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# Catalytic Hydrolysis of Urea with Fly Ash for Generation of Ammonia in a Batch Reactor for Flue Gas Conditioning and NO<sub>x</sub> Reduction

J. N. Sahu, P. Gangadharan, Anada V. Patwardhan, and B. C. Meikap\*

Department of Chemical Engineering, Indian Institute of Technology (IIT), Kharagpur, P.O. Kharagpur Technology, West Bengal, Pin – 721302, India

Ammonia is a highly volatile noxious material with adverse physiological effects, which become intolerable even at very low concentrations and present substantial environmental and operating hazards and risk. Yet ammonia has long been known to be used for feedstock of flue gas conditioning and NO<sub>x</sub> reduction. Urea as the source of ammonia for the production of ammonia has the obvious advantages that no ammonia shipping, handling, and storage is required. The process of this invention minimizes the risks and hazards associated with the transport, storage, and use of anhydrous and aqueous ammonia. Yet no such rapid urea conversion process is available as per requirement of high conversion in shorter time, so here we study the catalytic hydrolysis of urea for fast conversion in a batch reactor. The catalyst used in this study is fly ash, a waste material originating in great amounts in combustion processes. A number of experiments were carried out in a batch reactor at different catalytic doses, temperatures, times, and at a constant concentration of urea solution 10% by weight, and equilibrium and kinetic studies have been made.

## 1. Introduction

In the process of flue gas conditioning and NO<sub>x</sub> reduction, ammonia is used as a feedstock.<sup>1</sup> Unfortunately, ammonia presents significant danger to human health as a hazardous chemical. Its transportation, storage, and handling triggers serious safety and environmental regulatory requirements for risk management plans, accident prevention programs, emergency response plans, and release analysis.<sup>2</sup> An alternative approach to ammonia supply suggested in the late 1980s includes using urea feedstock to generate ammonia on site.<sup>3</sup>

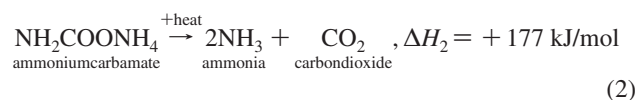
Urea is a stable, nonvolatile, environmentally benign material used primarily as fertilizer. Urea is a nontoxic chemical compound and presents essentially no danger to the environment, animals, plant life, and human beings. It is solid under ambient temperatures and pressures. Consequently, urea can be safely and inexpensively shipped in bulk and stored for long periods of time until it is converted into ammonia. It will not leak, explode, be a source of toxic fumes, require pressurization, increase insurance premiums, require extensive safety programs, or be a concern to the plant, community, and individuals who may be aware of the transportation and/or storage dangers of ammonia. The processes of this invention minimize the risks and hazards associated with the transport, storage, and use of anhydrous and aqueous ammonia. Using urea as the source of ammonia has the obvious advantages that no ammonia shipping, handling, and storage is required. The technology has proven to be a safe and relatively reliable alternative to either anhydrous or aqueous ammonia in recent commercial applications. Urea is available in solid form as prills or granulated material, and as urea solution in deionized water. Almost all urea to ammonia installations use solid urea. Ammonia is generated from urea by a hydrolysis reaction that yields a vapor phase mixture of ammonia, carbon dioxide, and steam.<sup>4</sup>

The hydrolysis of urea to ammonia is endothermic and proceeds rapidly above a temperature of approximately 120 °C. The basic chemistry employed in the process is the reverse of

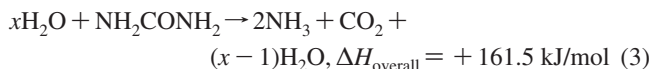
that employed in industrial production of urea from ammonia and carbon dioxide and employs two reaction steps as follows:<sup>1,5–9</sup>



The ammonium carbamate decomposed then to yield carbon dioxide and ammonia gases:



The first reaction in which urea hydrolyzes to form ammonium carbamate is mildly exothermic, while the second, 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. Excess water promotes the hydrolysis reaction, the overall reaction for which is as follows:



The completion of the reaction is favored by high temperature, stirring speed, and high reaction pressure. The overall reaction is endothermic, and the first reaction, that is, urea to ammonium carbamate reaction, is a slow reaction, and the second reaction is very fast and goes toward completion.<sup>8,9</sup>

A literature review indicates that no such rapid urea conversion process is available. Young<sup>10</sup> in patent describes a two-step acidic chemical process for converting urea to produce ammonia that employs polytronic acids, such as H<sub>3</sub>PO<sub>4</sub>. His two-step acid process, however, is substantially more complicated in its equipment requirements and method of operation and is not readily adapted to meet the critical rate of ammonia generation required. Jones<sup>11</sup> also describes the process: urea solution is pressurized to the pressure required to keep urea reaction products in the liquid phase and is contacted for a selected time with a conversion catalyst selected from the group

\* To whom correspondence should be addressed. Tel.: +91 3222 283958(O)/2283959. Fax: +91-3222-282250. E-mail: bcmeikap@che.iitkgp.ernet.in; bcmeikap@iitkgp.ac.in.

