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Catalytic oxidation of CO over $Cu_xCe_{1-x}O_{2-x}$/SBA-15/FeCrAl monolithic catalysts

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ABSTRACT

A series of $Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl with the $Cu_{0.5}Ce_{0.5}O_{1.5}$ contents from 10 wt% to 60 wt% and 50 wt% $Cu_xCe_{1-x}O_{2-x}$/SBA-15/FeCrAl ($x = 0–1$) catalysts were prepared. The structure of the catalysts was characterized using X-ray powder diffraction (XRD), X-ray photoelectron spectroscopy (XPS) and temperature-programmed reduction (TPR). The catalytic activity of the catalysts for the oxidation of CO was evaluated. The results indicated that for the $Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl catalysts CuO was observed when $Cu_{0.5}Ce_{0.5}O_{1.5}$ loading is more than 40 wt%. For the 50%$Cu_xCe_{1-x}O_{2-x}$/SBA-15/FeCrAl catalysts, when x is in the range of 0–0.9, CeO_2 or Cu-Ce oxides were observed. When x is more than 0.5, CuO was observed. There were the strong interactions among CuO, CeO_2 and the FeCrAl support, and they affected the catalytic activity of the catalysts. In all catalysts, the 50%$Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl catalyst exhibited the best catalytic activity. CO can be completely oxidized at 160 °C.

1. Introduction

Recently, the catalytic oxidation of CO is considered as immense importance due to its applications in pollution control devices for vehicle exhaust, indoor air cleaning, fuel cells, and carbon dioxide lasers [1,2]. Precious metal catalysts, such as Pt, Pd, Ru, Rh and Au, have been widely studied for CO catalytic oxidation and showed high catalytic activities [3,4]. However, the high cost and the limited availability of the precious metals has encouraged researchers to look for alternative catalysts. In particular, CuO-CeO_2 composite catalysts have been reported as highly active for CO oxidation. A strong interaction between copper and ceria has been reported to be responsible for the high activity of these CuO-CeO_2 catalysts [5].

The newly achieved SBA-15 has high surface area, uniform pore structure, and narrow pore size distribution [6]. These unique features offer new possibilities for obtaining highly dispersed metal catalysts. Therefore it has attracted much attention as a catalyst support. On the other hand, the conventional fixed-bed reactors loaded with catalyst pellets have high pressure drops and high gradient of temperature thus depressed the activity of the catalysts. Nowadays, monolithic catalysts, especially using the FeCrAl alloy foils as the catalyst supports, have increasingly drawn the attention of researchers. The monolithic catalysts have distinct advantages: the much lower pressure drops, the lower capacity of mass and heat transfer, the higher resistance to thermal shocks [7]. Especially, for the reactions with high space velocity, such as CO catalytic oxidation reaction, the metallic monolithic catalysts have a promising application.

In this work, a series of $Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl with the $Cu_{0.5}Ce_{0.5}O_{1.5}$ contents from 10 wt% to 60 wt% and 50%$Cu_xCe_{1-x}O_{2-x}$/SBA-15/FeCrAl ($x = 0–1$) metal monolithic catalysts were prepared. The structure of catalysts was characterized by XRD, XPS and TPR. The catalytic activity of catalysts for CO oxidation was evaluated.

2. Experimental

2.1. Catalyst preparation

The metallic monolithic catalysts were prepared using the FeCrAl alloy foils (OC404, Sandvik Steel, Sweden) as supports. The alloy foils were rolled into several cylinders in different diameters and 50 mm in length. The supports were pretreated successively in basic, acidic and ethanol solution, then thoroughly rinsed in deionized water, and finally the pretreated supports were calcined at 950 °C for 15 h in air. In order to improve the adhesion between the washcoat layers and the heat-treated metallic supports, a boehmite primer sol was used as first washcoat layer, then dried at room temperature in air and thereafter at 120 °C for 3 h, and calcined at 500 °C for 4 h, and the monolithic support (Al_2O_3/FeCrAl) was formed. SBA-15 was synthesized according to the method described in the literature [8]. The $Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15 and $Cu_xCe_{1-x}O_{2-x}$/SBA-15 samples were prepared by co-impregnation of pure SBA-15 with an aqueous solution of copper nitrate and cerium nitrate. Then, the slurry of the $Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15

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Table 1
Compositions and particle size of the catalysts.

