

## Research Article

# Synthesis of $\text{CeO}_2\text{-Fe}_2\text{O}_3$ Mixed Oxides for Low-Temperature Carbon Monoxide Oxidation

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In this study, the  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxide catalysts have been prepared by combustion method using gel-created tartaric acid. The ability of effective carbon monoxide (CO) oxidation to carbon dioxide ( $\text{CO}_2$ ) by  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  catalyst under low-temperature conditions was also demonstrated. The calcined  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  material has a porous honeycomb structure and good gaseous absorption-desorption ability. The solid solution of  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxides was formed by the substitution of  $\text{Fe}^{+3}$  ions at some  $\text{Ce}^{4+}$  ion sites within the  $\text{CeO}_2$  crystal lattice. The results also showed that the calcination temperature and the molar ratio of  $\text{Ce}^{3+}$  ions to  $\text{Fe}^{3+}$  ions (CF) affected the formation of the structural phase and the catalytic efficiency. The catalytic properties of the  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxide were good at the CF ratio of 1 : 1, the average crystal size was near 70 nm, and the specific surface area was about  $20.22\text{ m}^2\cdot\text{g}^{-1}$ . The full conversion of CO into  $\text{CO}_2$  has been accomplished at a relatively low temperature of 270 °C under insufficient  $\text{O}_2$  conditions.

## 1. Introduction

Every year, the world emits a large amount of CO gas from thermal power plants, metallurgical plants, vehicles, wood burning, and waste burning. According to research in several developmental countries, thousands of people are died each year due to CO gas poisoning [1, 2]. The easy cause of CO gas poisoning is because its colorless, odorless, and nontoxic

properties are difficult to recognize. CO gas is the incomplete oxidation product of carbon compounds at low temperatures and  $\text{O}_2$  deficiency [3–5]. Under suitable conditions, CO exhibits strong reducing properties for medium metal oxides, so it has been studied and used in the metallurgical industry early [6]. Because of its possible toxic property to humans, the study and treatment of CO gas together with its secondary  $\text{CO}_2$  product are very important. In some

technical and industrial fields, CO<sub>2</sub> has begun to be captured and applied to reduce the greenhouse effect [7]. There are two popular methods for CO treatment as follows: adsorbing to capture CO gas [8–10] or converting CO to CO<sub>2</sub> by metal oxide catalysts [11].

Nowadays, CeO<sub>2</sub> is one of the important metal oxides in gas conversion catalysis. Cerium (Ce) belongs to the rare earth family and orders the second in the lanthanide series. Ce reserves account for a small proportion of the earth's crust, so exploitation is difficult and expensive. Ce has the electron configuration [Xe]4f<sup>1</sup>5d<sup>1</sup>6s<sup>2</sup>, which it can exist in the oxidation states Ce<sup>+3</sup> ([Xe]4f<sup>1</sup>5d<sup>6</sup>s) and Ce<sup>+4</sup> ([Xe]4f<sup>5</sup>d<sup>6</sup>s). Thus, CeO<sub>2</sub> and Ce<sub>2</sub>O<sub>3</sub> are the compounds of Ce with oxygen, and CeO<sub>2</sub> is more stable due to the electron configuration of Ce<sup>+4</sup> ([Xe]4f<sup>5</sup>d<sup>6</sup>s) being similar to that of the inert xenon gas [12]. The flexible conversion of the two Ce<sup>+4</sup> and Ce<sup>+3</sup> states through the electron donor mechanisms on the surface of CeO<sub>2</sub> makes it widely used in catalytic reactions such as wastewater treatment [13–16], water-gas phase transition [17], and gas conversion [18–21]. CeO<sub>2</sub> oxides can be prepared by hydrothermal method [21, 22], coprecipitation [23–25], sol-gel [19, 26], impregnation [27], and combustion [18]. The choice of the fabrication method decides the crystal structure, morphology, and physicochemical properties of CeO<sub>2</sub>, leading to affect the CO conversion ability of the catalyst [21, 22, 28]. The surface of the fabricated CeO<sub>2</sub> crystals is often imperfect due to lattice defect, vacuous O<sup>2-</sup> ion (denoted V<sub>O</sub>) sites, and unsaturated bonds leading to the existence of Ce<sup>+3</sup> and Ce<sup>+4</sup> states [20, 29]. The ratio between the number of Ce<sup>+3</sup> ions to Ce<sup>+4</sup> ions (Ce<sup>+3</sup>/Ce<sup>+4</sup>) is related to the number of V<sub>O</sub> vacancies on the CeO<sub>2</sub> crystal surface, so it determines the oxygen storage capability and oxygen lease capability [25]. When the Ce<sup>+3</sup>/Ce<sup>+4</sup> ratio is large, the number of V<sub>O</sub> vacancies on the surface is also large [30]. Besides, the number of V<sub>O</sub> also depends on the crystal morphology, and it decreases gradually with nanorods > cubes nanoscale > nanopolyhedra [29]. The density functional theory (DFT) calculations show that for low-index faces, the energy to release an O<sup>2-</sup> ion from the lattice to create a V<sub>O</sub> vacancy of (111) face is the largest in comparison with the (110) and (100) face, so the number of V<sub>O</sub> on the faces decreases in the order (110) > (100) > (111) [29]. In addition, CeO<sub>2</sub> is a direct band-gap semiconductor with bandgap energies of 2.56 eV for the bulk sample and 3.23 eV for the nanoscale, corresponding to the absorption transition energy 2p (O<sup>2-</sup>) → 4f (Ce<sup>+4</sup>) [26, 30]. When an O<sup>2-</sup> ion on the crystal surface gets an excitation agent to promote the electron transition 2p → 4f, it will separate from the crystal lattice leaving a V<sub>O</sub> vacancy in company with the oxidation state transformation Ce<sup>+4</sup>(4f<sup>0</sup>) → Ce<sup>+3</sup>(4f<sup>1</sup>) [11]. V<sub>O</sub> vacancies are capable of adsorbing CO or O<sub>2</sub> molecules at these positions, so they play an important role in catalytic reactions. An O<sub>2</sub> molecule can be captured by a V<sub>O</sub> vacancy to form an O<sup>2-</sup> lattice ion [31]. The combination reactions of CO molecules with the absorbed O<sub>2</sub> molecules on the surface of the oxide catalyst are occurred by the Langmuir-Hinshelwood (L-H) mechanism or with the O<sup>2-</sup> ions of the lattice by the Mars-van Krevelen (M-K) mechanism [29]. The CeO<sub>2</sub> oxide catalyst