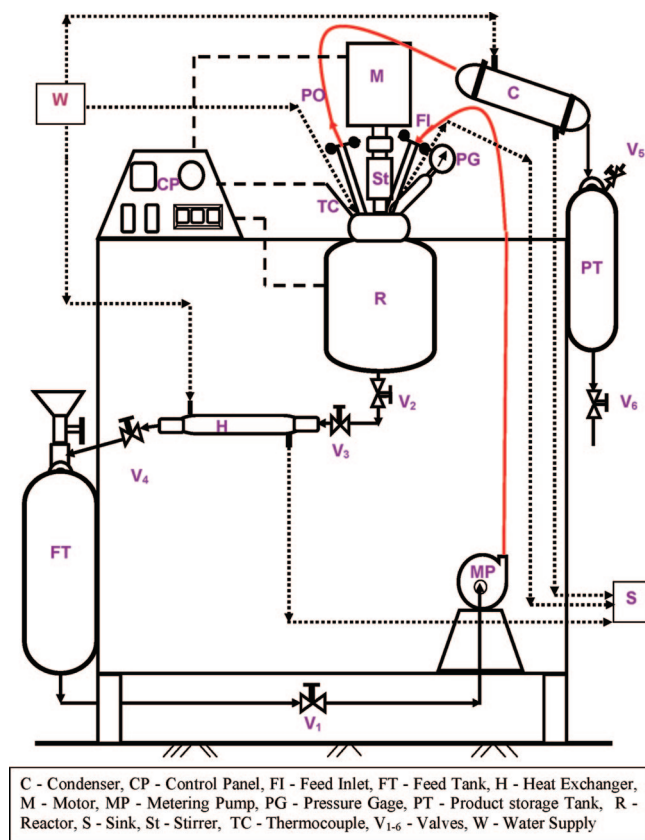


Figure 1. Schematic of experimental setup for urea hydrolysis.

of metals, metal oxides, or metal compounds comprising aluminum, chromium, cobalt, molybdenum, niobium, titanium, tungsten, and vanadium to provide a converted area solution. The actual chemical composition of the catalysts employed is not identified or specified, other than by an elemental name. Salts of vanadium, chromium, and molybdenum are classified as toxic, and their discharge into flue gas streams would be severely restrained from an environmental viewpoint.

The published information in the literature about catalytic hydrolysis of urea for production of ammonia is very little detailed and patented. However, there is no information available in the literature regarding the equilibrium study of catalytic urea hydrolysis for production of ammonia. Therefore, we decided to study more thoroughly the phenomenon of catalytic urea hydrolysis in the presence of fly ash as a catalyst for productions of ammonia for feedstock in different application areas flue gas conditioning and NO<sub>x</sub> reduction, etc., as in our early study without catalyst it shows a very slow rate of reaction.<sup>12,13</sup>

The objective of this work was to study the catalytic hydrolysis of urea for generation of ammonia in a batch reactor. The catalyst used in this study was fly ash, a waste material originating in great amounts in combustion processes. A number of experiments were carried out in a batch reactor at different catalytic doses, temperatures, times, and at a constant concentration of urea solution 10% by weight, and equilibrium and kinetic studies have been made.

## 2. Process

Figure 1 shows a schematic of a urea thermal hydrolyzer setup, and the photographic view is shown in Figure 2. The experimental setup mainly consists of a high pressure (100 kg/cm<sup>2</sup>) reactor, heat exchanger, condenser, pump, feed tank, control panel, and product storage tank. The reactor is of a type



Figure 2. Photograph of the experimental setup of a high pressure reactor.

of continuous stirred tank reactor (CSTR) having a capacity of 2000 cc volume, which was made up of stainless steel-316. A pump was used to feed the urea solution to the reactor at a controlled flow rate against a positive differential head between pump suction and discharge. The shell and tube type condenser was used to exchange heat between products (NH<sub>3</sub>, CO<sub>2</sub>, H<sub>2</sub>O vapor) and cooling water. The system also has a heat exchanger to cool the unreacted urea for recycle to the reactor where tap water at room temperature without any pressure is used for the purpose of cooling. Apart from the above, it also has two storage vessels: one for urea solution, and another for product storage. The whole setup was built in stainless steel to prevent corrosion. The reactor contains two openings: one is for feeding urea solution, and another is for withdrawing the product to condense the gaseous product from the reactor where tap water is used for the purpose of condensation. To measure the temperature and pressure of the reactor, a thermocouple and pressure gauge were attached to the reactor through the control panel. A cooling coil was placed inside the reactor to cool the reactor when required, and the same tap water was also used here as a coolant. There is a control panel to control the required temperature. To supply the necessary heat, an electrical heating coil outside the reactor wall was provided at a controlled rate by means of a control panel. The control panel consists of indicators connected by an electrical wire, mains ON/OFF switch, stirring motor ON/OFF switch with variable speed inverter drive and speed indicator, electrical heater ON/OFF switch, alarm system, fuses, and PID temperature controller. The whole setup is placed on a stand with moving arrangement.

