

The NO_x Storage and Desorption Activity of Mg/Al/Cu-NH₃·H₂O Hydrotalcites at Low Temperature

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Abstract

Ammonium containing catalyst Mg/Al/Cu-NH₃·H₂O hydrotalcite (HT) is prepared via co-precipitation, which is effective for NO_x storage under 160°C. XRD and FT-IR are employed to characterize its structure and show that ammonium is successfully incorporated into HT. Meanwhile, the HT still retains the typical structure of hydrotalcite-like compounds. NO_x storage activity tests indicate Mg/Al/Cu-NH₃·H₂O HT shows the significant NO_x removal activity at 156°C with a little Oxygen existence. TGA and DTG reveal the reaction of the released ammonium with NO_x and storage NO_x as nitrates will be responsible for the high NO_x storage activity. Additionally, the temperature programmed desorption test of NO_x adsorbed sample under 175°C proves NO_x is stored successfully into Mg/Al/Cu-NH₃·H₂O HT further.

Keywords

Hydrotalcites, NO_x Storage and Desorption, TG and DTG Analysis, FT-IR Characterization

1. Introduction

Nitrogen oxides (NO_x) emission which is resulted from the burning of fossil energy contributes heavily to the damage of environment and human beings, so it is a hot topic and concerned popularly [1]. Hydrotalcites with a general formula of $[M_{1-x}^{2+}M_x^{3+}(\text{OH})_2]^{x+}A_{x/m}^{m-} \cdot z\text{H}_2\text{O}$ not only have a high catalytic activity to NO_x removal but adsorb acid gas such as SO_x, CO_x and NO_x, because they are typically positively charged layers of brucite-like (Mg(OH)₂) [2] [3] [4]. The di-

valent or trivalent cations in the octahedral sites can be substituted with some catalytic activity cations and the ions in the interlayers can be substituted with some functional acid gas adsorbents, and so they received a great attention to be catalysts and adsorbents [5].

In the system with HTs or metal oxides as catalysts, reductant such as CO or NH₃ is absolutely necessary [6]. Ammonia has been employed as reductant for removing NO_x in industrial boilers and vehicle exhaust for years and many efforts have been made for improving NO_x and SO_x removal efficiency using ammonium as reductants. For example, Xinyan Zhang and his co-workers researched the selective catalytic reduction mechanism of N₂O by NH₃ over an Fe-Mordenite catalyst [7]. Huazhen Chang *et al.* prepared the fresh and sulfated MnO_x-CeO₂ catalysts and studied the performance of them for selective catalytic reduction of NO_x by NH₃ in a low temperature (T < 300°C) [8]. However, in the present technologies, NH₃ is needed to be supplied by an independent equipment and lead to the cost increase. On the other hand, the additional pollution resulted from ammonium itself is another serious problem [9]. Recently, the selective catalytic reduction of NO_x by urea (urea/SCR) has been widely accepted as the most efficient NO_x removal technologies to control the NO_x emissions in diesel engine. However, they still have serious drawbacks due to an additional urea tank to be refilled periodically for the urea/SCR [10]. In our previous research, Mg/Al/Cu/NH₄⁺ HT was prepared and an important result was found by means of sXAS x-ray adsorption scattering and CHN elementary analysis, *i.e.* ammonium can be induced into HT by incorporating with Cu²⁺ [9].

In this work, we report an efficient NO_x storage-decomposition hydrotalcite based on Mg/Al/Cu-NH₃·H₂O HT for low-temperature NO_x removal activity and the microscopic structure and thermal stability were investigated through combined lab-based tools. Besides the adsorption and storage activity removing NO_x is reported, and the temperature programmed desorption test of NO_x adsorbed sample under 175°C from room temperature to 400°C is investigated and the coherent mechanism is discussed.

