

Switching hydrogenation pathways of furfural *via* reduction-degree engineering of Ni-based catalysts

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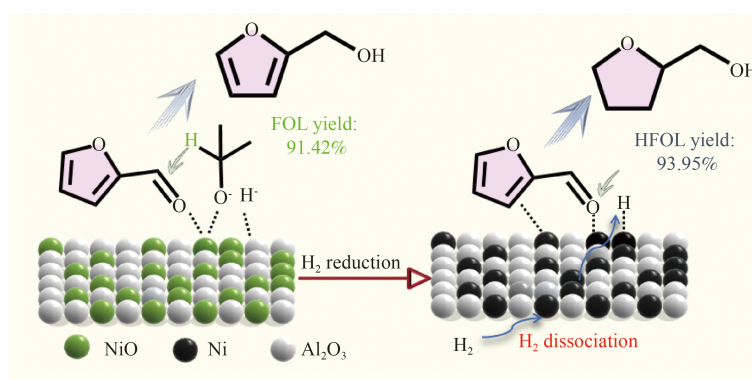
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Abstract: The selective hydrogenation of biomass-derived compounds is pivotal for the synthesis of high-value-added chemicals. The controllable hydrogenation in furfural for furfuryl alcohol or tetrahydrofurfuryl alcohol is highly desirable yet challenging. In this study, Ni₂Al-LDO (layered double oxide) metal oxide catalyst featuring highly dispersed surface NiO was synthesized *via* a structural topological transformation using layered double hydroxides. The

catalyst exhibited excellent performance in the hydrogenation of furfural, achieving 91.42% yield of furfuryl alcohol (FOL) at 160 °C and 1.4 MPa H₂. Gradual reduction of Ni₂Al-LDO produced Ni/NiO mixtures, enabling tunable shift from FOL to tetrahydrofurfuryl alcohol (HFOL) as the NiO content decreased and the Ni content increased. After a two-hour reduction at 700 °C, the HFOL yield reached 93.95%. CO₂-TPD, NH₃-TPD and FT-IR measurements revealed that variations in reduction degree on nickel influenced the adsorption behavior of furfural. NiO species could only adsorb the C=O group of furfural, isopropanol serves as the hydrogen source, and furfural undergoes hydrogenation to form furfuryl alcohol following the MPV conversion pathway. Whereas the flat adsorption mode on the metallic Ni surface facilitated the simultaneous adsorption and activation of both the furan ring and the carbonyl group of furfural. In addition, the hydrogen sources of the reaction systems also differ: the Ni₂Al-LDO catalyst is only capable of activating isopropanol, with the reaction proceeding *via* the MPV mechanism; in contrast, Ni₂Al-LDO-700 can activate both H₂ and isopropanol, where H₂ serves as the primary hydrogen source. This work elucidates a clear structure-activity relationship centered on the metal oxidation state and provides a practical reduction-engineering approach for designing adaptable catalysts in biomass upgrading.

Key words: furfural; selective hydrogenation; Ni-based catalysts; reduction degree; selectivity switching



With the rapid depletion of fossil energy sources and increasing environmental concerns, renewable biomass resources have attracted considerable attention as alternatives to traditional fossil fuels for the production of high-energy-density biofuels and high value-added

chemicals^[1–3]. Furfural (FAL) is one of the most important biomass-derived platform chemicals, owing to its wide range of high value-added derivatives. For instance, furfuryl alcohol (FOL), the partial hydrogenated product of FAL, is widely used in the production of resins, lysine, vitamins,

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and lubricants^[4–5]. Tetrahydrofurfuryl alcohol (HFOL), a deep hydrogenation product of furfural, is in high demand as a key precursor and environmentally friendly solvent in industrial, agricultural, and automotive sectors^[6–7]. However, controlling the selective hydrogenation of furfural toward either FOL or HFOL remains a significant challenge due to the complexity of parallel and sequential reaction networks^[8–9].

Traditionally, HFOL has been produced through a two-step hydrogenation process using commercial Cu-Cr catalysts under harsh conditions (140–180 °C, >4 MPa H₂), with FOL serving as an intermediate^[10]. Currently, many transition metals, such as the noble metals Pt^[11–12], Pd^[13–14], have been employed for furfural hydrogenation to produce HFOL under mild conditions. However, these catalysts often induce side reactions, such as hydroxyl or furan ring hydrogenolysis, and their high cost also severely limits their applicability in large-scale industrial processes^[15–16]. Consequently, research has increasingly focused on cost-effective non-precious metal catalysts, among which, nickel-based catalysts are highly attractive due to their proficient H₂ activation capability. However, Ni strongly adsorbs both C=O and C=C bonds, leading to unselective hydrogenation^[17–20].

To address this, modifying the catalyst surface structure to regulate the substrate adsorption configuration has emerged as a promising strategy for enhancing selectivity^[21–23]. For instance, Meng et al.^[24] demonstrated that different exposed facets of Ni, derived from hydrotalcite precursors, could direct furfural hydrogenation toward either FOL or HFOL with high selectivity. Similarly, Fu et al.^[25] achieved an exceptional HFOL yield over a Ni/C catalyst featuring abundant NiO species and exposed Ni(200) facets, highlighting the critical role of surface composition and microstructure. These findings confirm that tuning the microstructure of nickel particles can significantly influence their adsorption behavior.

Despite the remarkable advances achieved in the research of nickel-based catalysts, the hydrogenation of furfural still suffers from harsh reaction conditions and the preparation of such catalysts remains rather complicated^[26–28]. The rational design and development of novel catalysts with tunable selectivity toward a broad spectrum of high value-added hydrogenation products within a single catalytic system remains a considerable challenge. Furthermore, detailed mechanistic insights into how the catalyst structure influences the adsorption conformation of the substrate, such as the modulation of the reduction degree of nickel, are still lacking.

Herein, we report an approach to achieve switchable selectivity in furfural hydrogenation by precisely controlling the reduction degree of a flower-like Ni₂Al hydrotalcite-derived catalyst and Ni/NiO metal mixtures with different ratios were obtained by controlling the reduction time and temperature. The influence of nickel reduction degree on the adsorption configuration of furfural and the activation pathway of the hydrogen donor was systematically investigated. Notably, Ni₂Al-LDO exhibited

high selectivity for FOL (91.42%), while Ni₂Al-LDO-700 showed excellent selectivity for HFOL (93.95%). Based on structural characterization using XRD, CO₂-TPD and *in situ* FT-IR measurements, it was confirmed that variations in the reduction degree of nickel affect the adsorption sites of the substrate. This finding demonstrates that the Ni microstructure can be finely tuned within a single catalyst system to enable facile switching between the two target products. This work provides fundamental insights into the structure-activity relationship governed by metal oxidation state and offers a straightforward design principle for tunable catalysts in biomass upgrading.

