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The influence of microwave plasma pretreated CuO/TiO_2 catalysts in NO + CO reaction

Fei Gao[a], Bin Liu[b], Wenjing Sun[b], Yong Wu[b,c], Lin Dong[a,b,*]

[a] Jiangsu Key Laboratory of Vehicle Emissions Control, Center of Modern Analysis, Nanjing University, Nanjing 210093, PR China
[b] Key Laboratory of Mesoscopic Chemistry of MOE, School of Chemistry and Chemical Engineering, Nanjing University, Nanjing 210093, PR China
[c] College of Chemistry and Materials Science, Nanjing Normal University, Nanjing 210097, PR China

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ABSTRACT

NO reduction by CO reaction was comparatively studied over microwave plasma pretreated CuO/TiO_2 catalysts employing transmission electron microscopy (TEM), hydrogen temperature programmed reduction (H_2-TPR), and in situ Fourier-transform infrared spectroscopy (in situ FTIR). The catalytic performances indicated that a remarkable improvement in activity and selectivity was achieved after microwave plasma pretreatment, which was dependent on the microwave plasma pretreatment time. The results also suggested that a high active oxygen species (O^*) was formed on the surface of plasma pretreated CuO/TiO_2 catalysts, which led to the easy oxidation of NO to NO_2 at low temperature even without introducing any additional O_2 gas into the reaction system. Therefore, these high active oxygen species (O^*) should play an important role for the exaltation of the catalytic performances of the catalysts.

1. Introduction

Emissions of nitrogen oxides (NO_x) from both mobile and stationary sources are of great concern because of their impacts on the environment, including acid rain and photochemical smog formation. For cleaning these exhaust gases, catalytic removal of nitrogen oxide has drawn much attention in environmental catalysis, especially under lean-burn and diesel conditions. Much attention is paid to the improvement of the catalytic property via doping elements, exploring new preparation method, changing pretreatment conditions, etc. [1–3]. Among them, plasma-chemical method is considered as the one of the most potential candidates for enhancing the activity for the NO_x-reduction [4–19].

In general, these plasma-assisted methods include either single stage or two-step plasma catalytic reactor system. During the pretreatment process, the reaction is promoted to proceed. NO is primarily oxidized to NO_2, and partially hydrocarbon oxidized to reactive radicals like RO, ROO, CO, etc. Subsequently, these species react over the catalyst surface to eventually form N_2 and CO_2. As a result, non-thermal gas discharges such as corona or dielectric barrier discharges are able to initiate plasma-chemical reactions for the removal of nitrogen oxides by formation of highly reactive radicals like O, OH, or N in electron collisions with the main molecular components of exhaust gases [16–18,20–23]. Nevertheless, these reports are mainly focused on the favorable role of the plasma for activating gas reactants. It is well-known that catalytic reduction of NO by CO is a structure-sensitive reaction [19,24,25]. Additionally, rutile TiO_2 is the most stable phase as compare with anatase and brookite, and copper-based catalysts show the promising activity towards NO reduction. Therefore, it is necessary and important to further approach the change of catalyst surface with plasma-assisted processing.

Along this line, the microwave plasma is used for pretreating CuO/TiO_2 catalysts, and their catalytic performances in the "NO + CO" model reaction have been explored. In situ FTIR is performed to gain insight into the surface species before and after pretreatment. The results show that a remarkable improvement in both activity NO and N_2 selectivity has been achieved through the microwave plasma pretreatment, probably due to the important roles of a high active oxygen species (O^*) on the surface of CuO/TiO_2 during the microwave plasma processing.

2. Experimental

CuO/TiO_2 sample was prepared by impregnating TiO_2 support (a standard TiO_2 sample was obtained from *Catalysis Society of Japan*, rutile phase JRC-TiO_2-3, BET surface area 42 m^2/g) with an aqueous solution containing the requisite amount of $Cu(NO_3)_2$ (A.R., purchased from Nanjing (China) Chemical Reagents Fac-

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* Corresponding author at: Key Laboratory of Mesoscopic Chemistry of MOE, School of Chemistry and Chemical Engineering, Nanjing University, Nanjing 210093, PR China. Tel.: +86 25 83592290; fax: +86 25 83317761.
E-mail address: donglin@nju.edu.cn (L. Dong).

