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The selective oxidation of ammonia using copper-based catalysts: The effects of water

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ABSTRACT

The activity of copper oxide supported on alumina ($3.4Cu/Al_2O_3$) and a copper exchanged beta zeolite (3.0Cu/beta) for the selective oxidation of ammonia into nitrogen is presented. Both catalysts are active for the oxidation of ammonia into nitrogen and water, although the 3.0Cu/beta outperforms its alumina-based counterpart in terms of activity and selectivity to nitrogen. The activity of both catalysts decreases in the presence of water vapour. In the case of the copper supported on alumina catalyst, the activity could be restored by removal of water from the reactant stream. However, exposure of 3.0Cu/beta catalyst to extended use in the presence of water vapour irreversibly altered the catalyst and the initial high activity could not be restored.

1. Introduction

Ammonia is a well-known pollutant that has adverse effects on human health and on the atmosphere. The anthropogenic sources of ammonia have increased dramatically in the past years and indeed it is predicted that by 2010, ammonia will be the largest source of acidifying gas in Europe [1]. Several technologies exist that can convert or remove ammonia gas from waste streams including adsorption, scrubbing and biological purification [2–5]. There are disadvantages associated with each of these processes. For example, in biological purification careful control of reaction conditions is required due to the extreme sensitivity of the microorganisms to changes in parameters such as temperature, pH and amount of reactant.

The catalytic oxidation of ammonia into nitrogen and water offers several advantages over conventional ammonia abatement systems. One of the earliest reported works on the catalytic oxidation of ammonia concerned a detailed study over unsupported metal oxides at temperatures between 160 and 370 °C [6]. The products of reaction observed were N_2 and N_2O. Activity could be improved by supporting the metal oxide and several publications now exist showing that ammonia can be converted to nitrogen and water in the presence of oxygen over metal oxide supported catalysts. Examples include MoO_3/SiO_2 [7–9], V_2O_5/TiO_2 and CuO/TiO_2 [10,11], NiO/Al_2O_3 [12,13], CuO/Al_2O_3 [14–17] and supported Fe_2O_3 [18–21]. For most of these publications, by-products N_2O and NO are also formed in varying amounts.

One of the earliest reports concerning the catalytic oxidation of ammonia using exchanged zeolites was recorded by Golodets over a series of cation exchanged Y zeolites [22]. Copper exchanged ZSM-5 was also reported as a potential catalyst for the oxidation of ammonia and showed promising results [10,23]. More recently both Cu and Fe exchanged on ZSM-5 showed high activity for oxidation of ammonia with low levels of NO and N_2O formation [24,25]. Copper exchanged on beta zeolite has also been reported to be highly active and selective for this reaction [26]. Since water vapour is most likely to be present in waste streams which contain ammonia, it is important that ammonia oxidation catalysts have the ability to operate in the presence of water. Indeed, a feature of most catalysts reported to date is the fall in activity when water is introduced into the reactant stream. In general this effect does not adversely affect the selectivity to nitrogen [14]. This paper presents some results obtained regarding the oxidation of ammonia in our laboratory over a copper oxide supported on alumina and copper exchanged beta zeolite catalysts. The performance of these catalysts in converting ammonia in the absence and presence of water is presented in addition to the study of catalyst stability under these reaction conditions.

2. Experimental

Copper oxide supported (3.4 wt.% copper) on alumina was prepared using the conventional dry impregnation procedure. This catalyst was chosen as it was previously shown to be successful in

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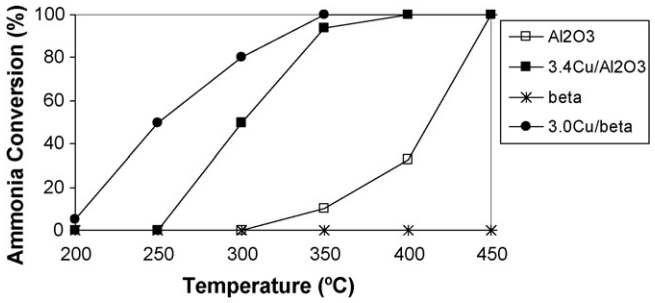