1 Experimental

1.1 Materials

Nickel nitrate (II) hydrate (Ni(NO₃)₂·6H₂O), aluminum nitrate nonahydrate (Al(NO₃)₃·9H₂O, AR), urea (CH₄N₂O, 99.5%), furfural (FAL, AR, 99%), furfuryl alcohol (FOL, AR, 98%), tetrahydrofurfuryl alcohol (HFOL, AR, 98%), isopropyl alcohol (C₃H₈O₂, AR, 98%) and *n*-octanol (C₈H₁₈O, 99.5%) were purchased from Shanghai Aladdin Biochemical Technology Co., Ltd. All the chemicals were used as received and did not need to be further purified.

1.2 Catalyst synthesis

A series of nickel-aluminum hydrotalcite catalysts were prepared using the hydrothermal method, with Ni₂Al-LDH (layered double hydroxides) as an example. A solution containing 0.01 mol of aluminum nitrate, 0.02 mol of nickel nitrate, and 0.084 mol of urea was dissolved in 60 mL of deionized water, stirred at room temperature for 1 h until a homogeneous solution was obtained, and then transferred to the liner of a hydrothermal autoclave. The mixture was aged at 140 °C for 7 h, naturally cooled to room temperature, and subsequently rinsed repeatedly with deionized water until the supernatant reached a pH value of 7. The resulting green precipitate was dispersed in 200 mL of methanol and stirred at room temperature for 2 h to effectively remove the residual ions. Methanol was used due to its lower surface tension, which helps mitigate structural aggregation during subsequent drying (24 h). The obtained hydrotalcite precursor was calcined at 500 °C in an argon gas stream (60 mL/min) for 2 h, yielding a gray-green solid catalyst. The catalyst was then reduced at different temperatures and then passivated in 1% O₂/Ar at room temperature. The resulting samples were named as Ni₂Al-LDO-*T-t*, where *T* represents the reduction temperature and *t* denotes the reduction duration.

1.3 Characterization

The morphology of the catalysts was analyzed using a JSM-7401F scanning electron microscope (SEM). X-ray diffraction (XRD) patterns were recorded using a Bruker X-

ray powder diffractometer (Germany) equipped with a Cu $K\alpha$ radiation source ($\lambda = 1.5418 \text{ \AA}$), operating at 40 kV and 40 mA, with a scanning step size of $0.02^\circ/\text{s}$ and a scanning range of $10^\circ\text{--}80^\circ$. Prior to testing, the catalysts were ground into a fine powder and placed in a ceramic sample holder (inner diameter: 10 mm).

Nitrogen physisorption experiments were conducted on the reduced catalysts at -196°C using a fully automated gas adsorption analyzer (ASAP-2460, Micromeritics). Before measurements, 200 mg of the sample was dehydrated under vacuum at 200°C for 6 h. The specific surface area was calculated using the multi-point Brunauer-Emmett-Teller (BET) method ($p/p_0 = 0\text{--}1$), while the pore size distribution was determined by the Barrett-Joyner-Halenda (BJH) method.

X-ray photoelectron spectroscopy (XPS) was performed using an ESCALAB 250Xi spectrometer (Thermo Fisher) with an Al $K\alpha$ X-ray source ($h\nu = 1486.6 \text{ eV}$). The X-ray source operated at an accelerating voltage of 15 kV and a power of 80 W.

High-resolution XPS spectra were recorded with a step size of 0.05 eV and a pass energy of 25 eV. All binding energies were calibrated to the C 1s peak of adventitious carbon at 284.6 eV, and the spectra were analyzed using CasaXPS software.

Hydrogen temperature-programmed reduction (H_2 -TPR), carbon dioxide temperature-programmed desorption (CO_2 -TPD), ammonia temperature-programmed desorption (NH_3 -TPD), and hydrogen temperature-programmed desorption (H_2 -TPD) experiments were conducted using a microreactor system (CATLAB, Hiden Analytical Ltd.), coupled with a quadrupole mass spectrometer (QIC20, Hiden Analytical Ltd.). For the H_2 -TPR experiments, 30 mg of catalyst was weighed and dehydrated under an Ar atmosphere (30 mL/min) at 200°C for 2 h. The catalyst was then cooled to 50°C , the atmosphere was switched to 10% H_2 , and the temperature was ramped to 800°C at a rate of $5^\circ\text{C}/\text{min}$, while the exhaust gases were analyzed by mass spectrometry. For the CO_2 -TPD, NH_3 -TPD and H_2 -TPD experiments, 30 mg of catalyst was pretreated by reduction or dehydration, cooled to 50°C , purged with 10% CO_2/Ar , 10% NH_3/Ar or 10% H_2/Ar for 1 h, followed by purging with Ar (50 mL/min) for 1 h. The temperature was then increased to 800°C at a rate of $5^\circ\text{C}/\text{min}$, and the exhaust gases were analyzed by a quadrupole mass spectrometer.

In situ diffuse reflectance Fourier transform infrared spectroscopy (DRIFTS) experiments were conducted using a Thermo iS50 spectrometer equipped with an MCT liquid nitrogen-cooled detector and an *in situ* reaction cell. The catalyst samples were reduced under an H_2 atmosphere at 500°C (30 mL/min) for 2 h (heating rate: $10^\circ\text{C}/\text{min}$), followed by cooling in an Ar flow (30 mL/min) to 30°C , and background spectra were collected. Pyridine (probe molecule) or substrates (furfural, isopropanol) were introduced into the reaction cell using Ar (30 mL/min) at 30°C . After saturation of adsorption, Ar was passed through to remove physically adsorbed substances, followed by the collection of successive *in situ* FT-IR spectra. After collecting the background spectrum, CO (probe molecule,

10 mL/min) was introduced to achieve saturation adsorption on the catalyst surface, followed by purging with Ar to remove physically adsorbed CO. Continuous *in situ* FT-IR spectra were collected throughout the process. The FT-IR spectra were recorded within the range of $1900\text{--}2200 \text{ cm}^{-1}$ with 64 scans and a resolution of 4 cm^{-1} .

1.4 Hydrogenation experiments

Selective hydrogenation of furfural was conducted in a 10 mL micromagnetic heating reactor (Haian Huawei Scientific Research Instrument). Furfural (FAL, 0.6 mmol), catalyst (30 mg) and isopropanol (3 mL) were added to a 10 mL autoclave. The autoclave was purged five times with H_2 (by pressurizing to 1 MPa and then venting to atmospheric pressure) to ensure complete removal of air, then filled with hydrogen to the specified pressure. The reactor was subsequently heated to the required reaction temperature, and the reaction was carried out at 1000 r/min (time). After the reaction was completed, the reactor was rapidly cooled in an ice-water bath. The reaction mixture was then separated and diluted with isopropanol, followed by the addition of *n*-octanol as an internal standard for further analysis. All key catalytic experiments were repeated at least twice under the same conditions, with the error of the experimental results within $\pm 3\%$.