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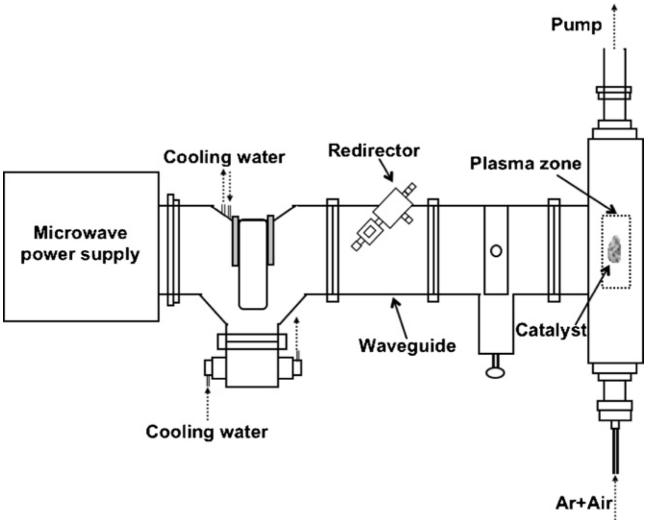


Fig. 1. The scheme of the microwave plasma instrument.

tory), followed by drying in air at 110 °C overnight and then calcining in flowing air at 450 °C for 2 h. The loading amount of CuO was 0.9 mmol Cu^{2+}/100 m^2 TiO_2 near the dispersion capacity (0.86 mmol/100 m^2 TiO_2) [26].

Microwave plasma pretreatment is carried out over the prepared CuO/TiO_2 samples. The scheme of the instrument was shown in Fig. 1. During the pretreatment, Argon-air gas (30% air by volume) plasma is excited in a large quartz tube where a microwave frequency at 2.45 GHz is applied with 80 Pa pressure and a maximum power of 800 W (reflected power at about 0). For simplicity, the sample treated by plasma was denoted as CuO/TiO_2-MP.

BET surface areas were measured by nitrogen adsorption at 77 K on a Micrometrics ASAP 2020.

TEM images of these samples were obtained by a JEM-2100 system. The samples were dispersed in ethanol and kept in an ultrasonic bath for 15 min, then deposited on a carbon-covered copper grid for measurement.

TPR was carried out in a quartz U-tube reactor connected to a thermal conduction detector (TCD) with H_2-Ar (7% H_2 by volume) as reductant, and 100 mg sample was used for each measurement. Prior to the reduction, the sample was pretreated in a nitrogen stream at 100 °C for 1 h. TPR started at room temperature at a rate of 10 °C min^{-1}.

In situ FTIR spectra of CO or/and NO molecules adsorbed on CuO/TiO_2 catalysts were recorded on a Nicolet 5700 FTIR spectrometer at a spectral resolution of 4.0 cm^{-1}. Considering CO as a case, a self-supporting wafer of a catalyst (about 10 mg), mounted in a commercial controlled environment chamber (HTC-3). Before adsorption, it was pretreated in a nitrogen stream at 100 °C for 1 h, then to a stream of CO–N_2 mixture (10% CO by volume) at a rate of 5 mL/min for 30 min. FTIR spectra were recorded at 25 °C and various target temperatures by subtracting the background of free CO molecular. NO adsorption and NO–CO co-adsorption were performed as CO adsorption.

The activities of the catalysts for NO + CO reaction were measured under steady state, involving a feed stream with a fixed composition, NO 3.33%, CO 6.67% and He 90% by volume as diluter. A quartz tube was employed as the reactor and 100 mg sample was used. The catalysts were pretreated in N_2 stream at 100 °C for 1 h and then heated to reaction temperature, after that, the mixed gases (total flow = 20 mL/min) were switched on. The reactions were carried out at 300 °C with a weight hourly space velocity (WHSV) of 12,000 mL/g h. Two columns and TCD were used for analyzing the production, column A with Porapak Q for separating N_2O and CO_2, and column B with 13× molecular sieve (30–60 M) for separating N_2, NO and CO.

