



Ni-induced structural modification and decreased catalytic activity of α -MnO₂ in toluene oxidation

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ABSTRACT

Ni-modified α -MnO₂ catalysts containing 5 and 10 mol.% Ni were synthesized by a redox precipitation method using KMnO₄ and ethanol, followed by calcination at 400 °C. The influence of Ni addition on the structural, textural, and redox properties of α -MnO₂ and its catalytic performance in toluene oxidation was investigated. X-ray diffraction analysis confirmed the formation of cryptomelane-type K₂Mn₈O₁₆ as the main phase. At 5 mol.% Ni, a slight shift of the characteristic α -MnO₂ reflection suggested partial Ni incorporation, whereas at 10 mol.% Ni, a secondary NiMnO₃ phase was observed. SEM observations revealed significant morphological changes from interconnected fibrous structures to fragmented rod-like particles. Nitrogen adsorption measurements showed that Ni addition reduced the specific surface area from 61 to 27–42 m² g⁻¹ and the pore volume from 0.37 to approximately 0.12 cm³ g⁻¹. H₂-TPR analysis showed that the main reduction peak shifted from approximately 284 °C to 317–321 °C after Ni addition, indicating lower low-temperature reducibility. Pristine α -MnO₂ exhibited the highest catalytic activity, with a T₅₀ of 274 °C and a CO₂ yield of 97.6% at approximately 390 °C. The corresponding T₅₀ values of 5Ni- α -MnO₂ and 10Ni- α -MnO₂ were 316 and 291 °C, with CO₂ yields of 75% and 86%, respectively. The lower catalytic performance of the Ni-modified catalysts was associated with the combined effects of reduced porosity, decreased reducibility, and Ni-induced structural changes.

Introduction

Volatile organic compounds (VOCs) are among the major atmospheric pollutants emitted from industrial activities, particularly petrochemical processes, as well as transportation, and solvent utilization [1]. Among them, toluene is a common aromatic pollutant and is selected as a representative VOC in environmental and laboratory studies because of its widespread occurrence and adverse effects on human health and the environment [2]. Catalytic oxidation is considered one of the most effective technologies for VOC abatement due to its high efficiency and ability to

convert pollutants into CO₂ and H₂O at relatively low temperatures [3, 4].

Manganese oxides have attracted considerable attention as cost-effective catalysts for VOC oxidation owing to their favorable redox properties, abundant surface oxygen species, and ability to accommodate multiple Mn oxidation states [5–8]. In particular, cryptomelane-type α -MnO₂ has been extensively investigated because of its characteristic 2×2 tunnel structure formed by edge- and corner-sharing MnO₆ octahedra [9–11]. This tunnel architecture can facilitate oxygen mobility and provides favorable active sites for the toluene oxidation reactions [12].

One common strategy to improve the catalytic performance of manganese oxides is their modification with transition-metal dopants, which can alter the electronic structure, generate oxygen vacancies, affect oxygen mobility, and improve the redox properties of MnO_2 [13]. Several transition metals, such as Co, Cu, Ce, Fe, and Ni have been reported to improve the catalytic activity of manganese oxide catalysts in toluene oxidation reactions [14–16]. Among these metals, nickel is of particular interest owing to its low cost, variable oxidation states, and strong interaction with Mn-based oxide structures [17–19]. However, the effects of Ni addition on the structure and catalytic behavior of cryptomelane-type $\alpha\text{-MnO}_2$ have not been fully clarified [17, 19, 20]. Therefore, understanding how Ni addition affects the structural, textural, and redox properties of $\alpha\text{-MnO}_2$ is important for elucidating its catalytic behavior.

In this work, Ni-modified $\alpha\text{-MnO}_2$ catalysts containing 5 and 10 mol.% Ni were synthesized by a redox precipitation method using KMnO_4 and ethanol. The effects of Ni addition on the crystal structure, morphology, textural properties, and reducibility of $\alpha\text{-MnO}_2$ were investigated by XRD, SEM, N_2 adsorption–desorption, and H_2 -TPR analyses. The catalytic performance of the prepared catalysts was evaluated in the oxidation of toluene, and the relationship between catalyst properties and activity was discussed.

Experimental

$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ($\geq 97\%$) was purchased from Sigma Aldrich. KMnO_4 ($\geq 99\%$) and Toluene ($> 99.5\%$) was purchased from Merck. All chemicals were used as received without further treatment.