Catalyst	Composition	D_{CuO}[a] (nm)	D_{CeO_2} or D_{CuCeO_x}[a] (nm)
1#	10%$Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl	n.d.[b]	9.9
2#	20%$Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl	n.d.[b]	9.7
3#	30%$Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl	n.d.[b]	9.7
4#	40%$Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl	34.1	9.8
5#	50%$Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl	28.6	9.8
6#	60%$Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl	35.0	9.6
7#	50%CuO/SBA-15/FeCrAl	25.4	–
8#	50%$Cu_{0.9}Ce_{0.1}O_{1.1}$/SBA-15/FeCrAl	22.9	10.7
9#	50%$Cu_{0.7}Ce_{0.3}O_{1.3}$/SBA-15/FeCrAl	28.1	9.7
10#	50%$Cu_{0.3}Ce_{0.7}O_{1.7}$/SBA-15/FeCrAl	n.d.[b]	9.8
11#	50%$Cu_{0.1}Ce_{0.9}O_{1.9}$/SBA-15/FeCrAl	n.d.[b]	10.5
12#	50%CeO_2/SBA-15/FeCrAl	–	10.6

[a] Calculated from XRD measurements.
[b] Not detected.

and $Cu_xCe_{1-x}O_{2-x}$/SBA-15 was coated on the Al_2O_3/FeCrAl monolithic supports, dried at room temperature in air and thereafter at 120 °C for 3 h, and then calcined at 500 °C for 4 h. The coating procedure should be repeated to achieve the desired coating amount. Finally, the $Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl with the $Cu_{0.5}Ce_{0.5}O_{1.5}$ content from 10 wt% to 60 wt% and $Cu_xCe_{1-x}O_{2-x}$/SBA-15/FeCrAl (x = 0–1) catalysts with the $Cu_xCe_{1-x}O_{2-x}$ content of 50 wt% were obtained. The compositions of the catalysts were listed in Table 1.

2.2. Catalytic activity measurements

CO catalytic oxidation experiments were performed in a conventional quartz flow reactor (i.d., 8 mm; length, 300 mm) at atmospheric pressure. The mixture gas was 1 vol.% CO in air, with a gas hourly space velocity of 36,000 ml/g h. The reaction was stabilized for 30 min at the required temperature, and the outlet products were measured with on-line gas chromatography (Beijing East & West Electronics Institute, GC-4000A). The catalyst temperature was controlled with a K-type thermocouple placed in the vicinity of the catalyst bed. In every case, carbon dioxide and water were the only reaction products detected along the whole experiments.

2.3. Catalysts characterization

The phase structure of the samples was characterized by XRD using a Rigaku D/Max 2500 VB2 + /PC diffractometer with a Cu Kα radiation operating at 200 mA and 40 kV. XPS experiments were carried out on an ESCALAB250 instrument (Thermo Electron Co.) using Al Kα as the exciting radiation at the constant pass energy of 50 eV. Binding energies were calibrated using the carbon present as a contaminant (C 1s = 285.0 eV). The surface atomic compositions of all samples were calculated from photoelectron peak areas for each element after correction for instrument parameters. Temperature-programmed reduction (TPR) experiments were performed using a Thermo Electron Corporation TPD/R/O 1100 series Catalytic Surfaces Analyzer equipped with a TC detector. Samples were preheated with 10 vol.% O_2/He mixture heating 20 °C/min up to 500 °C, then cooling down to room temperature in flowing N_2, and thereafter reduced with 5 vol.% H_2/N_2 mixture heating 10 °C/min up to 1000 °C. Water produced by the sample reduction was condensed in a cold trap before reaching the detectors. Only H_2 was detected in the outlet gas confirming the effectiveness of the cold trap.

3. Results and discussion

3.1. Catalytic activity

The catalytic activity over $Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl catalysts is presented in Fig. 1(A). It can be observed that the loading of $Cu_{0.5}Ce_{0.5}O_{1.5}$ had a significant influence on the catalytic activity of the catalysts. The catalytic activity was significantly enhanced with the increase in $Cu_{0.5}Ce_{0.5}O_{1.5}$ loading from 10 wt% to 40 wt%. As the loading of $Cu_{0.5}Ce_{0.5}O_{1.5}$ increased from 40 wt% to 50 wt%, there was only slight increase in catalytic activity. The 50%$Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl catalysts exhibited the highest catalytic activity with CO conversion of 10% at 66 °C. The CO can be completely oxidized at 160 °C.