1.5 Product analysis

The separated liquid samples were quantitatively analyzed by gas chromatography (SHIMADZU GC-2018, flame ionization detector, equipped with HP-5 column), and all products were qualitatively analyzed by gas chromatography-mass spectrometry (GC-MS, Agilent 8860-8977B). The standard curves of reactants and products were plotted by internal standard method, and the substrate conversion and product selectivity were calculated by the following equations.

$$\text{Conv}_{\text{FAL}} = \frac{n_{\text{FAL initial}} - n_{\text{FAL final}}}{n_{\text{FAL initial}}} \times 100\% \quad (1)$$

$$\text{Sel}_{\text{FOL}} = \frac{n_{\text{FOL}}}{n_{\text{FAL initial}} - n_{\text{FAL final}}} \times 100\% \quad (2)$$

$$\text{Sel}_{\text{HFOL}} = \frac{n_{\text{HFOL}}}{n_{\text{FAL initial}} - n_{\text{FAL final}}} \times 100\% \quad (3)$$

In equations (1)–(3), where $n_{\text{FAL initial}}$ and $n_{\text{FAL final}}$ are the initial and final molar amounts of furfural, respectively; n_{FOL} and n_{HFOL} are the molar amounts of the corresponding products formed.

2 Results and discussion

2.1 Catalyst characterizations

Three-dimensional (3D) flower-like $\text{Ni}_2\text{Al-LDO}$ precursors with uniform morphology were synthesized via the urea decomposition method, as shown in Figure 1(a),

(b)^[29]. After reduction, the flower-like morphology of the precursor was well preserved, and distinct open spaces between adjacent nanolayers were observed, as shown in the enlarged illustrations (Figure 1(c), (d)). Upon reduction, the surface roughness of the nanosheets increased, as shown in Figure 1(f), but the overall 3D hierarchical structure did not collapse. This preserved 3D layered hydrotalcite structure provides an effective contact area between the reaction substrate and hydrogen throughout the reaction system^[30], is expected to sufficient active sites to optimize hydrogenation performance.

The crystal structures of the synthesized samples were characterized using XRD. As shown in Figure 2(a), the Ni₂Al-LDO catalyst exhibited patterns corresponding to the NiO crystal planes ((200) and (220))^[31]. No characteristic diffraction patterns of Al₂O₃ were found, implying an amorphous phase. For samples reduced at 700 °C, the intensity of the Ni diffraction patterns ($2\theta = 44.5^\circ$, 51.8° , 76.3°) increased, whereas the NiO diffraction patterns disappeared completely after 2 h of reduction at 700 °C. This indicates that an increase in reduction temperature promotes the reduction of more Ni²⁺ to Ni⁰^[32]. The average crystallite sizes of NiO in Ni₂Al-LDO and Ni⁰ in Ni₂Al-LDO-700 were estimated using the Scherrer equation based on the NiO(200) and Ni(111) peaks, respectively (Table 1). The NiO crystallite size was calculated to be 4.04 nm. After reduction at 700 °C, the metallic Ni crystallite size increased to 8.46 nm, indicating the occurrence of metal

particle sintering under severe reduction conditions^[17].

The textural properties of the catalysts were investigated by N₂ physisorption. Both Ni₂Al-LDO and Ni₂Al-LDO-700 exhibit type IV isotherms with an H3-type hysteresis loop (Figure 2(b)), which is characteristic of mesoporous materials with slit-shaped pores. The corresponding pore size distribution curves (Figure 2(c)) show a unimodal peak centered in the mesoporous region, confirming the presence of uniform mesopores^[33]. The specific surface area of Ni₂Al-LDO was measured to be 173.3 m²/g, which decreased to 160.2 m²/g after reduction (Table 1). The layered structure of the catalysts contributed to their relatively high specific surface area^[34–35]. Additionally, the pore volume was calculated to be 0.6 cm³/g for both samples. The high surface area and well-developed mesoporosity are beneficial for the diffusion of reactant molecules, such as furfural, in liquid-phase reactions^[36].

The electronic valence state of Ni in the different samples was investigated using (XPS), as depicted in Figure 3 (a)–(h). According to the literature, the composite peaks obtained were deconvoluted into several peaks, which are summarized in Table S1. These results indicate that tuning the reduction parameters can effectively modulate the surface reduction degree Table S1 of nickel^[37]. It is important to note that even in the sample reduced at 700 °C, a significant portion of Ni²⁺ remains. This is attributed to the surface re-oxidation during the passivation step prior to XPS measurement and the possible presence of a thin oxidized layer upon air exposure^[38].

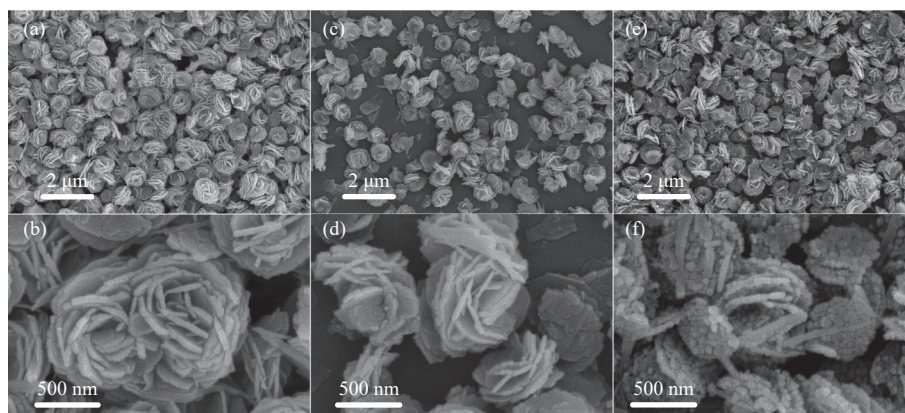


Fig. 1 SEM images of Ni₂Al catalyst at different magnifications ((a), (b) Ni₂Al-LDH precursor; (c), (d) Ni₂Al-LDO; (e), (f) Ni₂Al-LDO-700)

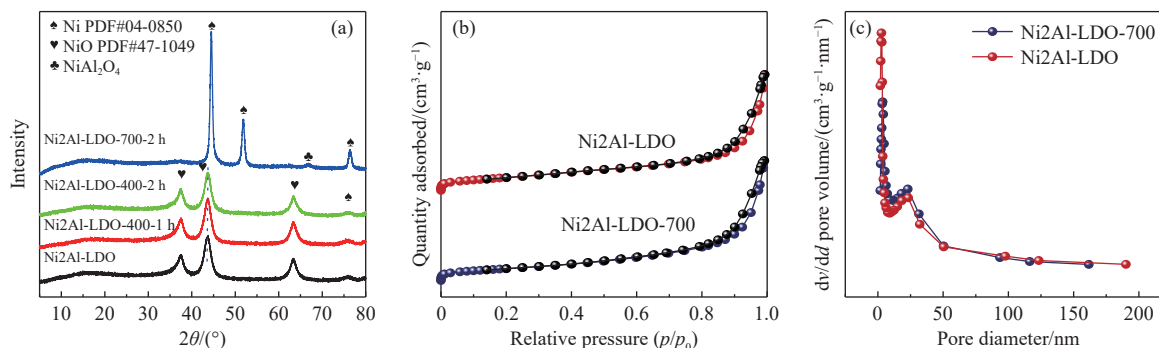


Fig. 2 (a) XRD patterns of different samples, (b) N₂ adsorption-desorption isotherms and (c) the corresponding pore size distribution curves of Ni₂Al-LDO and Ni₂Al-LDO-700