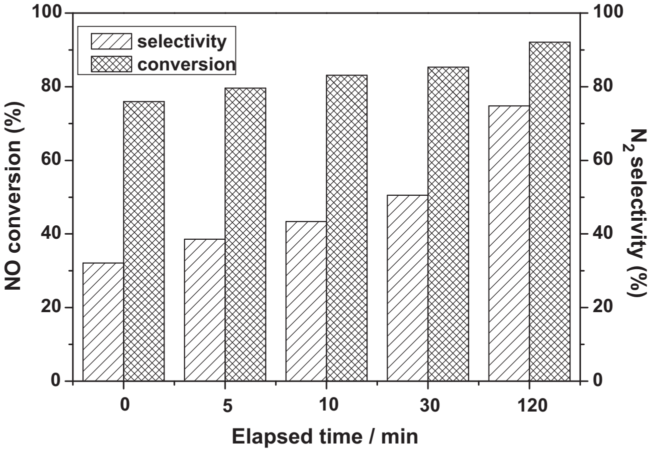


Fig. 2. Catalytic performances for NO + CO reaction over CuO/TiO_2 catalysts following different microwave plasma pretreatment times.

3. Results and discussion

3.1. Activity and N_2 selectivity results

Fig. 2 shows the NO conversion and N_2 selectivity for NO + CO reaction at 300 °C over CuO/TiO_2 and CuO/TiO_2-MP samples with different elapsed times of microwave plasma. For fresh CuO/TiO_2 sample, the NO conversion and N_2 selectivity are about 76%, 31%, respectively. The plasma treatment promoted the conversion of NO from 76% to 92% with the augment of microwave plasma processing time. When the microwave plasma treated time hold on 120 min, both activity and N_2 selectivity can reach nearly 100%, which doubles than the fresh one.

3.2. TEM and H_2-TPR results

Fig. 3A shows the low and high resolution TEM images of CuO/TiO_2 sample. From the low magnification image, it is seen that particle size is in the range of 30–40 nm and in well distribution. As evidenced by selected area electron diffraction pattern (SAED), this sample is polycrystalline. HRTEM image represents only one kind of periodicity of observed fringes (0.32 nm), which is compatible with the (1 1 0) plane of rutile TiO_2 [27,28]. For CuO/TiO_2-MP sample after microwave plasma pretreatment for 120 min (Fig. 3B), TEM and HRTEM images exhibit the similar particle distribution and interplaner spacing (0.32 nm). These results indicate that the microstructure of CuO/TiO_2 samples are still relatively stable, and no further changes have been taken place at the atomic scale during the microwave plasma processing.

Fig. 4 shows the H_2-TPR profiles of CuO/TiO_2 and CuO/TiO_2-MP samples. Obviously, two reduction peaks at about 172 °C and 221 °C are present on all samples, which are attributed to the reduction of dispersed copper oxide and crystalline copper oxide species, respectively [29,30]. However, a new low temperature reduction peak at about 158 °C is only revealed for CuO/TiO_2-MP samples. It is suggests that a "new oxygen species" has been produced through microwave plasma treatments, which react with hydrogen gas more easily. Interestingly, the intensity of this low temperature reduction peak increases with the pre-treating time. Simultaneously, the intensity of the reduction peak at 172 °C decreases accordingly. Whereas, the changing of the peak at 221 °C is nearly imperceptible. All these suggest that the activity of the oxygen species of the dispersed copper oxide on the catalysts surface is sig-

