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CHARACTERISTICS OF DEPOSIT FORMATION IN SCR SYSTEM FOR VARYING TEMPERATURE AND FLOW RATE OF EXHAUST GASES -A STUDY USING XRD

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ABSTRACT

To mitigate environmental pollution due to NO_x emitted from the exhaust of diesel engines, the automobile manufacturers are adopting a technique called "Selective Catalytic Reduction (SCR) System" for modern automobiles. The reducing agent NH₃ is generated by spraying Urea-Water Solution (UWS). When exhaust temperature is low, the overdosing of UWS is necessary; but it results in deposits of urea and its by-products. The temperature and Reynolds number have significant effects on ammonia generation and urea deposits. In the present work, an investigation is done for deposit formation considering temperature and flow rate as variables. The results show that the urea deposits obtained at open reactor vessel and in hot air test bench are different with their crystallinity, quantity and type. However, EDAX indicates urea as a major constituent in the deposits at low exhaust temperatures.

KEYWORDS: SCR, Deposit Formation, Temperature, Flow Rate, XRD

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1. INTRODUCTION

The emission of nitrogen oxides (NO_x) from the exhaust of diesel engines can be reduced by a technique called Selective Catalytic Reduction (SCR). In SCR, of Urea Water Solution (UWS) (32.5% aqueous urea solution) is injected in the form of an atomized spray to obtain reducing agent ammonia (NH₃). The UWS transforms into solid urea and water vapour. Solid urea undergoes thermolysis and produces NH₃ and HNCO. HNCO further reacts with water vapour and, gives CO₂ and NH₃ [1]. The reduction of NO_x takes place over the catalyst through various favourable reactions [2, 3]. Even though SCR technique is more promising, the major drawback associated within it is deposit formation which is a challenge faced by OEMs [3]. As urea deposits deplete, there will be changes in the stoichiometric NH₃/NO_x ratio which leads to NH₃ slip to environment when exhaust gas temperature increases. The operating conditions like engine exhaust temperature, flow velocity, spray characteristics such as UWS droplet velocity, diameter, injector cone angle, spray pattern etc. were found to have impact on deposit formation significantly [4]. Air assisted urea injection and mixers placed in the flow stream of the exhaust system were found to be effective in reducing deposits and also to accelerate UWS decomposition [5, 6, 7, 8]. More details on urea decomposition and deposit formation are discussed in the following sections.

2. DEPOSIT FORMATION IN SCR SYSTEM

2.1. Urea Decomposition Mechanism

The chemical composition of deposits in the Urea-SCR system is different compared to original urea. The remnant urea undergoes different treatments and changes with its crystallinity [9]. After the evaporation of water from UWS, pure solid urea is continually heated, with which it goes through stages of decomposition. According to the literature, urea melts at 130°C and decomposes at about 152°C [10]. At temperatures above 150°C, about 20% of the mass transforms to gaseous NH_3 and HNCO . At 150-225°C, the remaining solid urea converts into Biuret (formed due to prior reaction of cyanic acid, HNCO , with intact urea) according to pyrolysis reaction and the quantity nearly to be 30%. 66% of urea mass converts to gaseous forms when solid reaches 225°C. Beyond 225°C, there is an increasing amount of Cyanuric acid, Ammelide, Ammeline and Melamine production which are more difficult for removal. Cyanuric acid (CYA) requires above 350°C before it begins to form NH_3 [4, 5, 11]. However, these characteristics are different when UWS is injected to flowing hot exhaust gas and wall interaction of droplets takes place.

There can be some more side reactions that might occur in gas phase during the flow of exhaust gas. The Isocyanic acid produced in the first step of the urea decomposition may react with unreacted urea forming Biuret, Triuret and Melamine (Figure 1) which are likely to contribute for deposit formation [7,12]. The conversion of melamine to oligomeric species is possible transformation of the solid residue during urea thermolysis [13, 14]. To characterize the chemical composition of urea deposits formed in Urea-SCR system over a wide range of operating temperatures, it is essential to study the depletion characteristics.

2.2. Types of Urea Deposits and Effect of Exhaust Gas Temperature on Deposit Formation

The major reason for deposit formation is non-ideal behavior of urea decomposition like incomplete decomposition of Isocyanic acid (HNCO), Conversion of Isocyanic acid to form Cyanuric acid(CYA) and various other reactions [15, 7]. CYA is primarily a result of urea decomposition at temperatures in excess of 152°C. Biuret is a product that forms when HNCO reacts with undecomposed urea. Above 190°C, biuret decomposes into CYA. Ammelide and Ammeline are produced primarily from CYA and Ammelide appear initially at approximately 175°C, but the rate of reaction is slow [11]. The route map of formation of by-products of urea is indicated in Figure 1.