2. Experimental Section

2.1. Hydrotalcites (HTs) Preparation

The Mg/Al/Cu HT incorporated with NH₃ (Mg/Al/Cu-NH₃·H₂O HT) were prepared via co-precipitation. 0.01 M aluminum nitrate nonahydrate, 0.03 M magnesium nitrate hexahydrate, 0.01 M copper nitrate hexahydrate, were dissolved in DI water (Solution 1). 0.01 M sodium hydroxide and 0.02 M sodium carbonate were dissolved in DI water (Solution 2). Solution 1 and solution 2 were slowly added dropwise into a flask containing 0.03 mol ammonia and 50 ml of water during vigorous stirring. The pH was controlled at 9 - 10 by controlling their addition rate at 60°C. The slurry was stirred for an additional 2 h and aged quiescently at 80°C for 18 h. The obtained precipitate was filtered, washed with distilled water until pH was 7, and then vacuum dried at 80°C for 12 h into

Mg/Al/Cu-NH₃·H₂O HT powder. The chemical reagents (AR) above obtained from Shanghai Sinopharm Chemical Reagent Co., Ltd.

2.2. NO_x Storage and Decomposition Tests

The experiments were designed to obtain information on the NO_x storage and decomposition activity of the samples when atmospheric pressure N₂ with a flow rate of 54 - 60 l/h was fed into the reactor as a carrier gas. The gas mixture consisted of 10% NO and 90% N₂ with the flow rate of 2.6 mL/min. An approximately 300 mg sample was placed in the center of a quartz reactor tube (Figure 1) with mass space-velocity of 237 h⁻¹.

The sample which has been placed for 24 h in the drier was held in a small instrument covered with quartz cloth, and a K-type grounded thermocouple for temperature measurements was placed in the center of the tube which is blown by nitrogen gas before and after every test. The reactor was heated via a furnace and the temperature was controlled by a thermocouple connected with a temperature control device from 30°C - 600°C at room temperature and 60% humidity [11] [12]. Also, the reactor's exit was connected to an Germany MRU MGA infrared gas analyzer, which was used to analyze the inlet and outlet NO_x, NO and NO₂ concentration. The measurements last for 20 minutes.

The following equation was used to calculate the decomposition and storage rate of NO_x.

$$\eta = (\text{NO}_{\text{xi}} - \text{NO}_{\text{xo}}) / \text{NO}_{\text{xi}} \times 100\% \quad (1)$$

NO_{xi}: the inlet NO_x concentration; NO_{xo}: the outlet NO_x concentration.

The desorption performance of the samples after adsorbing NO_x for 30 minutes was obtained by putting it into the reactor once again with transferring N₂ at room temperature as a balanced gas to blow for 10 minutes at the flow rate of 900 ml/min. Rising and keeping the temperature according to the program with a flow rate of 900 ml/min N₂ as carrier gas, and then check the outlet NO and NO₂ concentration varies with temperature.

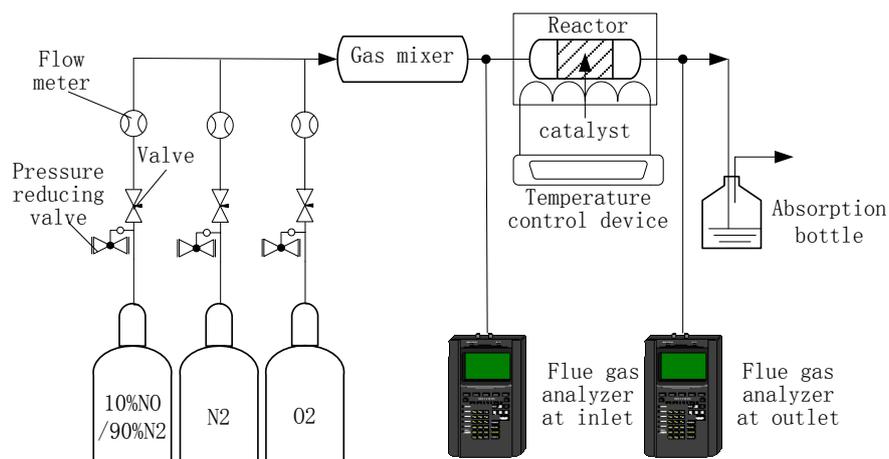


Figure 1. Catalytic test system.