## 3. Experimental Technique

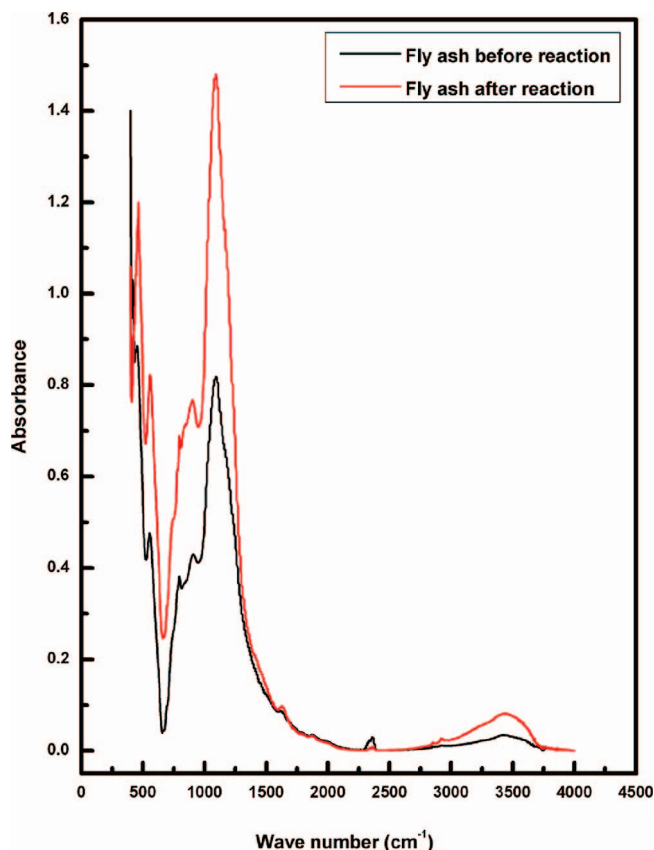
**3.1. Method of Experiment.** An IFFCO-made urea sample was used to conduct the experiment, which has been collected

**Table 1. Chemical Composition of Fly Ash**

component	% elemental composition	% oxide composition
Si	47.71	54.17
Al	26.61	30.08
Fe	7.07	4.57
K	3.43	2.50
Ti	3.06	2.12
Ca	2.73	1.11
Rb	2.35	1.20
Na	2.15	1.02
Mg	1.87	0.91
Ba	1.35	0.65
S	0.49	0.52
others	0.08	0.05
loss on ignition	1.10	1.10

from market. A first urea solution of concentration 10 wt % was prepared. In each case, the volume of solution was taken as 500 mL. The solution of particular concentration was then fed into the feed tank. A metering pump was used to feed the urea solution to the reactor at high pressure at controlled flow rate against a positive differential between pump suction and discharge. The experiments are conducted without stirrer. Heat was supplied by heating an electrical coil outside of the reactor wall at a controlled rate by means of a control panel. The decomposition of urea takes place slowly starting around at 110 °C. As the reaction starts, the product, which is a gaseous mixture of ammonia, carbon dioxide, and water vapor, goes through the condenser. In the condenser, the gaseous product mixture gets condensed where tap water at room temperature without any pressure was circulated through the condenser. The product was then stored in the product storage tank and was collected in a beaker with boric acid solution as it is an absorbing material for ammonia solution. Boric acid solution is prepared by dissolving 4 g of boric acid in 100 mL of warm distilled water. The absorbing ammonia solution was then taken out, and its volume was measured. After it gets absorbed with boric acid, three samples each of 10 mL volume were taken for titration. Three drops of methyl orange indicator is mixed in each sample. It is then titrated with hydrochloric acid. Boric acid is so weak that it does not interfere with acidimetric titration. By comparing the initial concentration and final concentration, the equilibrium conversion was determined. Finally, the rate of reaction was determined from the slope of the line in the graph, which was plotted between time versus concentration data. A graph was plotted between  $\ln(C_A)$  and  $\ln(r_A)$ . The slope and intercept give the value of order of the reaction and rate constant, respectively.

**3.2. Characterization of Urea.** Urea and biuret were detected and quantified using HPLC (high performance liquid chromatography). The HPLC equipment consisted of a Perkin Elmer chromatograph series 200 UV/vis LC detector (Perkin Elmer, USA) equipped with an isocratic pump series 200 and a rheodyne injector with a 20  $\mu$ L loop, and a variable-wavelength ultraviolet/visible detector was used. The equipment was controlled by Total chrom software that controlled the solvent gradient, data acquisition, and data processing. The column was a reversed-phase agilent column. Separation in a C-18 column (250 mm  $\times$  4.6 mm diam), 5  $\mu$ m pore size, and a UV detector set at a wavelength of 199 nm were used. The mobile phase used here 0.1 M phosphate buffer pH 6.7 at flow rate 1 mL/min, and the column temperature was  $25 \pm 1$  °C. HPLC provides the following features: high resolving power, speedy separation, continuous monitoring of the column effluent, accurate quantitative measurement, repetitive and reproducible analysis using the same column, and automation of the analytical procedure and data handling.

**Figure 3.** FTIR result of fly ash before and after hydrolysis reaction.

**3.3. Catalyst: Fly Ash.** The fly ash used in the present study was obtained from the Kolaghat thermal power plant, West Bengal, India. The collected materials were then washed with hot distilled water four to five times to remove the soluble organic matters that may be present in them. The washed materials were then dried in a hot air oven at 105 °C for 24 h. The dried materials were then stored in desiccators for use.

**3.4. Characterization of the Fly Ash. 3.4.1. Pore Structure Characterization.** The pore structure of fly ash was analyzed using  $N_2$  adsorption and scanning electron microscopy (SEM). The BET surface area, total pore volume, and DFT (density functional theory) pore size distribution were determined from nitrogen adsorption/desorption isotherms measured at  $-194$  °C (boiling point of nitrogen gas at atmospheric pressure) by a Quantachrome Autosorb-I. Prior to gas adsorption measurements, the fly ash was degassed at 200 °C in a vacuum condition for a period at least 4 h. The BET surface area was determined by means of the standard BET equation applied in the relative pressure range from 0.05 to 0.3. The total pore volume was calculated at a relative pressure of approximately 0.98, and at this relative pressure all pores were completely filled with nitrogen gas. The DFT pore size distribution of fly ash sample was obtained on the basis of nitrogen adsorption isotherms, using Autosorb software package with medium regularization.

