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Selective catalytic reduction of NO_x with NH_3 over different copper exchanged zeolites in the presence of decane

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ABSTRACT

Copper-exchanged in to zeolites containing large, medium and small pores were studied for selective catalytic reduction (SCR) of NO with NH_3 in the presence and absence of decane. Cu/ZSM-5 and Cu/ERI exhibited higher NO_x conversion compared to Cu/FER and Cu/MOR. On the other hand NO_x conversion over Cu/FER was more stable in the presence of decane compared to Cu/MOR and Cu/ZSM-5 catalysts. Fresh and used Cu/zeolite catalysts were analyzed by H_2-TPR, NH_3-TPD, ESR and TGA. Copper species with different reducibility and isolated Cu^{2+} ions in different coordinations were identified by H_2-TPR and the ESR techniques respectively. The initial NO_x conversion activity of Cu/zeolites seems to depend on the number of isolated Cu^{2+} species, which were affected by the zeolite structure. Whereas, the catalysts resistance to hydrocarbon poisoning depended on the pore geometry of the zeolites. During NH_3-SCR, the presence of large pores and cages in the zeolite lead to hydrocarbon deposit and retention by the zeolites which blocks the active sites and also decreases the active intermediates needed for NO_x conversion. Cu/ZSM-5 with higher number of isolated Cu^{2+} species showed higher initial NO_x conversion compared to other Cu/zeolite catalysts. Cu/FER on the other hand with small pore and cage diameter with one-dimensional channel structure showed higher hydrocarbon poisoning resistance.

1. Introduction

The use of the catalytic after treatment device seems to be inevitable due to the increasingly stringent limitations on the emission of the pollutant NO_x from various sources, both in developed and developing countries. Several technologies are being developed and practiced to reduce NO_x to N_2. Among these technologies three way catalysts (TWC), NO_x adsorbers, urea-selective catalytic reduction (SCR), and HC-SCR are most promising and therefore extensively pursued [1–4]. For majority of automotive applications operating under lean conditions, that is exhaust gas with excess oxygen, selective catalytic reduction (urea-SCR) of NO_x with urea seems to be the most promising technology [5].

Several varieties of NH_3-SCR catalysts are being developed and are being used in practice to suit a particular application [6]. For example, catalysts such as vanadia supported on titania are extensively used in NO_x reduction in stationary applications, where the aftertreatment system, most of the time, operates under steady state conditions. On the other hand for most challenging automotive applications which operate under transient conditions application of vanadia-titania catalyst becomes more difficult. This is due to several factors some of them related to the performance of these catalysts under narrow temperature window, poisoning of these catalysts by sulfur etc. For the past two decades metal exchanged zeolites have been extensively studied for NH_3-SCR for such challenging automotive applications [7]. Among several tens of these metal exchanged zeolites, Fe and Cu ion exchanged zeolites in particular were proved to be the suitable candidates for NH_3-SCR [8–10].

Transition metal zeolite based catalysts in general are found to efficiently convert NO_x to N_2 with ammonia as a reductant. However application of transition metal exchanged zeolite catalysts poses unique challenges, such as catalyst degradation due to hydrothermal dealumination/decatanation, HC and S poisoning etc., which are detrimental to NO_x conversion [11–14]. Although hydrocarbons are used as reductant for the reduction of NO in HC-SCR reaction, they act as poison in NH_3-SCR reaction. The internal combustion engines emit a significant amount of unburned hydrocarbons especially during cold start, which gets adsorbed onto the NH_3-SCR catalyst, causing a decrease in NO_x conversion. Furthermore, the adsorbed hydrocarbons may form carbonaceous deposits, requiring high temperatures to remove by oxidation in air. Therefore, catalysts that can avoid or minimize hydrocarbon adsorption and deposits at low temperature, or more preferably, oxidize unburned hydrocarbons at the lower temperatures, are highly desired. Despite the known fact that, the fuel derived hydrocarbons tend to poison metal ZSM-5 catalyst, there are very scarce reports on the influence of hydrocarbon on the NH_3-SCR activity.