Fig. 1. Ammonia conversion with temperature for the indicated catalysts (0.54% NH_3, 8% O_2, He balance, 25 mg catalyst, W/F = 0.015 g s ml^{-1}).

converting ammonia into nitrogen and water [15]. The required amount of nitrate salt was dissolved in a volume of water enough to fill the pores of pre-calcined alumina (Rhône-Poulenc RP535, pore volume 1.3 ml g^{-1}, surface area 134 m^2 g^{-1}). The resultant solid was dried for 1 h at 80 °C and finally calcined at 450 °C for 5 h in air. The copper content of the catalyst (3.4 wt.%) was verified using Atomic Absorption Spectroscopy. The copper exchanged beta zeolite was prepared using a procedure similar to that reported by Iwamoto et al. [27]. Dried beta zeolite (Zeolyst International, Si/Al = 25,) was added to an aqueous copper acetate solution and stirred for 24 h. Subsequently the pH of the suspension was adjusted to pH 7 by the gradual addition of 8% NH_4OH solution and allowed to stir for a further hour. The resulting suspension was filtered and the filter cake washed thoroughly with distilled water. The solid was dried at 80 °C and calcined at 450 °C for 5 h in air. The final copper content of the zeolite was measured by Atomic Absorption Spectroscopy and was found to be 3 wt.%. All catalysts were sieved to a particle size between 106 and 225 μm before testing.

The crystallinity of the catalysts was investigated using XRD. This was performed on a Philips diffractometer with nickel filtered Kα Cu radiation (λ = 1.543 Å) between the X-ray source angles of 5° and 70°. All samples were ground with a mortar and pestle prior to analysis. The catalysts were tested for ammonia oxidation using a continuous flow system operated at ambient pressure. The reactor was a conventional plug flow reactor composed of quartz glass with the catalyst mounted between two pieces of loosely packed quartz wool. All catalysts (25 mg) were pre-treated at 420 °C for 90 min in a flow of 20 ml min^{-1} helium before testing. Typically the following feed composition was used: 0.54% NH_3, 8% O_2 with a balance of helium. The catalysts and supports were tested at different temperatures from 200 to 500 °C at 50 °C increments. Catalysts were also tested at different flow rates and in these experiments, the temperature was maintained at 325 °C (unless otherwise stated) and the flow rates were 20, 50, 100, 150 and 200 ml min^{-1}. The composition of the gases leaving the reactor was measured using a Hewlett Packard HP5971A Mass Spectrometer (M.S.). For experiments with added water vapour, the rig was slightly modified to incorporate a saturator filled with molecular sieves soaked in water.

Temperature programmed reduction in a 5% H_2/N_2 flow (heating rate 10 °C min^{-1}) was performed using a thermal conductivity detector (TCD) to monitor consumption of hydrogen. Samples were pre-treated at 600 °C for 1 h in helium and then cooled to room temperature. For temperature programmed desorption of ammonia experiments, the catalysts were pre-treated in a similar way. A flow of 4% NH_3/He was then passed over the pre-treated catalyst for 25 min. Following this ammonia adsorption procedure, the reactor was purged with helium for 30 min to remove residual/physisorbed ammonia. The sample was then heated to 600 °C at a rate of 10 °C min^{-1} in 20 ml min^{-1} helium and the ammonia desorption recorded continuously by the TCD. In some cases, the sample was treated with water vapour prior to the ammonia desorption step. A flow of 1% H_2O/He (using a water saturator) was passed over the catalyst for 25 min. The sample was purged with helium for 25 min before carrying out the ammonia desorption step.