material becomes thermal stable, has a fast catalytic rate, and has a low catalytic temperature when combined with some rare metals such as Pt [32], Pd [33], Au [34], and Ag [35]. However, a current trend uses the low-cost transition metal iron (Fe) element to make Fe<sub>2</sub>O<sub>3</sub>-CeO<sub>2</sub> mixed oxides. In nature, iron exists in both Fe<sup>2+</sup> and Fe<sup>3+</sup> oxidation states. The radius of Fe<sup>3+</sup> ion (0.64 Å) is smaller than that of Ce<sup>+4</sup> ion (1.01 Å), so the substitution of Fe<sup>3+</sup> ions in some Ce<sup>+4</sup> ion positions can shrink the CeO<sub>2</sub> crystal lattice, the 2θ diffraction angle positions of the (111) face is shifted towards the larger angle, and the formed possibility of V<sub>O</sub> vacancies is also increased [36]. The optical absorption transition properties of Fe<sub>2</sub>O<sub>3</sub> oxide include the direct charge transition O<sup>2-</sup>(2p) → Fe<sup>3+</sup>(3d) in the ultraviolet region and the indirect charge transition between Fe<sup>3+</sup>(3d) states in the visible region [37]. The conversion ability between Fe<sup>3+</sup> and Fe<sup>2+</sup> ions become even more flexible in Fe<sub>2</sub>O<sub>3</sub>-CeO<sub>2</sub> composition, which is demonstrated through photochemical reactions treating organic pigments [15]. The O<sup>2-</sup> ions of the Fe-O-Ce bonds react better with CO than that of the Ce-O-Ce bonds [38], even so than those of Fe-O-Fe bonds. The energy separates an O<sup>2-</sup> atom from the surface to create a V<sub>O</sub> vacancy of about 3.04 eV [39]. The redox potential of the Fe<sup>+3</sup>/Fe<sup>+2</sup> pair (0.71 eV) is smaller than that of the Ce<sup>+4</sup>/Ce<sup>+3</sup> pair (1.61 eV), which facilitates the electron donor-acceptor processes and the oxidation state transformation of Ce<sup>+4</sup>/Ce<sup>+3</sup> and Fe<sup>+3</sup>/Fe<sup>+2</sup> pairs [20]. Other studies show that the combination of Ce and Fe elements forms CeO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> mixed oxide, which can reduce the reactive catalytic temperature of 50% (T<sub>50</sub>) and 100% (T<sub>100</sub>) CO conversion in comparison with single oxide catalysts of CeO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub>, depending on the fabrication method [31, 40, 41].

From the above-mentioned characteristics, it shows that the CeO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> mixed oxide material has many advantageous photochemical properties that need further studying and applying for life. This study was carried out with the purpose of making CeO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> mixed oxide materials from popular Fe metal, reducing the content of rare earth Ce and the reactive catalytic temperature of complete CO conversion to CO<sub>2</sub> while applying to treat the exhaust gas in simple incinerators.

## 2. Chemicals and Experiments

**2.1. Preparation of Mixed Oxide Catalysts.** High-purity chemicals were used as Fe(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O (99.98%), Ce(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O (99.98%), and tartaric acid (TA) (99.98%). TA was dissolved with twice distilled water at 80 °C to get A solution. The above nitrate salts were also dissolved to give B solution such that the molar ratio of CF mixture to TA solution always was 1:3. A regulator matter was utilized to keep the pH of the solution equal to 2. The B solution was slowly added to the A solution, stirred, and heated at 80 °C for 2 h to obtain a pale-yellow homogeneous gel solution. The gel was then dried at 100 °C for 4 h to obtain a porous shape sample. The obtained sample was analyzed by TGA to investigate the phase transition of the sample in accordance with the calcination temperature. The samples calcined in turn at 450, 550, 650, 750, 850, and 950 °C for 2 h to obtain mixed

oxides which denote as CF450, CF550, CF650, CF750, CF850, and CF950. To investigate the influence of the CF molar ratio on the structural phase formation of mixed oxides, the weight of the nitrate salts in the B solution was calculated so that the CF molar ratio of the obtained gels is in turn 9:1; 3:1; 1:1; 1:3, and 1:9. These gels, calcined at 650 °C for 2 h, received the mixed oxides of CF91, CF31, CF11, CF13, and CF19, respectively.

## 2.2. Analytics

**2.2.1. Differential Thermal Analysis and Thermogravimetric Analysis.** Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) were performed on a Labsys Evo 1600 system. Samples were measured in air, heating rate 10 °C.min<sup>-1</sup>.

**2.2.2. X-Ray Diffraction Analysis.** X-ray diffraction (XRD) measurements of the studied samples were performed on a Siemens D5000 X-meter.

**2.2.3. Scanning Electron Microscopy Image.** The surface morphology of the materials was recorded by scanning electron microscopy (SEM). Samples were measured on a Hitachi S-4800 instrument operating at 10 kV with a magnification of 80000-100000 times.

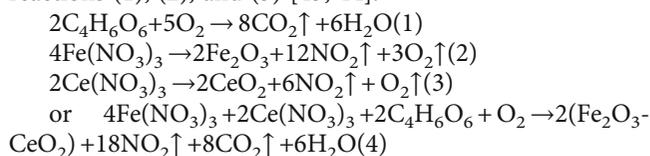
**2.2.4. Determine the Specific Surface Area.** The sample surface area was determined by the Brunauer-Emmett-Teller (BET) method on the Autosorb IQ Station measuring system. BET equation:  $P/[V(P_o - P)] = 1/V_m C + [(C - 1)/V_m C] \cdot P/P_o$ , where  $P$  is the equilibrium pressure,  $P_o$  is the saturated vapor pressure of the adsorbed gas at experimental temperature,  $V$  is the volume of the adsorbed gas at pressure  $P$ , and  $V_m$  is the saturated gas volume of monolayer adsorption per 1 gram of adsorbent, and  $C$  is the BET constant. The specific surface area BET ( $S_{BET}$ ) of the material is calculated according to the equation:  $S_{BET} = (V_m/M) \cdot N A_m d$ , where  $d$  is the density,  $M$  is the molar mass of the adsorbent, respectively,  $N$  is the Avogadro number ( $N = 6.023 \times 10^{23}$  molecules.mol<sup>-1</sup>), and  $A_m$  is the cross-sectional area of 1 molecule occupied on the surface adsorbent.

**2.2.5. Fourier Transform Infrared Spectra.** The Fourier transform infrared (FTIR) spectra of the samples were recorded in the range of wavenumbers from 400 cm<sup>-1</sup> to 4000 cm<sup>-1</sup> on the Impact 410 spectrometer.

**2.2.6. Determination of CO Conversion.** The TPSR temperature surface response program was conducted with a Siemens temperature controller. The CO conversion of the catalytic material performing on the microcurrent device was calculated by the following formula:  $H (\%) = (C_f/C_o) \cdot 100\%$ , where  $H$  is the CO (%) conversion and  $C_o$  and  $C_f$  are the CO concentrations before and after catalysis, respectively.

## 3. Results and Discussions

**3.1. DTA and TGA Spectra of CeO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> Mixed Oxides.** The DTA and TGA spectra of the gel sample with the CF/TA ratio of 1:3 presented in Figure 1. The DTA curve showed the temperature increased from 100 °C to 250 °C. The sample weight decreased by 38.89%, with an endothermic peak at 217 °C. This reduction may be due to the physical evaporation of the adsorbed water on the gel surface [42, 43]. In the temperature range from 250 °C to 550 °C, the sample weight decreased by 34.83% on the TGA curve corresponding to the exothermic peak of 328 °C on the DTA curve, which was caused by the pyrolysis processes of the nitrate salts released O<sub>2</sub> and NO<sub>2</sub> gas; the combustion process of the TA released CO<sub>2</sub> gas and H<sub>2</sub>O according to the following reactions (1), (2), and (3) [43, 44]:



When the temperature increased from 550 °C to 900 °C, the sample weight was almost unchanged, showing that the Fe<sub>2</sub>O<sub>3</sub>-CeO<sub>2</sub> mixed oxide was formed and stable.