Table 1 Textural properties of Ni2Al-LDO and Ni2Al-LDO-700 samples

Entry	Catalyst	$S_{\text{BET}}/(\text{m}^2 \cdot \text{g}^{-1})$	$v_{\text{total}}^{\text{a}}/(\text{cm}^3 \cdot \text{g}^{-1})$	Crystallite size ^b /nm
1	Ni2Al-LDO	173.3	0.6	NiO: 4.04
2	Ni2Al-LDO-700	160.2	0.6	Ni: 8.46

^a: Determined from the aggregation of N_2 adsorbed at a relative pressure (p/p_0) of 0.99; ^b: Calculated by Ni(111) or NiO(200) reflections based on Scherrer equation.

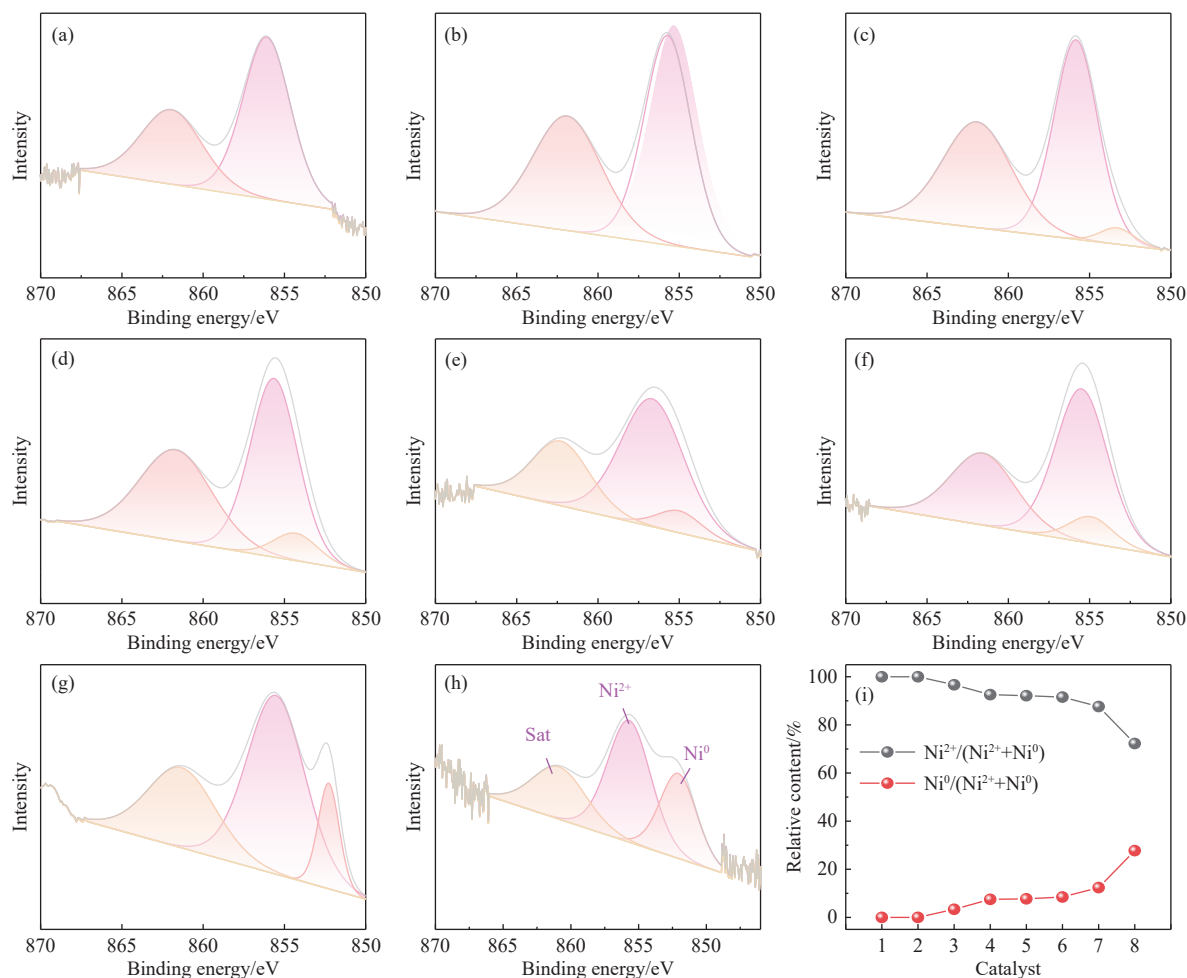


Fig. 3 Ni 2p spectra of (a) Ni2Al-LDO, (b) Ni2Al-LDO-400-0.5h, (c) Ni2Al-LDO-400-1h, (d) Ni2Al-LDO-400-1.5h, (e) Ni2Al-LDO-400-2h, (f) Ni2Al-LDO-500-2h, (g) Ni2Al-LDO-600-2h and (h) Ni2Al-LDO-700-2h catalysts; (i) the relative proportions of Ni^0 and Ni^{2+} derived from the XPS spectral fitting, where labels 1 to 8 on the horizontal axis correspond to samples (a) through (h)

2.2 Catalytic performance

The catalytic hydrogenation of furfural over a series of hydrotalcite-derived catalysts with varying Ni/Al ratios was first investigated. In the case of the Ni2Al-LDO catalyst (Figure 4(a)), FOL was the dominant product regardless of the Ni/Al ratio, achieving a yield of over 90% at complete conversion. This suggests that the NiO sites in these catalysts are highly active and selective for the partial hydrogenation of the carbonyl group. In contrast, the fully reduced Ni2Al-LDO-700 catalysts exhibited a strong dependence on nickel loading (Figure 4(b)). At a low Ni/Al ratio (e.g., 1:2), the primary product remained FOL.

However, as the Ni/Al ratio increased to 2:1, the yield of tetrahydrofurfuryl HFOL dramatically rose to 93.95% at full conversion. This indicates that a sufficient density of reduced metallic Ni^0 sites is crucial for driving the deep hydrogenation of the furan ring. Based on these results, the catalyst with a Ni/Al ratio of 2:1, which displayed the highest HFOL yield, was selected for subsequent studies on the effect of reduction degree.

To systematically tune the reduction degree, the Ni2Al-LDO catalyst was subjected to reduction at varying temperatures and durations, guided by H_2 -TPR analysis (Figure S1) which indicated a main reduction onset around 400 °C. The catalytic performance as a function of reduction temperature and duration is summarized in Figure 5.