3. Results

XRDs were carried out on the prepared catalysts, 3.4Cu/Al_2O_3 and 3.0Cu/beta, and both resembled the XRDs of their parent supports. The supports, Al_2O_3 and beta zeolite, demonstrated poor performance for ammonia oxidation especially at temperatures below 400 °C. This can be seen from Fig. 1. Indeed, the beta zeolite presented no activity at all at temperatures below 450 °C. The introduction of copper onto these supports resulted in a dramatic increase in ammonia conversion especially for the copper exchanged beta zeolite, 3.0Cu/beta. Total ammonia conversion was observed at 350 °C. The selectivities to the various products, N_2, NO and N_2O, are presented in Table 1. Both copper supported catalysts presented high selectivities to nitrogen. 3.4Cu/Al_2O_3 presented 93% selectivity to N_2 at 300 °C, however this decreased at higher reaction temperatures with a corresponding increase in NO and N_2O formation. On the other hand, 3.0Cu/beta presented over 95% selectivity to N_2 at all temperatures investigated.

The influence of water on ammonia oxidation was investigated over 3.4Cu/Al_2O_3 and 3.0Cu/beta. The reactant stream for ammonia oxidation in wet conditions was 0.54% NH_3, 8% O_2, 1% H_2O and a helium balance. Table 2 lists the activities of these two catalysts at 325 °C in dry and wet conditions at various W/Fs. The addition of water vapour to the feed resulted in a significant decrease in ammonia conversion for both catalysts. The loss in ammonia conversion in the presence of water was over 50% for 3.0Cu/beta and a similar loss in activity was observed for the alumina-based catalyst at a W/F 0.0075 g s ml^{-1}. Selectivity to nitrogen was not affected by the introduction of water for the copper exchanged beta zeolite whereby only traces of NO and N_2O were detected. For 3.4Cu/Al_2O_3, the selectivity to nitrogen increased slightly in the

Table 1
Selectivity (%) to various products with temperature for the indicated catalysts

Temperature (°C)	Al_2O_3			3.4Cu/Al_2O_3			Beta			3.0Cu/beta		
	N_2	NO	N_2O	N_2	NO	N_2O	N_2	NO	N_2O	N_2	NO	N_2O
200	–	–	–	–	–	–	–	–	–	97	2	1
250	–	–	–	–	–	–	–	–	–	95	2	3
300	–	–	–	93	3	4	–	–	–	96	2	2
350	37	57	5	75	10	15	–	–	–	96	2	2
400	41	54	5	51	23	26	–	–	–	98	1	1
500	45	51	4				77	23	0			

Reaction conditions: 0.54% NH_3, 8% O_2, He balance, 25 mg catalyst, W/F = 0.015 g s ml^{-1}.

Table 2
Ammonia conversion (%) in dry and wet conditions using 3.4Cu/Al_2O_3 and 3.0Cu/beta

W/F (g s ml^{-1})	3.4Cu/Al_2O_3		3.0Cu/beta	
	Dry	Wet	Dry	Wet
0.0075	41	23	69	34
0.01	47	25	100	41
0.015	69	27	100	54
0.03	71	45	100	83
0.075	82	51	100	87

Reaction conditions: 0.54% NH_3, 8% O_2 (1% H_2O), He balance, 25 mg catalyst, 325 °C.

Table 3
Selectivity (%) to N_2, NO and N_2O in dry and wet streams using $3.4Cu/Al_2O_3$

W/F (g s ml^{-1})	N_2 selectivity		NO selectivity		N_2O selectivity	
	Dry	Wet	Dry	Wet	Dry	Wet
0.0075	85	90	8	6	7	4
0.01	88	93	6	5	6	2
0.015	89	94	5	4	6	2
0.03	88	93	6	5	6	2
0.075	89	94	5	4	6	2

Reaction conditions: 0.54% NH_3, 8% O_2 (1% H_2O), He balance, 25 mg catalyst, 325 °C.

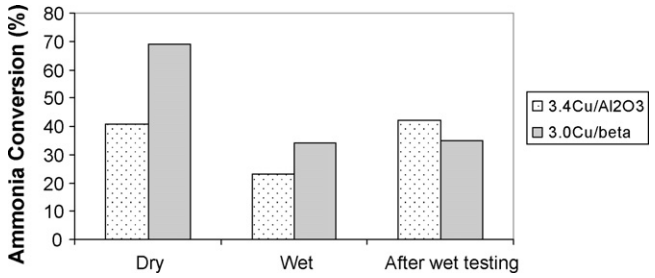

Fig. 2. Ammonia conversion in dry conditions, in the presence of 1% H_2O and in subsequent dry conditions (0.54% NH_3, 8% O_2 (1% H_2O), He balance, 25 mg catalyst, test temperature = 325 °C, W/F = 0.0075 g s ml^{-1}).

presence of water with a corresponding drop in selectivity to NO and especially N_2O. This is presented in Table 3.