**3.2. FTIR Spectra of CeO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> Mixed Oxides.** The appearance of vibrations characterized to the bonds of CeO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> mixed oxide determined via the FTIR spectra. In Figure 2, the FTIR spectra of all samples in the broad absorption range from 3000 cm<sup>-1</sup> to 3500 cm<sup>-1</sup> appeared a peak at about 3378 cm<sup>-1</sup> characterized by the stretching vibration of the O-H group because of the physically adsorbed water; however, this peak intensity decreased gradually as the calcination temperature of samples increased. A peak at about 2337 cm<sup>-1</sup> characterized the stretching vibration of adsorbed CO<sub>2</sub> molecules from the air for CF450, CF550, and CF650 samples, and this peak was completely suppressed for CF750 and CF850 samples. The peaks at 1630, 1380, and 1121 cm<sup>-1</sup> represented the C=O and C-O stretching vibrations of the -COO- group [42, 43]. At the high calcination temperature, the process of CO<sub>2</sub> separation occurred, so the absorption intensity of the band related to -COO- decreased and expanded into a large halo as on the CF850 sample. It is noteworthy that the appearance of the peak at 529 cm<sup>-1</sup> with strong absorption intensity for samples calcined at 850 °C was typical for Fe-O vibrations, the peaks of 643 and 443 cm<sup>-1</sup> were characteristic of Ce-O vibrations [42, 43, 45], and the above peaks appeared clear for the CF650 sample. This also shows that the characteristic phases of the CeO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> mixed oxide were soon formed, and it was also consistent with the result of the TGA-DTA analysis.

**3.3. The Role of Calcination Temperature and Metal Ion Molar Ratio in the Structural Phase Formation of CeO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> Mixed Oxides**

**3.3.1. The Role of Calcination Temperature.** The thermogravimetric analysis shows that the suitable calcination

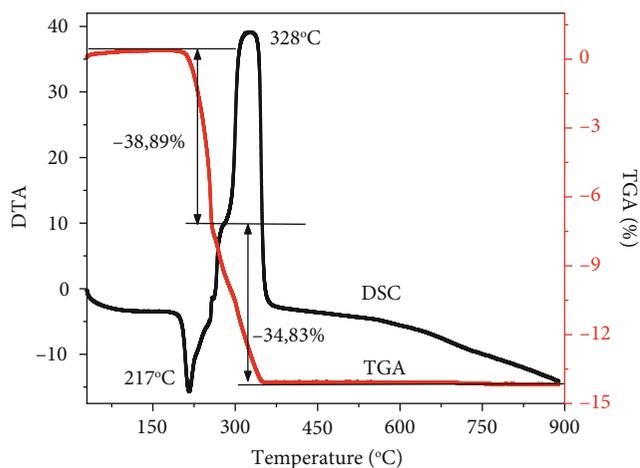


FIGURE 1: TGA and DTA spectra of the gel sample with the CF/TA ratio of 1:3.

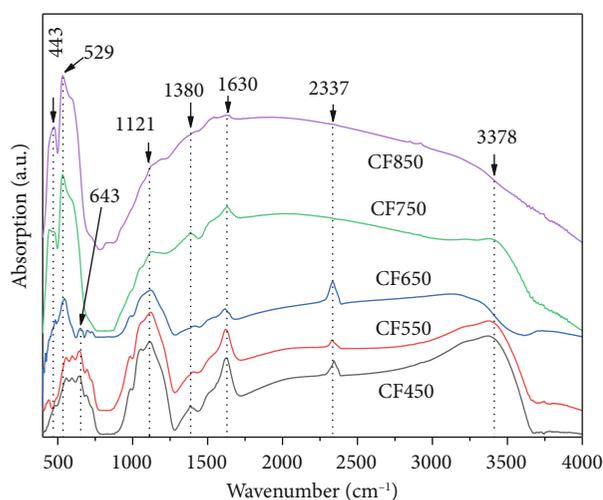


FIGURE 2: FTIR spectra of gel samples with the CF/TA ratio of 1:3 calcined at different temperatures for 2 h.

temperature range was from 450 to 900 °C. Figure 3 presents the XRD pattern of the  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxides calcined at 450, 550, 650, 750, 850, and 900 °C for 2 h. At 450 °C and 550 °C, the XRD pattern of CF450 and CF550 only appeared the (111), (200), and (220) faces corresponding to the  $2\theta$  diffraction angles at 28.96°, 33.11°, and 47.47° (JCPDS No. 34-0394), in which the (111) face had the greatest intensity. These faces characterized the face-centered cubic structure of  $\text{CeO}_2$  in mixed oxides [36]. Meanwhile, at these calcination temperatures, there were almost no faces that characterized the structural phase of  $\text{Fe}_2\text{O}_3$ . This may be because the samples had not reached good crystallinity, and the crystal structure was not complete. At 650 °C, the reflection faces characterizing the structural phase of  $\text{Fe}_2\text{O}_3$  also occurred clearly and intertwined with those of  $\text{CeO}_2$  as the represented spectrum of the CF650 sample in Figure 3(b). The (311), (222), (400), (331), (420), (422), and (511) faces corresponded to  $2\theta$  diffraction angles of 56.32°, 59.02°, 69.42°,

76.66°, 79.92°, 88.39°, and 95.04° belonged to the  $\text{CeO}_2$  crystal structure, but the (012), (110), (024), and (116) faces assigned to  $2\theta$  angles of 24.92°, 35.64°, 49.46°, and 54.02°, respectively, representing the hexagonal structure phase of  $\text{Fe}_2\text{O}_3$  (JCPDS No. 33-0664) [14, 19, 27].

The diffraction intensity on all faces became narrower and stronger as the calcination temperature increased. This meant that the samples had better crystallinity, a complete crystal structure, and larger crystal sizes [46]. However, when the samples calcined to 750 °C and 850 °C, a separating tendency of mixed oxide phase into two single oxide phases of  $\text{CeO}_2$  and  $\text{Fe}_2\text{O}_3$  was happened because of these more thermal stable oxides. Based on the XRD pattern and Brass' formula, it is able to determine the  $a$  lattice constant via the (111) face of all the  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxides, as seen in Table 1.

The results showed that there was a slight increase of the  $a$  lattice constant from 5.3385 to 5.3530 Å, corresponding to a slight decrease of the  $2\theta$  diffraction angle from 28.96 to 28.89 Å. The increase of the  $a$  lattice constant together with the left shift of the  $2\theta$  angle indicated that the calcination sample at 450 °C had formed in a solution of  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxide [23]. It is explained that the  $\text{Fe}^{3+}$  (0.64 Å) ion radius is smaller than the  $\text{Ce}^{4+}$  (1.01 Å) ion radius, so when the  $\text{Ce}^{4+}$  ions are replaced by  $\text{Fe}^{3+}$  ions at some  $\text{CeO}_2$  lattice sites, these substitutions cause the shrink of the crystal lattice [36]. In contrast, the partial separation of the  $\text{CeO}_2$  phase widened the crystal lattice and shifted the  $2\theta$  angle position of the (111) face towards a smaller angle as illustrated by CF450, CF650, and CF850 patterns in the small inset of Figure 3(b). Thus, it can be seen that the change in calcination temperature affected the formation of mixed oxide solution. At 650 °C, the crystal was complete with the appearance of both  $\text{CeO}_2$  and  $\text{Fe}_2\text{O}_3$  phases. When the calcination temperature of the sample increased, the mixed oxides had good crystallization and tended to the segregation of single oxide phases.

**3.3.2. The Role of Metal Ion Molar Ratio.** XRD pattern of  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxides with different CF molar ratios calcined at 650 °C for 2 h, as seen in Figure 4. When the molar quantity of  $\text{Ce}^{3+}$  ions was more than that of  $\text{Fe}^{3+}$  ions (CF91 and CF31 samples), the XRD pattern manifested the (111), (200), (220), (311), (222), (400), (331), (422), and (511) faces corresponded to the  $2\theta$  diffraction angles of 28.82°, 33.11°, 47.47°, 56.32°, 59.07°, 69.41°, 76.64°, 79.91°, 88.39°, and 95.04° and characterized the crystalline structure phase of  $\text{CeO}_2$  (for CF91 and CF31 samples in Figure 4(a)) [14, 19, 27].