Fig. 1(B) shows catalytic performance for CO oxidation over the 50%$Cu_xCe_{1-x}O_{2-x}$/SBA-15/FeCrAl catalysts. As observed, the catalytic activity of CO oxidation over both 50%CeO_2/SBA-15/FeCrAl (12#) and 50%CuO/SBA-15/FeCrAl (7#) were quite low. For catalyst 12#, T_{10} and T_{90} were 360 °C and 537 °C, respectively; while for catalyst 7#, T_{10} and T_{90} were 214 °C and 319 °C, respectively. The activities of 50%$Cu_xCe_{1-x}O_{2-x}$/SBA-15/FeCrAl (x = 0.1–0.9) catalysts were much higher than that of catalyst 12# or 7#. The catalytic activities of the 50%$Cu_xCe_{1-x}O_{2-x}$/SBA-15 (x = 0.1–0.9) catalysts were closely related to the Cu/Ce molar ratios. When x = 0.3 and 0.5, the catalysts exhibited the similar catalytic activity. However, further increasing the Cu content ($x > 0.5$) resulted in the decrease of the catalytic activity. On the basis of the activity results, it indicated that the excessive copper would decrease the activity of catalysts.

3.2. XRD

The XRD patterns of FeCrAl metal support and $Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl monolithic catalysts are presented in Fig. 2(A). For

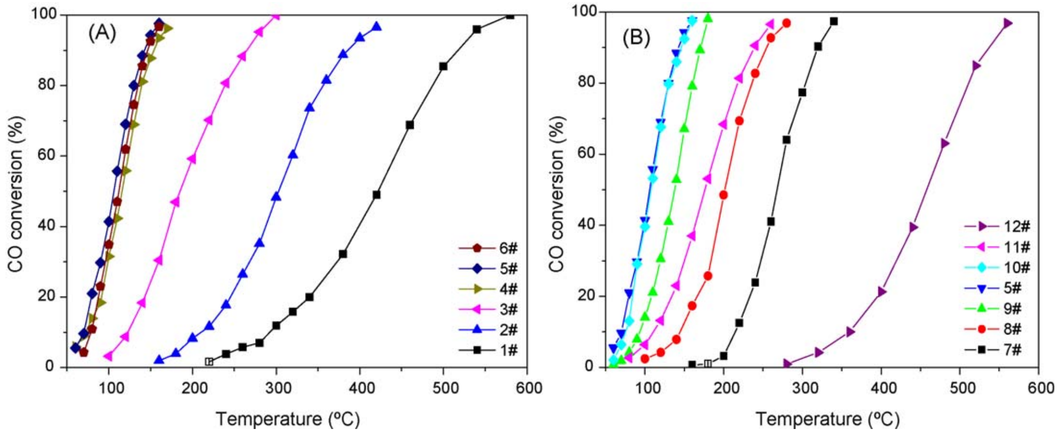

Fig. 1. CO catalytic oxidation over the monolithic catalysts: (A) $Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl catalysts; (B) 50%$Cu_xCe_{1-x}O_{2-x}$/SBA-15/FeCrAl catalysts.

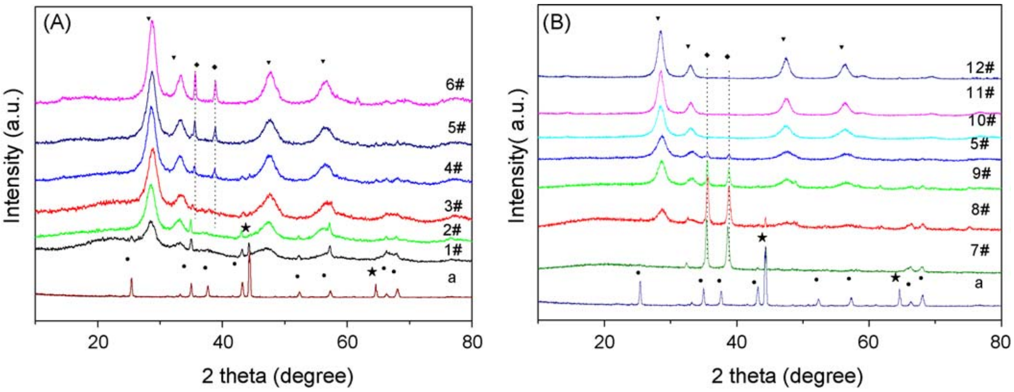

Fig. 2. XRD spectra of the samples: (A) FeCrAl foil pre-oxidized at 950 °C for 15 h and $Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl catalysts; (B) FeCrAl foil pre-oxidized at 950 °C for 15 h and 50%$Cu_xCe_{1-x}O_{2-x}$/SBA-15/FeCrAl catalysts (★) FeCr; (▼) CeO_2; (●) α-Al_2O_3; (◆) CuO.