2.3. Characterization

The characteristics such as powder X Ray diffraction (XRD) patterns for Mg/Al/Cu-NH₃·H₂O HT at room temperature were performed using a Brukers X-Ray Diffractometer equipped with Cu K α radiation ($\lambda = 0.15406$ nm, 40 kW, 4 mA). The patterns were acquired over a 2θ range of 5° - 80° within increment of 0.02° and scan speed of 0.5 s [13]. Fourier transform infrared (FT-IR) spectra were recorded at room temperature from 4000 to 350 cm⁻¹ using an iS10 Thermo Fisher spectrometer with a total reflection measuring head. 16 scans were collected with a resolution of 2 cm⁻¹. Thermogravimetric (TG) and DTG were carried out on a SII Nano TG-DTG 6300 instrument. Analysis was done from 50°C to 700°C at a heating rate of 10°C·min⁻¹ under nitrogen (100 mL·min⁻¹) [14].

3. Results and Discussion

3.1. XRD Characterization

Powder XRD of Mg/Al/Cu-NH₃·H₂O HT is in agreement with the standard hydrotalcite peaks. The diffraction peaks which 2θ located at 11.63°, 23.12°, 35.6°, 38.78°, 46.46° and 61.83° are assigned to characteristic Mg/Al HT layered structure [15], apparently, 2θ located at 29.37°, 48.93° and etc attributed to another new crystallinity resulted from the corporation of Cu²⁺ with NH₃·H₂O. Layer-layer d-spacing which is calculated from (003) peaks is about 0.75nm correlating with the reported hydrotalcite [16] (Figure 2).