This may be because with the calcination condition to 650 °C, and the used  $\text{Fe}^{3+}$  ion content was smaller in comparison with the  $\text{Ce}^{4+}$  ion content, so  $\text{Fe}^{3+}$  ions were dissolved in the crystal lattice of  $\text{CeO}_2$  oxide [23]. When the molar quantity of  $\text{Ce}^{3+}$  ion and  $\text{Fe}^{3+}$  ions was equal, the characteristic faces of the  $\text{Fe}_2\text{O}_3$  phase appeared and interwove with those of  $\text{CeO}_2$ , and the XRD pattern of CF11 was presented again in Figure 4(b). The presence of the typical (110) face of  $\text{Fe}_2\text{O}_3$  shows that the mixed oxide has

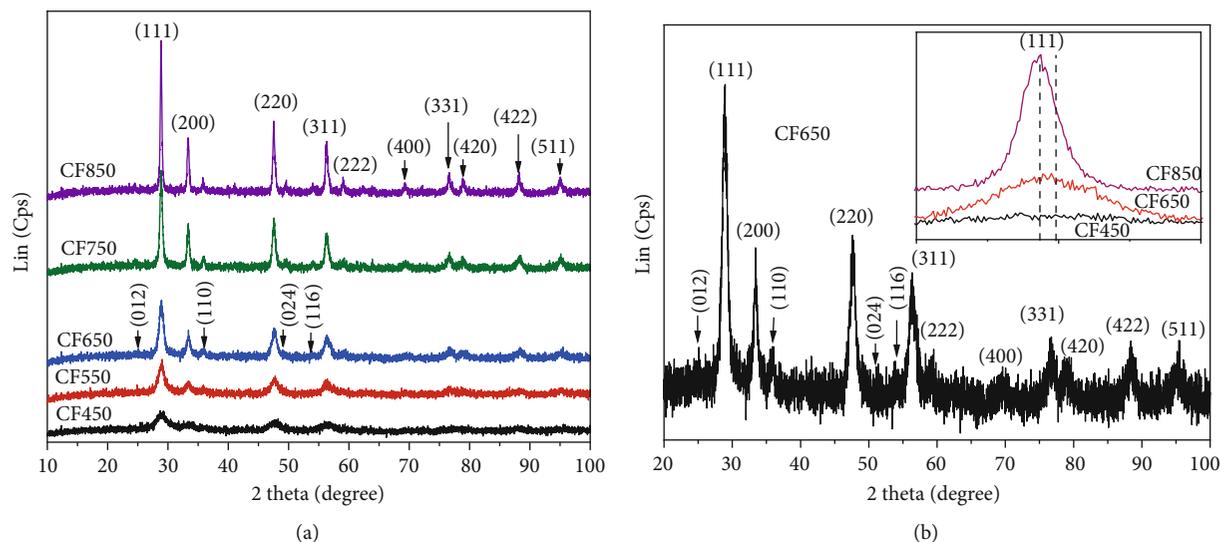


FIGURE 3: XRD pattern of  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxides. Samples with the CF molar ratio of 1 : 1 calcined at different temperatures for 2 h (a) and CF650 sample was redrawn (b).

TABLE 1:  $2\theta$  diffraction angle and  $a$  lattice constant of  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxides calcined at different temperatures for 2 h.

Sample	CF450	CF550	CF650	CF750	CF850
Diffraction angle $2\theta$ (degree)	28.96	28.94	28.92	28.89	28.88
Lattice constant $a$ (Å)	5.3385	5.3422	5.3458	5.3512	5.3530

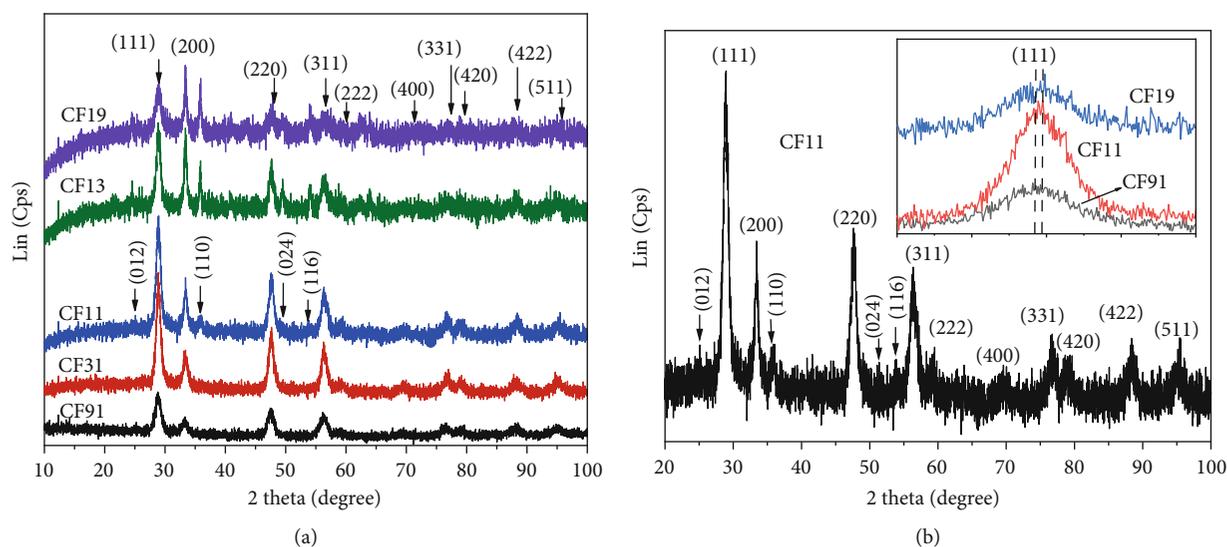


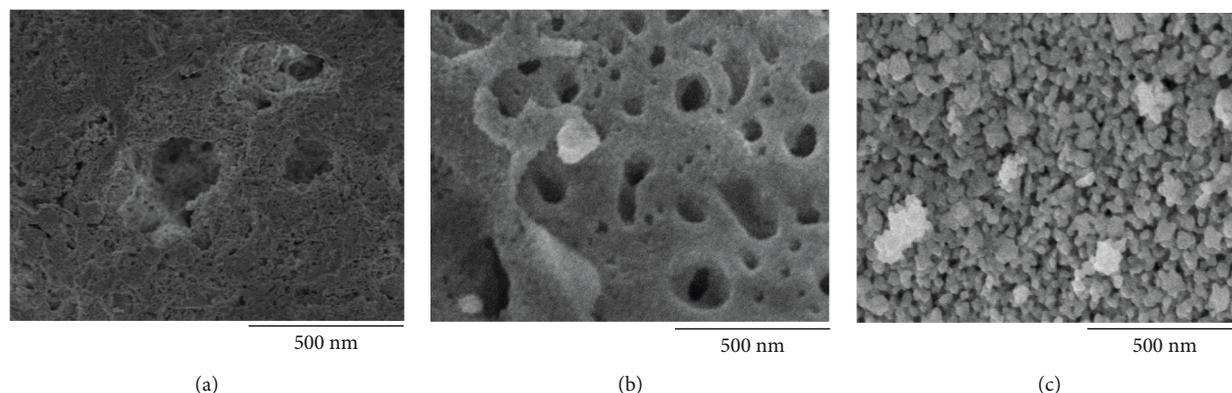
FIGURE 4: XRD patterns of  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxides. Samples with different CF molar ratios calcined at  $650^\circ\text{C}$  for 2 h (a) and CF11 sample was redrawn (b).