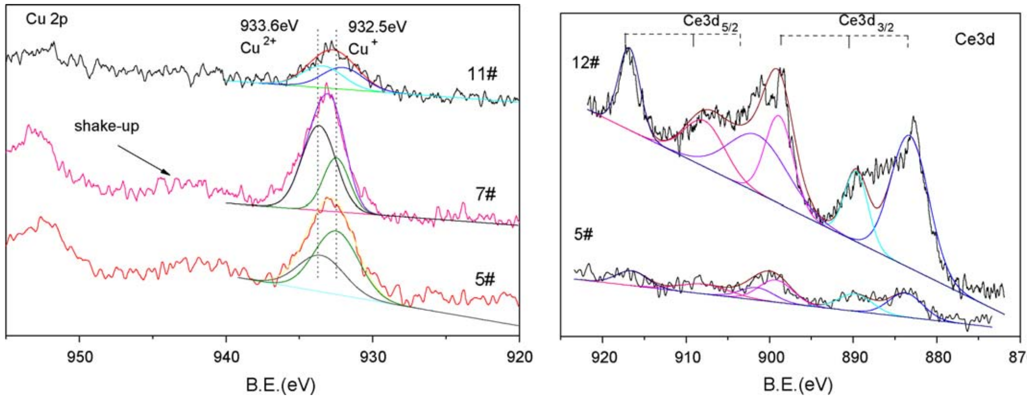

Fig. 3. XPS spectra of catalysts 5#, 7#, 11#, 12#.

FeCrAl metal support, after the heat treatment at 950 °C for 15 h, the characteristic peaks of FeCr (JCPDS 34-0396) and α-Al_2O_3 (JCPDS 88-0826) were observed. For all $Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl catalysts, the diffraction peaks of CeO_2 (JCPDS 43-1002) or Cu-Ce oxides were observed. The diffraction peaks of CuO at 35.5° and 38.7° [9] were only observed when the $Cu_{0.5}Ce_{0.5}O_{1.5}$ contents were more than 40 wt%. It may be due to the well dispersion of CuO on the catalysts [10–12]. Mean crystallites sizes were calculated from the line broadening of the most intense peak of a given crystalline phase using the Scherrer equation. These data are summarized in Table 1. Considering the metal particle sizes and the pore diameter of the support SBA-15, as given in the literature [8], it can be suggested that the majority of particles might be located outside the pores.

The XRD patterns of the 50%$Cu_xCe_{1-x}O_{2-x}$/SBA-15/FeCrAl monolithic catalysts are presented in Fig. 2(B). The diffraction peaks of CeO_2 can be seen when $x = 0$ (catalyst 12#). When x is in the range of 0.1–0.9, CeO_2 or Cu-Ce oxides were observed. When $x \geq 0.5$, the peaks of CuO were observed. From the results of the catalyst activity, 12# and 7# catalysts showed poor catalytic

Table 2
Binding energies (eV) of core electrons and surface atomic composition of samples.