3.2. NO_x Storage Performance

The previous research has provided direct evidence that ammonium incorporated

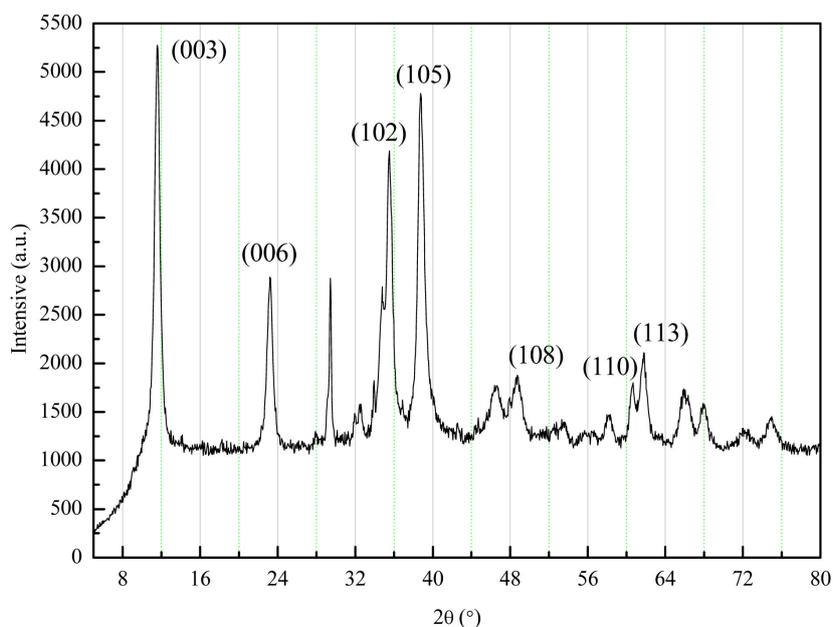
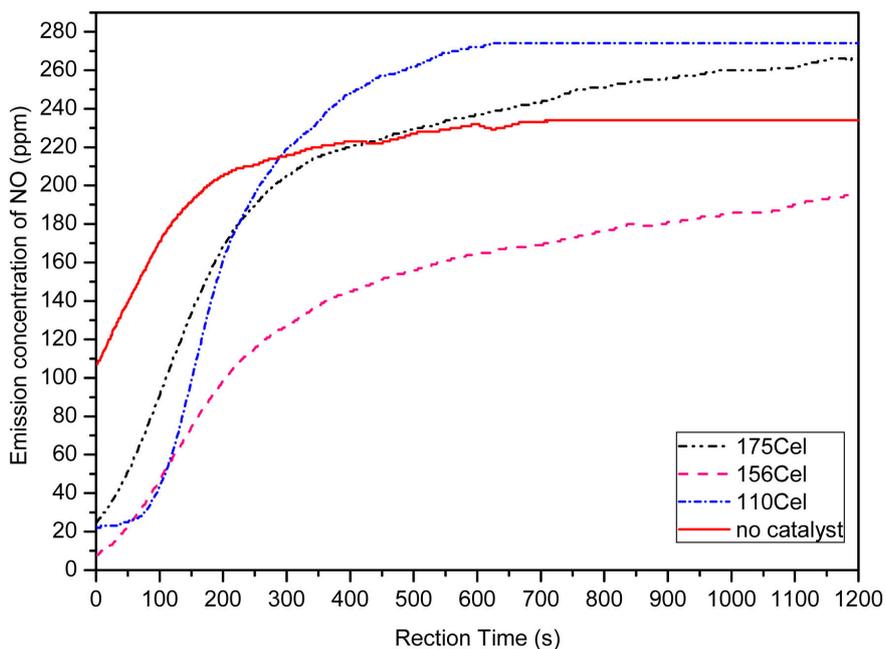
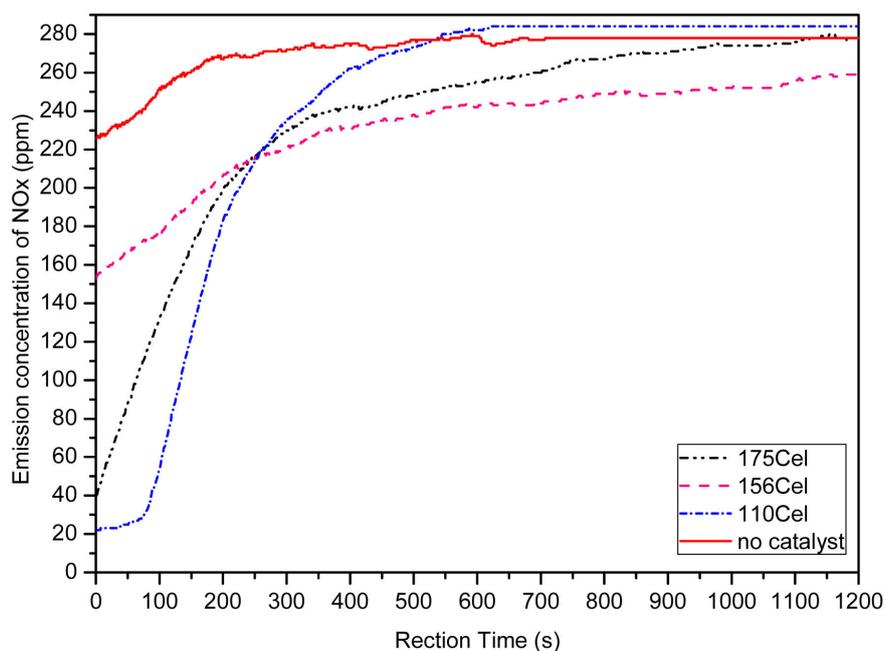


Figure 2. XRD spectra.

into Mg/Al/Cu/NH₄⁺ HT could release under 156°C from HT [9], and play an important role for NO_x removal. Herein, NO and NO_x removal performances of Mg/Al/Cu-NH₃·H₂O HT at 110°C (the concentration of O₂ is zero), 156°C (the concentration of O₂ is 1.2% - 1.7%), 175°C (the concentration of O₂ is zero) and no catalyst (the concentration of O₂ is 1.2% - 1.7%) are focused on as shown in **Figure 3(a)** and **Figure 3(b)**.



(a)



(b)

Figure 3. NO emission concentration with removal time (a) and NO_x emission concentration with removal time (b).