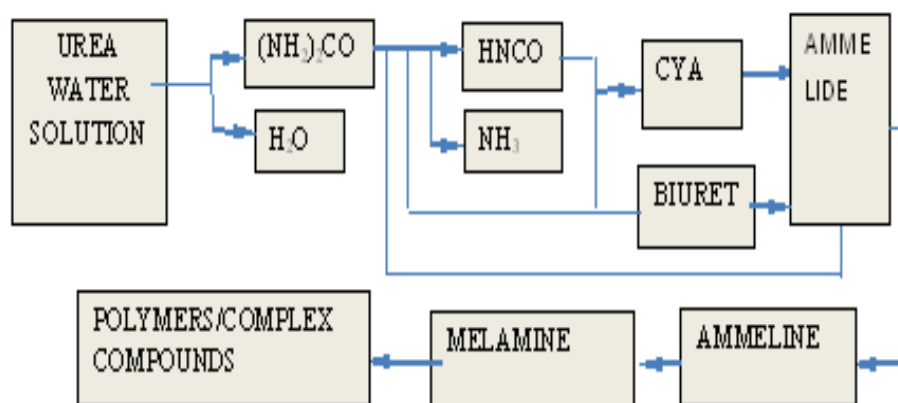


Figure 1: Reactions and By-products Involved in Urea Thermolysis [11].

2.3. Studies on Urea Decomposition Characteristics

Thermo Gravimetric Analysis (TGA) is a method used by some researchers to know the chemical variation of urea during heating [16]. Thermal decomposition of urea in an open vessel at different temperatures showed that a variety of decomposition products by undesirable ways can form at different temperatures [17]. An observation of urea depletion involves 3-stage urea decomposition. In the first stage, i.e. room temperature to 190⁰C, only urea decomposition predominates with Biuret as a major by-product and Ammelide in a slower rate. In the second stage, i.e. 190–250⁰C, urea decomposition continues and Biuret decomposition starts forming. The formation of CYA and Ammelide increases during this stage. The Melamine and Ammeline start forming in smaller amounts. Sublimation of all the products take place after they reach their maximum level by mass. In the third stage, i.e. 250–360⁰C, sublimation and decomposition takes place simultaneously. Above 360⁰C, urea sublimates directly and depletion of rest of the mass takes place. In laboratory environment, heating of solid urea sample is done in a gas chamber gradually. The urea residue monitored closely and then gas components are analysed to evaluate various species [11, 18].

2.4. Need of Studies on Deposit Formation and Characterization of Deposits

By the review of literature, it is found that limited studies were done on decomposition characteristics of urea in flowing exhaust gas at low temperatures. Further, different studies are done which include Gravimetric Analysis, deposit characterization and depletion characteristics of by-products under certain operating conditions [19]. Analytical investigation using HPLC, DSC, etc. are done at some particular operating conditions of an engine [17, 20]. Some more studies in literature [19, 21, 22], show investigations have been done on some operational points. The deposits formed in SCR are heat transfer dependent which is decided by forced convection variables like velocity and temperature. So, the objective of present work is to characterize the deposits at various temperatures and flow conditions which helps in deciding dosage strategy by knowing NH₃ leakage to environment during depletion of deposits. In our study, the deposits formed at temperature range 150-250⁰C for 3 different flow rates 34.74 kg/hr, 86.86 kg/hr and 121.6kg/hr are considered for structural changes and characterization.

3. EXPERIMENTAL PROCEDURE

3.1. Experimental Setup

Many researchers [23, 24] conducted tests in a hot air test rig and obtained the results equivalent to results of actual SCR unit with an error less than 10%. So, hot air generated from heater- blower setup (Figure 2) is used instead of actual engine exhaust with a flow rate 0-500kg/hr and temperature is sensed using three thermocouples. Deposit collection is done using stainless foils steel (SS). Test conditions are: 1) 150⁰C-34.74, 86.86, 121.60 kg/hr 2) 200⁰C-34.74, 86.86, 121.60kg/hr 3)250⁰C-34.74, 86.86, 121.60kg/hr. Time of experiments is 20 minutes and UWS dosage is 72(mg/sec).Collected deposits are tested in XRD as per procedure given in Appendix.