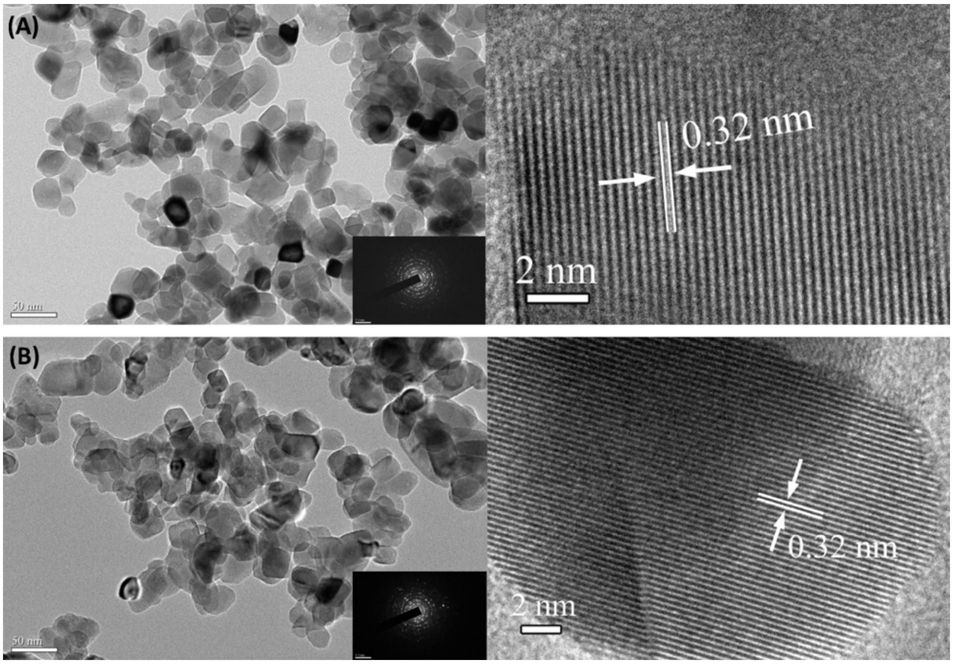


Fig. 3. TEM images of Cu/TiO_2 sample before (A) and after (B) pretreat with microwave plasma for 120 min (inset: the diffraction pattern).

nificantly improved with the microwave plasma pretreatment time increasing. This can be understood by taking into consideration that TiO_2 is an n-type semiconductor, and tends to produce a high concentration of n-type defects, i.e. Ti^{3+} ions and oxygen vacancies on the surface, under a reduction atmosphere. As a result, Ti^{3+} ions can donate electric charges to Cu^{2+} ions, and the unstable surface Ti^{3+} are returned to Ti^{4+}. Therefore, the reduction of copper oxide species is promoted [31,32]. The enhancement of the reducibility of copper oxide species should be related to the interaction between the copper oxide species and TiO_2 support. Apparently, compared with the crystalline CuO species, the dispersed copper oxide species are in close contact with the surface of TiO_2, thus resulting in the strong interaction between them, which lead to the lowest reduction temperature.

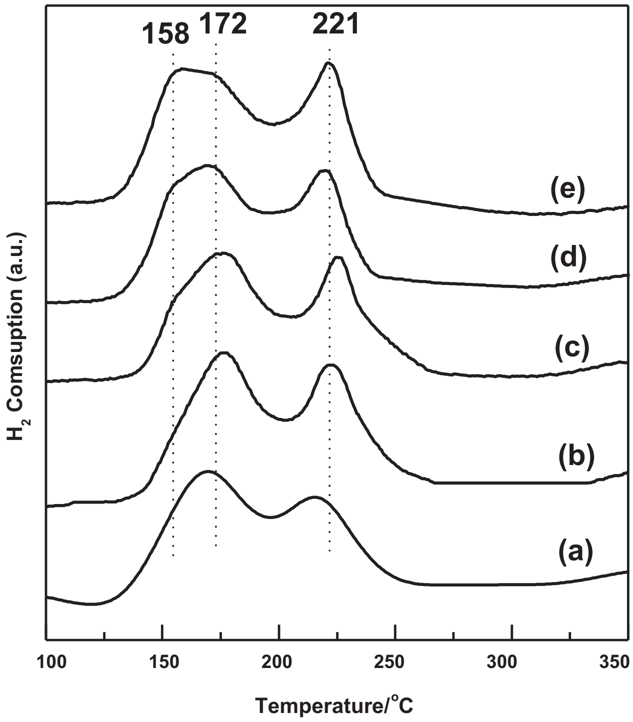
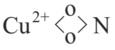


Fig. 4. TPR profiles of CuO/TiO_2 catalysts following different microwave plasma pretreatment times: (a) CuO/TiO_2 and (b–e) CuO/TiO_2-MP with microwave plasma treating 5, 10, 30, and 120 min.