Dry testing was carried out after the $3.4Cu/Al_2O_3$ and the 3.0Cu/beta catalysts had been exposed to wet conditions in an effort to restore the original activity (activity in dry conditions). For this test, the catalysts were tested at 325 °C in dry conditions followed by testing in wet conditions at 325 °C. Water was then removed from the reactant stream and the catalyst was pre-treated in helium for 90 min at 450 °C. They were subsequently retested at 325 °C for ammonia oxidation in dry conditions. Fig. 2 presents ammonia conversion obtained from testing in dry, wet and subsequent dry conditions for $3.4Cu/Al_2O_3$ and 3.0Cu/beta at a W/F of 0.0075 g s ml^{-1}. For $3.4Cu/Al_2O_3$, ammonia conversion dropped on the introduction of water. However, when the water vapour was removed from the reactant stream, ammonia conversion was restored. It would appear that $3.4Cu/Al_2O_3$ was not irreversibly altered following exposure to water vapour. In the presence of water vapour, selectivity to nitrogen increased for $3.4Cu/Al_2O_3$ as seen in Table 4. However, when the water vapour was removed from the feed stream, selectivity to nitrogen was restored to a value similar to that attained in dry conditions. For the 3.0Cu/beta catalyst, when the water vapour was removed from the reactant stream, ammonia conversion was not restored. It would appear that 3.0Cu/beta was irreversible altered following exposure to water vapour. Selectivity to nitrogen remained unaffected in the presence of water vapour, as seen in Table 4 and dropped slightly after wet testing.

Fig. 3 compares the TPR profiles of fresh $3.4Cu/Al_2O_3$ and $3.4Cu/Al_2O_3$ after 1 h of wet ammonia oxidation testing. Both presented similar reduction profiles as the copper reduction temperature was

Table 4
Selectivity to N_2 (%) for $3.4Cu/Al_2O_3$ and 3.0Cu/beta in dry and wet streams

Catalyst	Dry conditions	Wet conditions	After wet testing
$3.4Cu/Al_2O_3$	89	94	88
3.0Cu/beta	97	97	92

Reaction conditions: 0.54% NH_3, 8% O_2 (1% H_2O), He balance; 25 mg catalyst; 325 °C, W/F = 0.0075 g s ml^{-1}.

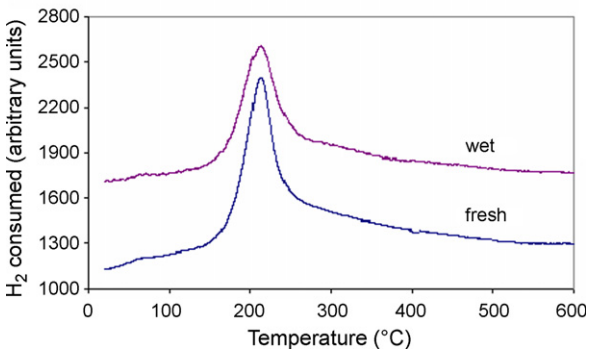

Fig. 3. TPR profiles of fresh $3.4Cu/Al_2O_3$ and $3.4Cu/Al_2O_3$ after 1 h of wet testing (5% H_2/N_2, flow rate = 20 ml min^{-1}, ramp rate = 10 °C min^{-1}, 35 mg catalyst).