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Over zeolite catalysts presence of hydrocarbons are generally detrimental as the zeolite acid sites leads to the coke formation blocking the active sites. This effect can be expected to be more pronounced at low temperatures on Cu/zeolite catalysts than at higher temperatures, where HC oxidation minimizes such coke formation. The pore structure and topologies are responsible for generating unique transition metal active sites and a proper choice of the zeolite could increase the population of such sites leading to an active catalyst. On the other hand the rate of HC poisoning depends on zeolite pore structure, acidic properties, reaction temperature and reactant nature and the choice of the zeolite with proper pore structure could also minimize the reversible effects such as HC poisoning [15]. Recently a few articles addressed the above issues on the zeolite based NH_3-SCR catalysts with particular pore structure [16,17]. Comparative study among the catalysts with different zeolite structures are scarce and are the subject of discussion of this article. In the present study a comparison of copper active sites and NO_x conversion with NH_3 in the absence and presence of decane over different Cu exchanged zeolites differing in their pore geometries and acidic properties were studied.

2. Experimental

The copper exchanged zeolites were prepared by ion-exchange method using 0.25 M $Cu(CH_3COO)_2$ aqueous solution at 80 °C and were continuously stirred for 24 h. The thus obtained materials were filtered and washed with deionized water followed by drying in air at 110 °C for 10 h and calcined in air at 500 °C for 5 h to obtain Cu/zeolite catalysts. The Cu loading in the catalysts were estimated by ICP-AES analysis and documented in Table 1.

The acidity of the samples were measured by ammonia temperature-programmed desorption (NH_3-TPD) technique. A quadrapole mass spectrometer (Bel Japan) was used to detect desorbed NH_3. Ca. 100 mg of the sample was out gassed at 500 °C for 1 h in a helium flow followed by ammonia adsorption at 100 °C for 1 h. Subsequently, the sample was flushed with helium for 30 min at 100 °C to remove physically adsorbed ammonia. Ammonia desorption is carried out by rising the temperature to 600 °C with a heating rate of 10 °C/min.

The H_2-TPR experiments were performed on a custom made reactor using 100 mg of the calcined catalyst. Before TPR measurement, the catalysts were pretreated at 500 °C for 1 h in air ($50\,cm^3\,min^{-1}$) and then the furnace temperature was lowered to 25 °C. The feed containing 10 vol% H_2 in Ar was fed at a flow rate of $50\,cm^3\,min^{-1}$. H_2-TPR was performed by heating the sample from 25 to 700 °C at a heating rate of $10\,°C\,min^{-1}$. The hydrogen consumed in the TPR was measured by a TCD.

Electron spin resonance (ESR) analysis was carried out with a JEOL TE300 instrument. All samples were heated at 200 °C for 2 h in vacuum. The ESR measurements were carried out in a vacuum at room temperature. Spin concentrations were estimated by double integration of the first-derivative spectrum and comparison with a known concentration of crystals of $CuSO_4{\cdot}5H_2O$ as a standard.

The catalysts are evaluated for the SCR of NO with NH_3 in a fixed bed reactor with a simulated exhaust gas containing 300 ppm NO, 300 ppm NH_3, 10 vol% O_2, 6 vol% H_2O, 1 ppm SO_2 and 10 ppm decane (when used) all diluted in He. The gas mixture was fed to 5 mg of the catalyst diluted with 50 mg of SiO_2 at a flow rate of $90\,cm^3\,min^{-1}$ (SV = ca.75,000 h^{-1} catalyst volume based). The steady-state activity was measured at each reaction temperature by decreasing the temperature in 20 °C steps from 400 to 140 °C. NO_x conversion was allowed to equilibrate for 20 min before changing the catalyst temperature to next level. The time on stream was measured by exposing the catalyst to reaction gas mixture containing decane (10 ppm) at 280 °C for 10 h. The products were analyzed by gas chromatography (Shimadzu, GC-8A TCD detector) with a Molecular Sieve 5A column to separate N_2 and CO and a Porapak Q column to separate N_2O and CO_2. The concentration of NO was continuously monitored using a NO_x analyzer.