3.3. CO or/and NO adsorption results

To approach the relationship between the catalytic performances and the surface species, in situ FTIR for CO or/and NO adsorption on fresh and plasma treated samples are performed. Fig. 5 shows the FTIR spectra of CO adsorption on CuO/TiO_2 and CuO/TiO_2-MP samples at different temperatures. Noticeably, similar IR spectra are obtained on both samples in the range of 2000–2200 cm^{-1}. At low temperature (below 100 °C), a typical band for CO adsorbed on Cu^+ appears at 2095 cm^{-1} (Fig. 5A(a) and B(a)) [33–35]. While, this band shifts to 2116 cm^{-1} with the temperature increasing. On the other hand, increasing the temperature to 150 °C result in the maximum intensity of Cu^+–CO, and further increasing the temperature to 300 °C lead to the weakness and disappearance of this species. Generally, the adsorption of CO molecules on Cu^{2+}, Cu^+, and Cu^0 gives rise to peaks with characteristic vibrational frequencies at about 2220–2150 cm^{-1}, 2160–2080 cm^{-1}, and below 2130 cm^{-1} respectively, and at the ambient temperature, Cu^+–CO is the most stable adsorption [33–35]. From these information, it is deduced that copper species go through stepwise reduction, namely from Cu^{2+} to Cu^+ to Cu^0, similar as our previously results [36,37].

As shown in Fig. 6, introduction of NO to the samples results in the appearance of several bands with very low intensities. For both fresh and treated catalysts at 50 °C, two bands appeared at 1886 and 1291 cm^{-1}, which are attributed to the frequencies of NO molecular adsorbed on Cu^{2+} species [38–40], and the overlapping component of the NO_2 asymmetric vibration with chelating nitro  $Cu^{2+}\langle\begin{smallmatrix}O\\O\end{smallmatrix}\rangle N$, respectively [41–43]. Importantly, a strong peak at 1352 cm^{-1} is present on CuO/TiO_2-MP sample, which is assigned to NO_2 stretch of a distorted $[NO_3{}^-]_2$ adsorbed on Cu^{2+} [41,44,45].

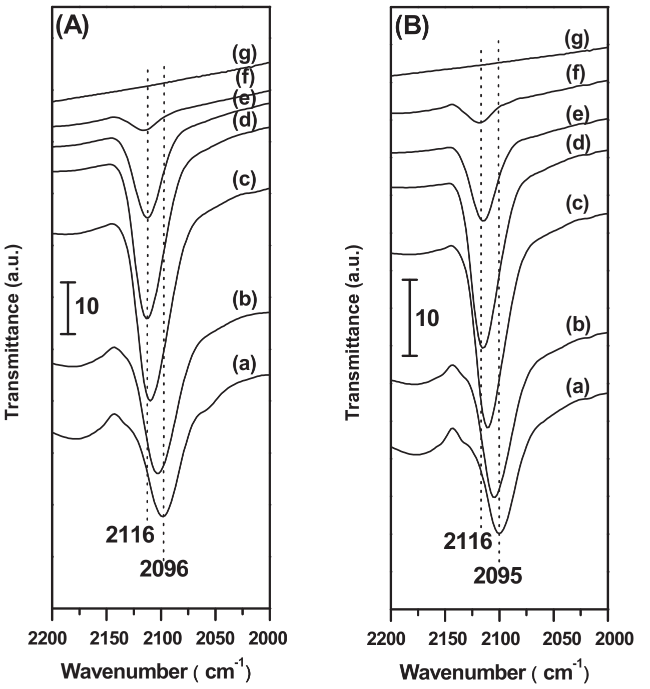
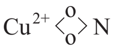
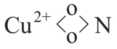


Fig. 5. FTIR spectra of CO adsorbed on CuO/TiO_2 (A) and CuO/TiO_2-MP (B) samples: (a) 50 °C, (b) 100 °C, (c) 150 °C, (d) 200 °C, (e) 250 °C, (f) 300 °C, and (g) pretreatment at 100 °C.

This band is quite weak in CuO/TiO_2 sample. With the temperature increasing, the bands at about 1880 and 1291 cm^{-1} are weakened gradually and disappeared at 200 °C. Nevertheless, the evolution of the band at 1350 cm^{-1} is extremely different in two samples. For CuO/TiO_2 sample, this weak band increases firstly and then decreases. While in CuO/TiO_2-MP sample, the tendency of the band at 1350 cm^{-1} changes imperceptible with the temperature increasing. This information on the changes of these adsorbed NO_x species indicate that plasma treatment enhanced the activity of surface oxygen from dispersed copper species significantly, which favors the strong interaction of NO with copper oxide.