separated into phases [47]. When the molar quantity of  $\text{Ce}^{3+}$  ions was smaller than those of  $\text{Fe}^{3+}$  ions (for CF13 and CF19 samples), the diffraction intensity on the (110) face became more narrow and stronger. This phenomenon was known that the molar quantity of  $\text{Fe}^{3+}$  ions in mixed oxides was high, which led to the split gradually mixed oxides into

two individual oxide phases of  $\text{CeO}_2$  and  $\text{Fe}_2\text{O}_3$  [23]. The  $a$  lattice constants of  $\text{CeO}_2$  were calculated from the (111) face in Table 2. As a result, there was a slight decrease of  $a$  lattice constant from  $5.3639\text{ \AA}$  to  $5.3385\text{ \AA}$ , while the  $2\theta$  diffraction angle of the (111) face shifted towards the greater diffraction angle from  $28.82^\circ$  to  $28.98^\circ$ , respectively. This

TABLE 2:  $2\theta$  diffraction angle and  $a$  lattice constant of  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxides with different CF molar ratios calcined at  $650^\circ\text{C}$  for 2 h.

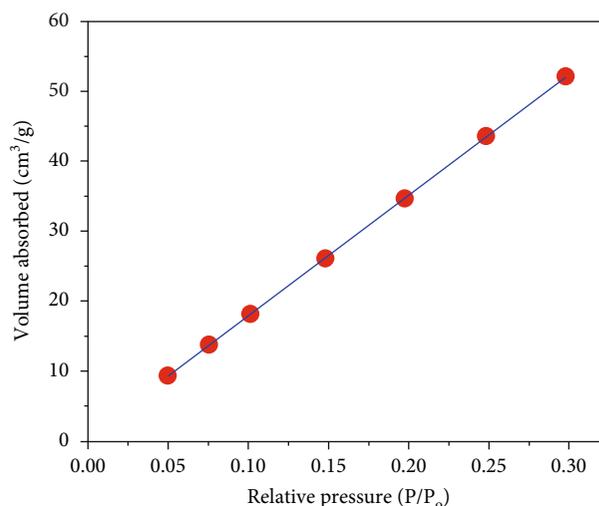
Sample	CF91	CF31	CF11	CF13	CF19
Diffraction angle $2\theta$ (degree)	28.82	28.87	28.92	28.94	28.98
Lattice constant $a$ (Å)	5.3639	5.3548	5.3458	5.3421	5.3385

FIGURE 5: SEM images of  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxides with the CF ratio of 1 : 1 calcined at  $450^\circ\text{C}$  (a),  $650^\circ\text{C}$  (b), and  $850^\circ\text{C}$  (c) for 2 h.

shift can observe clearly by CF91, CF11, and CF19, as seen in the small inset in Figure 3(b) [20, 23, 24, 36]. The above results showed that the change in CF molar ratio also affected the formation of the crystal structure phase of the mixed oxides. With a CF molar ratio of 1 : 1, there appeared characteristic reflection faces for the structural phases of  $\text{CeO}_2$  and  $\text{Fe}_2\text{O}_3$ . When the molar quantity of  $\text{Fe}^{3+}$  ions were high, the  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxide tended to separate into two oxide phases of  $\text{CeO}_2$  and  $\text{Fe}_2\text{O}_3$ .

**3.4. Morphology of  $\text{Fe}_2\text{O}_3\text{-CeO}_2$  Mixed Oxides.** SEM images of the  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxides were shown in Figure 5 with the CF molar ratio of 1 : 1 which were calcined at temperatures of  $450^\circ\text{C}$ ,  $650^\circ\text{C}$ , and  $850^\circ\text{C}$  for 2 h. It is observed that the surface of the CF450 sample was porous. This is the characteristic of materials that are prepared by the combustion method using organic compounds to create gels (Figure 5(a)) [48]. For the CF650 sample, the porous property became clear to many honeycomb-like cavities. It can be explained that the molecular structure of TA consists of strongly polar functional groups  $\text{HOOC}(\text{HO})\text{CC}(\text{OH})\text{COOH}$  that played the role of stretching and uniformly dispersing  $\text{Ce}^{3+}$  and  $\text{Fe}^{3+}$  ions in solution [49]. At  $650^\circ\text{C}$ , the decomposition of the organic component and the pyrolysis of nitrate salts led to a decrease in volume and mass, creating a system of space-connected microcapillary tubes (Figure 5(b)). This created a porous property of mixed oxides as well as the obtained result in other reports [50]. The average crystal size of the oxide was determined to be about 70 nm. For the CF850 sample, the crystals tended to break the spatial porous block into discrete nanosized particles (Figure 5(c)) [42].

From the dependence of  $P/[V(P_0 - P)]$  on  $P/P_0$  in the BET equation, the adsorption-desorption isotherm representing the relation between the adsorbed volume and the relative pressure was shown in Figure 6. The linear shape

FIGURE 6: Nitrogen adsorption-desorption isotherms of  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxide with the CF molar ratio of 1 : 1 calcined at  $650^\circ\text{C}$  for 2 h.

of the isotherm in the low  $P/P_0$  value range indicated that monolayer adsorption occurred on the surface of the porous catalyst, and it is also suitable for the linear region in the range of  $P/P_0$  from 0.05 to 0.35 of this material [51]. The specific surface area of the  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxide, CF650 sample, was determined at about  $20.22\text{ m}^2\cdot\text{g}^{-1}$ .

**3.5. Process of CO Conversion to  $\text{CO}_2$ .** The process of CO oxidation by  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxide catalysts and the curves of CO conversion according to temperature is described via Figure 7. Under the catalytic temperature condition below  $300^\circ\text{C}$  and deficient  $\text{O}_2$  gas, the activities of CO oxidation took place [6]. The CF91, CF31, CF13, and CF19