The oxidation of various coked Cu/zeolite samples were carried out in a Shimadzu, DTG-50 simultaneous DTA-TG instrument. Weight loss was determined by heating a known amount of the sample in 100 ml/min air flow to 600 °C with a heating rate of 10 °C/min.

3. Results and discussion

3.1. Characterization

3.1.1. H_2-TPR

In NH_3-SCR over transition metal zeolites the redox properties of the metal species are an important factor for determining the extent of NO_x conversion. In Cu/zeolites the redox property such as the ease of reduction depends on the copper content and zeolite structure [18,19]. With increase in copper content the reducibility increases. H_2-TPR profiles of calcined Cu/zeolite catalysts are shown in Fig. 1. All catalysts exhibited two distinct reduction peaks referred to as low and high temperature reduction peaks appearing

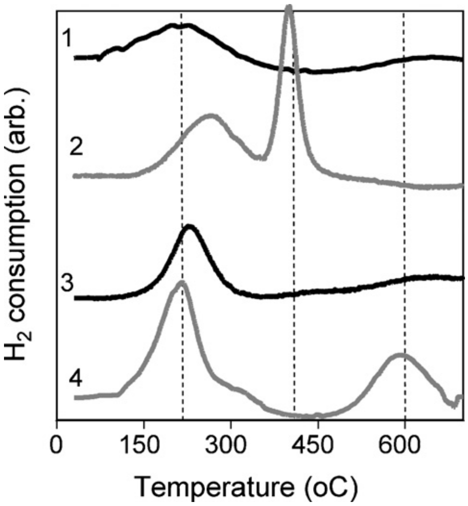

Fig. 1. H_2-TPR profiles of (1) Cu/MOR, (2) Cu/ZSM-5, (3) Cu/ERI and (4) Cu/FER catalysts.

Table 1
Physico-chemical properties of Cu/zeolites used in this study.

Zeolite	Si/Al	Channel dimensions (Å)	Zeolite void characteristic		Cu content (wt%)	NH3 uptake (mmol/g)	[a]Isolated Cu^{2+} (mmol/g)	Amount of coke formed (wt%)
Cu/ZSM-5	12	5.4 × 5.6	3D	10MR	2.6	0.98	0.15	2.57
Cu/MOR	9	6.7 × 7.0	1D	12MR	2.1	0.79	0.12	2.45
Cu/FER	9	4.5 × 5.5	2D	10MR	3.1	1.46	0.14	1.25
Cu/ERI	3	3.6 × 5.2	3D	8MR	2.0[b]	0.65	–	1.51

[a] Estimated from ESR.
[b] Estimated from TPR.

below and above 360 °C respectively. The shape of the low temperature reduction peak is similar for all catalysts, where as the second reduction peak appeared narrow in Cu/ZSM-5 and Cu/FER compared with Cu/MOR and Cu/ERI catalysts. For Cu/ZSM-5 the reduction of Cu^{2+} to Cu^+ occurred at higher temperatures and that of Cu^+ to CuO at lower temperatures, relative to other Cu/zeolite catalysts. The low temperature reduction peak can be attributed to the reduction of Cu^{2+} to Cu^+ and the high temperature reduction peak to Cu^+ reduction to CuO [20]. Generally the catalysts with more facile reduction of Cu^{2+} to Cu^+ are expected to be more active as this is a key step generating the reaction intermediates, for example, NO oxidation to NO_2. The catalyst with Cu^+ to Cu^0 reduction occurring at higher temperature is expected to give more stable copper active species as Cu^0 and if Cu^0 forms at low temperature is expected to sinter as it is not bound to the zeolite framework. The substantial difference in behavior of the monovalent Cu ions reduction in FER and MOR and ERI compared to ZSM-5 is due to high negative framework charge because of low Si/Al ratio of these employed zeolites leading to strong bonding of the Cu ions to framework oxygen. Wichterlova et al. [21] reported such reduction behaviour in the reducibility of copper species in different zeolites. The reduction temperature of Cu^{2+} and Cu^+ also depends on the presence of copper at the ion exchanged positions as compared to the presence of CuO like oxide phase.