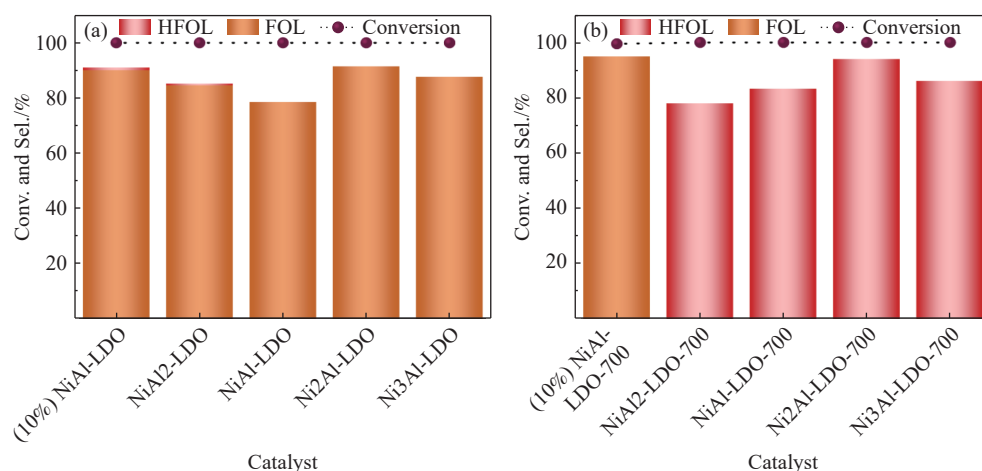


Fig. 4 Effects of Ni/Al molar ratio on FAL hydrogenation over (a) Ni_xAl-LDO and (b) Ni_xAl-LDO-700
Reaction conditions: 60 mg of FAL, 3 mL of isopropanol, 30 mg of catalyst, 1.6 MPa H₂, 140 °C, 3 h.

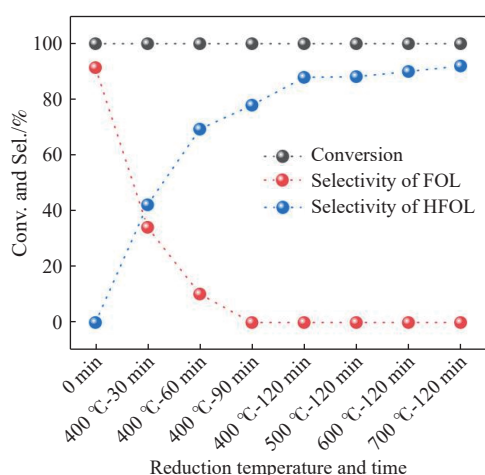


Fig. 5 Effects of reduction degree of catalysts on FAL hydrogenation

Reaction conditions: 0.6 mmol of FAL, 3 mL isopropanol, 30 mg of catalyst, 1.6 MPa H₂, 140 °C, 3 h.

Therefore, Ni₂Al-LDO catalysts were subjected to reduction at various temperatures and durations, starting from 400 °C, to systematically examine the influence of reduction degree. According to Figure 5, the unreduced catalyst exhibited a single activity toward furfuryl alcohol with a yield of 91.42%. As the reduction time increased from 400 °C, the selectivity for furfuryl alcohol decreased while that for tetrahydrofurfuryl alcohol increased, reaching a yield of 77.96% after 90 min of reduction. When the reduction time was extended to 2 h and the temperature increased from 400 to 700 °C, NiO was completely reduced (as confirmed by XRD), the HFOL yield was further optimized to 93.95% at 100% conversion. These results demonstrate a clear correlation between the catalyst reduction degree (Ni⁰/NiO ratio) and the hydrogenation pathway: NiO-rich surfaces favor carbonyl hydrogenation to FOL, while Ni⁰-rich surfaces promote subsequent furan ring hydrogenation to HFOL.

The distinct catalytic behaviors of Ni₂Al-LDO (FOL-selective) and Ni₂Al-LDO-700 (HFOL-selective) were further compared under different reaction conditions

(Figure 6). For the Ni₂Al-LDO catalyst, the complete furfural conversion required relatively harsh conditions. In contrast, the fully reduced Ni₂Al-LDO-700 catalyst achieved 100% conversion under much milder conditions, and the HFOL yield remained consistently high (>90%) across a wide range of temperatures, pressures, and reaction times. This highlights the superior activity and outstanding performance of the metallic Ni⁰ sites for the deep hydrogenation reaction

Finally, the stability of the two catalysts was evaluated over five continuous reaction cycles (Figure 7). At the end of each reaction cycle, the liquid phase was separated from the catalyst by centrifugation, followed by rinsing the catalyst three times with ethanol and water, and drying it at 40 °C for 12 h before reuse. Each experiment ensured the use of 30 mg of catalyst, with any loss supplemented by fresh catalyst. The recovery rate was calculated based on the ratio of fresh catalyst to used catalyst. As shown in Figure 7, the furfural conversion of the Ni₂Al-LDO catalyst remained above 93.8% over five cycles, while the selectivity for furfuryl alcohol slightly decreased from 87.79% to 82.09%, with minimal loss of activity. Compared to the fresh catalyst, the used Ni₂Al-LDO catalyst exhibited a similar crystal structure (Figure S2), and the NiO grain size, calculated to be 4.89 nm (Table S2), was negligible increase to that of the fresh catalyst, suggesting that Table S2 Ni₂Al-LDO demonstrated good stability in liquid-phase reactions. In contrast, the Ni₂Al-LDO-700 catalyst maintained a 100% conversion rate after five cycles; however, its HFOL selectivity decreased from 91.14% to 81.04%. Significantly, this selectivity could be largely restored to 89% upon re-reduction of the spent catalyst, suggesting that the deactivation is primarily due to the oxidation of metallic Ni⁰ during the reaction/repeat cycle rather than permanent structural degradation. XRD of the spent Ni₂Al-LDO-700 (Figure S3) confirmed a decrease in metallic Ni peak intensity and a slight increase in Ni⁰ crystallite size (to 10.35 nm, Table S2), indicating some surface oxidation and mild particle sintering. Overall, both catalysts demonstrated satisfactory recyclability, which can be attributed to the flower-like structure of layered double hydroxides.