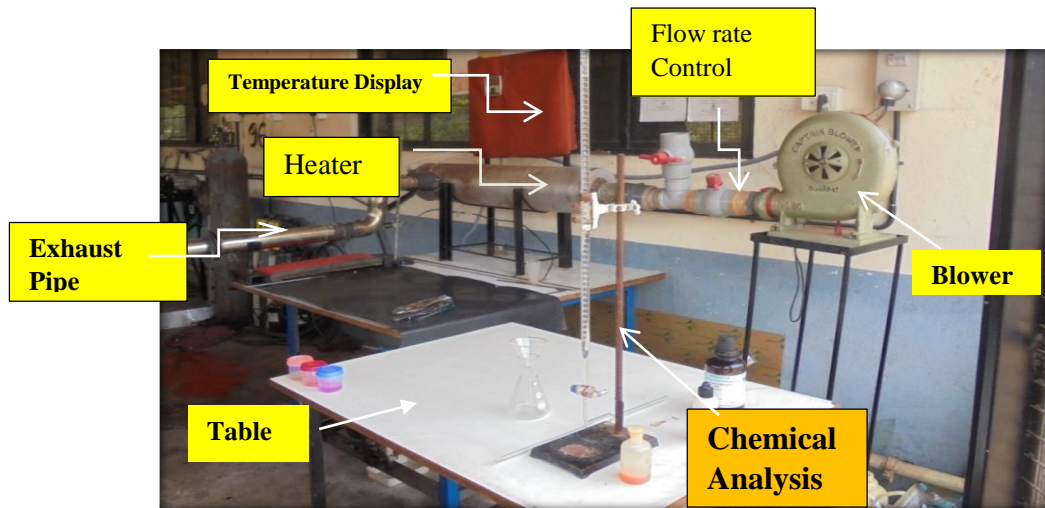


Figure 2: Experimental Setup for Deposit Formation Study.

4. RESULTS AND DISCUSSION

4.1. Structural Changes Deposits with Flow Rate and Temperature

The structural changes of deposits are observed physically for the different cases with variations in temperature and flow rates. When temperature is low (150°C), urea from UWS droplet segregates and forms deposits of solid form. When flow rate is low, deposits form near the injection zone and it propagates as flow rate increases. At higher temperature 200°C , the amount of deposit is remarkably low. At lower flow rate 34.74kg/hr , the solid urea is segregating in spray zone, whereas the liquid urea extends towards the end of foil (Figure 3(c)). For increased flow rates, the deposits disintegrate and they are more prone to evaporate or sublime or undergo pyrolysis (Figure 4(d)).

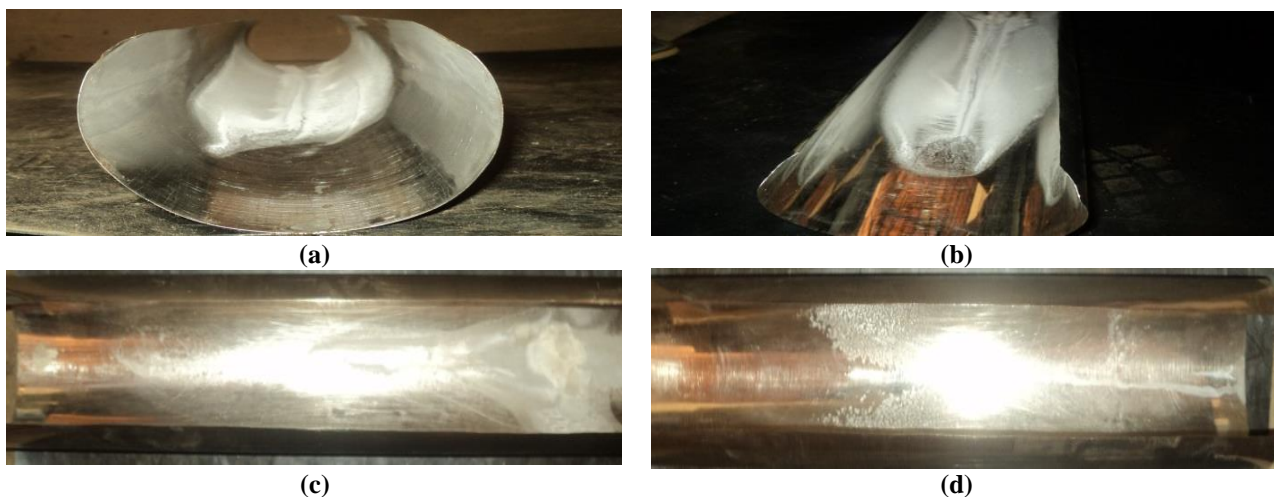


Figure 3: Experimental Observations of Deposit Formation (Right) Regions for (a) 150°C - 34.74 kg/hr , (b) 150°C - 121.64kg/hr , (c) 200°C - 34.74 kg/hr , (d) 200°C - 121.6 kg/hr , after 20 Minutes of UWS injection.

4.2. Identification of Variation in Crystallinity and Identification of By-products of Urea

The details of recording and processing of X-ray signal are very well dealt in literature [25]. The detection of compounds like Cyanuric acid (CYA), Biuret, Ammeline, Ammelide and Triuric acid is paramount as they have different depleting

characteristics compared to original urea [17, 19]. The standard JCPDS files were referred and peaks were compared starting from the highest intensity peaks of detection compounds.