The nature and relative population of the surface species under the simulative reaction conditions have been examined by CO and NO co-adsorption FTIR spectroscopy (Fig. 7). As compare with the spectra of these activated catalysts with NO adsorption, the exposure of samples to NO–CO mixtures also produce a variety of nitrates/nitrites species, which is due to the preferential adsorbability of NO with an unpaired electron [46]. For CuO/TiO_2 sample (Fig. 7A), the  $\mathrm{Cu^{2+}}\overset{O}{\underset{O}{\langle\rangle}}\mathrm{N}$ species at 1298 cm^{-1} dominate the spectra at low temperature, but disappear above 150 °C. At the same time, the band intensity of $Cu^{2+}[NO_3^{-}]_2$ species at 1368 cm^{-1} are grown up gradually. In fact, this peak is very weak, indicating the amount of this species is negligible on the catalyst surface, which is similar to the single NO adsorption FTIR results. In addition, a band at 2107 cm^{-1} for CO banding to Cu^{+} is also represented at 100 °C and disappeared at 300 °C, which should be resulted from the stepwise reduction of surface copper oxide by CO. This is also an indication that Cu^{+} species should play an important role in NO reduction. However, the band at 1886 cm^{-1} for NO molecular adsorbed on strongly associated Cu^{2+} species has not been detected during the whole heating process, which is quiet different from the results of CuO/TiO_2-MP. For CuO/TiO_2-MP sample (Fig. 7B), the band of Cu^{2+}–NO can be detected at 50 °C and gets weaker with increasing temperature. Moreover, it is worth to note that  $\mathrm{Cu^{2+}}\overset{O}{\underset{O}{\langle\rangle}}\mathrm{N}$ species decrease in intensity and disappear dramatically with temperature increasing to 150 °C. The distorted nitrates at 1370 cm^{-1} almost remained unchange below 200 °C, but decrease a little at higher than 200 °C. This result illuminate that the formation of nitrate is possibly resulted from the oxidation of NO to NO_2 from active oxy-