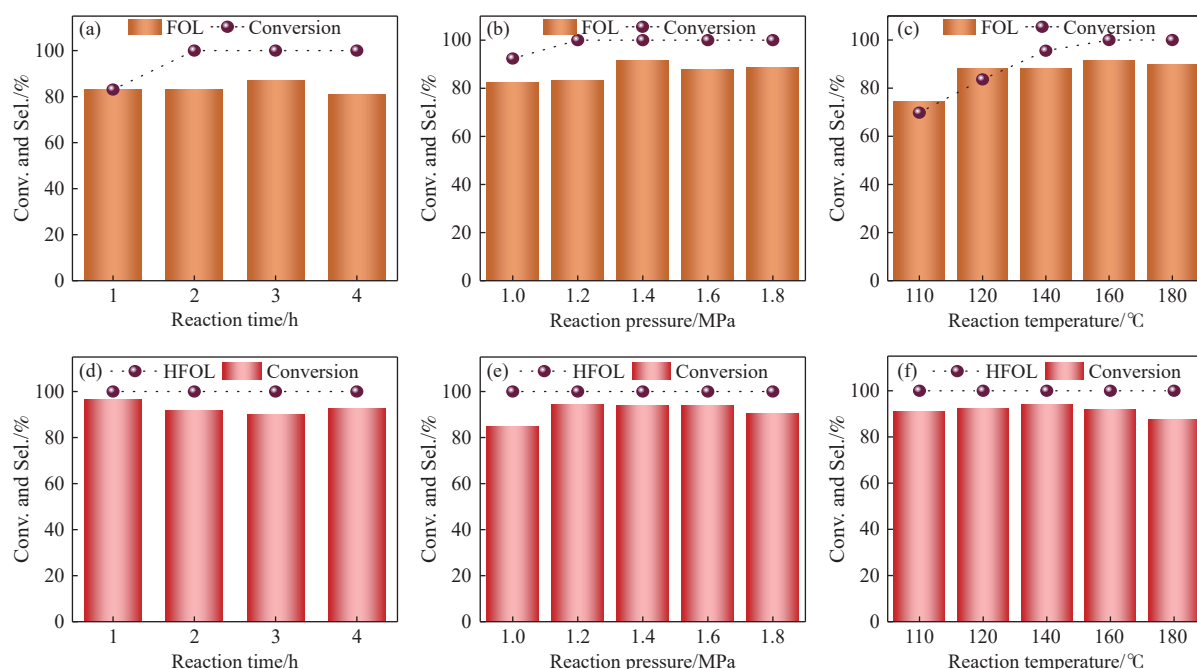


Fig. 6 Effects of reaction time, reaction pressure and reaction temperature on FAL hydrogenation reaction
(a)–(c): Ni2Al-LDO, (d)–(f): Ni2Al-LDO-700.
Reaction conditions: 0.6 mmol FAL, 3 mL of isopropanol, 30 mg of catalyst.

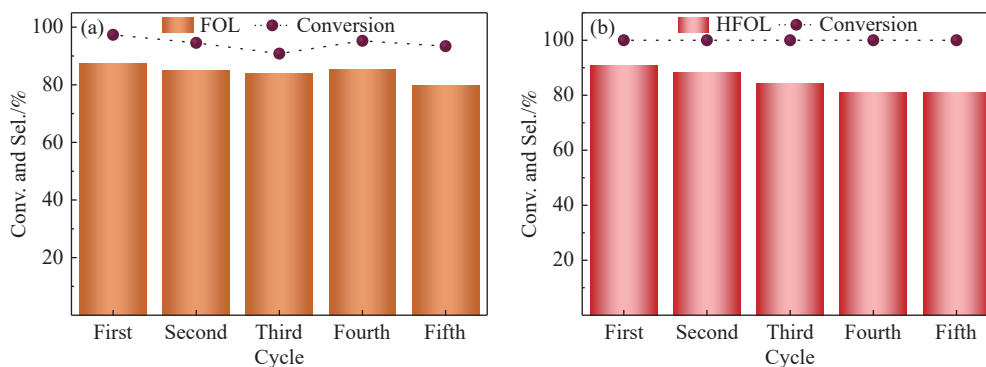


Fig. 7 Performance of (a) Ni2Al-LDO and (b) Ni2Al-LDO-700 catalysts during five consecutive runs
Reaction conditions: 0.6 mmol FAL, 30 mg of catalyst, 3 mL isopropanol.

2.3 The activation of hydrogen and isopropanol

Since isopropanol is used as the solvent and the reaction atmosphere consists of H_2 , it is essential to first determine the hydrogen donor for the reaction to elucidate the hydrogenation mechanism. As shown in Figure 8(a), control experiments were conducted under a N_2 atmosphere (1.4 MPa) at 160 °C for 3 h. In contrast, the catalytic activity of Ni2Al-LDO under a hydrogen atmosphere showed no significant difference compared to that under a nitrogen atmosphere. This indicates that the two catalysts exhibit different hydrogenation behaviors. Furthermore, the two catalysts exhibit different hydrogen consumption behaviors. As shown in the H_2 -TPD profile in Figure 8(b), Ni2Al-LDO-700 exhibits two strong hydrogen adsorption peaks: one in the low-temperature region (centered at 119.2 °C) and the other in the high-temperature region (centered at 196.3 °C). These peaks are attributed to the

dissociation of H–H bonds on metallic Ni particles^[39]. In contrast, Ni2Al-LDO shows no detectable H_2 adsorption, indicating its inability to dissociate hydrogen. This result is consistent with its inactivity under pure H_2 atmosphere in the control experiment.

The adsorption behavior of isopropanol on the catalyst was investigated using *in situ* FT-IR and CO_2 -TPD. As shown in Figure 9, three C–H stretching vibration peaks were observed after isopropanol adsorption on the catalyst: $\nu(CH_3)$, $\nu(CH_2)$, $\nu(CH)$, corresponding to 2970.4, 2935.1 and 2868.4 cm^{-1} , respectively. These results indicate that the prepared catalysts effectively adsorb isopropanol.

To assess the catalysts' ability to abstract hydride from isopropanol (a key step in the MPV mechanism), their surface basicity were evaluated *via* CO_2 -TPD (Figure 9(b)). Furthermore, the acidic nature of the C–H bond in isopropanol was confirmed by CO_2 -TPD analysis, as shown in Figure 9(b).

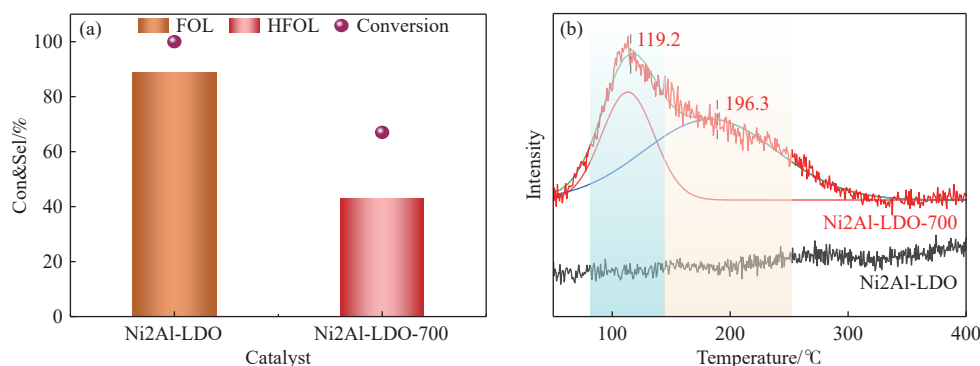


Fig. 8 (a) Performance of Ni₂Al-LDO and Ni₂Al-LDO-700 catalysts under N₂ atmosphere (Reaction conditions: 0.6 mmol of FAL, 30 mg of catalyst, N₂ 1.4 MPa, 160 °C, 3 h), (b) H₂-TPD profiles of Ni₂Al-LDO and Ni₂Al-LDO-700 catalysts