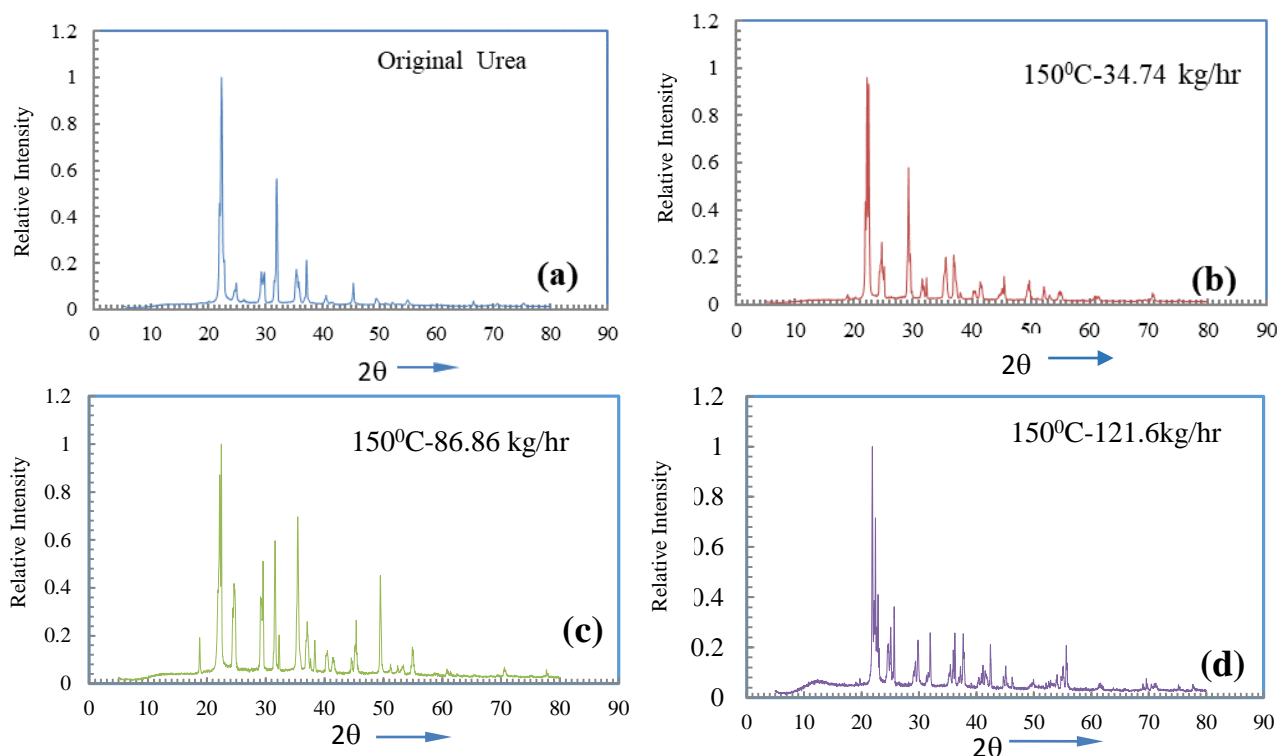


Figure 4: X-Ray Diffraction Pattern for (a) Original Urea (b) 34.74 kg/hr (c) 86.86 kg/hr (d) 121.6 kg/hr.

4.2.1. Effect of Flow Rate on Characteristics of Deposits at 150°C

Figure 4 (a) represents the relative intensity vs “2θ” of X-ray diffraction studies on original urea. Figures 4(b-d) represent the X-ray diffraction pattern of samples tested at 150°C for different flow rates as mentioned. Studies reveal that at 150-170°C the liberation of NH₃ leads to change in composition of residual urea. Likewise, it is evident from XRD results that the some peaks are deviating from that of original urea. Figure 4(b) shows the spectra of condition of flow rate of 34.74kg/hr. Additional peaks are observed as indicated. The peak lists corresponding to this conditions are at angles, 22.15°, 24.67°, 25.1°, 29.10°, 32.29°, 35.48°, 36.9°, 37.09°, 45.47°. According to it, most of the peaks resemble the original urea by observation. Additionally, we could see peaks for 25.01°, 36.9° and 45.47°. This indicates the change in crystallinity. As the flow rate increases, the amount of heat transfer to droplets and to solid urea increases due to increased dispersion of the droplets.

