Enhancing the Activity and Stability of CuO/OMS-2 Catalyst for CO Oxidation at Low Temperature by Modification with Metal Oxides

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In this study, mixed oxides of Mn-Cu and Fe-Cu on OMS-2 support having an octahedral structure were synthesized by the refluxing and impregnation methods. The characteristics of the materials were analyzed by XRD, FTIR, SEM, EDX, and H2-TPR. In the CO oxidation test, CuFeOx/OMS-2 had slightly higher catalytic activity but is significantly more stable than CuMnOx/OMS-2 and CuO/OMS-2. Due to its lower reduction temperature in H2-TPR analysis, the Mars-Van-Krevelen mechanism for CuFeOx/OMS-2 (Cu2+–O–Fe3+ ↔ Cu+–□–Fe2+) could take place more energetically than CuO/OMS-2 and CuMnOx/OMS-2 (Cu2+–O2−–Mn4+ ↔ Cu+–□–Mn3+). In addition, the interaction between Fe and Cu in the catalyst could improve the durability of the surface oxides structure in comparison with that between Mn and Cu. With the high specific rate and TOF of 28.6 mmol/h.g and 0.508, respectively, CuFeOx/OMS-2 has a great potential as an effective catalyst for low-temperature oxidation application in CO and possible VOCs removal.

1. Introduction

Air pollution by CO has recently been considered to be one of the serious problems in the developing countries due to the increasing consumption of fuels and especially biomass with low combustion efficiency [1]. There are many ways for CO treatment, in which catalytic CO oxidation is considered as one of the most effective and energy-saving techniques [2–4]. In general, precious metal catalysts based on Pt-group have high efficiency in CO oxidation at low temperature, but they are expensive and precious metals [2, 5–7]. To reduce the cost, several studies have been done to look for oxides of transition metals (e.g., Mn, Co, Ce, Cu, Mo, and Fe). Among them, copper oxide and its combination with other metals have been of special interest and hopcalite (CuMnOx) is one of the most popular catalysts for low-temperature oxidation of CO [8, 9].

Octahedral molecular sieve (OMS-2), a cryptomelane type of manganese oxides, is a promising catalyst for applications in oxidation reactions due to its high activity, high...
selectivity, and good catalytic durability [10]. Liu et al. [11] combined CuO and OMS-2 for CO oxidation at low temperature and reported that the good dispersion and the interaction between copper oxide and manganese oxide in CuO/OMS-2 catalyst produce active sites for the oxidation of CO, which can be explained by Mars-Van-Krevelen mechanism, whereby the redox pair $\text{Cu}^{2+} - \text{O}_2$ $\rightarrow$ Mn$^{4+}$ took place. After that, the CuMnO$_2$/OMS-2 catalyst was prepared for preparing for the interaction between copper oxide and manganese oxide on the surface of OMS-2 support and, therefore, improving the catalytic activity in CO oxidation at low temperature [12]. However, the role of redox pairs in the transition metal oxide catalyst structure and the effect of adding metal oxides on the stability of CuO catalysts on OMS-2 support have not been well understood yet.

On the other hand, the combination of Cu and Fe oxides for CO oxidation at low temperatures was also reported in several studies [13]. Cao et al. [14] reported that mesoporous CuO-Fe$_2$O$_3$ composite produced by the surfactant-assisted method with wormhole-like mesostructure gave high catalytic activity and stability for the oxidation of CO. Amini et al. [15] synthesized mesoporous CuFe$_2$O$_4$ nanopowders via the sol-gel method and declared that the addition of Cu into iron oxide affected its properties and yielded high catalytic stability for CO oxidation. Yeste et al. [16] used Cu-Fe mixed oxides (CuFe$_2$O$_4$) fabricated by the coprecipitation method at low temperature as an effective catalyst for CO oxidation with high stability. Rezaei et al. [17] prepared mesoporous nanopowders of iron and copper mixed oxides by the simple one-pot solid-state method, and the results showed that the spinel structure gave the best CO oxidation ability without obvious deactivation. Hence, there is a great potential for the combination of CuFeO$_x$ catalysts with OMS-2 support not only to enhance the oxidation ability at a lower temperature, but also to improve the stability of the catalyst. However, to the best of our knowledge, the application of such CuFeO$_x$/OMS-2 material synthesized by a facile impregnation method has not been reported yet.