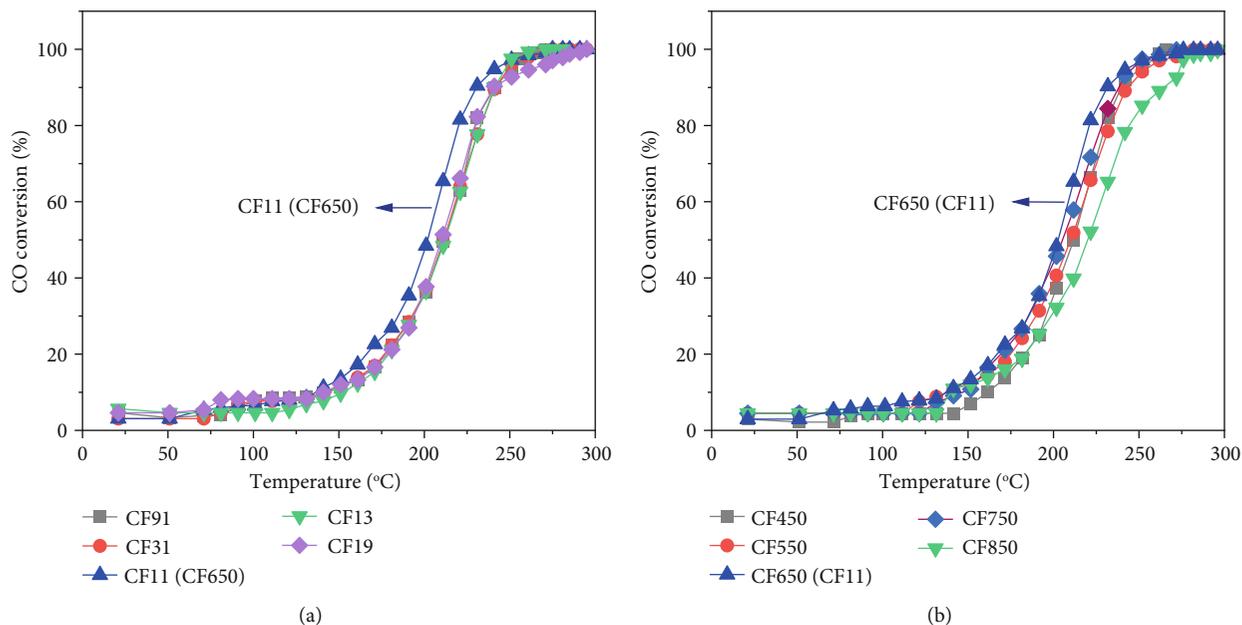
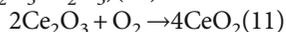
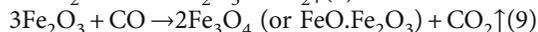
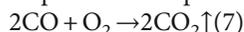


FIGURE 7: CO conversion of CeO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> samples according to temperature. Samples with different CF molar ratios calcined at 650 °C for 2 h (a) and samples calcined at different temperatures for 2 h (b).

samples with the different molar quantities of Ce<sup>3+</sup> and Fe<sup>3+</sup> ions shown that the CO conversion curves were similar variation, and the ability of CO conversion was lower than that of the CF11 sample (Figure 7(a)). For the CF11 sample (also named as CF11(CF650) or CF650(CF11)), the CO conversion curve was left-skewed, meaning that at the same catalytic temperature, the CO conversion of the CF11 sample was higher and finished earlier than other samples. This can be because the molar quantity of Ce<sup>3+</sup> and Fe<sup>3+</sup> ions was the same, the quantity of O<sup>2-</sup> ions in the -Ce-O-Fe- bonds was more dominant than that in two -Ce-O-Ce and -Fe-O-Fe- bonds of other samples, so the catalytic reactions of CF11 sample also happened better than the others [38]. The CO conversion for all samples can be explained by the following reactions:

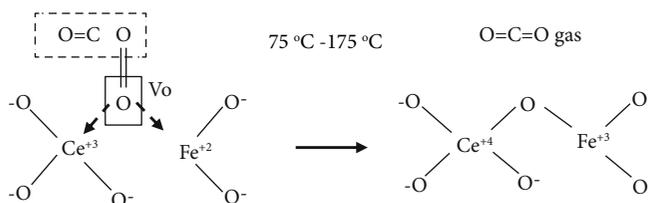


When the catalytic temperature increased from 75 °C to 175 °C, the amount of CO gas was converted about 12%; this may be because a part of CO gas was oxidized by O<sub>2</sub> in (7) [11]. In addition, these reactions can also occur between adsorbed CO and O<sub>2</sub> gases on the surface of the CeO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> catalyst to emit CO<sub>2</sub> by the J-H mechanism. When the temperature increased from 175 °C to 260 °C, the CO conversion was rapid for all samples but reached the fastest 50% at 211 °C for CF11. The intensification of CO oxidation can be because CO combines with an O<sup>2-</sup> at the lattice sites in -Ce-O-Fe-, -Ce-O-Ce- and -Fe-O-Fe- bonds to release CO<sub>2</sub> by the M-K mechanism in (8) and (9) [27, 29]. The Fe<sub>3</sub>O<sub>4</sub> product was a mixture of two FeO·Fe<sub>2</sub>O<sub>3</sub> oxides, meaning that only a part of Fe<sup>3+</sup> ions were reduced to Fe<sup>2+</sup> ions. This low-temperature reaction was also carried out early by another researcher [6].

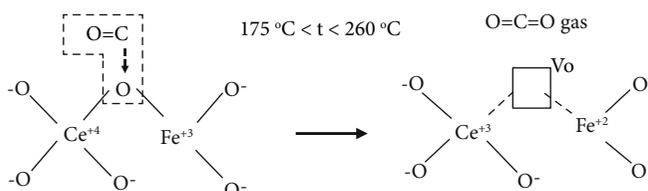
When the temperature was over 260 °C, the CO conversion process slowed down because it needed the recovery of the CeO<sub>2</sub> oxidizing agent from Ce<sub>2</sub>O<sub>3</sub> according to (10) and (11) and then reached 100% at 275 °C faster for the CF11 sample. The recovery here was due to the redox potential of Fe<sup>3+</sup>/Fe<sup>2+</sup> (0.711 V) < Ce<sup>4+</sup>/Ce<sup>3+</sup> (1.61 V) [20], so a tendency happened the redox process Ce<sup>4+</sup> + Fe<sup>2+</sup> → Ce<sup>3+</sup> + Fe<sup>3+</sup> [23, 52], or this was also the process of transferring an O<sup>2-</sup> ion from Ce<sup>4+</sup> ion to a neighboring Fe<sup>2+</sup> ion, creating the charge balance [38]. The (11) reaction turned Ce<sup>3+</sup> into initial Ce<sup>4+</sup>, which was an important agent that helped Fe ion form a closed-loop bridge of oxidation states by the (9) and (10) reactions as follows: Fe<sub>2</sub>O<sub>3</sub> → Fe<sub>3</sub>O<sub>4</sub> → FeO → -Fe<sub>2</sub>O<sub>3</sub>. The participation of Fe<sup>3+</sup> ions in the mixed oxide made the deformation of lattice structure and increased the reaction centers of gases storage and release. Therefore, the conversion of CO to CO<sub>2</sub> became more efficient under low temperatures and O<sub>2</sub> deficiency [4, 5]. It can be seen that thanks to the redox process between Fe<sup>2+</sup> and Ce<sup>4+</sup>, the catalyst system was restored to its original properties and was not degraded.

With the same catalytic mechanisms above, among all CeO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub> mixed oxide samples calcined at different temperatures for 2 h, the CO conversion of the CF650 sample was better than all of those (Figure 7(b)). The CF650 curve was also skewed to the left and rapidly reached 100% at 270 °C. It is possible that for the samples CF450 and CF550, the crystallization of the oxides had not been complete, and there were still carbonate components of organic combustion products as analyzed in the TGA-DGT and FTIR spectra, which interfered with the process of CO oxidation. For the CF750 and CF850 samples, the crystal grain size increased, and the surface area decreased, leading to a decrease in the contact and oxidation capacity. Moreover, at high calcination temperature, there was

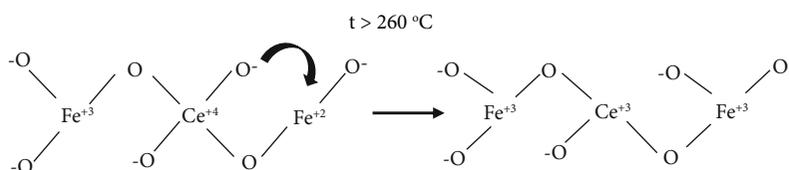
For the J-H mechanism, a CO molecule reacted with adsorbed an  $O_2$  molecule at  $V_O$  vacancy to emit a  $CO_2$  molecule (7):