Figure 4(c) shows spectra of condition 150°C-86.86kg/hr. The peaks observed are at angles 18.77°, 22.18°, 22.47°, 24.79°, 29.56°, 31.63°, 35.43°, 38.35°, 49.5°, 54.90°. Additionally, we could see peaks for 38.35°, 49.5° which indicate the change in crystallinity. Figure 4(d) reveals the XRD spectra for the condition 121.6kg/hr. Peaks are observed at angles 22.21°, 22.42°, 22.63°, 22.88°, 23.05°, 24.62°, 25.09°, 25.67°, 29.83°, 31.94°, 35.97°, 36.23°, 37.70°, 37.86°, 42.43°, 55.03° and 55.64°.

Comparing all XRD patterns of earlier conditions, the additional peaks represent there are further changes in crystallinity of urea deposits as the flow rate increases. It is evident that at low temperatures, the urea decomposition can follow 3 different routes as shown in Figure 1. The samples are collected at three different flow rates at 150°C and checked for the presence of

various urea-derived compounds. Table 1 gives the JCPDS file name, 2θ (the highest peaks (degrees)), corresponding d-spacing and presence of various by-products for 3 different flow rates.

Table 1. Comparison of presence of various urea related compounds in the samples tested at 150°C.

Compound	JCPDS File No	' 2θ '	'd' A°	34.74kg/hr	86.kg/hr	121.6kg/hr
Ammelide	00-031-1527	27.88	3.197	No	No	No
Ammeline	00-031-1526	27.94	3.19	No	No	No
Biuret	00-011-0720	11.41	7.75	No	No	No
CYA	00-023-1637	29.78	2.998	Marginal	Yes ,Marginal	Yes, relatively higher intensity
Melamine	00-024-1654	26.2	3.399	No	No	No
Triuret	01-072-1631	28.52	3.127	No	No	No
Urea	00-008-0822	22.15	4.010	Major constituent	Major constituent	Major constituent

It is observed that urea is the major constituent in all samples tested at temperature 150°C. The intermediate route of formation of Biuret [16, 1] is not found for any of the flow conditions. An intermediate compound Cyanuric acid is found for all conditions with varying intensities.

4.2.2. Effect of Flow Rate on Characteristics of Deposits at 200°C

The urea related compounds formed are analytically estimated by several researchers and they revealed that decomposition characteristics are completely different by the type of compounds formed [20]. Schaber et al.[11] found that at temperatures in excess of 190°C, Cyanuric acid(CYA), Ammelide and Ammeline are produced primarily from Biuret. But these compounds are obtained when tests are done at the open reactor vessel wherein the effect of air flow rate is not considered. The deposits obtained at 200°C at 3 different flow rates are compared with the original urea in present study.

The variation of XRD peaks in comparison with the original urea sample can be seen in Figures 5(a-d). Schaber et al.[11] revealed that at temperatures in excess of 190°C, alternate reactions involving the decomposition of Biuret substantially increases the decomposition. But, the presence of Biuret is found for higher flow rates 86.86 kg/hr and 121.6 kg/hr(Table 2). The presence of Biuret is found by XRD comparison with standard XRD obtained from JCPDS file of high temperature Biuret. So, it is inferred that at higher flow rates, Biuret forms; but at low flow rates, only urea depletion takes place. The variations in crystallinity given in Figures 5 (a-d).

The Table 2 gives 2θ values and d-spacing of various compounds and occurrence of them for various flow rates. It indicates the decomposition products like CYA found at lower flow rates. Biuret and its by-products like Ammelide and Ammeline are also observed at higher flow rates.