In this study, CuFeO$_x$/OMS-2 catalyst was synthesized by adding Fe$^{3+}$ into the structure of CuO catalyst to compare the efficiency of its redox pair ($\text{Cu}^{2+} - \text{Fe}^{3+} \rightarrow \text{Cu}^{3+} - \text{Fe}^{2+}$) to that of CuMnO$_2$/OMS-2 ($\text{Cu}^{2+} - \text{O}_2^\cdot \rightarrow \text{Cu}^{3+} - \text{O}^\cdot$) in CO oxidation at low temperature. Furthermore, this study was also conducted to demonstrate a multidimensional view of the Mars-Van-Krevelen mechanism to find out potential catalysts for efficient oxidation of CO at low temperatures.

2. Materials and Methods

2.1. Preparation for Catalysts. Analytical-grade chemicals such as Cu (NO$_3$)$_3$, Fe (NO$_3$)$_3$, Mn (NO$_3$)$_2$, and standard solutions of iron, and copper and manganese used in this study were purchased from Sigma Aldrich and Merck and used directly without further purification.

OMS-2 support was synthesized by the refluxing method according to DeGuzman et al. [10]. The synthesis process can be described basically as follows. At first, 11.33 g of MnSO$_4$·H$_2$O (analytical-grade, Xilong, China) was dissolved in 120 mL of deionized (DI) water (solution A), while 7.57 g of KMnO$_4$ (analytical-grade, Xilong, China) was dissolved in 38 mL of DI water (solution B). The two solutions were then mixed thoroughly and added with 4 mL of concentrated HNO$_3$, which was followed by refluxing at 100°C for 24 h. The brown solid product was subsequently separated by filtration and washed several times with DI water until the pH of about 7 was reached. The OMS-2 product was finally obtained after drying at 120°C overnight and calcined at 400°C for 4 h.

The catalysts, including CuMnO$_x$/OMS-2 and CuFeO$_x$/OMS-2 with 15 wt.% of CuMnO$_x$ and CuFeO$_x$, were then prepared by impregnation method using the prepared OMS-2 support [11, 12]. The OMS-2 support was impregnated in a solution containing 250 mL of DI water and appropriate amounts of Cu(NO$_3$)$_2$ and Mn(NO$_3$)$_2$ or Fe(NO$_3$)$_3$ salts. Afterward, the mixture was heated at 80°C while being stirred to remove water and followed by drying at 100°C and annealing at 400°C for 4 h.

2.2. Material Characterization. Wide-angle X-ray diffraction (XRD) patterns, which were collected by an XRD 300W diffractometer (D2 Phaser, Bruker) with a CuK$_\alpha$ radiation wavelength of 1.5406 Å having a step size of 0.05° and a step time of 30 s, were used to analyze the material crystalline structure. The morphology and particle size of CuMnO$_x$/OMS-2 and CuFeO$_x$/OMS-2 were evaluated using scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDS) (JSM-IT200, JEOL) and transmission electron microscopy (TEM) on a Philips Tecnai G$^2$ F20 TEM microscope operated at 40 kV. The samples were prepared by ultrasonically suspending the materials in ethanol, and the suspension was then applied to a copper grid and dried in an oven. Another method of determining specific surface area and pore size of the materials was the Bruauer–Emmett–Teller (BET) analysis. The analysis was conducted at 77 K using a BET-202A system (Porou Materials Inc.), in which nitrogen was used as an adsorbate for probing the surface of the materials. It should be noted that BET analysis was carried out after the materials were degassed at 150°C for 4 hours to remove the water from the oxide meso-/micropores thoroughly. An inductively coupled plasma atomic emission spectrometry (ICP-AES, iCAP 6500, Thermo Scientific) was utilized to measure the amount of CuMnO$_x$ and CuFeO$_x$ adhering to the OMS-2 support by determining the Cu, Fe, and Mn contaminations in Mn(NO$_3$)$_2$, Fe(NO$_3$)$_3$, and Cu(NO$_3$)$_2$ solutions.