Figure 3(a) and **Figure 3(b)** clearly shows that the changes of NO and NO_x emission concentration with the removal time when simulation gas NO_x flows through Mg/Al/Cu-NH₃·H₂O HT at different temperatures, separately. The results of graph (a) indicate the NO removal rates of HT with 45.6%, 63.6% and 34.7%, separately at 110°C, 156°C and 175°C are higher than that of “No catalyst” within 290s, which agrees with the physical adsorption action of HTs [17]. The results registered from 290s to 1200s show the NO emission concentration at 156°C under 1.2% - 1.7% O₂ existence is lower than that of “no catalyst” can be attributed to the consumption of some NO being oxidized into NO₂, which reacts with the ammonium incorporated into Mg/Al/Cu-NH₃·H₂O HT released from HT at this temperature. In contrast, the results of NO emission concentration at 110°C and 175°C are higher than that of “No catalyst” reveal that not only NO can't react with ammonium efficiently, but NO adsorbed in the structure of HTs will release in that period as well. Graph (b) reflects that Mg/Al/Cu-NH₃·H₂O HT is effective for NO_x removal within 500 s at different temperature with or without O₂ existence, and the optimum NO_x removal condition is 156°C with a little O₂ existence. The amount of NO and NO_x storage conversion with about 0.13 mmol/g and 0.10 mmol/g, separately proves Mg/Al/Cu-NH₃·H₂O HT has higher storage conversion amount at the calculation of the data at 1200 s. The amount of NO storage conversion with 0.60 mmol/g is three times higher than that of NiMgAl hydrotalcite with 0.18 mmol/g at the calculation of the data at 5400 s [18]. It can be concluded the Mg/Al/Cu-NH₃·H₂O HT can realize physical and chemical adsorption for NO_x at the same time with O₂ existence at 156°C, NO_x can be stored in HT in the form of nitrates, while, only physical adsorption for NO_x without O₂ existence.

3.3. NO Desorption Test

NO_x temperature-programmed desorption of sample 175°C was carried out in a conventional flow system equipped with a temperature controller under N₂ as a carrier gas at a flow rate of 0.9 l/min. 300 mg sample was loaded in a quartz tube reactor, which was heated at 5°C/min from 100°C to 400°C. The NO, NO₂ and NO_x emission concentration is the averaged value of multiple measurements in the span of 60 minutes. It can be seen from **Figure 4**, the sample begins to release NO at about 160°C under heating and reaches the highest releasing speed at 265°C - 270°C. There are two peaks on the decomposition curves, the first one is assigned to the decomposition of NH₄NO₂, and the second one at 365°C - 370°C is assigned to the decomposition of metal nitrates such as magnesium nitrate. The thermal decomposition reaction equation of magnesium nitrate is as below [19]:



The sample begins to release NO₂ at about 250°C and reaches the highest releasing speed at 310°C - 315°C, which is attributed to the decomposition of NH₄NO₃. In the view of the releasing of NO and NO₂, the optimum thermal decomposition temperature range is 270°C - 310°C.