Pristine $\alpha\text{-MnO}_2$ was prepared according to the procedure reported in our previous work [13]. Ni-modified $\alpha\text{-MnO}_2$ catalysts were synthesized using a modified redox precipitation method. Briefly, 1.8 g of KMnO_4 was dissolved in 200 mL of deionized water to obtain solution A. Solution B was prepared by dissolving an appropriate amount of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in a mixture of 25 mL deionized water and 25 mL absolute ethanol. The amount of nickel nitrate was adjusted to obtain nominal Ni loadings of 5 and 10 mol.%, calculated as $\text{Ni}/(\text{Mn} + \text{Ni})$. Then, the solution A was added dropwise into solution B at a rate of 30–35 drops min^{-1} under continuous stirring. The resulting suspension was aged at room temperature for 16 h, filtered, washed with deionized water until neutral pH, and dried at 110 °C for 16 h. The dried solid was then calcined in air at 400 °C for 4 h with a heating rate of 1

°C min^{-1} . The obtained catalysts were denoted as 5Ni- $\alpha\text{-MnO}_2$ and 10Ni- $\alpha\text{-MnO}_2$ according to their nominal Ni contents.

The crystalline phases of the catalysts were identified by X-ray diffraction (XRD) using a SmartLab diffractometer (Rigaku, Japan) equipped with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$), operating at 40 kV and 40 mA. Diffraction patterns were recorded in the 2θ range of 5–70°. The morphology of the catalysts was examined using scanning electron microscopy (SEM). Nitrogen adsorption–desorption measurements were carried out at –196 °C using an ASAP 2020 analyzer (Micromeritics, USA). Prior to analysis, the samples were degassed at 150 °C for 2 h. The reducibility of the catalysts was evaluated by H_2 -temperature programmed reduction (H_2 -TPR). Prior to the measurement, the catalyst sample (approximately 30 mg) was treated in an air stream at 300 °C for 1 h to remove adsorbed species and then cooled to ambient temperature. The analysis was subsequently carried out from 30 to 900 °C at a heating rate of 10 °C min^{-1} under a flowing mixture of 10 vol.% H_2 in Ar (30 mL min^{-1}). Hydrogen consumption during the reduction process was continuously monitored using a thermal conductivity detector (TCD).

The catalytic activity for toluene oxidation was evaluated in a fixed-bed continuous-flow reactor following the procedure reported in our previous studies [13, 21]. The catalyst (0.1 gram) was pretreated in flowing air at 250 °C for 2 h prior to the reaction. Catalytic tests were performed between 100 and 400 °C under a feed gas containing toluene (1000 ppm) in air with a total flow rate of 100 mL min^{-1} . The concentrations of reactants and products were analyzed online using gas chromatography, and the catalytic performance was expressed as the CO_2 yield. Toluene conversion to CO_2 (expressed as CO_2 yield) was calculated according to the equation previously reported [22].

Results and discussion

Fig. 1A shows the X-ray diffraction patterns of pristine $\alpha\text{-MnO}_2$ and Ni-modified $\alpha\text{-MnO}_2$ catalysts. The diffraction peaks of the undoped sample can be indexed to cryptomelane-type $\text{K}_2\text{Mn}_8\text{O}_{16}$ ($\alpha\text{-MnO}_2$), which possesses a characteristic 2×2 tunnel structure formed by edge- and corner-sharing MnO_6 octahedra [23]. The major reflections observed at $2\theta \approx 12.7^\circ$, 17.9° , 28.7° , 37.5° , 41.9° , 49.8° , 60.0° , and 69.7° correspond well to the cryptomelane phase (JCPDS card no. 44-0141), confirming the formation of tunnel-structured $\alpha\text{-MnO}_2$.

