation was optimized using a quadratic programming to maximize the conversion. The optimum region for the production of ammonia on the response surface plot shown in Fig. 10 corresponds



**Fig. 9.** Combined effect of temperature and stirring speed on conversion at initial concentration of 5 mol/l.



**Fig. 10.** Optimum region for the ammonia production on the initial urea concentration–temperature–conversion response surface plot.

to a temperature of 130  $^{\circ}\text{C}$ , a feed concentration of 4.16 mol/l and a stirring speed of 400 rpm. The detail of the conversion optimization data is reported in Table 5.

## 6. Conclusion

The objective of the present study was to find out the optimum condition to maximize the conversion of ammonia by developing a model equation. The response surface modelling based on three variables central composite design was used to determine both the individual as well as the combined effect of different reaction variables such as temperature, feed concentration and stirring speed on the conversion of ammonia from urea. The regression analysis and optimization of variables are done by using design expert software for predicting the response in all experimental regions. The experimental values are found to agree well with that of predicted from the model with a correlation coefficient of 93%. It was found that the predicted conversion from the model is a direct function of temperature, concentration and stirring speed. However, the stirring speed has the greatest effect on the conversion with concentration and temperature exerting least and moderate effect respectively. Finally, the quadratic equation was optimized using quadratic programming to maximize the conversion of urea within experimental range studied. The optimum production condition has been found to be temperature 130  $^{\circ}\text{C}$ , feed concentration 4.16 mol/l, and stirring speed 400 rpm and the corresponding conversion, 63.24%.

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