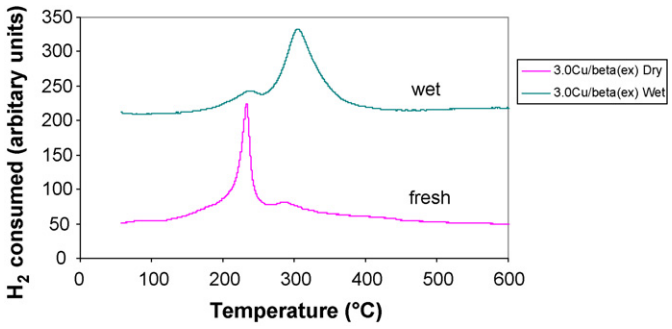

Fig. 4. TPR profiles of fresh 3.0Cu/beta and 3.0Cu/beta after 1 h of wet testing (5% H_2/N_2, flow rate = 20 ml min^{-1}, ramp rate = 10 °C min^{-1}, 35 mg catalyst).

unchanged at 216 °C. The catalyst was pre-treated as normal after wet testing.

Fig. 4 presents the TPR profiles of fresh 3.0Cu/beta and 3.0Cu/beta after 1 h of wet testing at 325 °C. The catalyst was pre-treated as normal after wet testing. The main reduction peak of fresh 3.0Cu/beta was seen at 234 °C. A second smaller peak was noted at the higher temperature of 297 °C. Following wet testing the TPR profile of the catalyst significantly changed. The principal reduction peak associated with 3.0Cu/beta after wet testing was at 309 °C with a second peak at 243 °C.

Fig. 5 compares the ammonia TPD profile of fresh $3.4Cu/Al_2O_3$ with the ammonia TPD profile of $3.4Cu/Al_2O_3$ treated with water vapour after ammonia adsorption. The fresh $3.4Cu/Al_2O_3$ catalyst exhibited two broad regions at approximately 120 and 245 °C. On the introduction of water vapour before the ammonia desorption step, only one relatively small ammonia desorption peak is observed at 350 °C.

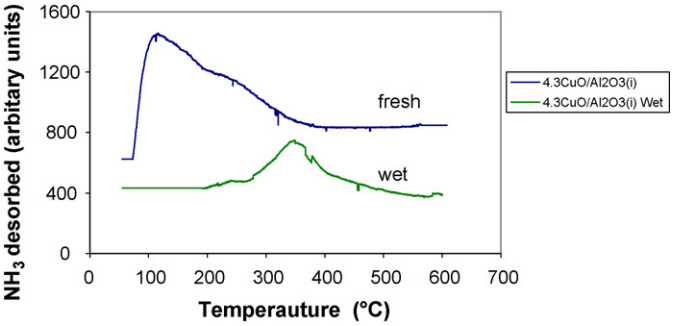

Fig. 5. Comparison of TPD of NH_3 with and without water treatment for $3.4Cu/Al_2O_3$ (4% NH_3/He (1% H_2O/He), 20 ml min^{-1}, ramp rate = 10 °C min^{-1}, 150 mg catalyst).

4. Discussion

The two supports presented in this work, Al_2O_3 and beta zeolite, demonstrated poor performances for ammonia oxidation especially at low temperatures (<400 °C). Al_2O_3 was the most active in terms of both ammonia conversion and selectivity to nitrogen and the beta zeolite presented no activity at all below 450 °C. The introduction of copper onto these supports resulted in a significant increase in ammonia conversion (see Fig. 1). The 3.4Cu/Al_2O_3 catalyst presented almost complete conversion at 350 °C and the selectivity to nitrogen was 75%. Increasing the temperature of reaction resulted in complete ammonia conversion but greater formation of unwanted by-products NO and N_2O. The 3.0Cu/beta catalyst presented the best activity in terms of converting ammonia. A promising feature of this catalyst was the very high selectivity to nitrogen with little or no NO detected. Low levels of N_2O were observed, however, in comparison to 3.4Cu/Al_2O_3 a marked improvement was observed in terms of minimising by-product formation. This high selectivity to nitrogen and high activity makes the copper exchanged beta catalyst one of the most active catalysts reported to date in the literature for the decomposition of ammonia to nitrogen in the presence of oxygen.