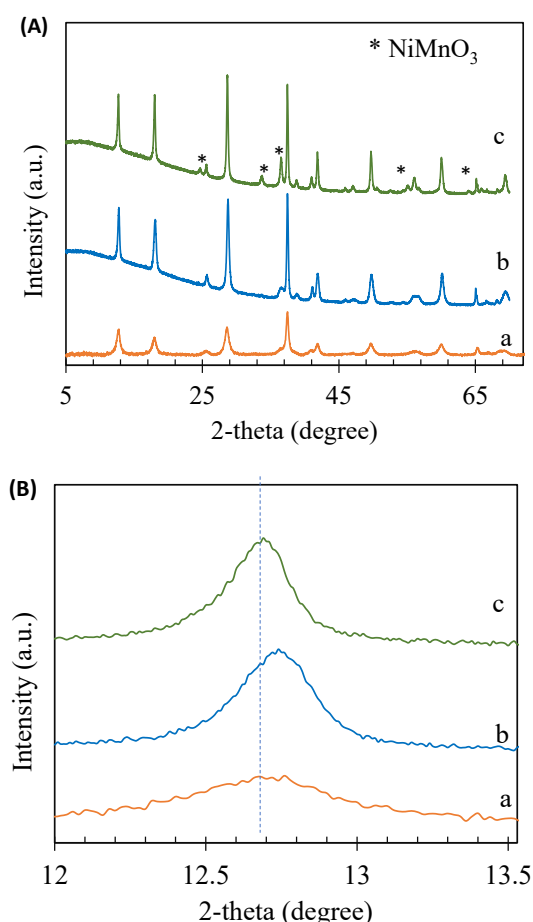


Fig 1: (A) X-ray diffraction patterns of pristine α - MnO_2 and Ni-modified α - MnO_2 catalysts; (B) enlarged view of the diffraction peaks in the 2θ range of 12–13.5°. a) α - MnO_2 , b) 5Ni- α - MnO_2 , and c) 10Ni- α - MnO_2 .

For the 5Ni- α - MnO_2 sample, no additional crystalline phase is detected. However, a slight shift of the characteristic reflection at $2\theta \approx 12.7^\circ$ toward higher diffraction angles is observed (Fig. 1B). According to Bragg's law, this shift corresponds to a decrease in the interplanar spacing, indicating a slight lattice contraction. This observation is consistent with partial incorporation of Ni species into the cryptomelane-type MnO_2 structure, resulting in slight lattice distortion. The observed lattice contraction may be associated with differences in the ionic radii and local coordination environments of Ni and Mn species.

When the nominal Ni content is increased from 5 to 10 mol.%, additional reflections at $2\theta \approx 24.5^\circ$ and 33.7° appear and can be assigned to the NiMnO_3 phase (JCPDS card No. 12-0269). The appearance of this secondary phase indicates that the capacity of the cryptomelane framework to accommodate Ni is limited, and excess Ni tends to segregate as NiMnO_3 at higher Ni content. Meanwhile, the position of the α -

MnO_2 reflection near 12.7° in the 10Ni- α - MnO_2 sample approaches that of pristine α - MnO_2 , further suggesting that additional Ni is preferentially accommodated in the secondary phase rather than further incorporated into the α - MnO_2 lattice.

The diffraction peaks of the Ni-containing samples are also sharper and more intense than those of pristine α - MnO_2 , suggesting increased crystallinity and larger crystallite sizes after Ni addition. These structural changes may influence the redox and catalytic properties of the resulting materials.

Fig. 2 presents the SEM images of pristine α - MnO_2 and Ni-modified α - MnO_2 catalysts. Pristine α - MnO_2 sample exhibits a heterogeneous morphology consisting of both large fibers and aggregates of smaller nanofibers interconnected to form larger secondary particles. This fibrous morphology is characteristic of tunnel-structured α - MnO_2 . The addition of nickel significantly alters the morphology of the catalyst. For the 5Ni- α - MnO_2 , the fibrous structure becomes less pronounced and is partially replaced by rod-like particles accompanied by fragmented particles (Fig. 2B). The observed changes may be related to the influence of Ni on the nucleation and crystal growth of α - MnO_2 during synthesis and subsequent calcination.

With further increasing Ni content, the degree of fragmentation becomes more evident, resulting in the formation of smaller and more irregular particles (Fig. 2C). This morphological change coincides with the formation of the secondary NiMnO_3 phase observed by XRD, suggesting that phase segregation at higher Ni content may further modify the growth of α - MnO_2 crystals and disrupt the original fibrous architecture. The progressive transformation from interconnected fibrous assemblies to fragmented particles may also influence the textural properties and surface accessibility of the catalysts.

The N_2 adsorption–desorption isotherms of α - MnO_2 , 5Ni- α - MnO_2 , and 10Ni- α - MnO_2 are shown in Fig. 3. All samples exhibit a distinct hysteresis loop at high relative pressures ($P/P_0 \approx 0.8$ –1.0), indicating capillary condensation within mesopores. According to the IUPAC classification, the obtained isotherms can be assigned to type IV, characteristic of mesoporous materials [24]. The pristine α - MnO_2 catalyst exhibits a specific surface area (SSA) of $61 \text{ m}^2 \text{ g}^{-1}$ and a total pore volume (V_p) of $0.37 \text{ cm}^3 \text{ g}^{-1}$. After Ni addition, the SSA value decreases to 27 and $42 \text{ m}^2 \text{ g}^{-1}$ for 5Ni- α - MnO_2 and 10Ni- α - MnO_2 , respectively. Simultaneously, the V_p value decreases to approximately $0.12 \text{ cm}^3 \text{ g}^{-1}$. These results indicate

that Ni modification substantially alters the textural properties and reduces the accessible porosity of α -MnO₂. Interestingly, 10Ni- α -MnO₂ exhibits a

higher surface area than 5Ni- α -MnO₂, which may be associated with the more pronounced particle fragmentation observed by SEM.