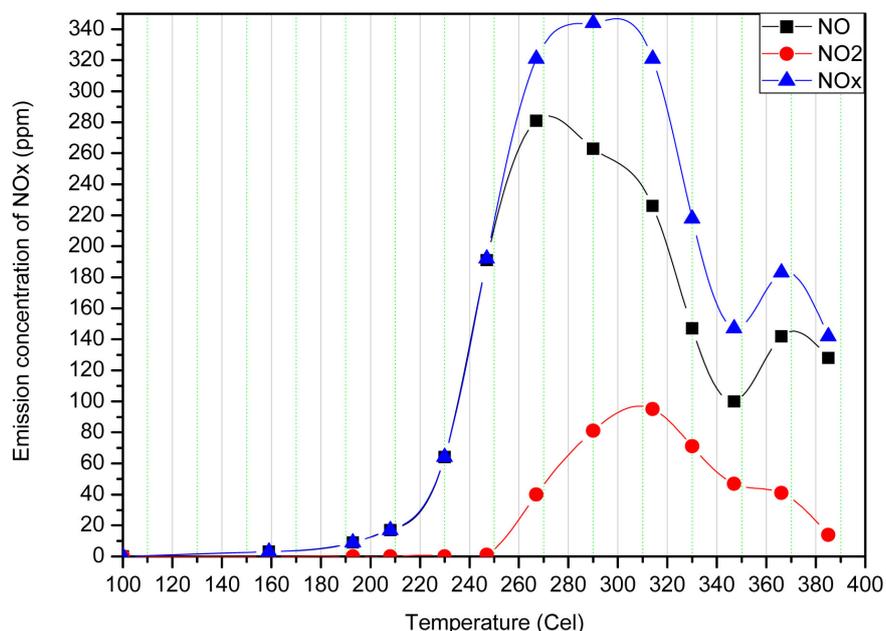


Figure 4. Desorption curves.

3.4. Thermal Analysis

Thermogravimetric analysis (TGA) and (DTG) traces of Mg/Al/Cu-NH₃·H₂O HT without adsorbing NO_x (0 sample) and after NO_x adsorption for 1200 seconds at 110°C (110°C sample) and 156°C (156°C sample), separately are obtained under nitrogen atmosphere. **Figure 5(a)** and **Figure 5(b)** describe the degradation process of the three samples. The samples present three stages of weight loss at TGA. The first stage, which occurs at a temperature below 159°C, is associated with the removal of the small molecules such as water and ammonium weakly adsorbed in the interlayer, the second stage of the thermal decomposition is observed between 159°C and 325°C, and there is an apparent thermal decomposition over 325°C with 156°C sample. Mg/Al/Cu-NH₃·H₂O HT without adsorbing NO_x displays a peak in DTG trace below 159°C attributed to the removal of ammonium or water which has been proved in the former research [9], however, the samples after adsorbing NO_x at 110 and 159°C don't exhibit any peaks which reveals that the small molecules has released from HTs when they are heated. It can be seen from the second stage, there is only one peak at 244°C for every sample, which is assigned to the decomposition and removal of hydroxyl groups in the brucite-like layers, as well as the CO₃²⁻, and other interlayer anions decomposition occurs over 200°C, 156°C sample showing weaker peak than the other two can be assigned to part of groups such as OH⁻ or CO₃²⁻ released from HT with the temperature increases. Meanwhile, in the case of the 156°C sample, a strong peak at about 368°C resulted from the decomposition of nitrates stored in HT is incoherent with the result of NO_x storage test.

3.5. FT-IR Analysis

The infrared adsorption spectra in **Figure 6** display the characteristic bands for