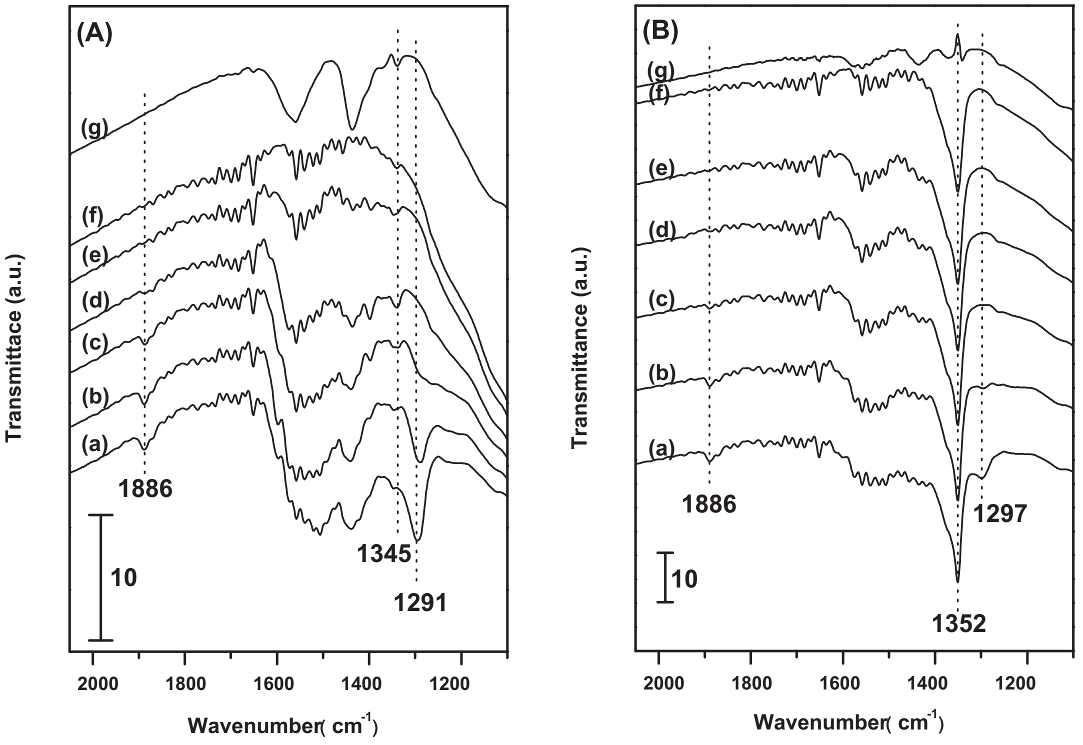


Fig. 6. FTIR spectra of NO adsorbed on CuO/TiO_2 (A) and CuO/TiO_2-MP (B) samples: (a) 50 °C, (b) 100 °C, (c) 150 °C, (d) 200 °C, (e) 250 °C, (f) 300 °C, and (g) pretreatment at 100 °C.

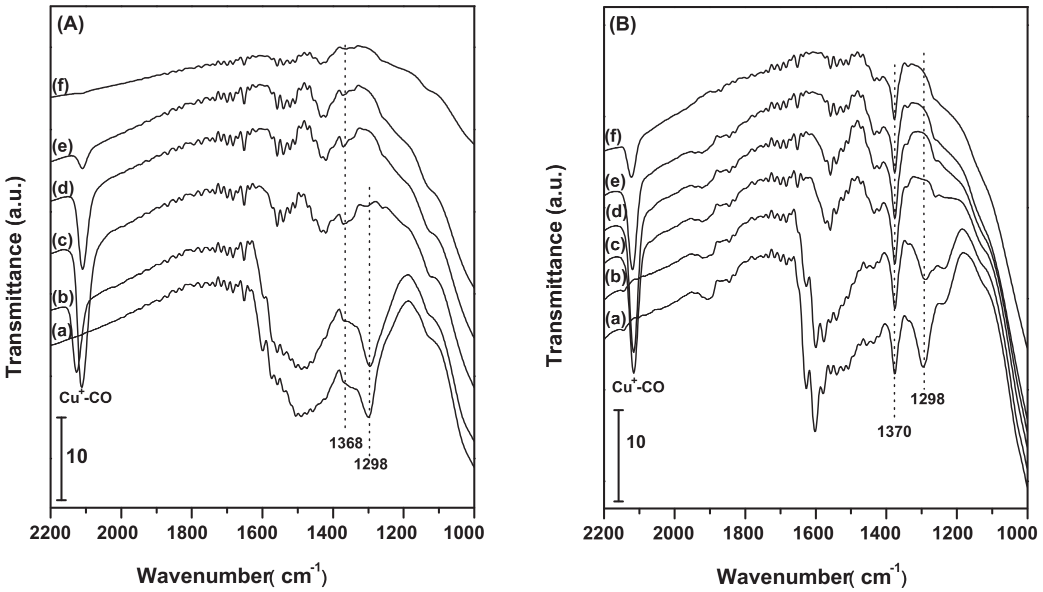


Fig. 7. FTIR spectra of NO-CO co-adsorbed on Cu/TiO$_2$ (A) and CuO/TiO$_2$-MP (B) samples: (a) 50 °C, (b) 100 °C, (c) 150 °C, (d) 200 °C, (e) 250 °C, and (f) 300 °C.

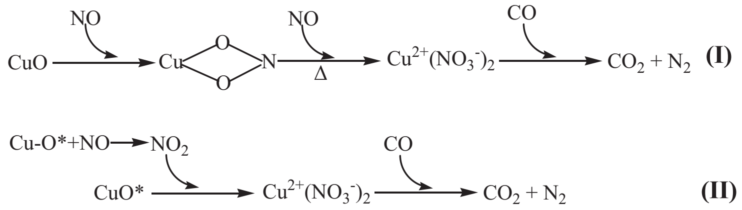
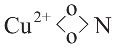


Fig. 8. A diagram of CO + NO reaction over CuO/TiO$_2$ samples without and with microwave plasma pretreatment.

gen species via plasma treatment, where NO$_2$ is easily transformed to NO$_3{}^-$. Simultaneously, the band for Cu$^+$–CO species at 2108 cm^{-1} appear, and is gradually weakened but still can be detected when the temperature reach to 300 °C. These results suggest that the redox properties of the sample surface have been changed during microwave plasma processing, thus affecting the catalytic properties of CuO/TiO$_2$ samples. On the other word, it is reasonable to suggest that all these difference should be due to the new oxygen species (O*).