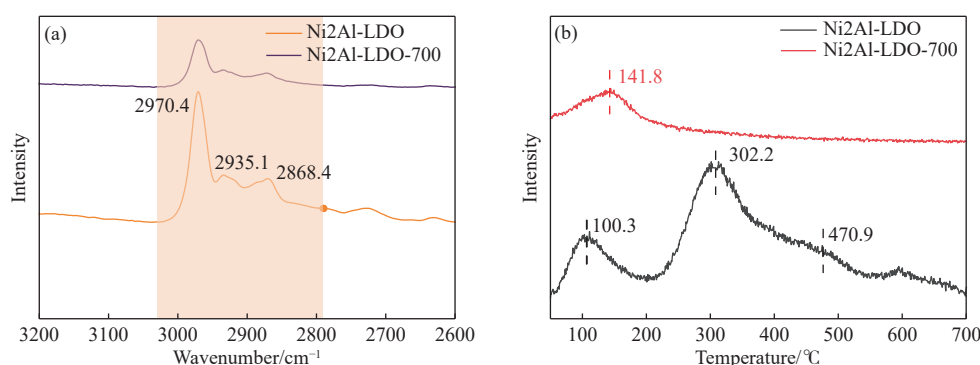


Fig. 9 (a) *In situ* FT-IR spectra of isopropanol adsorption on the catalysts, (b) CO₂-TPD profiles of Ni₂Al-LDO and Ni₂Al-LDO-700

Both catalysts exhibit CO₂ desorption peaks, with three distinct peak regions centered at 100.3, 302.2 and 470.9 °C, corresponding to weak, medium, and strong basic sites, respectively^[40]. According to previous studies, weak, medium, and strong basic sites originate from hydroxyl groups, M⁺–O[–] species, and low-coordinated oxygen anions, respectively^[41]. It is evident that the Ni₂Al-LDO catalyst possesses a higher density of basic sites compared to Ni₂Al-LDO-700, resulting in stronger C–H adsorption of isopropanol. However, isopropanol binding to the basic sites occurs in both catalysts. Based on these results, for the unreduced catalyst, it cannot dissociate hydrogen but exhibits basicity. The furfural hydrogenation process follows the Meerwein-Ponndorf-Verley (MPV) pathway, with isopropanol serving as the hydrogen donor and the basic sites facilitating hydrogen transfer. In contrast, for the reduced catalyst, Ni enables hydrogen dissociation, and H₂ acts as the primary hydrogen source^[42].

2.4 Adsorption configuration of furfural and reaction mechanism

The adsorption configuration of the substrate plays a crucial role in determining the hydrogenation reaction pathway and selectivity. The acid sites on the catalyst

surface were initially characterized using pyridine infrared spectroscopy (Py-FTIR, Figure 10(a)) and temperature-programmed desorption of ammonia (NH₃-TPD, Figure 10(b)). As shown in Figure 10(a), both samples exhibit a distinct characteristic band of pyridine adsorption at 1445.9 cm⁻¹, which is attributed to the Lewis acid sites of the metal oxides (NiO and Al₂O₃)^[43]. As presented in Figure 10(b), the NH₃ desorption peaks at 100.7 and 500.2 °C correspond to two broad peaks, which are associated with weak (W) and strong (S) acid sites, respectively. A comparative analysis indicates that the unreduced Ni₂Al-LDO possesses a higher overall acidity, particularly in the strong acid region, than the reduced Ni₂Al-LDO-700.

The adsorption behavior of furfural on the catalyst was further investigated (Figure 10(d)), and the surface electron density of Ni along with the bonding state of CO was analyzed using *in situ* FT-IR (Figure 10(c)). As shown in Figure 10(c), for the Ni₂Al-LDO-700 catalyst, CO is adsorbed on monometallic Ni in both linear (2074.4 and 2046.8 cm⁻¹) and bridge (1960.3 cm⁻¹) configurations^[44].

These frequencies correspond to CO adsorption on metallic Ni, confirming that the catalyst possesses both low and high coordination adsorption sites, which are capable of adsorbing the furan ring and carbonyl group of furfural on the catalyst surface^[45]. In contrast, for the Ni₂Al-LDO catalyst, no significant CO adsorption is observed, as the chemical state of nickel is predominantly NiO.

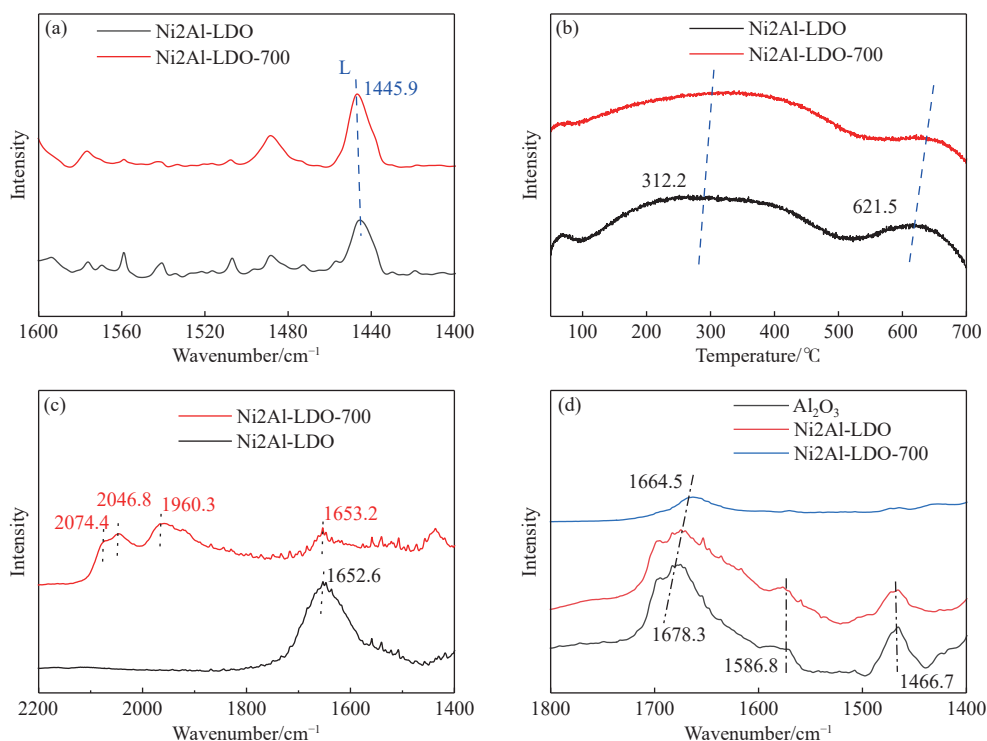


Fig. 10 (a) Pyridine-adsorption infrared spectra, (b) NH_3 -TPD profiles, (c) *In situ* CO DRIFT spectra, (d) Gas-phase infrared spectra of furfural adsorbed on Al_2O_3 , Ni2Al-LDO and Ni2Al-LDO-700 catalysts