For the M-K mechanism, a CO molecule combined with an  $O^{2-}$  ion of the crystal lattice to release a  $CO_2$  molecule and a  $V_O$  vacancy (8) and (9):



The transformation of  $Ce^{+4}$  to  $Ce^{3+}$  and  $Fe^{+2}$  to  $Fe^{3+}$  (10):



The transformation of  $Ce^{+4}$  from  $Ce^{+3}$  after an  $O_2$  molecule was captured by a  $V_O$  vacancy (11):

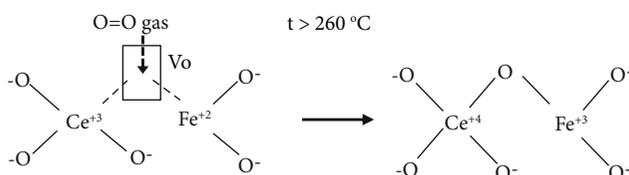


FIGURE 8: The mechanisms of CO oxidation and the oxidation state conversion of metal ions between  $Ce^{+4}/Ce^{+3}$  and  $Fe^{+3}/Fe^{+2}$  pairs on the  $CeO_2-Fe_2O_3$  catalyst surface.

a tendency of phase separation into two single oxides as mentioned in the XRD pattern, so their oxidation ability became less than that of mixed oxide catalysts [23]. Thus, the calcination temperature changed the crystallinity and affected the CO catalytic ability of the  $CeO_2-Fe_2O_3$  mixed oxides. From the above arguments, it is able to describe the mechanisms of CO oxidation, capture  $O_2$ , and release  $O^{2-}$  ions, creating  $V_O$  vacancies on the surface of  $CeO_2$  crystal, as shown in Figure 8.

For the J-H mechanism, a CO molecule reacted with adsorbed an  $O_2$  molecule at  $V_O$  vacancy to emit a  $CO_2$  molecule (7):

For the M-K mechanism, a CO molecule combined with an  $O^{2-}$  ion of the crystal lattice to release a  $CO_2$  molecule and a  $V_O$  vacancy (8) and (9).

The transformation of  $Ce^{+4}$  to  $Ce^{+3}$  and  $Fe^{+2}$  to  $Fe^{+3}$  was in (10).

The transformation of  $Ce^{+4}$  from  $Ce^{+3}$  after an  $O_2$  molecule was captured by a  $V_O$  vacancy (11):

TABLE 3:  $T_{50}$  and  $T_{100}$  temperatures of  $CeO_2-Fe_2O_3$  samples. Samples with different CF molar ratios calcined at  $650^\circ C$  for 2 h (a) and samples calcined at different temperatures for 2 h (b).

a			b		
Sample	$T_{50}$ ( $^\circ C$ )	$T_{100}$ ( $^\circ C$ )	Sample	$T_{50}$ ( $^\circ C$ )	$T_{100}$ ( $^\circ C$ )
CF91	211	275	CF450	211	275
CF31	211	275	CF550	211	280
CF11	200	270	CF650(CF11)	200	270
CF13	211	275	CF750	205	275
CF19	211	295	CF850	221	275

The CO conversion temperatures at  $T_{50}$  and  $T_{100}$  of all  $CeO_2-Fe_2O_3$  samples are shown in Table 3. It can show that the change of the CF molar ratio and calcination temperature of samples affected the CO conversion of  $CeO_2-Fe_2O_3$  mixed oxide catalysts. The  $T_{50}$  and  $T_{100}$  temperatures of all

TABLE 4:  $T_{50}$  and  $T_{100}$  temperatures of  $\text{CeO}_2$ ,  $\text{Fe}_2\text{O}_3$ , and  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  oxides cited in some other references.

Oxide	Fabricated method	$T_{50}$	$T_M/T_{100}$ ( $T_M < T_{100}$ )	Reference
$\text{CeO}_2$	Polyol method	250°C	350°C	[57]
$\text{CeO}_2$	Combustion method	x	~240°C <sup>a</sup>	[58]
$\text{CeO}_2$	Hydrothermal method	216	~230°C	[22]
$\text{CeO}_2$	Combustion method	330°C	~410°C	[52]
$\text{CeO}_2$	Combustion method	~210°C	~260°C <sup>b</sup>	[48]
$\text{CeO}_2$	Co-precipitation method	500 K	700 K	[23]
$\text{CeO}_2$	Microwave and combustion method	362°C	~475°C	[41]
$\text{CeO}_2$	Hydrothermal method	x	~610 K	[55]
$\text{CeO}_2$	Surfactant-templated method	~230°C	~300°C	[46]
$\text{CeO}_2$	Thermal decomposition method	300°C	x	[40]
$\text{CeO}_2$	Solvothermal reaction method	~320°C	~400°C	[56]
$\text{Fe}_2\text{O}_3$	Co-precipitation method	620 K	750 K	[23]
$\text{Fe}_2\text{O}_3$	Co-precipitation method	290°C	450°C	[54]
$\text{Fe}_2\text{O}_3$	Surfactant-assisted method	~220°C	270°C	[53]
$\text{CeO}_2\text{-Fe}_2\text{O}_3$	Co-precipitation method	500 K	600 K	[23]
$\text{CeO}_2\text{-Fe}_2\text{O}_3$	Cyclic molecular designed dispersion method	~465 K	548 K	[27]
$\text{CeO}_2\text{-Fe}_2\text{O}_3$	Co-precipitation method	x	25°C <sup>c</sup>	[25]
$\text{CeO}_2\text{-Fe}_2\text{O}_3$	Hydrothermal method	166°C	~280°C	[19]
$\text{CeO}_2\text{-Fe}_2\text{O}_3$	Co-precipitation method	480 K	~575 K	[24]
$\text{CeFe}_{10}$	Thermal decomposition method	203°C	~280°C	[40]
$\text{Ce}_x\text{Fe}_{1-x}\text{O}_{2-\delta}$	Co-precipitation method	250°C	~450°C	[31]
$\text{Ce}_{0.98}\text{Fe}_{0.03}\text{O}_2$	Microwave and combustion method	298°C	~375°C	[41]

<sup>a,b,c</sup>The maximum CO conversion catalytic temperatures ( $T_M$ ) of 30%, 70%, and 96.17%, respectively. x is the reaction catalyst temperature that is unknown or out of measurement scale.

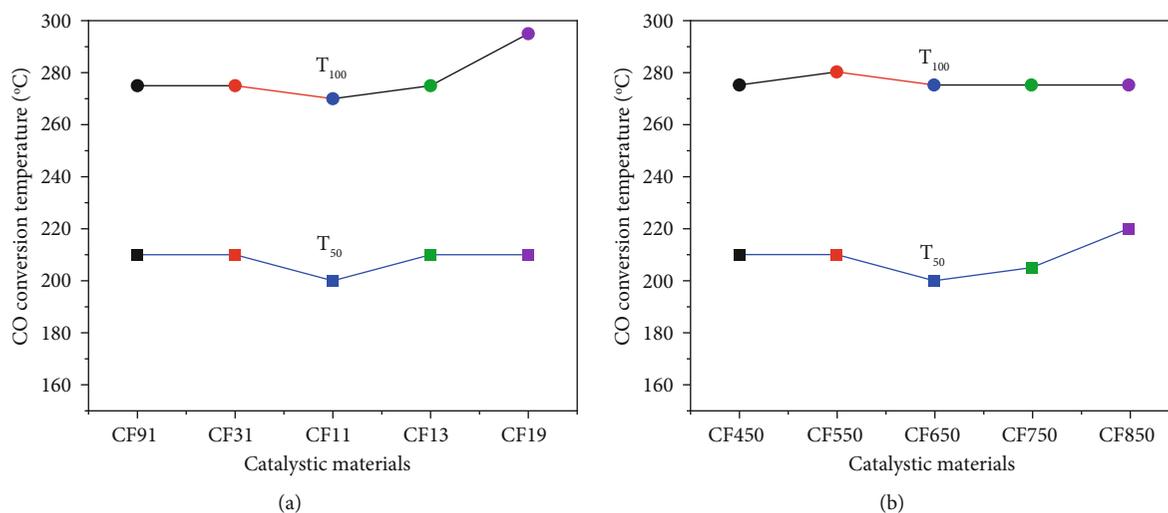


FIGURE 9:  $T_{50}$  and  $T_{100}$  temperatures of  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  samples. Samples with different CF molar ratios calcined at 650 °C for 2 h (a) and samples calcined at different temperatures for 2 h (b).