2.3. Measurement of Catalytic Activity. A quartz tubular fixed bed reactor with an internal diameter of 10 mm, which was employed for CO oxidation under atmospheric pressure, was loaded up with 200 mg of the catalysts. During the oxidation, the reaction temperature was monitored continuously by a thermocouple placed in the middle of the catalyst bed. A mixture of 1 vol.% CO and 1 vol.% O$_2$, as the reactants with a total gas flow rate of 30 mL min$^{-1}$ (space
velocity of 12,000 mL·g⁻¹·h⁻¹), balanced in nitrogen, was passed through the reactor. The CO and CO₂ were analyzed by a gas chromatograph (GC, Trace-GC 1310, Thermo Scientific) equipped with a flame ionization detector (FID) and a TR-WaxMS GC column. The carbon-containing gaseous products were conducted to a methanation converter filled with Raney-Ni catalyst before going to the detector to ensure obtaining signals with high intensity [11].

3. Results and Discussion

3.1. Material Synthesis and Characterization. The morphology is one of the important properties of the catalysts, which could show the shape, size, and even crystalline property of the catalyst particles. Figure 1 shows the TEM image of CuFeOₓ/OMS-2 catalyst at low and high resolutions. It was apparent that the CuFeOₓ/OMS-2 has nanorod-shaped morphology, with a diameter of approximately 13 nm and length from 300 to 500 nm. This result is consistent with that in the study of Zhang et al. [18] reported on related OMS-2 materials.

In addition, CuMnOₓ/OMS-2 and CuFeOₓ/OMS-2 catalysts were also analyzed with the SEM-EDS, and the results are shown in Figures 2 and 3. For CuMnOₓ/OMS-2 sample (Figure 2), the Cu and Mn components were evenly distributed on the surface of OMS-2 support. It was discovered that the results were similar to those of CuFeOₓ/OMS-2 sample (Figure 3). There was an agreement in the calculation and the analysis results of CuMnOₓ and CuFeOₓ contents adhering to OMS-2 support, which were determined by ICP-AES. The atomic percentage of elements on the surface of the catalysts is summarized in Table 1.

The XRD patterns of the catalysts are displayed in Figure 4. It has been observed that the diffraction peaks at 2θ of 12.6°, 17.9°, 28.7°, 37.5°, 49.9°, and 60.1° directly contributed to the crystalline phase of cryptomelane (KMn₈O₁₆), proving that the nanorod OMS-2 material had a cryptomelane-type structure [19], corresponding to the XRD patterns of OMS-2 material in some works [18, 19]. Although appearing at relatively low intensity, the diffraction peaks at 2θ of 32.6°, 35.5°, and 38.8° in XRD patterns of Cu-containing catalysts could be attributed to its low crystalline phase of copper oxide [11]. Meanwhile, peaks at 23° and 25.5° in the XRD pattern of CuFeOₓ/OMS-2 catalyst are indexed to the crystalline phase of ferric oxide.

Figure 5 illustrates the FTIR patterns of catalysts including OMS-2, CuO/OMS-2, CuMnOₓ/OMS-2, and CuFeOₓ/OMS-2. In CuMnOₓ/OMS-2 catalyst, the FTIR patterns, to a large extent, were characterized by the vibration peaks of Cu-O-Cu at 410, 420, and 430 cm⁻¹ and of Cu-O at 440 cm⁻¹ [20]. Similarly, the FTIR pattern in CuFeOₓ/OMS-2 catalyst was also characterized by the vibrations of the same peaks above. Nevertheless, differences between the vibrations below 500 cm⁻¹ were observed, which determined that these vibrations were of Fe-O. Moreover, these results were also found to be consistent with the XRD patterns of both materials.