The high ammonia conversion and selectivity to nitrogen attained by copper exchanged beta zeolite would suggest that the copper species formed during catalyst preparation have an important role to play in ammonia oxidation. In the exchange procedure, copper exchanges for two exchangeable protons or cations, depending on the zeolite type. This preparation method should result in a well-dispersed copper component within the zeolite, as it is replacing the non-framework cations to maintain a neutral framework. In ion exchange, copper is theoretically in the form of Cu^{2+} ions, located in close proximity to two framework Al atoms. Copper can also be found close to framework silica and these are referred to as 'non-exchangeable' sites also known as silica vacancy defects [28]. In addition, copper on zeolites can form copper oxide particles upon calcination [29]. Therefore, besides single transition metal ions, transition metal ions at non-exchangeable sites and polynuclear species can also exist.

Addition of water to the feed stream reduced ammonia conversion for the 3.4Cu/Al_2O_3 catalyst as illustrated in Table 2. This was accompanied by a slight increase in nitrogen selectivity. This increase in nitrogen selectivity could be attributed to the fall in N_2O selectivity as water vapour was seen to suppress its formation. From the temperature programmed desorption experiments, it was observed that 3.4Cu/Al_2O_3 exhibited two ammonia desorption regions at 80–150 and 210–300 °C (Fig. 5). When water (1%) was passed over a catalyst with pre-adsorbed ammonia, the resulting NH_3 TPD presented a profile which showed the absence of the low temperature desorption peak. However, the high temperature desorption peak remained. These findings suggest that some of the ammonia adsorbed on 3.4/Al_2O_3, particularly the weakly adsorbed ammonia, is displaced by water vapour. This illustrates the importance of weakly adsorbed ammonia for the ammonia oxidation reaction and the existence of competitive adsorption between water and ammonia for active sites.

The effect of water vapour on the copper species was investigated by testing both 3.4Cu/Al_2O_3 and 3.0Cu/beta in dry, wet and subsequent dry condition. A fall in activity was observed in wet conditions for both catalysts (see Fig. 2). For the 3.4Cu/Al_2O_3 catalyst the activity level was restored once the water was removed from the system. TPR profiles of fresh 3.4Cu/Al_2O_3 and 3.4Cu/Al_2O_3 following reaction in wet conditions were identical demonstrating that this catalyst was not irreversibly altered after wet testing. Therefore, it seems that ammonia conversion is reduced in wet conditions solely due to competitive adsorption between ammonia and water on active sites of 3.4Cu/Al_2O_3 and the stability of the catalyst is maintained throughout the reaction.

For the 3.0Cu/beta catalyst, removal of water from the reactant stream did not result in a restoration of ammonia conversion and nitrogen selectivity to levels observed in dry conditions (see Fig. 2 and Table 4). From the TPR profiles presented in Fig. 4, two reduction peaks are observed for the fresh 3.0Cu/beta zeolite. The principal reduction peak at 234 °C (low temperature) and a second peak at 297 °C (high temperature). After water exposure, the high temperature peak increased significantly at the expense of the low temperature peak. This suggests that there are changes to the copper species which are promoted by wet ammonia oxidation conditions. The most likely explanation is that active dispersed Cu^{2+} species aggregate to form larger CuO species (high temperature TPR peak). These species are less active in terms of the NH_3 oxidation reaction. These changes may account for the loss in ammonia conversion and the fall in selectivity to nitrogen. Therefore, for the 3.0Cu/beta catalyst, ammonia conversion is reduced in wet conditions not only due to competitive adsorption between ammonia and water on active sites but also due to the instability of the active copper species. There is a rearrangement of copper species, which is promoted in the presence of water and this leads to an irreversible change in the activity of these solids.

5. Conclusion

Beta zeolite containing 3 wt.% copper is extremely active for the decomposition of ammonia to nitrogen in the presence of oxygen. In fact, it is one of the most active catalysts investigated to date in the literature. Another promising feature of this catalyst is its ability to convert ammonia almost exclusively to nitrogen with only trace levels of N_2O and NO observed.

However, this copper beta exchanged catalyst, in comparison to copper supported on alumina, is not stable when exposed to wet oxidation conditions. From TPR and activity testing, it seems that this loss in activity is due to the conversion of an easily reducible copper species into a copper species that reduced at higher temperature.

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