samples are quite low, so it is considered an advantage of this fabrication method. Of all the samples, the  $T_{50}$  and  $T_{100}$  temperatures of the CF11 (CF650) samples are 200 °C and 270 °C, respectively, which are lower than those of the other samples (Table 3(a) and 3(b)).

Some publications have shown that the  $T_{50}$  and  $T_{100}$  of oxide catalysts are different depending on the fabrication method, as shown in Table 4. In some cases, if the single  $\text{CeO}_2$  or  $\text{Fe}_2\text{O}_3$  oxide is used as a catalyst, the  $T_{50}$  and  $T_{100}$  also decrease quite low [22, 53], but the rest is very high

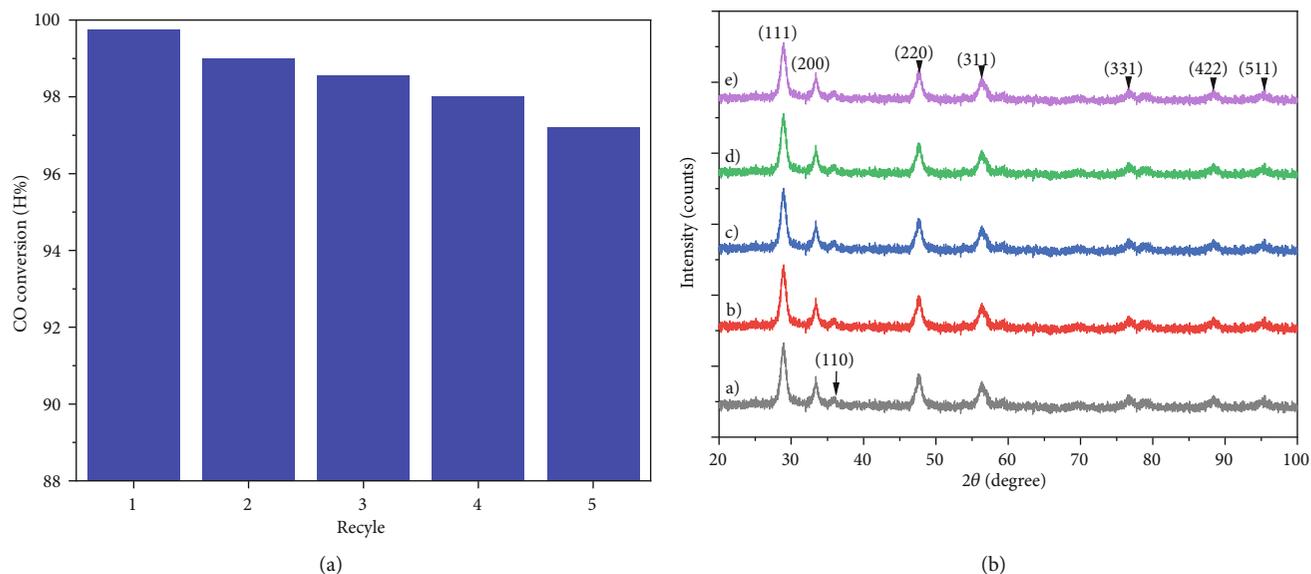


FIGURE 10: CO conversion of sample CF650(CF11) at  $T_{100}$  after 5 recycles (a) and corresponding XRD patterns (A) 1<sup>st</sup>, (B) 2<sup>nd</sup>, (C) 3<sup>rd</sup>, (D) 4<sup>th</sup>, and (E) 5<sup>th</sup> (b).

[23, 41, 46, 52, 54–56]. For  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  catalyst materials, several studies have achieved immediate maximum CO conversion (M) at room temperature but are not capable of 100% CO conversion [25]. Some results are quite similar [19, 27, 40], but the others are very high [23, 24, 31, 41].

The correlation between  $T_{50}$  and  $T_{100}$  temperatures of the  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxides is shown in Figure 9. It is seen that the  $T_{50}$  and  $T_{100}$  are quite low for all samples and the conversion ability is the best corresponding to the CF11 sample calcined at  $650^\circ\text{C}$  for 2 h.

Figure 10 shows five oxidation cycles at  $T_{100}$  that were used to study the structural and mixed catalytic stability of sample CF11(650). After 5 oxidation cycles, the CO conversion efficiency was lowered to 97.18% (Figure 10(a)). This reduction in CO oxidation performance might be attributed to phase separation in the mixed oxide, as evidenced by the presence of the  $\text{Fe}_2\text{O}_3$  phase at peak (110) (Figure 10(b)).

The studied results showed that  $\text{Fe}^{3+}$  ions joined in  $\text{CeO}_2$  crystal lattice, which has caused the deformation of the lattice structure, increasing the quantity of  $V_{\text{O}}$  vacancies. The  $V_{\text{O}}$  vacancies acted as the reaction centers, thereby promoting easier oxidation processes. The  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxide catalysts are fabricated by the combustion method using gel-created TA matter for CO conversion is effective.

#### 4. Conclusion

$\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxides have been prepared successfully by the combustion method using gel-created tartaric acid. The solid solution of  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxides formed a molar ratio of  $\text{Ce}^{+3}$  ions to  $\text{Fe}^{3+}$  ions of 1:1 at  $650^\circ\text{C}$  for 2 h with a uniform average crystal size of 70 nm and a surface area of  $20.22\text{ m}^2\text{g}^{-1}$ . In particular, the transformation of metal-ion states in  $\text{Fe}^{3+}/\text{Fe}^{2+}$  and  $\text{Ce}^{4+}/\text{Ce}^{3+}$  pairs through the redox processes have formed a closed loop

of Fe-ion oxidation states:  $\text{Fe}_2\text{O}_3 \rightarrow \text{Fe}_3\text{O}_4 \rightarrow \text{FeO} \rightarrow \text{Fe}_2\text{O}_3$ , and maintains the catalytic properties of the  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxides. The participation of Fe-metal ions in  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxide solution enhanced the density of  $V_{\text{O}}$  vacancies and promoted the catalytic reactions of CO conversion. The choice of  $\text{Ce}^{3+}$  to  $\text{Fe}^{3+}$  molar ratio of 1:1 has halved the needed Ce content. The complete conversion of CO into  $\text{CO}_2$  has taken place at a low temperature of  $270^\circ\text{C}$  under deficient  $\text{O}_2$  conditions. The studied results can open a prospect of using  $\text{CeO}_2\text{-Fe}_2\text{O}_3$  mixed oxide catalysts for simple CO emission incinerators.

#### Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

#### Conflicts of Interest

The authors declare that they have no conflicts of interest.

#### Acknowledgments

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