The redox characteristics of the catalysts are often determined by H₂-TPR. Figure 6 compares H₂-TPR profiles of CuO/OMS-2, CuMnOₓ/OMS-2, and CuFeOₓ/OMS-2 catalysts at different temperature ranges. In all catalysts, γ₁ and γ₂ peaks present the two reduction steps of manganese oxide, where γ₁ peak is for the first reduction step of MnO₂/Mn₃O₄ to Mn₃O₄ and γ₂ peak is for the second reduction step of Mn₃O₄ to MnO [11]. There were shifts of γ₁ and γ₂ peaks to lower reduction temperature in CuMnOₓ/OMS-2 and CuFeOₓ/OMS-2 as compared to CuO/OMS-2 catalyst, and the peaks of CuFeOₓ/OMS-2 were the lowest ones (Table 2). It can be stated that the presence of Cu and Fe encouraged the reducibility of manganese oxide via an H-spillover effect [21]. Furthermore, the α peak resulted in the reduction of the CuOₓ species having strong interaction with manganese and ferric oxides, while the β peak was responsible for the reduction of highly dispersed CuO species on the catalyst surface, which had a weak interaction with the surface manganese and ferric oxides [22]. In general, the α and β peaks of CuMnOₓ/OMS-2 and CuFeOₓ/OMS-2 were at lower temperatures than those of CuO/OMS-2, particularly for CuFeOₓ/OMS-2 at the lowest temperatures (Table 2), which is due to the strong interaction of oxides in the catalyst structure. The β peak is also assigned to the reduction of large CuO particles as well as the reduction of MnO₂ to Mn₃O₄ [11, 23]. There was another peak observed at 200°C for CuFeOₓ/OMS-2 catalyst, which is ascribed to the reduction of FeO₃ to Fe₂O₃ [21]. Besides, when applying mesoporous CuO-Fe₂O₃ for CO oxidation, Cao et al. [14] found that this reduction peak is at a lower temperature than that of iron oxide, possibly due to the strong interaction of iron oxide and copper oxide in the structure of CuO-Fe₂O₃ composite.

3.2. CO Oxidation Test. Table 3 lists CO oxidation performances of catalysts, composed of OMS-2, CuO/OMS-2, CuMnOₓ/OMS-2, and CuFeOₓ/OMS-2, in several studies. It is important to notice that the obtained catalysts were made into pellets and sieved to enhance the stability under the effect of a high gas flow rate in the reactor. From the results in Table 3, it is obvious that all CuO-supported OMS-2 catalysts showed very high activities for CO oxidation at a room temperature of roughly 35°C, while OMS-2 had no activity. The specific reaction rate of CO oxidation of CuFeOₓ/OMS-2 catalyst was 28.6 mmol CO·h⁻¹·g⁻¹, which is slightly higher than those of CuMnOₓ/OMS-2 (27.8 mmol·h⁻¹·g⁻¹) and CuO/OMS-2 (27.2 mmol·h⁻¹·g⁻¹) catalysts. The TOF of the catalysts is in the order of CuFeOₓ/OMS-2 (0.508) > CuMnOₓ/OMS-2 (0.495) > CuO/OMS-2 (0.484), indicating that the presence of Fe and Mn components in the CuO structure could enhance the catalytic activity of CuO/OMS-2 catalyst. It could be explained by the existence of two redox pairs Cu²⁺–O₂⁻–Fe³⁺ ↔ Cu⁰–O₂⁻–Fe⁴⁺ and Cu₂⁺–O²⁻–Mn⁴⁺ ↔ Cu⁰–O₂⁻–Mn³⁺ + O₂ in structures of CuFeOₓ/OMS-2 and CuMnOₓ/OMS-2 catalysts, respectively. On the other hand, a higher catalytic activity of CuFeOₓ/OMS-2 catalyst as against CuMnOₓ/OMS-2 catalyst could be due to the lower redox potential of Fe⁴⁺/Fe³⁺ compared to that of Mn⁴⁺/Mn³⁺. Consequently, the reduction process of
Figure 1: TEM images of CuFeO$_x$/OMS-2 material at low and high resolutions.

Figure 2: SEM-mapping and EDS images of CuMnO$_x$/OMS-2 catalyst.
CuFeO\textsubscript{x} took place more smoothly than that of CuMnO\textsubscript{x}, and thus it is more active and easily activated for CO oxidation at low temperatures.

Durability, which is an important aspect, is often of great interest when studying the catalyst for chemical reactions. Here, the catalytic durability was evaluated via the specific
rate of catalyst in CO oxidation at room temperature after 48 h of operation (Figure 7). The stability of the CuO/OMS-2 increased significantly after adding manganese oxide and especially iron oxide into the structure of CuO. After 48 h of reaction, the activity of CuFeOₓ/OMS-2 only reduced by 16.8%, which was much lower than those of 55.4% and 50.0% for CuMnOₓ/OMS-2 and CuO/OMS-2, respectively. This study.