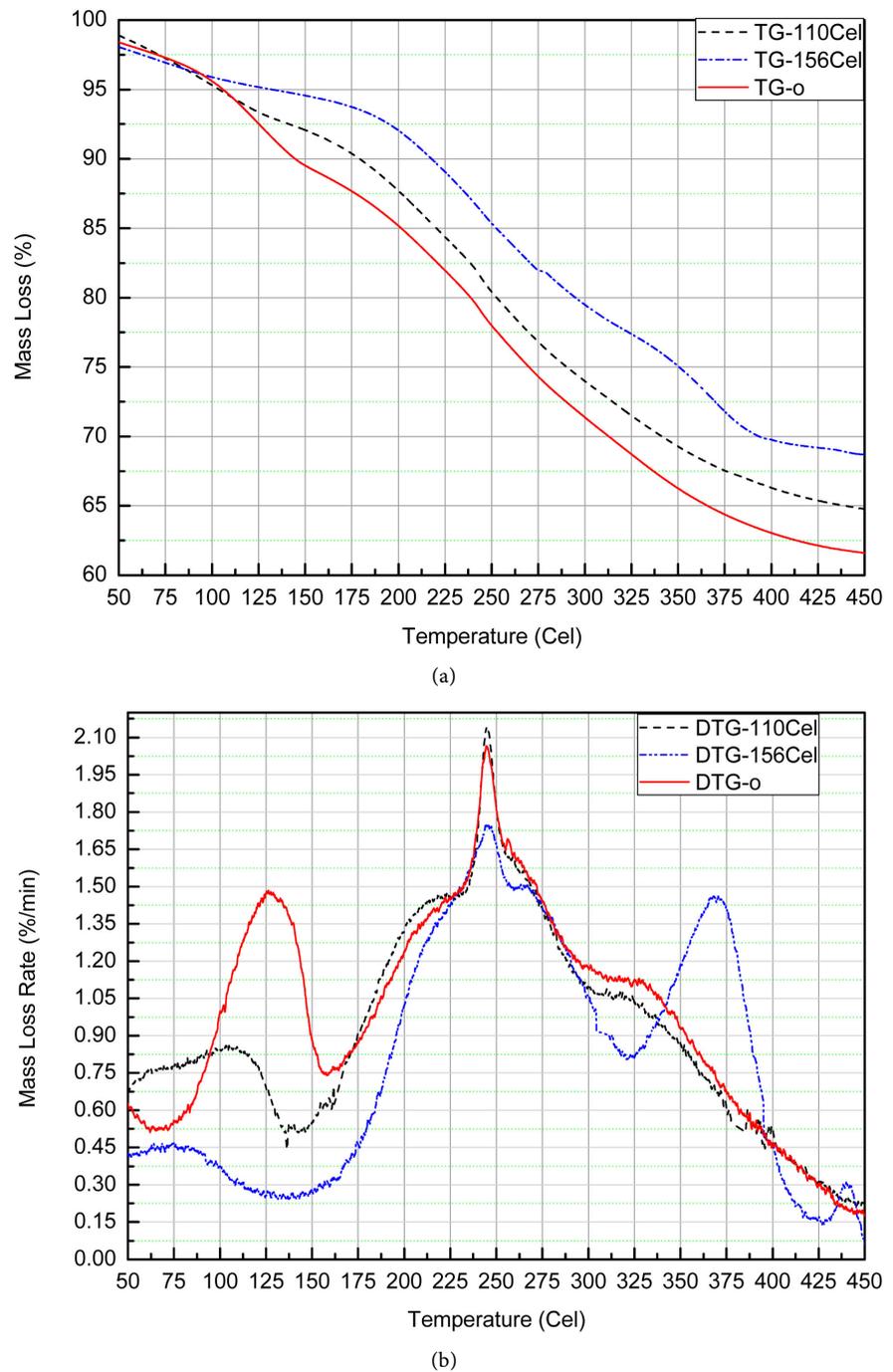


Figure 5. (a) TGA and (b) DTG trace.

the OH and CO bonds that are abundantly present in the mineral. Both Mg/Al/Cu-NH₃-H₂O HT and Mg/Al/Cu-NH₃-H₂O HT after adsorbing NO_x at 156°C present the similar spectrum with the typical hydrotalcites [20]. Didier Tichit and his co-workers investigated the FT-IR spectra of Mg/Al HT, and found the bands between 3500 and 3700 cm⁻¹ can be attributed to hydroxyl groups stretching vibration, and the broad bands at 3400 cm⁻¹ is assigned to the ν (NH) [21]. So 3325, 3408 and 3463 cm⁻¹ at **Figure 6** are visible to be attributed