Generally, for selective catalytic reduction (SCR) pathway, oxygen gas normally will be introduced into the reaction system for oxidizing NO to NO$_2$, because NO$_2$ had higher reactivity than NO to nitrogen. Furthermore, the conversion of NO$_x$ is increased with the O$_2$ concentration [47]. The fact indicates that the presence of O$_2$ does not inhibit NO reduction but promotes it. Li et al. suggest that the NO$_x$ species are more easily adsorbed on the catalyst surface due to the presence of oxygen [47]. Chuang et al. have investigated the FTIR spectra of NO + O$_2$ co-adsorption on CuO/Al$_2$O$_3$ surface [41]. The results show that many adsorption species can be formed, particularly conducive to the formation of Cu^{2+}[NO$_3{}^-$]$_2$ species with introducing oxygen, which is an important intermediate for SCR pathway. They suggest that the reducing species, such as CO and C$_x$H$_y$, are introduced into the reaction system, can not only reduce Cu^{2+} to Cu$^+$ but also remove (NO$_3{}^-$)$_2$ to form N$_2$ and N$_2$O. In our current model reaction system (NO + CO), although no additional O$_2$ has been added, the Cu^{2+}[NO$_3{}^-$]$_2$ can also be formed over the microwave plasma pretreatment samples even at room temperature.

Hence, on the basis of this viewpoint and considering the above discussions, the possible reaction pathway for the formation of NO$_x$ species can be illustrated in Fig. 8. For CuO/TiO$_2$ sample without microwave plasma pretreatment exposed in NO + CO mixtures, it is assumed that NO is preferentially adsorbed on the surface of CuO/TiO$_2$ to produce  with the corresponding bands at 1291 cm^{-1} firstly. While prolonging the exposure time of sample CuO/TiO$_2$ with NO and increasing the temperature, a little amount of Cu^{2+}[NO$_3{}^-$]$_2$ species (∼1350 cm^{-1}) will be formed (Fig. 8I). These adsorbed species react with CO activated by Cu$^+$ species and produce N$_2$ and CO$_2$. While, for the catalysts treated by microwave plasma (CuO/TiO$_2$-MP), a great amount of Cu^{2+}[NO$_3{}^-$]$_2$ species can be formed immediately over the catalyst surface. Combined with the results of TPR, a large number of higher active oxygen species (O*) has been formed on the surface of CuO/TiO$_2$-MP sample through the microwave plasma pretreatment, due to the enhancement of the reducibility of the dispersed copper oxide species by the TiO$_2$ support. When import of NO + CO to the reaction system, preferentially adsorbed NO species bond with O* to form NO$_2$ firstly, and then the NO$_2$ integrate with copper oxide to produce Cu^{2+}[NO$_3{}^-$]$_2$ species at lower temperature (Fig. 8II). Therefore, microwave plasma pretreatment induce the formation of the higher active oxygen species (O*), which should play an important role in the oxidation of NO to NO$_2$, as a result of improving the activity and N$_2$ selectivity.

4. Conclusion

The present work investigated the influence of microwave plasma pretreatment on the activities of CuO/TiO$_2$ catalysts in “NO + CO” model reaction. CuO/TiO$_2$ catalyst can remain it original morphology and particle size after plasma treatment. At 300 °C, the microwave plasma pretreatment improved both the NO conversion and the N_2 selectivity, which was dependent on the treatment time. This promotion in the activities of CuO/TiO_2 catalysts is attributed to the presence of the high active oxygen species (O^*), which can affect the redox properties of the catalysts and facilitate the NO adsorption on the surface as distorted nitrate species. As a result, these factors can explain the enhancement of activity and selectivity through microwave plasma pretreatment.

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