Table 2: The reduction temperature of peaks in H₂-TPR profile of catalysts.

| Catalyst            | α  (°C) | δ  (°C) | β  (°C) | γ₁ (°C) | γ₂ (°C) | Note       |
|---------------------|---------|---------|---------|---------|---------|------------|
| CuO/OMS-2           | 180     | —       | 275     | 350     | 400     | [11]       |
| CuMnOₓ/OMS-2        | 170     | —       | 245     | 310     | 330     | This study |
| CuFeOₓ/OMS-2        | 160     | 200     | 230     | 290     | 320     | This study |

Figure 5: FTIR patterns of OMS-2, CuO/OMS-2, CuMnOₓ/OMS-2, and CuFeOₓ/OMS-2 samples in the range of (a) 500–4000 cm⁻¹ and (b) 400–500 cm⁻¹.

Figure 6: TPR patterns of catalysts with different temperature ranges: (1) CuO/OMS-2, (2) CuMnOₓ/OMS-2, and (3) CuFeOₓ/OMS-2.
82.4% in CuMnO$_x$/OMS-2 and CuO/OMS-2 catalysts, respectively. Although they are similar at the beginning, the specific CO oxidation rate by CuFeO$_x$/OMS-2 was 2 times higher than that by CuMnO$_x$/OMS-2 and 5 times higher than that by CuO/OMS-2 after 48 h of reaction. The addition of CuO into the lattice of Fe$_x$O$_y$ reduces the temperature for complete CO oxidation [15]. Besides, Fe$_x$O$_y$ plays two roles of (i) catalyst under excess oxygen condition and (ii) oxidant (by lattice oxygen) under low oxygen condition [14]. The improvement in the stability of the CuO catalyst by adding Fe is consistent with those reported in the literature for mixed oxides of Cu and Fe [14–17]. Since iron is cheaper than the others, CuFeO$_x$/OMS-2 is more applicable as a commercial catalyst. It is also noticed that the stability of the catalysts is a very important factor affecting its ability in practical application.

### 4. Conclusions

The catalysts of CuFeO$_x$ and CuMnO$_x$ were impregnated on OMS-2 support and applied for the oxidation of CO at a low temperature of ~35°C. The addition of Mn and Fe into CuO was proven as an effective way to enhance the activity of CuO/OMS-2 catalyst for CO oxidation, where the specific reaction rate increases from 27.2 to 27.8–28.6 mmol CO h$^{-1}$ gCu$^{-1}$. The stability of the material was improved by adding Mn and greatly enhanced by adding Fe. The catalytic activity decreased after 48 h of the reaction was recorded to be 82.4% and 55.4% for CuO/OMS-2 and CuO/OMS-2, respectively, but only 16.8% for CuFeO$_x$/OMS-2 catalyst in terms of specific rates. The high catalytic activity and stability of CuFeO$_x$/OMS-2 make it very promising as a stable and effective catalyst for CO oxidation at low temperature in practical applications.

### Data Availability

The experimental data used to support the findings of this study are included in the manuscript. The other data are available from the corresponding author upon request.

### Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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**Table 3: Performance of catalysts for CO oxidation.**

| Catalyst          | Surface area (m$^2$/g) | Reaction temperature (°C) | Specific rate (mmol CO h$^{-1}$ gCu$^{-1}$) | TOF  | Note/reference |
|-------------------|------------------------|----------------------------|---------------------------------------------|------|----------------|
| OMS-2             | 48.5                   | ~35                        | 0                                           | —    | This study     |
| CuO/OMS-2         | —                      | 20                         | 24.5                                        | 0.436| [11]           |
| CuO/OMS-2         | 41.4                   | ~35                        | 27.2                                        | 0.484| [12]           |
| CuMnO$_x$/OMS-2   | 52.76                  | ~35                        | 27.8                                        | 0.495| This study     |
| CuFeO$_x$/OMS-2   | 52.40                  | ~35                        | 28.6                                        | 0.508| This study     |
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