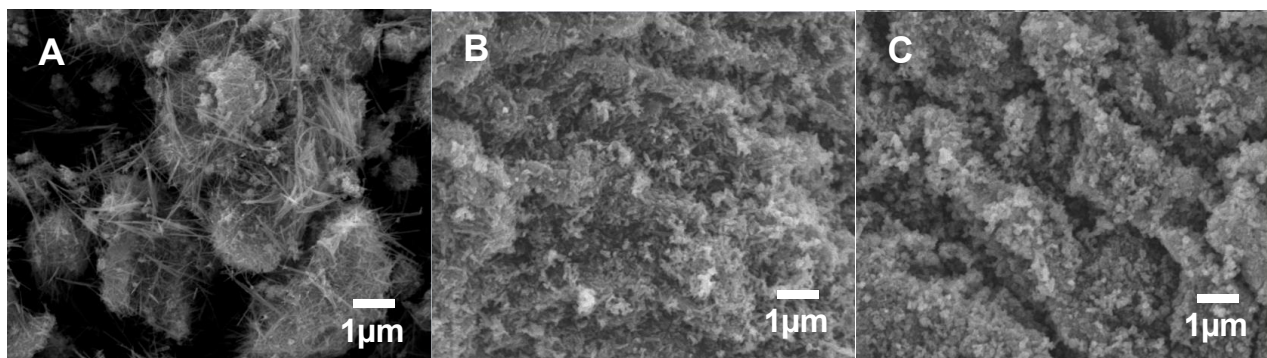


Fig 2: SEM images of (A) α -MnO₂, (B) 5Ni- α -MnO₂, and (C) 10Ni- α -MnO₂ catalysts

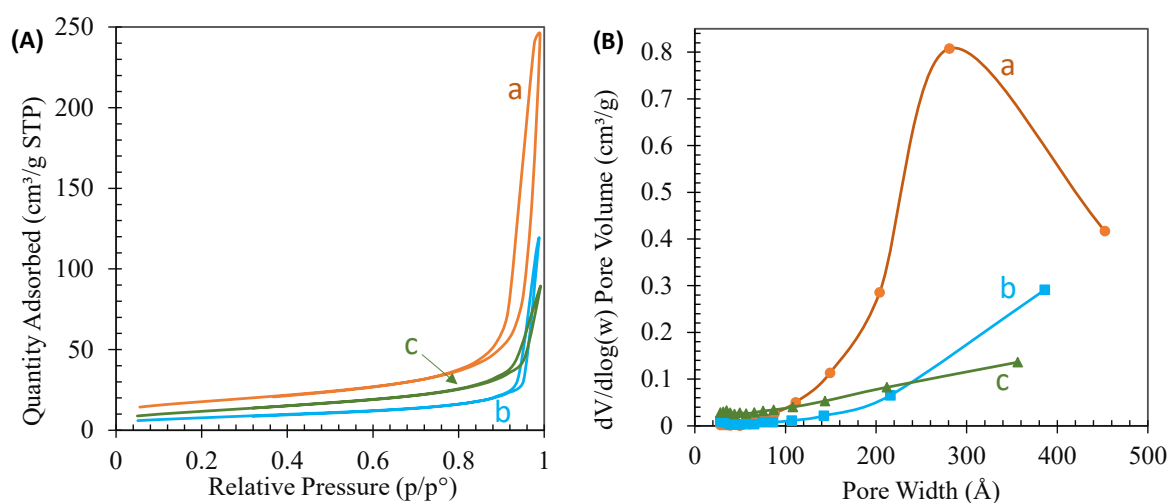


Fig 3: (A) N₂ adsorption-desorption isotherms and (B) pore size distributions of (a) α -MnO₂, (b) 5Ni- α -MnO₂, and (c) 10Ni- α -MnO₂ catalysts

The pore size distribution curves (Fig. 3B) further reveal that all samples possess predominantly mesoporous structures with pore widths mainly distributed in the range of 150–450 Å. Pristine α -MnO₂ exhibits the largest pore volume, with a pronounced maximum centered at approximately 280–300 Å. In contrast, Ni-modified samples show a substantial decrease in pore volume over the entire pore-size range. The intensity of the distribution peak decreases significantly after Ni addition, indicating a substantial reduction in the accessible mesopore volume. Furthermore, the broad pore distribution observed for α -MnO₂ becomes less pronounced in the Ni-containing samples, further indicating that Ni addition significantly modifies the pore architecture of α -MnO₂.