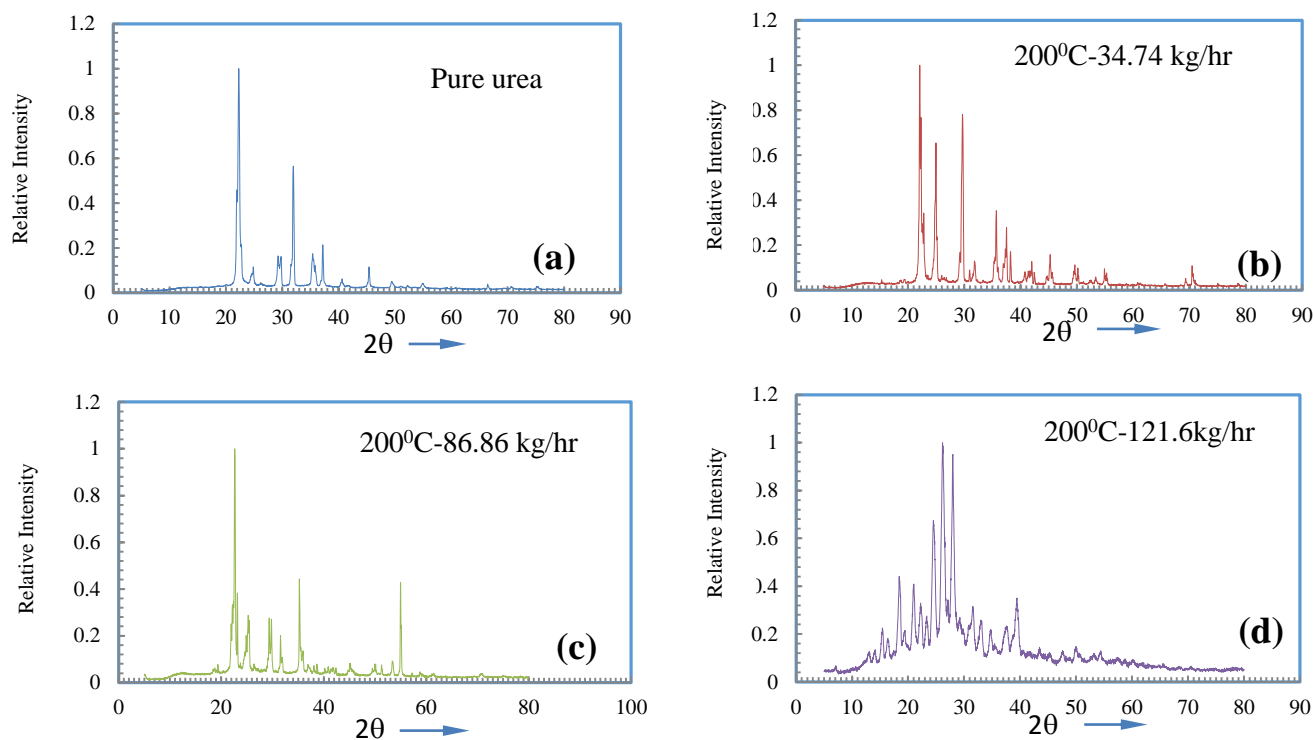


Figure 5: X-Ray Diffraction Pattern for (a) Original Urea (b) 34.74 kg/hr (c) 86.86 kg/hr (d) 121.6 kg/hr.

Table 2. Comparison of presence of various urea related compounds in the samples tested at 200°C.

Compound	'2θ'	'd' Å'	34.74kg/hr	86 kg/hr	121.6 kg/hr
Ammelide	27.88	3.197	No	Yes	Yes
Ammeline	27.94	3.19	No	Yes	Yes
High temp. Biuret	11.41/39.31	2.29	No	Yes, relatively higher intensity	Yes
CYA	29.78	2.998	Yes, higher intensity	Yes, relatively higher intensity	Yes, with lower intensity
Melamine	26.2	3.399	No	No	Yes
Triuret	28.52	3.127	No	No	Yes, higher intensity
Urea	22.15	4.010	Major constituent	Major constituent	Major constituent

The study of nature of deposits revealed that at temperature in excess of 200°C, deposits are of solidified form of urea melt. The effect of increased temperature results in the formation of by-products like Ammeline, Ammeline, etc. At lower flow rate 34.74kg/hr, it is found that urea is a major constituent and Cyanuric acid is one among the by-products. Usually, Biuret is the immediate by-product at temperatures above 170°C. However, Biuret is not observed here. At lower flow rates, the alternate route of urea decomposition without the occurrence of Biuret is observed (Figure 1). Once the flow rate increases, the effect of flow rate is substantially observed with the presence of Biuret. At higher flow rates of 121.6 kg/hr, along with Cyanuric acid and Biuret, Triuret is also observed. It is evident from Table 2, that the by-products of urea decomposition take different routes. By overall, results indicate the presence of Ammelide, Ammeline and Biuret for higher flow rates. However, Biuret is not observed in the samples tested at lower flow rates. The same sample is further investigated for other by-products. The presence of Cyanuric acid is observed at all flow rates.

4.2.3. Effect of Flow Rate on Characteristics of Deposits at 250°C

From earlier Figures 4 (b-d) and Figures 5(b-d) the characteristics of deposits at 150°C and 200°C are different from that at the open reactor conditions. The by-products form relatively faster at higher exhaust gas flow rates due to heat transfer by forced convection. If the temperature is increased further up to 250°C, the production of CYA, Ammeline and Ammelide appear before 250°C [11]. The samples obtained at 250°C at different flow rates are tested for different by-products. Characterization indicates the change in the crystallinity (Figure 6) and most of these by-products are observed at all flow rates. Melamine is observed at lower flow rates and urea decomposition is not complete even at 250°C (Table 3).