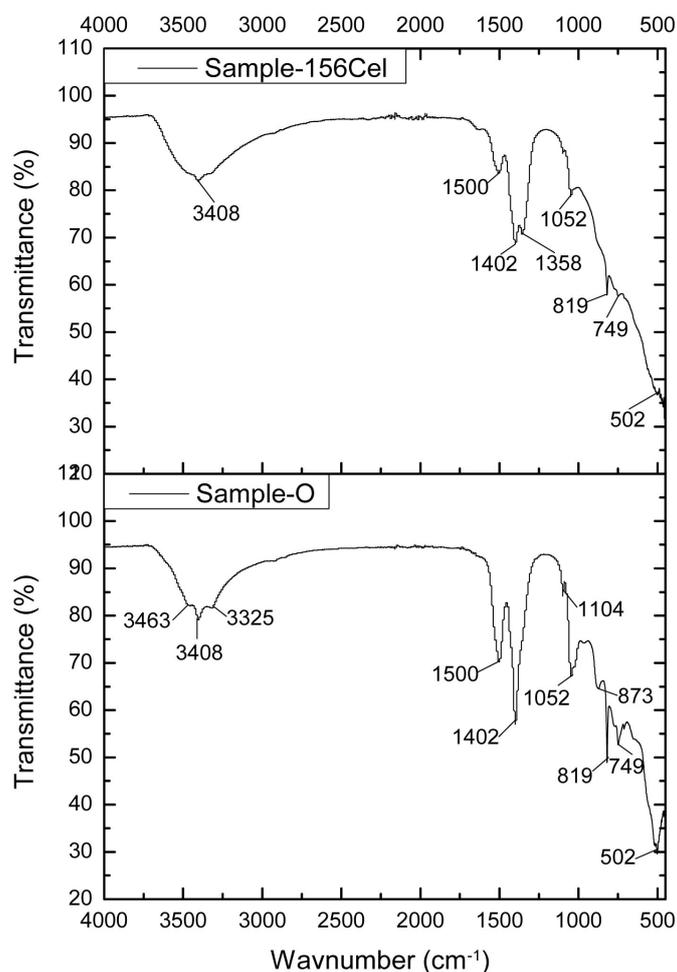


Figure 6. FT-IR spectra.

to the stretching vibrations of the amino group, which proves ammonium is incorporated by bonding with Cu^{2+} . Compared to $\text{Mg/Al/Cu-NH}_3 \cdot \text{H}_2\text{O}$ HT, the amino group band spectra of $\text{Mg/Al/Cu-NH}_3 \cdot \text{H}_2\text{O}$ HT after adsorbing NO_x at 156°C nearly disappeared, which indicates ammonium bounded with metal ions released when heating. In regard to the weak band at $1634 - 1642 \text{ cm}^{-1}$ assigned to interlaminar water molecules deformation mode doesn't appear in this specie, it can be ascribed the amount of interlayer water is little, which proves the mass loss at TGA below 159°C mainly result from the release of ammonium further more and the new phase in XRD should be resulted from ammonia [22]. The peak around 1402 cm^{-1} is caused by the asymmetric stretching vibration of the C-O bond of CO_3^{2-} group. Compared with the CO_3^{2-} group in free state (1415 cm^{-1}), this peak noticeably shifts to a lower wave number, which reveals that the CO_3^{2-} inserted between layers are not truly free ions, however, due to the hydrogen bonds with amino group isn't as strong as interlaminar water molecules, herein, the peak of CO_3^{2-} group shifts to 1402 cm^{-1} , not to 1380 cm^{-1} as reported. So, it can be concluded that and ammonium is easier to induce into the structure than water molecules, and this is very important to prepare ammo-

mium storage HTs. The weak band at 1358 cm^{-1} on the sample- 156°C should be assigned to the surface nitrite or nitrate transferred by the reaction between ammonium and NO_x [23]. The other absorption bands below 800 cm^{-1} are associated with the stretching and bending modes of metal-oxygen bonds.

4. Conclusion

The Mg/Al/Cu- $\text{NH}_3\cdot\text{H}_2\text{O}$ HT is prepared by co-precipitation, and the NO_x storage activity is discussed at 110°C , 156°C and 175°C with or without oxygen. Meanwhile, NO_x temperature-programmed desorption test of the NO_x adsorbed sample at 175°C is also investigated. Based on XRD result, we determine that a new phase resulted from the corporation of Cu^{2+} with $\text{NH}_3\cdot\text{H}_2\text{O}$ existed in HT. NO_x storage performance test proved the optimum NO_x removal condition is 156°C with a little O_2 existence. TGA and DTG analysis indicate the nitrates which are formed by ammonium released from HT before 156°C reacting with NO_x will decompose over 350°C . The FT-IR results agree with TGA and DTG analysis further. From NO_x temperature-programmed desorption test of the NO_x adsorbed sample at 175°C , we found the adsorbed NO will release over 160°C and NO_2 will release over 240°C continuously, so it is proved Mg/Al/Cu- $\text{NH}_3\cdot\text{H}_2\text{O}$ HT has the significant storage activity under 156°C .

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