3.1.2. ESR

The coordination of the copper species to the zeolite framework, and therefore the nature of active copper species, is influenced by the zeolite structure. The ESR spectra of Cu/zeolites could be useful in identifying such coordination and location of metal ions in the zeolites. Several reports are documented on the structural environment of paramagnetic copper sites in zeolites by ESR spectroscopy [22–25]. The coupling between the 3d unpaired electron and the nuclear spin (*I*D3/2) of Cu^{2+} results in a fourfold splitting of the EPR line in both of parallel and perpendicular components. The ESR spectra of Cu^{2+} in different (MOR, ZSM-5 and FER) zeolites along with g and A values are depicted in Fig. 2, and the amount of total isolated Cu^{2+} species are given in Table 1. As seen in the figure the Cu^{2+} ions are located in similar coordination atmosphere in MOR and ZSM-5, despite the significant difference in the framework structure of these two zeolites in agreement with the earlier reports [26]. Three different coordinations are reported in the literature for the Cu^{2+} ions siting in Cu-ZSM-5 zeolites [27,28]. These are: square planar ($g = 2.27$), square pyramidal ($g = 2.33$) and octahedral ($g = 2.38$). The coordination of copper species in Cu/MOR and Cu/ZSM-5 catalysts can be attributed to Cu^{2+} cations in five coordinated square pyramidal and four coordinated square planar environments. The walls of the channels in these zeolites are formed by folded six rings of oxygens on which four, five or six coordinated Cu^{2+} complexes can be formed [29]. The Cu^{2+} species in Cu-FER seems to be present in square pyramidal coordination only. Cu/FER showed the presence of only one type of isolated Cu^{2+} ion in agreement with the earlier reports by Attfield et al. [24].

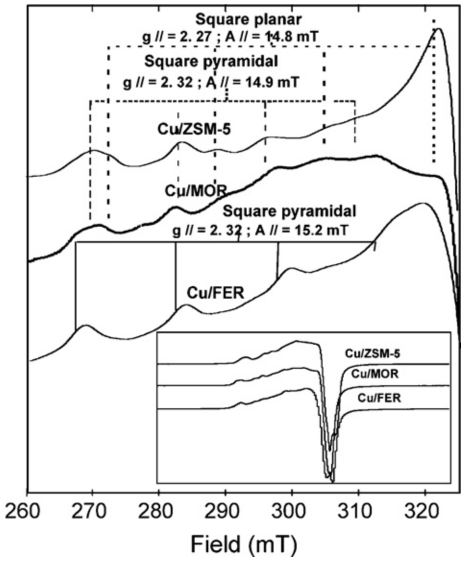

Fig. 2. ESR spectra of Cu/zeolites, showing square planar and square pyramidal copper species. The full ESR spectra are shown in the inlet.

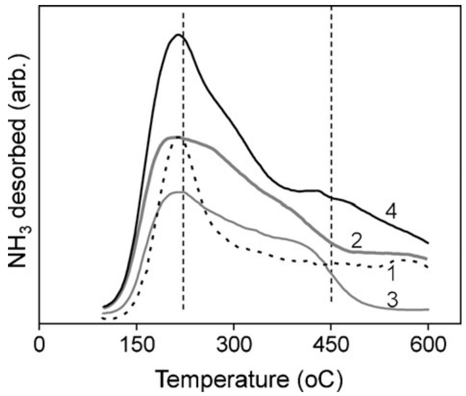

Fig. 3. NH3-TPD profiles of (1) Cu/MOR, (2) Cu/ZSM-5, (3) Cu/ERI and (4) Cu/FER catalysts.