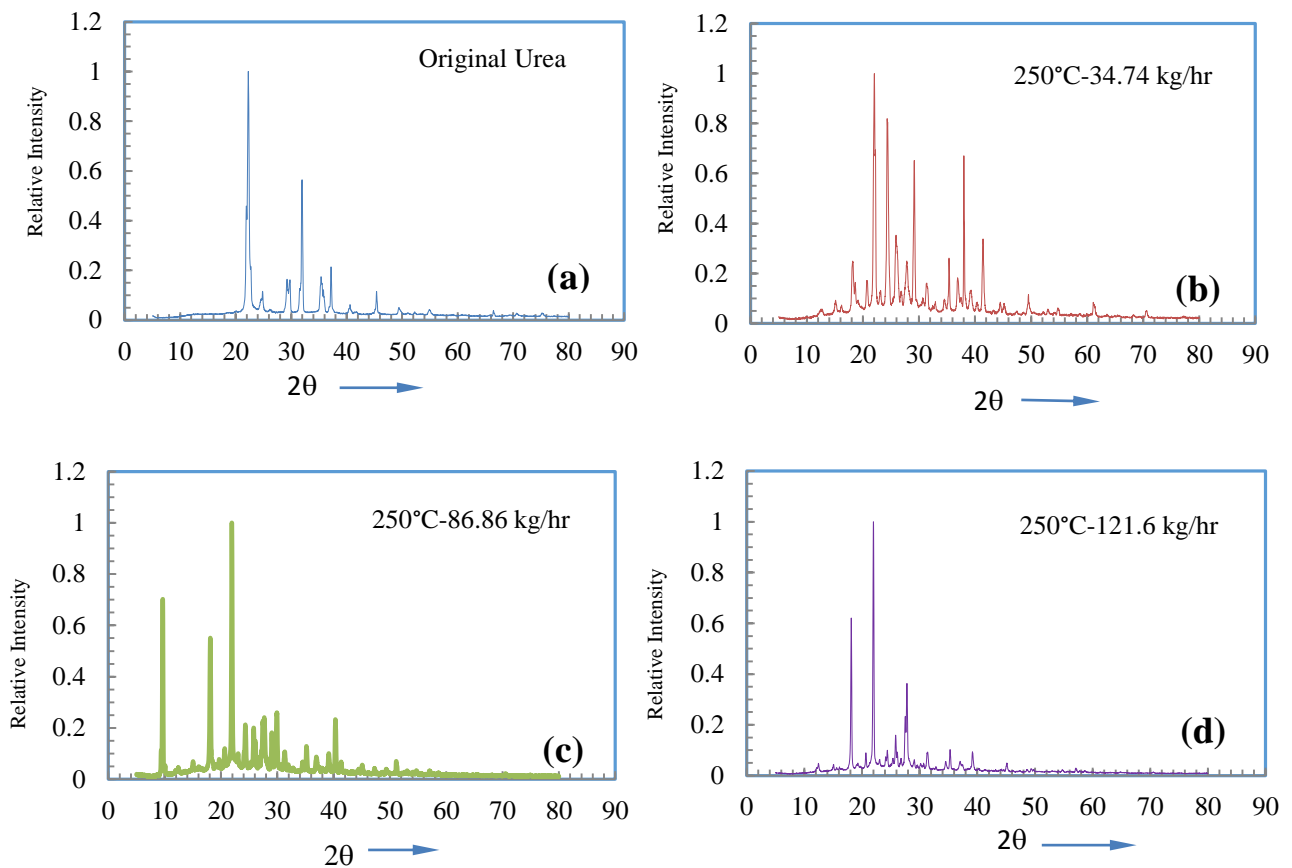


Figure 6: X-ray diffraction patterns for (a) Original urea (b) 34.74 kg/hr (c) 86.86 kg/hr (d) 121.6 kg/hr.

Table 3. Comparison of presence of various urea related compounds in the samples tested at 250°C.

Compound	'2θ'	'd' A°	34.74kg/hr	86.kg/hr	121.6kg/hr
Ammelide	27.88	3.197	Yes	Yes	Yes, Strong peak
Ammeline	27.94	3.19	Yes	Yes, with lower intensity	Yes, with lower intensity
High temp Biuret	11.41/39.31	2.29	Yes	Yes, with lower intensity	Yes, with lower intensity
CYA	29.78	2.998	Yes	Yes, with lower intensity peaks	Yes, with lower intensity peaks
Melamine	26.2	3.399	Yes, with lower intensity	No	No
Triuret	28.52	3.127	Yes, with lower intensity	Yes, with lower intensity	Yes with lower intensity
Urea	22.15	4.010	Yes, with some additional peaks	Yes, with some additional peaks	Yes peaks are shifting from actual urea

By overall, the intermediate routes of formation of Biuret are not found in some flow conditions. The elemental analysis done for different samples using EDAX indicates that urea as a major deposit as constituent elemental concentrations of carbon, nitrogen and oxygen comparable to that of pure urea in all cases. So, the major constituent is urea, even if it is treated at elevated exhaust temperatures up to 250°C.