Scanning electron microscopy (SEM) analysis was carried out on the fly ash; SEM images were recorded using JEOL JSM-6300F field emission SEM. A thin layer of platinum was sputter-coated on the samples for charge dissipation during SEM imaging. The sputter coater was operated in an argon atmosphere using a current of 6 mA. The coated samples were then transferred to the SEM specimen chamber and observed at an accelerating voltage of 10 kV, 8 spot size, 4 aperture, and 5 mm working distance.



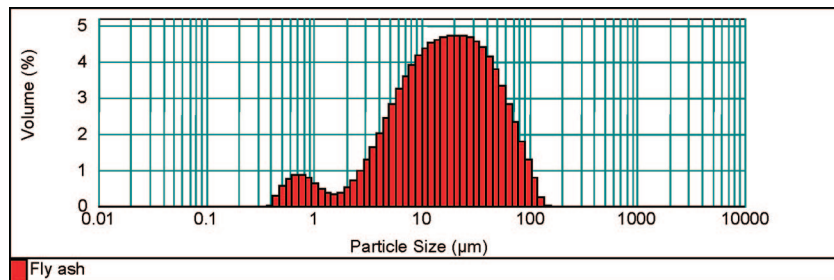


Figure 4. Particle size distribution of fly ash using Malvern Master Size.

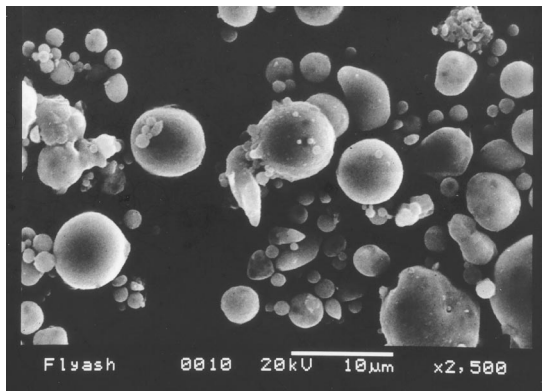


Figure 5. Scanning electron micrograph (2500×) of fly ash.

**3.4.2. Surface Chemistry Determination.** The surface chemistry of the fly ash was determined using Fourier transform infrared radiation (FTIR). FTIR analysis was applied on the same fly ash to determine the surface functional groups, by using FTIR spectroscopy (FTIR-2000, Perkin-Elmer), where the spectra were recorded from 400 to 4000  $\text{cm}^{-1}$ .

**3.4.3. Particle Size Analysis.** Particle size analysis of fly ash samples was done using a Malvern instruments, mastersizer 2000 (UK). Samples were dispersed in water with the help of an ultrasonic magnetic stirrer before feeding into the instrument. Through use of laser diffraction technology, the technique of laser diffraction is based on the principle that particles passing through a laser beam will scatter light at an angle that is directly related to their size. As the particle size decreases, the observed scattering angle increases logarithmically.

**3.5. Reagents.** All of the chemicals used in the study were from Merck (India) Ltd. and Qualigens Glaxo (India) Ltd., analytical grade.

## 4. Results and Discussion

### 4.1. Physical and Chemical Characterization of the Fly Ash.

**4.1.1. Chemical Properties.** Fly ash pH may influence the conversion efficiency. Distinctly acidic catalyst may react with the material to urea and may hamper the surface properties of the catalyst. The pH of fly ash was measured by using the method recommended by Al-Ghouti et al.<sup>14</sup> as follows: 3 g of fly ash was mixed with 30 mL of distilled water and agitated for 24 h. The pH value of the mixture was then recorded with a pH meter. For our experiment, the pH of fly ash was 6.75. The fly ash consists mainly of minerals such as silica, aluminum, iron, magnesium, and calcium. Major oxide of fly ash was analyzed by X-ray fluorescence (XRF) technique. The chemical properties of the fly ash used are shown in Table 1. The spectra of the fly ash were measured by an FTIR spectrometer within the range of 400–4000  $\text{cm}^{-1}$ . The FTIR spectrum obtained for the fly ash after and before reaction plotted was shown in Figure 3.

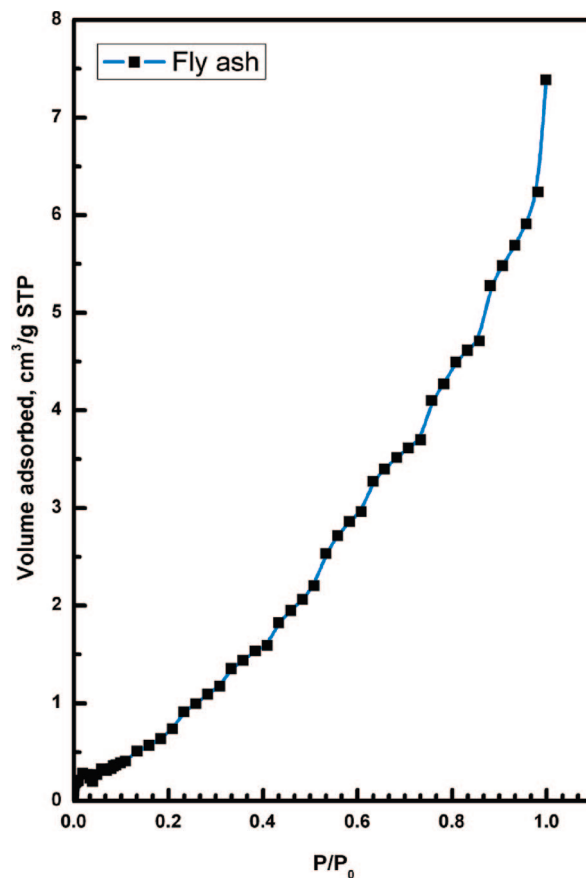


Figure 6.  $\text{N}_2$  adsorption isotherm of fly ash.