Samples	Cu $2p_{3/2}$			Ce $3d_{5/2}$		Cu/(Cu + Ce)		Si 2p		O 1s	
	B.E. (eV)[a]		at. (%)	B.E. (eV)	at. (%)	Nominal	XPS	B.E. (eV)	at. (%)	B.E. (eV)	at. (%)
	Cu^+	Cu^{2+}									
1#	932.5(43.0)[b]	933.6(57.0)	0.77	882.3	0.16	0.5	0.83	99.5	17.20	531.3	81.87
2#	932.3(29.4)	933.6(70.6)	1.21	882.4	0.27	0.5	0.82	99.5	16.40	531.5	82.11
3#	932.4(35.8)	933.9(64.2)	1.94	883.0	0.31	0.5	0.86	99.0	14.71	531.4	83.04
4#	932.6(39.1)	933.8(60.9)	2.65	882.8	0.30	0.5	0.90	99.8	17.46	531.6	79.59
5#	932.5(62.5)	933.5(37.5)	2.62	883.8	0.46	0.5	0.85	99.7	13.21	531.7	83.70
6#	932.4(41.3)	933.4(58.7)	3.23	882.8	0.43	0.5	0.88	99.5	13.33	531.0	83.01
7#	932.5(32.7)	933.7(67.3)	2.71	–	–	1	1	99.8	16.96	531.4	80.33
8#	932.6(43.0)	933.7(57.0)	2.91	882.7	0.23	0.9	0.92	99.3	18.40	531.2	78.45
9#	932.4(43.4)	933.6(56.6)	3.91	882.5	0.22	0.7	0.95	99.3	12.54	531.2	83.33
10#	932.7(56.7)	934.0(43.3)	2.35	882.4	0.68	0.3	0.77	99.2	14.04	531.0	82.93
11#	932.0(49.8)	933.4(50.2)	1.15	882.1	1.60	0.1	0.42	99.2	19.69	531.3	77.56
12#	–	–	–	882.8	1.93	0	0	99.3	12.62	531.6	85.45

[a] The binding energy (BE) values were corrected using the C 1s peak at 285.0 eV.
[b] The value in the brackets refers to the relative content of the components of the Cu $2p_{3/2}$ spectra.

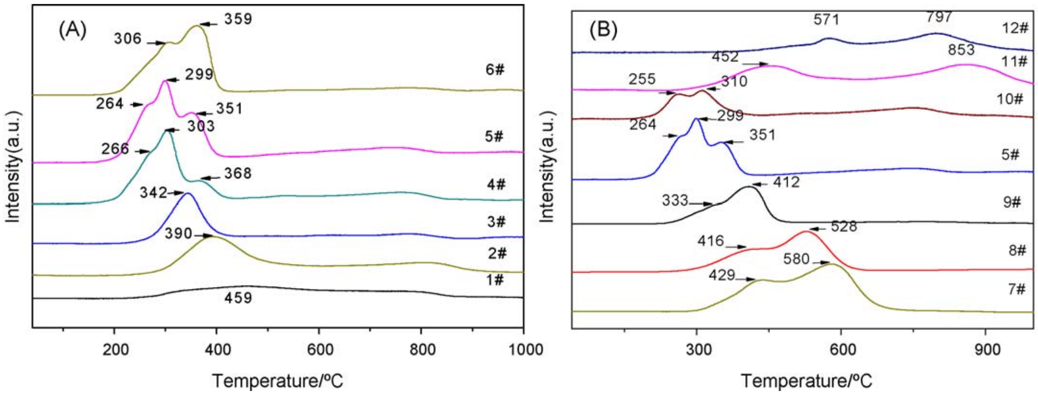

Fig. 4. H_2-TPR profiles of the samples: (A) $Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl catalysts; (B) 50%$Cu_xCe_{1-x}O_{2-x}$/SBA-15/FeCrAl catalysts.

activity. It indicated that the catalysts only containing CeO_2 or CuO had lower catalytic activity.

3.3. XPS

XPS spectra of Cu 2p and Ce 3d of 5#, 7#, 11# and 12# catalysts are shown in Fig. 3. The XPS data are listed in Table 2. The presence of a higher Cu $2p_{3/2}$ binding energy (BE) and the pronounced shake-up peak (940.0–945.0 eV) are characteristics of CuO, while a lower Cu $2p_{3/2}$ binding energy (932.2–933.1 eV) and the absence of the shake-up peak are characteristics of Cu_2O [13,14]. It suggested that there were Cu^+ and Cu^{2+} species on the surface of catalysts. Zhu et al. [15] proposed that the reduction of Cu^{2+} to Cu^+ resulted from the properties of ceria with the oxygen storage and release capacity. Liu [16] reported the Cu^+ species result from the strong interaction of the copper clusters with cerium oxide.

There were six peaks (883.2, 889.6, 898.9, 901.4, 907.8, and 916.8 eV) of Ce 3d in the catalysts, which can be assigned to Ce^{4+} species by comparison with data reported in the literature [14]. The peak of O 1s was at about 531.4 eV [17]. From Table 2, it can be seen that the copper appears to enrich on the surface of the catalysts, since the Cu/(Cu + Ce) atomic ratio is considerably large than the nominal composition of the samples. The results were similar with the literature [18,19].