CONCLUSION

The deposits obtained from the tests at varying temperature and flow rate conditions in hot air test rig are collected and characterized using XRD for identification of various by-products of urea. The characteristics of the deposits found to be different for the samples at open reactor vessel and hot air test bench. It is observed that both temperature and flow rate have substantial effect on deposit formation and depletion characteristics. EDAX indicates urea as a major deposit as concentrations of carbon, nitrogen and oxygen comparable to that of pure urea.

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APPENDIX

Sample Preparation for XRD Study

- Obtain a few tenths of a gram (or more) of the material, as pure as possible
- Grind the sample to a fine powder, typically in a fluid to minimize inducing extra strain (surface energy) that can offset peak positions, and to randomize orientation. Powder less than $\sim 10\ \mu\text{m}$ (or 200-mesh) in size is preferred
- Place into a sample holder or onto the sample surface

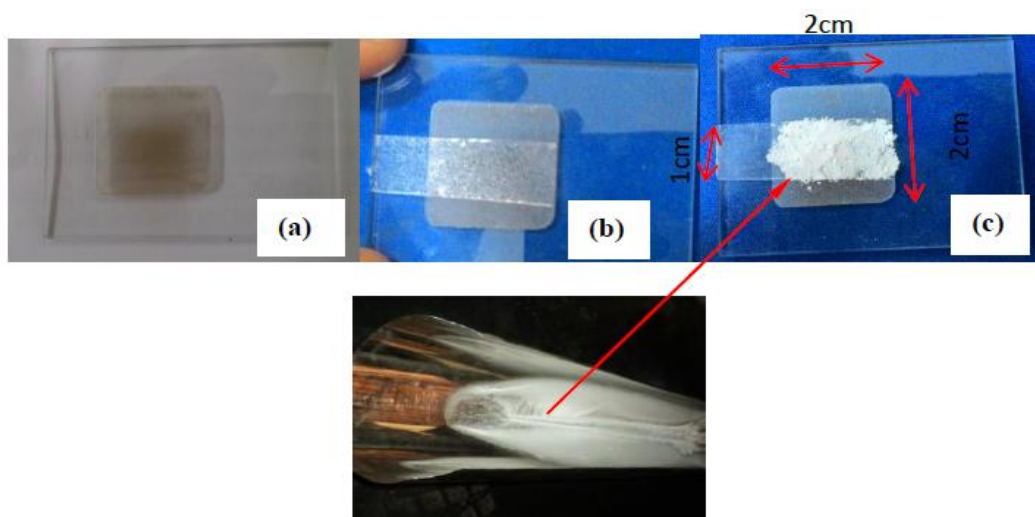


Figure 1: (a) Glass plate (b) Glass plate with double sticky tape (on blue background) (c) Powder of test material (on blue background).



Figure 2: Powder XRD (Voltage-20-40V, Current-2-15mA along with chiller water circulation)

After all the above steps transfer the powder of urea deposit into a sample holder and spread uniformly onto a glass slide assuring perfect level sprinkle on double sticky tape. Usually the substrate is amorphous to avoid interference. Care must be taken to create a flat upper surface and to achieve a random distribution of lattice orientations.

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Dr. Sadashiva Prabhu S is working as Asst.Professor (Sl.Grade) in the Department of Mechanical and Manufacturing Engineering at Manipal Institute of Technology, Manipal. His areas of expertise are Thermal Engineering, Materials Engineering and High Temperature Materials. He has total 15 years teaching experience and he has 14 publications in National and International Journals and Conferences.



Dr. Nagaraj S Nayak, currently working as Deputy Manager, at KBAT Technologies Pvt.Ltd. Pune. He holds Doctorate degree from IIT-Delhi and Post Doctorate degree from University of Wisconsin Madison, USA, and University of Texas in Mechanical Engineering and has more than twenty years of experience in the fields of Industry, Teaching and Research. His research areas include Engine Tribology, Combustion, Thermal Engineering, and Energy Engineering. He published two text books and he has more than 40 publications in National and International Journals and Conferences.



Dr. N Kapilan is currently working as Professor and Head of Department of Mechanical Engineering, Nagarjuna College of Engineering and Technology, Bangalore. He has more than 21 years of Teaching and Research experience. He published more than 54 papers in Journals and, authored one Technical Book and four Book Chapters. He has filed one patent and received research funding more than US \$ 125,000 from various funding agencies and professional societies. He has organised several international and national conferences, workshops, and seminars. He is actively involved in ASHRAE and ISHRAE Student Chapter Activities.