**4.1.2. Physical Properties.** Particle size distribution analysis was done using a Malvern analyzer. From Figure 4, it shows that there are no particles above the size of 150  $\mu\text{m}$ . The average bulk density can be calculated by the water displacement method. In this method, the volume of water displaced is observed by a particular amount of fly ash. The average bulk density is 0.8138  $\text{g/mL}$ . Figure 5 shows the scanning electron microscopy (SEM) images of the fly ash.

**4.1.3. Pore Structure Characterization.** The  $\text{N}_2$  adsorption isotherm is shown in Figure 6. The fly ash exhibits type I isotherm. Type I isotherm corresponds to essentially microporous fly ash. The isotherm has characteristics; at low pressure region, significant uptake of nitrogen occurred. This means that nitrogen molecules are adsorbed in the microporous structure. The adsorption in micropores was interpreted according to the pore filling mechanism, and thus results in highly adsorbed volume. Surface area and total pore volume of fly ash are also obtained from the  $\text{N}_2$  adsorption isotherm. The surface area and total pores volume for the fly ash are found to be 5.184  $\text{m}^2/\text{g}$  and 0.0094  $\text{cm}^3/\text{g}$ , respectively. The DFT result given in

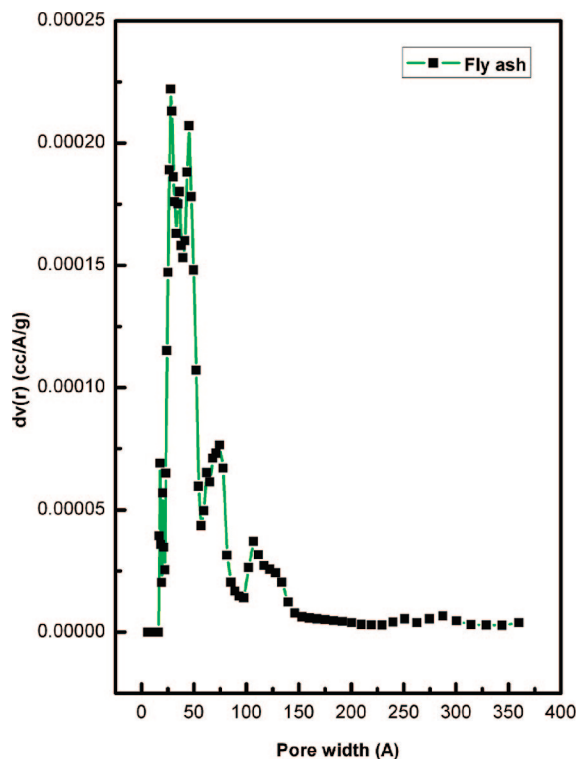


Figure 7. Determination of pore size distribution of fly ash using DFT.

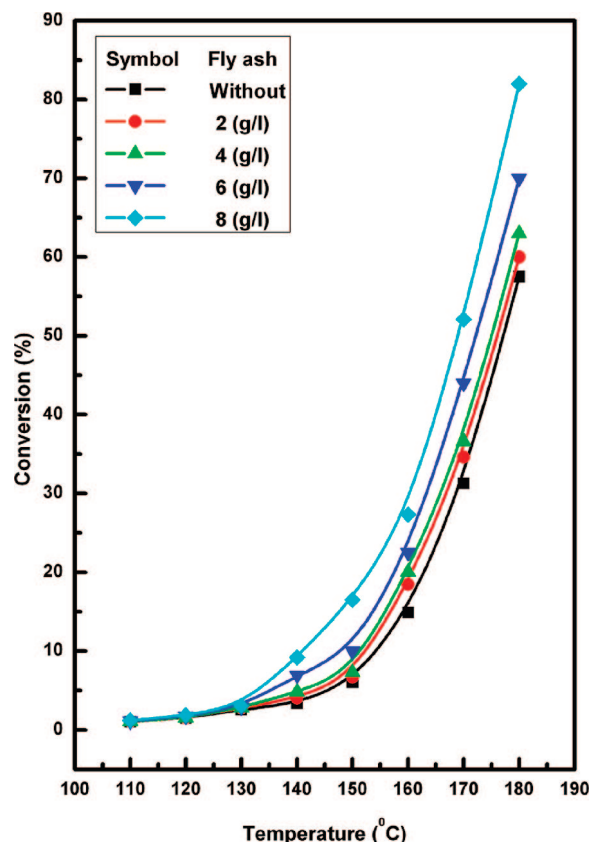


Figure 9. Effect of temperature on conversion of urea at constant initial feed concentration of 10 wt % with different fly ash doses.