3.1.3. TPD

Acidity of the zeolites is one of the important parameter that determines the extent of NO_x reduction with ammonia over zeolite based catalysts. The relative position of the ammonia desorption peaks of NH_3-TPD curves, with respect to temperature gives information on the acid sites strength of the zeolites. With the NH_3-TPD experimental approach employed, generally two ammonia desorption peaks are observed on acidic zeolite materials. The ammonia desorption peak below 250 °C is considered as weakly bound ammonia and above 250 °C is considered as strongly bound ammonia arising from ammonia bound to protons of the zeolite [30]. Ammonia desorption profiles over Cu/zeolite SCR catalysts employed in this work measured by NH_3-TPD are shown in Fig. 3 and the amount of ammonia uptake is given in Table 1. The weak acid sites of Cu/MOR shows an NH_3 desorption peak in a narrow temperature window and Cu/FER showed higher intensity peak in a broader temperature window. The intensity of high temperature peak on Cu/zeolite catalysts significantly decreased, compared to the zeolite material alone, due to compensation of Bronsted acid sites by Cu^{2+} [31]. The participation of weakly bound ammonia in SCR reaction is ambiguous. According to Long and Yang, zeolites Bronsted acid sites provide sites for generating NH_4^+ ions [32], which reacts with NO_x to produce N_2. Among all the catalysts Cu/FER catalyst showed significantly more strongly bound ammonia and therefore is expected to show higher NO_x conversion.

3.2. Catalytic activity

Fig. 4 shows the NO_x conversion over Cu/zeolite SCR catalysts with ammonia as a reductant in 160–400 °C temperature range in the absence of hydrocarbon decane in the feed gas. Among the Cu/zeolite catalysts Cu/ZSM-5 and Cu/ERI showed significantly higher NO_x conversion below 300 °C compared with Cu/FER and Cu/MOR catalysts. The NO_x conversion (Fig. 4) did not correlate with the copper content (Table 1) of the Cu/zeolite catalysts. For instance, Cu/FER with higher 3.1 wt% copper showed lower NO_x conversion compared with 2.6 wt% copper containing ZSM-5. Comparison of

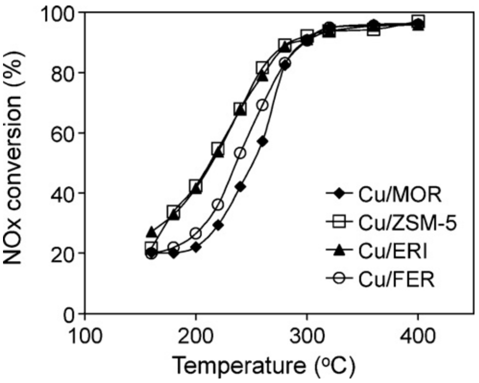

Fig. 4. NO_x conversion over Cu/zeolites with out decane in the feed gas. Reaction conditions NO = 300 ppm, NH_3 = 300 ppm, H_2O = 10 vol%, O_2 = 6 vol%, balance = He, SV = 75,000 h^{-1}.

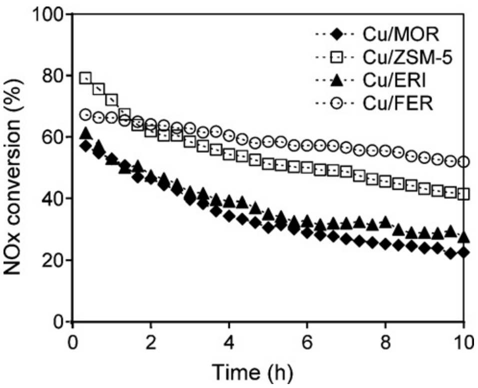

Fig. 6. Time on stream NO_x conversion with decane in the feed gas over Cu/zeolites at 240 °C. Reaction conditions NO = 300 ppm, NH_3 = 300 ppm, decane = 10 ppm, H_2O = 10 vol%, O_2 = 6 vol%, balance = He, SV = 75,000 h^{-1}.