As shown in Figure 10(d), gas-phase furfural adsorbed on Al_2O_3 exhibits a distinct Lewis acid adsorption peak for ν (C=O) at 1678.3 cm^{-1} , while ν (C=C) and furan ring vibrations appear at 1586.8 and 1466.7 cm^{-1} , respectively^[46]. Notably, the chemisorption of furan rings on the catalyst surface leads to the disappearance of the ν (C=C) and furan ring vibration peaks^[47]. As depicted in the figure, both Ni2Al-LDO and Ni2Al-LDO-700 catalysts exhibit corresponding adsorption peaks at 1671.2 and 1664.5 cm^{-1} , which are red-shifted compared to Al_2O_3 , indicating that the C=O group undergoes activated adsorption on both catalysts. Moreover, the Ni2Al-LDO catalyst displays characteristic furan ring bands at 1586.8 and 1466.7 cm^{-1} , suggesting that only the C=O group of furfural is adsorbed on the catalyst surface. In contrast, the spectral bands of ν (C=C) and furan ring vibrations on Ni2Al-LDO-700 are negligible, indicating that furfural is adsorbed in a parallel

configuration on the catalyst surface^[48]. These findings demonstrate that Ni2Al-LDO-700 exhibits a stronger adsorption capacity for both the furan ring and the C=O group compared to Ni2Al-LDO, making it more favorable for the complete hydrogenation of furfural molecules. Conversely, the Ni2Al-LDO catalyst primarily facilitates the chemisorption of the C=O group, which may hinder the hydrogenation of the furan ring.

In summary, the comprehensive reaction mechanism dictated by the nickel reduction degree for the two catalysts are illustrated in Figure 11. For the Ni2Al-LDO catalyst, NiO functions as a Lewis acid site that adsorbs the C=O group of furfural, while basic sites in the catalyst adsorb the α -hydrogen from isopropanol. This interaction facilitates the transfer of proton hydrogen from the hydrogen donor to the acceptor, resulting in the formation of furfuryl alcohol.

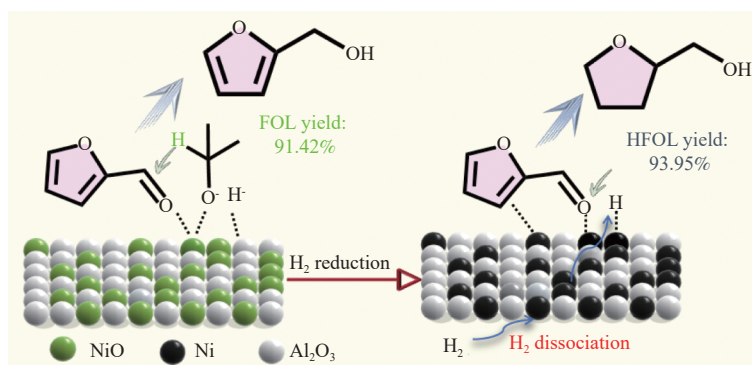


Fig. 11 Mechanism of the influence of different reduction degree of Ni-based catalysts over hydrogenation selectivity of furfural

In contrast, for the Ni₂Al-LDO-700 catalyst, the NiO reduction degree is negligible, the primary active sites are metallic Ni, which enable simultaneous adsorption on both the furan ring and the C=O group of furfural. Additionally, Ni facilitates the dissociation of H₂ into H–H, leading to the complete hydrogenation of furfural to tetrahydrofurfuryl alcohol. The influence of Ni reduction degree on product selectivity is attributed to the adsorption capability of NiO. NiO can adsorb only the C=O group of furfural as a Lewis acid site, and the hydrogenation pathway follows the MPV mechanism. In contrast, when NiO is fully reduced to metallic Ni, multiple adsorption sites are exposed, enabling simultaneous interaction with both the furan ring and the C=O group. Thus, variations in Ni reduction degree directly modulate the adsorption configuration of the catalyst on the substrate, thereby influencing the reaction pathway and product distribution.

3 Conclusion

In summary, the hydrogenation selectivity of furfural can be precisely switched between FOL and HFOL by controlling the reduction degree of a flower-like Ni₂Al hydrotalcite-derived catalyst. The Ni₂Al-LDO catalyst, rich in NiO, exhibited high selectivity (91.42%) towards FOL at 160 °C and 1.4 MPa H₂. Upon reduction at 700 °C for 2 h, the catalyst transformed into a Ni⁰-dominant state, which exclusively produced HFOL with a yield of 93.95% under identical conditions. Experimental and theoretical analyses revealed that the switchable selectivity originates from the distinct adsorption configurations of furfural dictated by the catalyst's composition. On the NiO-rich catalyst, Lewis acidic Ni²⁺ sites selectively adsorb and activate the carbonyl group of furfural and adjacent basic sites cooperate to facilitate a hydride transfer from isopropanol *via* the MPV mechanism, yielding FOL. In contrast, on the metallic Ni⁰ surface, furfural enables simultaneous interaction with both the carbonyl group and the furan ring. This configuration, coupled with efficient H₂ dissociation on Ni⁰, drives the complete hydrogenation to HFOL. This work provides fundamental insight into how the oxidation state of a non-precious metal catalyst governs substrate adsorption and reaction pathways. The strategy of tuning the metal/oxide ratio through controlled reduction offers a general approach for designing selective catalysts for biomass upgrading.

Declaration of Competing Interest

The authors declare no conflict of interest.

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镍基催化剂表面氧覆盖度对糠醛选择性加氢路径的调控规律研究

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摘 要: 生物质选择性加氢是获得高附加值化学品的必要条件。本研究采用层状双氢氧化物的结构拓扑转化工艺, 制备了 Ni-Al 混合金属氧化物催化剂, 它具有丰富的高度分散的表面 NiO 物质, 对糠醛(FAL)的半加氢表现出优异的催化性能, 在 160 °C 和 1.4 MPa H₂ 条件下, 糠醇(FOL)的收率达到 91.42%。对 Ni-Al 金属氧化物进行不同程度还原, 得到 Ni 与 NiO 混合物, 随着 NiO 占比下降, Ni 占比提高, 可以控制糠醇产率降低, 四氢糠醇产率上升。在 700 °C 还原 2 h 后, 对四氢糠醇产率达到 93.95%。基于 CO₂-TPD、NH₃-TPD 和异丙醇、糠醛原位红外光谱, 确定了镍的氧覆盖度的变化影响了糠醛的吸附位点, NiO 只能吸附 C=O, 而在 Ni 表面较好地采用扁平吸附模式, 吸附并激活呋喃环和羰基, 高度选择性地转化为四氢糠醇, 从而影响了产物的不同。

关键词: 糠醛; 选择性加氢; 镍基催化剂; 还原程度; 选择性反转

中图分类号: O643.36

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