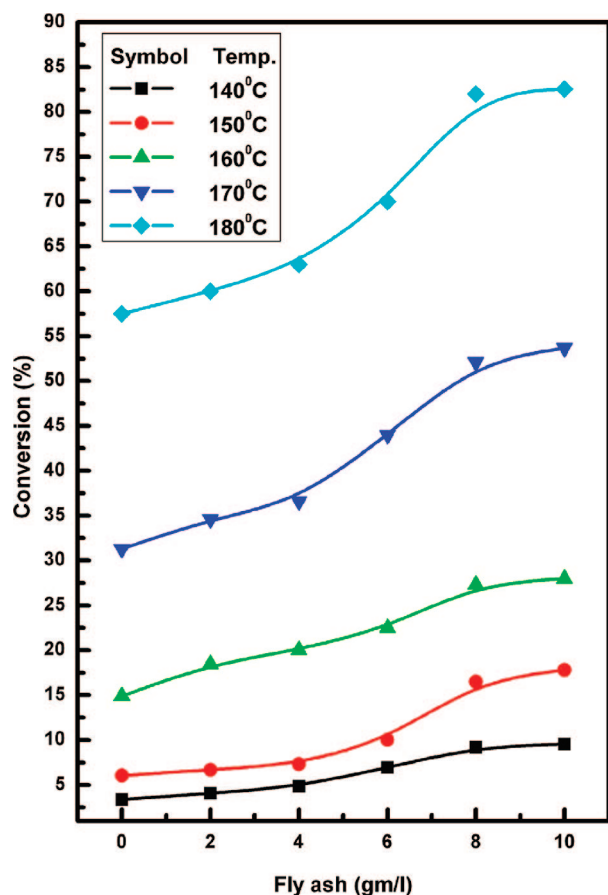


Figure 8. Effect of fly ash doses on conversion of urea at constant initial feed concentration of 10 wt % with different temperatures.

Figure 7 shows that the fly ash consists mainly of micropores of pore width up to 200 Å.

**4.2. Study of Equilibrium Conversion.** Experiment has been conducted for hydrolysis of urea in a batch reactor with the presence of fly ash as a catalyst at different doses of fly ash and temperatures. From the initial concentration and final concentration, the conversion was determined, and the equilibrium study of catalytic hydrolysis has been studied. Here, the effect of equilibrium conversion on doses of fly ash and temperatures has been studied and is shown in Figures 8 and 9.

**4.2.1. Effect of Fly Ash Doses.** The effect of fly ash dosage on the percentage conversion has been shown in Figure 8. It can be seen from the figure that initially the percentage conversion increases very sharply with the increase in doses of fly ash, but beyond a certain value of 8–10 g/L, the percentage conversion reaches almost a constant value. This trend is expected because as the doses of fly ash increase the number of catalytic particles increases, and thus more urea is attached to their surfaces. The conversion capacities increased from 3.38% to 9.53%, 6.04% to 17.8%, 14.89% to 27.98%, 31.3% to 53.68%, and 57.5% to 82.56% at 140, 150, 160, 170, and 180 °C, respectively, with the increase in the fly ash doses from 0 to 10 g/L at a constant initial feed concentration of 10 wt %. A maximum conversion of 82.56% was observed at fly ash dosage of 10 g/L at 180 °C for an initial feed concentration of 10 wt %.

**4.2.2. Effect of Temperatures on Conversion.** Experiments were performed at different temperatures ranging from 110 to 180 at 10 °C temperature intervals with a constant initial feed concentration of 10 wt % with different doses of fly ash. It can be seen from Figure 9 that the conversion is a function of temperature. It increases exponentially with increase in temperature. For 2 g/L doses of fly ash, the conversion increases from 1.07% to 60% when temperature increases from 110 to

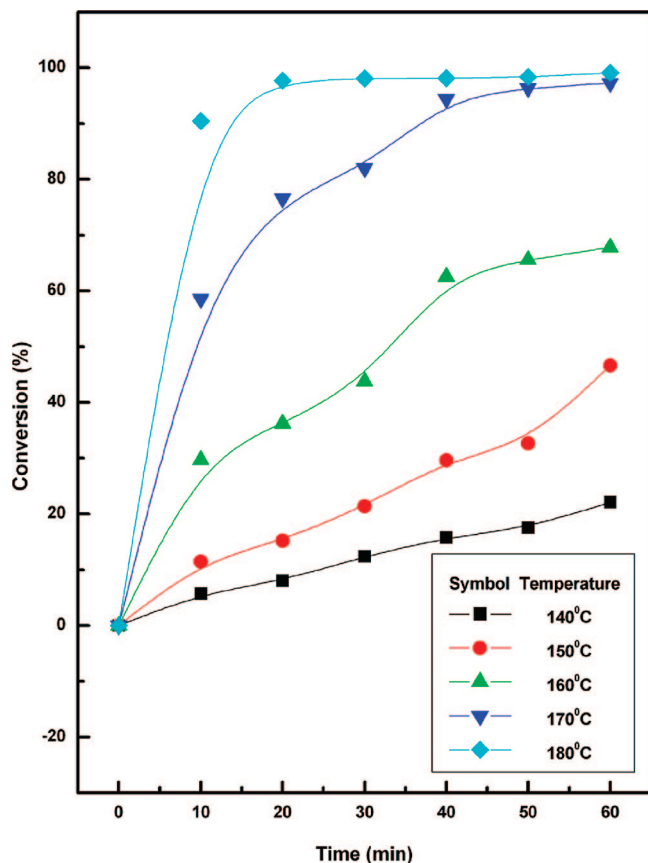


Figure 10. Effect of time on conversion at different temperature for constant initial feed concentration 10 wt % solution.

180 °C in 10 °C intervals. Similarly, a trend has been observed for 4, 6, and 8 g/L doses of fly ash solution, and the conversions are 63%, 70%, and 81.98%, respectively, at 180 °C. Also, it was observed that the initial reaction rate is slower, and it becomes rapid at around 140 °C. At this temperature, the production of ammonia is higher than that at lower temperature.