The reducibility of the catalysts was investigated by H₂-temperature programmed reduction (H₂-TPR) using a gas mixture containing 10 vol.% H₂ in Ar. The corresponding H₂-TPR profiles are presented in Fig. 4.

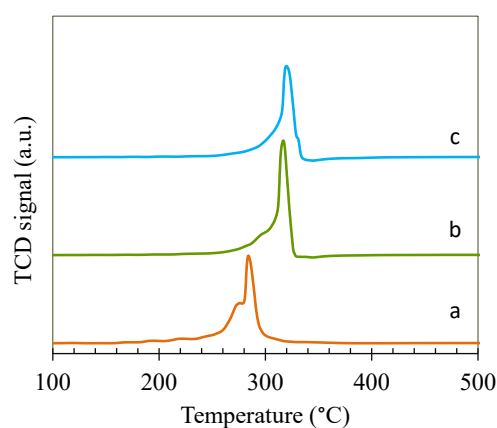


Fig 4: H₂-TPR profiles of (a) α -MnO₂, (b) 5Ni- α -MnO₂, and (c) 10Ni- α -MnO₂ catalysts

The H₂-TPR profile of pristine α -MnO₂ shows two partially overlapped reduction peaks in the temperature range of 200–300 °C. These features are

commonly attributed to the stepwise reduction of manganese oxides, corresponding to the transformation of MnO_2 into lower-valence manganese oxides (Mn_2O_3 and/or Mn_3O_4), followed by further reduction to MnO [12, 25]. The relatively low reduction temperatures indicate that the lattice oxygen of $\alpha\text{-MnO}_2$ can be readily removed under reducing conditions. After Ni addition, the main reduction peak shifts toward higher temperatures, reaching approximately 317 and 321 °C for 5Ni- $\alpha\text{-MnO}_2$ and 10Ni- $\alpha\text{-MnO}_2$, respectively. This shift indicates that Ni addition decreases the low-temperature reducibility of manganese oxide species. For 5Ni- $\alpha\text{-MnO}_2$, the shift to higher reduction temperature may be associated with the partial incorporation of Ni into the cryptomelane-type MnO_2 structure and the accompanying lattice distortion suggested by XRD. For 10Ni- $\alpha\text{-MnO}_2$, the formation of the secondary NiMnO_3 phase may additionally contribute to the altered reduction behavior. The increased crystallinity of the Ni-modified samples may also contribute to their lower reducibility. Overall, these results indicate that Ni addition suppresses the low-temperature reducibility of $\alpha\text{-MnO}_2$.

Catalytic performance

The catalytic activities of $\alpha\text{-MnO}_2$ and Ni-modified $\alpha\text{-MnO}_2$ catalysts toward toluene oxidation were evaluated in air over the temperature range of 100–400 °C. The catalytic performance is presented in Fig. 5 in terms of toluene conversion to CO_2 as a function of reaction temperature. For all catalysts, the conversion of toluene increased progressively with increasing reaction temperature, indicating enhanced oxidation kinetics at elevated temperatures. However, significant differences in catalytic performance were observed among the catalysts. Pristine $\alpha\text{-MnO}_2$ exhibited the highest catalytic activity, achieving a CO_2 yield of 97.6% at approximately 390 °C. At the same temperature, the CO_2 yields of 5Ni- $\alpha\text{-MnO}_2$ and 10Ni- $\alpha\text{-MnO}_2$ are approximately 75% and 86%, respectively. The T_{10} values are 212, 214, and 238 °C, while the corresponding T_{50} values are 274, 316, and 291 °C for $\alpha\text{-MnO}_2$, 5Ni- $\alpha\text{-MnO}_2$, and 10Ni- $\alpha\text{-MnO}_2$, respectively. Although 5Ni- $\alpha\text{-MnO}_2$ exhibits a T_{10} value comparable to that of pristine $\alpha\text{-MnO}_2$, its substantially higher T_{50} indicates a slower increase in catalytic activity with increasing temperature. In contrast, 10Ni- $\alpha\text{-MnO}_2$ shows a higher T_{10} but a lower T_{50} than 5Ni- $\alpha\text{-MnO}_2$, indicating a later onset of CO_2 formation followed by a more rapid increase in catalytic activity. Overall, the T_{50} values and high-temperature CO_2 yields indicate the activity order $\alpha\text{-MnO}_2 > 10\text{Ni-}\alpha\text{-MnO}_2 > 5\text{Ni-}\alpha\text{-MnO}_2$.