3.4. H_2-TPR

Fig. 4(A) shows the H_2-TPR profiles of $Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl monolithic catalysts. It can be seen that there is only one reduction peak when the $Cu_{0.5}Ce_{0.5}O_{1.5}$ contents were lower than 40%, and the peak gradually moved to lower temperature with the increase in the $Cu_{0.5}Ce_{0.5}O_{1.5}$ contents. When the $Cu_{0.5}Ce_{0.5}O_{1.5}$ content became higher, there appeared two or three reduction peaks.

H_2-TPR profiles of 50%$Cu_xCe_{1-x}O_{2-x}$/SBA-15/FeCrAl catalysts are shown in Fig. 4(B). It is obvious that the catalysts with different Ce and Cu atomic ratios have different profiles. When $x = 0$ (catalyst 12#), the catalyst had two reduction peaks. The peak at 571 °C was attributed to the reduction of the most easily reducible surface-capping oxygen of ceria, while the other peak at about 797 °C was due to the reduction of CeO_2. Compared to the reduction peaks of pure CeO_2 [20], the peaks of the CeO_2 in the catalyst 12# had a little shift, which substantiated the interaction between the CeO_2 and the support. It is well known that the reduction of pure CuO is characterized by a single peak at about 380 °C. Our work shows that there are two reduction peaks at 424 °C and 586 °C for catalyst 7#, which also substantiate the interaction between the CuO and the support. The two peaks may correspond to the reduction of Cu^{2+} to Cu^+ and Cu^+ to Cu^0, respectively. It is obvious that the reduction temperature of copper species in the 50%$Cu_xCe_{1-x}O_{2-x}$/SBA-15/FeCrAl ($x = 0.1$–0.9) catalysts is much lower than that of CuO. The presence of two or three reduction peaks in the catalysts indicated more than one copper oxide species in the catalysts.

Many researchers observed two reduction peaks in the CuO-CeO_2 catalysts. Gómez-Cortés et al. [21] reported two reduction peaks which were assigned to highly dispersed CuO clusters and somewhat larger CuO particles. Águila et al. [22] also observed two reduction peaks for CuO-CeO_2/SiO_2 catalysts. These peaks can be attributed to the reduction of small CuO clusters with and without interaction with CeO_2 respectively. In addition, Zheng et al. [10] observed there were three reduction peaks: the first peak was attributed to reduction of non-crystalline CuO strongly interacting with CeO_2; the second one was ascribed to the reduction of larger CuO particles weakly associated with CeO_2; and the third one represented the reduction of bulk copper oxide (crystalline forms) which associated with CeO_2 to some extend. Mariño et al. [23] also observed three reduction peaks. However, the last peak was assigned to the reduction of some form of bulk CuO less associated with ceria or to some extent of surface reduction of ceria by hydrogen split from the reduced copper moieties over its surface.

Based on the above analysis, it can be suggested that the only one reduction peak at lower $Cu_{0.5}Ce_{0.5}O_{1.5}$ content may be attributed to the highly dispersed copper oxide strongly interacting with the ceria. The two reduction peaks probably represented the highly dispersed copper oxide strongly interacting with the ceria and the reduction of bulk CuO, respectively. For the catalysts 4# and 5#, we can ascribe the first reduction peak to the highly dispersed copper oxide strongly interacting with the ceria surface, the second peak to the reduction of larger CuO particles weakly associated with CeO_2 and the third peak to the reduction of bulk CuO.

4. Conclusions

A series of $Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl and 50 wt%$Cu_xCe_{1-x}O_{2-x}$/SBA-15/FeCrAl ($x = 0$–1) catalysts were prepared using FeCrAl alloy as support. Their catalytic performance for catalytic oxidation of CO was tested. The catalytic activities of the catalysts were strongly influenced by the $Cu_{0.5}Ce_{0.5}O_{1.5}$ contents and the Cu/Ce molar ratios. The 50%$Cu_{0.5}Ce_{0.5}O_{1.5}$/SBA-15/FeCrAl exhibited the highest catalytic activity. CO can be completely oxidized at 160 °C with a GHSV of 36,000 ml/g h. Using FeCrAl alloy foils as supports benefits the dispersion of CuO. CuO phase was observed with higher Cu contents. The copper can be enriched on the catalysts surface in all the catalysts. The strong interaction among the Cu, Ce and the FeCrAl support affected the redox performance and catalytic activity of the catalysts.

Acknowledgements

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