NH_3-TPD and NO_x conversion further reveals that no apparent correlation was found between the NO_x conversion and the ammonia storage capacity and strength among the different Cu/zeolites catalysts. From the H_2-TPR profiles one would also expect Cu/FER to show higher NO_x conversion as it shows relatively more easily reducible copper species (Cu^{2+} to Cu^+) and has higher amount of Bronsted acid sites. However Cu/ZSM-5 and Cu/ERI which showed slightly higher Cu^{2+} to Cu^+ reduction showed better NO_x conversion. The increasing trend in NO_x conversion among the catalysts to some extent can be correlated to the number of isolated copper species as estimated from ESR analysis. Cu/ZSM-5 with higher number of isolated Cu^{2+} species (0.15 mmol/g) showed higher NO_x conversion compared to Cu/FER and Cu/MOR with less number of such isolated Cu^{2+} species (0.13 and 0.12 mmol/g). When comparing the different zeolites, from the limited characterization and SCR performance data it can be suggested that the zeolite structure has a greater influence on the activity of the copper species than the reducibility of the copper species and amount and strength of acid sites of the zeolite.

NO_x conversions were evaluated in the presence of n-decane and the comparison of NO_x conversion with and without decane in the feed gas are presented in Fig. 5. Cu/ZSM-5 showed significant decrease in NO_x conversion in the presence of decane compared with other Cu/zeolite catalysts. Cu/FER was found to show minimum decrease in NO_x conversions, compared with other Cu/zeolites, in the whole temperature range when HC is present in the feed gas. The NO_x conversions at each temperature, plotted in Fig. 5 were measured at a fixed reaction time of 20 min and no attempt was made to clean the catalyst during the temperature ramping down and therefore may not represent a true steady state NO_x conversion. Fig. 6 shows the time on stream of NO_x conversion in the presence of decane at 240 °C over different Cu/zeolite catalysts. NO_x conversion over all copper exchanged zeolites decreased gradually with time on stream and a true steady state NO_x conversion was not achieved even after 10 h of time on stream, presumably due to slow and continuous accumulation of HC deposits. The decrease in NO_x conversion was more pronounced over Cu/ZSM-5 catalyst compared to other Cu/zeolite catalysts employed in this study. During this 10 h of SCR reaction in the presence of decane, among all catalysts, Cu/FER showed minimal loss of NO_x conversion as observed from Fig. 5.

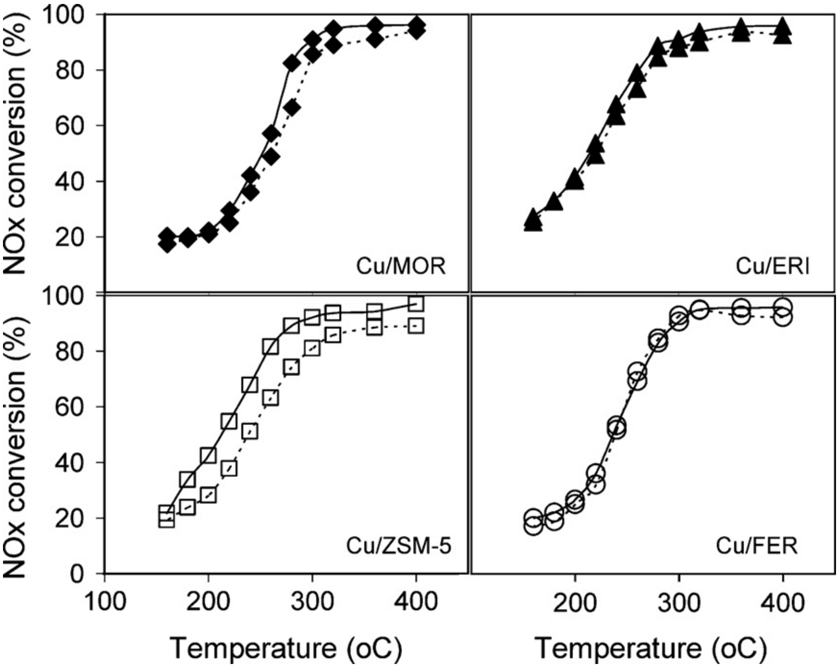

Fig. 5. NO_x conversion over Cu/zeolites with out (solid traces) and with (dashed traces) with decane in the feed gas. Reaction conditions NO = 300 ppm, NH_3 = 300 ppm, decane = 10 ppm, H_2O = 10 vol%, O_2 = 6 vol%, balance = He, SV = 75,000 h^{-1}.