The results clearly demonstrate that Ni addition does not improve the catalytic performance of $\alpha\text{-MnO}_2$ for toluene oxidation.

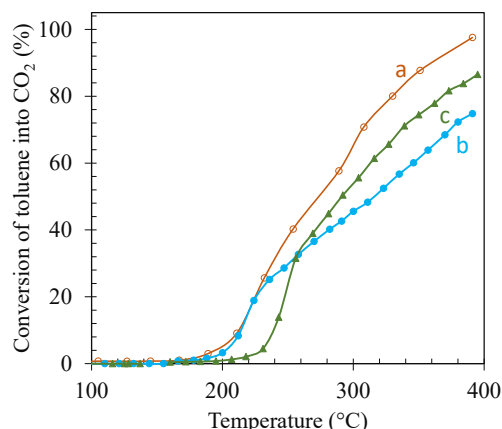


Fig 5: Light-off curves for toluene oxidation over a) $\alpha\text{-MnO}_2$, b) 5Ni- $\alpha\text{-MnO}_2$, and c) 10Ni- $\alpha\text{-MnO}_2$ catalysts

Although the T_{10} value of 5Ni- $\alpha\text{-MnO}_2$ remains comparable to that of pristine $\alpha\text{-MnO}_2$, the markedly higher T_{50} values and lower high-temperature CO_2 yields of the Ni-modified samples indicate an overall decrease in catalytic performance after Ni addition. This decrease can be correlated with their lower surface area and pore volume, together with their lower low-temperature reducibility, as evidenced by the shift of the H_2 -TPR reduction peak toward higher temperatures. In addition, the increased crystallinity after Ni addition may also contribute to the lower catalytic performance. Interestingly, 10Ni- $\alpha\text{-MnO}_2$ exhibits higher catalytic activity than 5Ni- $\alpha\text{-MnO}_2$ despite the formation of the secondary NiMnO_3 phase. This behavior may be partly attributed to the higher specific surface area of 10Ni- $\alpha\text{-MnO}_2$ ($42 \text{ m}^2 \text{ g}^{-1}$) compared with 5Ni- $\alpha\text{-MnO}_2$ ($27 \text{ m}^2 \text{ g}^{-1}$), which may provide greater accessibility to surface active sites. The more pronounced particle fragmentation observed by SEM for 10Ni- $\alpha\text{-MnO}_2$ may also contribute to this higher surface area. Since the reduction temperatures of 5Ni- $\alpha\text{-MnO}_2$ and 10Ni- $\alpha\text{-MnO}_2$ are relatively similar (317 and 321 °C, respectively), the higher activity of 10Ni- $\alpha\text{-MnO}_2$ is unlikely to originate from improved reducibility. Therefore, the catalytic performance appears to result from the combined effects of the textural, structural, and redox properties of the catalysts.

Conclusion

Ni-modified $\alpha\text{-MnO}_2$ catalysts were successfully synthesized by a redox precipitation method followed by calcination at 400 °C. XRD results suggested partial

incorporation of Ni into the cryptomelane structure at the lower Ni content (5 mol.%), whereas a secondary NiMnO₃ phase was formed at the higher Ni content (10 mol.%). Ni addition also increased the crystallinity of α -MnO₂. SEM and N₂ adsorption analyses revealed significant changes in morphology, accompanied by decreases in surface area and pore volume. H₂-TPR results further indicated a reduction in low-temperature reducibility after Ni addition. In the catalytic oxidation of toluene, pristine α -MnO₂ exhibited the highest activity, with a T₅₀ of 274 °C and a CO₂ yield of 97.6% at approximately 390 °C. The corresponding T₅₀ values of 5Ni- α -MnO₂ and 10Ni- α -MnO₂ were 316 and 291 °C, respectively, while their CO₂ yields at approximately 390 °C were 75% and 86%. The lower activity of the Ni-modified catalysts may be associated with the combined effects of reduced porosity, lower low-temperature reducibility, and structural changes induced by Ni addition.

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