**4.3. Study of Reaction Kinetics.** Experiments have been conducted for hydrolysis of urea in a batch reactor at different temperature, reaction pressure, and concentration, and a kinetic study has been done. From the initial feed concentration and final concentration, the conversion was determined, and the kinetic study of hydrolysis reaction has been studied. Assuming no backward reaction, the rate constant for forward reaction is calculated. As in the reaction, excess water is used, and the concentration of water is neglected. Let  $-r_A$  be the rate of reaction,  $C_{A0}$  be the initial concentration, and  $C_A$  be the concentration at any time. The rate of reaction for forward reaction can be written as

$$-r_A = \frac{-dC_A}{dt} = k(C_A)^n \quad (4)$$

Multiplying  $\ln$  on both sides in eq 4, we have:

$$\ln(-r_A) = \ln(k) + n \ln(C_A) \quad (5)$$

Hence, if a graph is plotted taking  $\ln(C_A)$  on abscissa and  $\ln(-r_A)$  on ordinate, a straight line will come whose y-intercept is represented by a rate constant.

**4.3.1. Effect of Times on Concentration.** From the concentration time data, a kinetic study has been done at a constant fly ash dose of 8 g/L. Figure 10 depicts that the concentration of urea decreases as time increases at a constant temperature; also, it depicts that the higher temperature possesses less

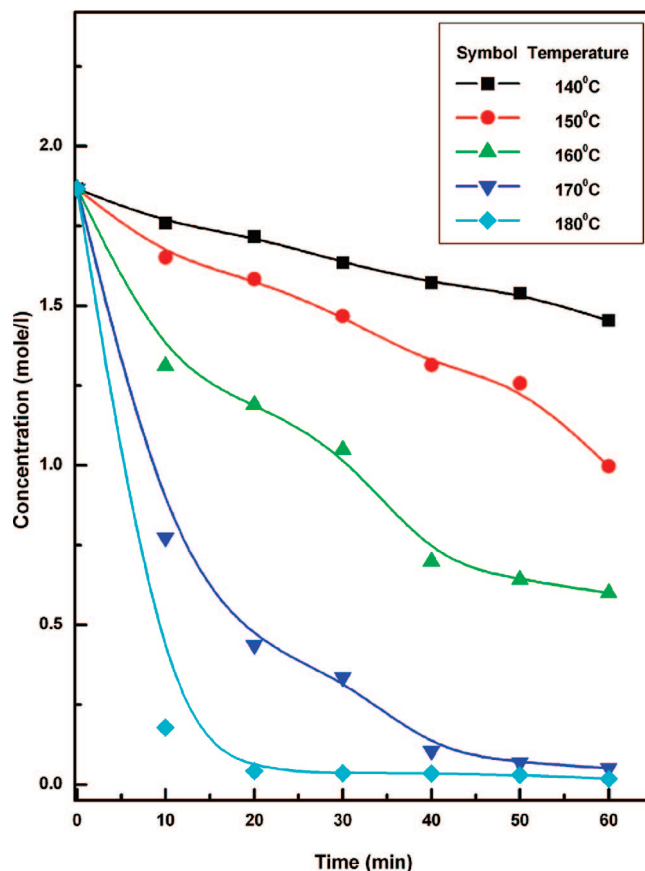


Figure 11. Effect of time on concentration at different temperature.

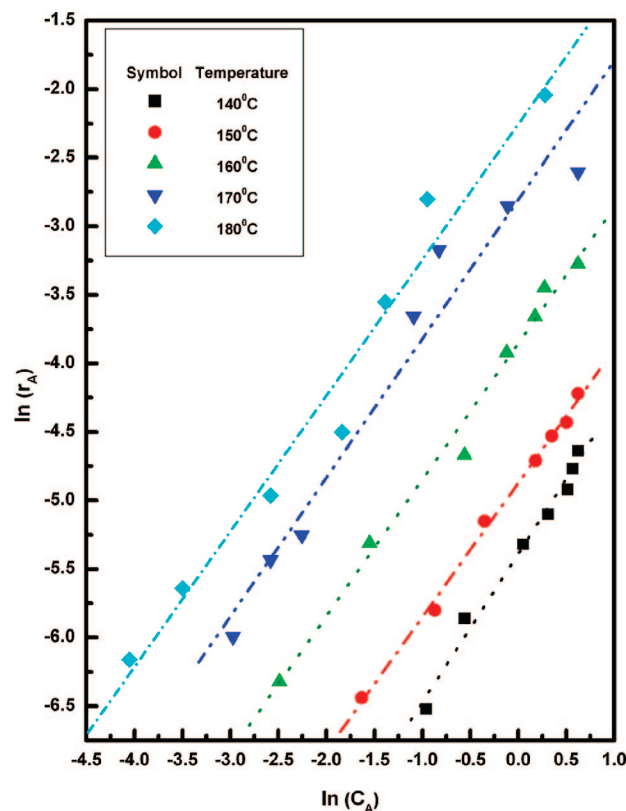


Figure 12. Effect of concentration on reaction rate at different temperature.

concentration of urea as compared to low temperature. It was observed from Figure 11 that the conversion increases with times. For 10 wt % initial concentrations, the conversion reaches

**Table 2. Kinetic Data for Urea Hydrolysis in the Presence of Fly Ash as a Catalyst**

temperature in °C	$k$ (min <sup>-1</sup> )	$n$ (calculated)	$n$ , average	$R^2$
140	0.00457	1.085	1	0.984
150	0.00764	0.977		0.996
160	0.02120	0.994		0.987
170	0.06044	1.013		0.953
180	0.10483	0.989		0.971

from 0% to 99% at a constant temperature of 180 °C when time increases from 0 to 60 min, so 60 min is required to achieve 99% conversion for 10 wt % solution at 180 °C.