Two main routes of NO_x performance decrease in the presence of HCs are (i) the HC deposit (or coke) formation which physically blocks the access of the active sites to the reactants and (ii) unselective consumption of SCR reaction intermediates such as adsorbed nitrates. The amount of HC deposits retained by the catalysts (Table 1), at the end of 10 h of time on stream under SCR conditions, were estimated from weight loss profiles of TGA in air. Cu/ZSM-5 and Cu/MOR retained significant amount of HC deposits compared with Cu/ERI and Cu/FER catalysts. Cu/FER retained the least amount of HC among all catalysts in accord with its NO_x performance degradation profile (Fig. 6). Formation and retention of HC deposits, therefore its effect on NO_x conversion in Cu/zeolite catalysts, can be mostly explained based the pore structure of different zeolites used.

MOR and ZSM-5 are considered as large and medium pore zeolites respectively and have relatively larger channels and its intersections compared with ERI and FER zeolites. For example, ZSM-5 is considered as three dimensional pores system where one straight channel with an elliptical cross section of 5.2–5.7 Å intersects with circular 5.4 Å diameter channel resulting in an elongated cavity of 9 Å diameter. The three dimensional structure of ZSM-5 and its larger channels and wider intersection favors the effective diffusion of decane there by leading to more HC deposit accumulation in Cu/ZSM-5. Furthermore the oligomerisation and hydrogen transfer reactions can extensively take place in ZSM-5 zeolite depositing coke in the catalyst. Similar explanation holds for the effect of HC on Cu/MOR catalyst NO_x conversion. FER, on the other hand, has two intersecting channel systems, one formed by circular 10-membered rings (5.2 Å diameter) and the second by 8-membered rings (of 4.7–3.7 Å cross-section), resulting in roughly spherical cavities with a diameter approximately 7 Å. These cavities are connected to the larger channels by smaller eight member ring windows. FER can be considered as a uni-dimensional pore system for long chain molecules like decane, while its full two-dimensional pore system can be exploited by shorter molecules like NO_x and NH_3. The high concentration of acid sites in Cu/FER compared with Cu/ZSM-5 should favor the hydride transfer reactions which are mainly responsible for carbonaceous deposits. However based on the comparison of TGA analysis of HC deposits this did not seem to take place as indicated by relatively small amount of retained hydrocarbon deposits in Cu/FER (Table 1). This is due to the narrow pore size of Cu/FER, where single file diffusion prevails resulting in restricted movement of large molecules like decane. Also one can expect that the HC in FER is mostly retained in the 10 membered ring channels. Furthermore FER zeolites smaller intersections do not allow the formation of heavy hydrocarbon deposits leading to less coke formation therefore resulting in relatively very small decrease in NO_x conversion. ERI has slightly larger 8-membered ring channels than FER and is a three dimensional zeolite with cages as opposed to one-dimensional FER zeolite. The larger degradation observed over ERI compared with FER is due to these differences in the structure.

4. Conclusions

Copper-exchanged zeolites containing large, medium and small pores were studied for SCR of NO with NH_3 in the presence and absence of decane. Cu/ZSM-5 and Cu/ERI were found to show better NO_x conversion than Cu/FER and Cu/MOR. No correlation was found between the NO_x conversion and ammonia storage and reducibility of the catalyst. Based on the characterization it can be said that zeolite structure seems to play a major role in determining the number of active sites, isolated Cu^{2+} species. All catalysts showed degradation when decane is present in the SCR feed gas. Among these different catalysts, having different pore structure, Cu/FER showed the minimal degradation. In the presence of hydrocarbons during SCR, the large pore diameter and the presence of large cages in the zeolite leads to hydrocarbon deposits, which blocks the active sites and also decreases the active intermediates needed for NO_x conversion. Cu/FER with relatively small pores and one-dimensional pore structure showed more hydrocarbon poisoning resistance and showed minimal degradation in NO_x conversion.

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