The effect of rate on concentration at a fixed temperature is shown by Figure 12. From the figure, it is observed that the slope of the plot is approximately equal to one. This indicates that the forward reaction is first order type. Further, the intercept of the figure gives the value of forward rate constant, which is a function of temperature. It is observed from the figure that with increases in temperature from 140 to 180 °C, the forward rate constant increases from 0.00457 to 0.10483 min<sup>-1</sup>. The variation with temperature is very high because of the fact that there was stirring and catalyst. Table 2 shows the values of rate constant with temperature.

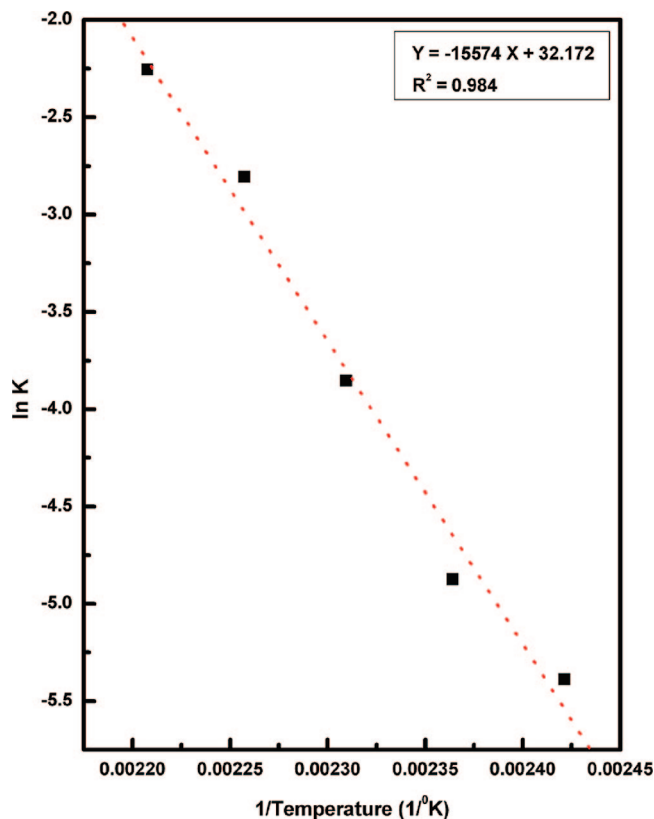
From the above result, it is observed that the order of the forward reaction is close to one, and with an increase in temperature the rate constant increases. So finally, the temperature dependency of forward rate constant was explained by the Arrhenius theory. The rate equation can be written as follows:

$$-r_A = kC_A \quad (6)$$

where  $-r_A$  is the reaction rate,  $k$  is the forward rate constant, and  $C_A$  is the concentration of urea solution.

According to the Arrhenius theory, the temperature dependency of the forward rate constant can be written as

$$k = Ae^{\frac{-E}{RT}} \quad (7)$$

**Figure 13.** Effect of temperature on rate constant.

where  $A$  is the frequency factor and  $E$  is the activation energy.

From Figure 13, the activation energy and frequency factor were determined as 129.48 kJ/mol and  $9.378 \times 10^{13}$  min<sup>-1</sup>, respectively.

## 5. Conclusions

The objective of this work was to study the catalytic hydrolysis of urea in the presence of fly ash as a catalyst for generation of ammonia in a batch reactor. A number of experiments were carried out in a batch reactor at different temperatures, taking different concentrations of fly ash doses from 2 to 10 g/L, and both an equilibrium and a kinetic study have been made. From the equilibrium study, it is concluded that with an increase in temperature and fly ash doses, the equilibrium conversion increases, and a maximum conversion of 99% is possible with 10 wt % concentration of feed solution and at 180 °C with 10 g/L fly ash dose. Also, it can be concluded that the initial reaction rate is slower, and it becomes rapid at around 140 °C. At this temperature, the production of ammonia is higher than that at lower temperature. It was observed that initially the percentage conversion increases very sharply with the increase in doses of fly ash, but beyond a certain value of 8–10 g/L, the percentage conversion reaches almost a constant value, so the optimum condition of fly ash doses is 8 g/L. Further, the reaction rate constant is a function of temperature. It has also been found that the forward reaction is a pseudo first-order reaction with a rate constant that varies from 0.00457 to 0.10483 min<sup>-1</sup> within the temperature range of 140–180 °C, respectively. The activation energy and frequency factor of urea hydrolysis reaction are found to be 129.48 kJ/mol and  $9.378 \times 10^{13}$  min<sup>-1</sup>, respectively. Finally, it can be concluded that the catalytic hydrolysis of urea to form ammonia and carbon dioxide behaves as a first-order reaction with respect to urea.

## Acknowledgment

We would like to gratefully acknowledge the financial support that the National Thermal Power Corp. (NTPC), New Delhi, India, has extended toward this work.

## Nomenclature

- $A$  = frequency factor (min<sup>-1</sup>)
- $C_A$  = final concentration of urea solution (mol/L)
- $C_{A0}$  = initial concentration of urea solution (mol/L)
- $E$  = activation energy (kJ/mol)
- $k$  = forward rate constant (min<sup>-1</sup>)
- $n$  = order of forward reaction, dimensionless
- $R$  = ideal law gas constant (kJ/kg·mol·K)
- $r_A$  = rate of reaction
- $T$  = reaction temperature (K)
- $t$  = reaction time (min)

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