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Supported $CuO/\gamma\text{-}Al_2O_3$ as heterogeneous catalyst for synthesis of diaryl ether under ligand-free conditions

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ABSTRACT

$CuO/\gamma\text{-}Al_2O_3$ was employed as catalyst in the coupling reaction of iodobenzene with *m*-cresol to give 3-methyldiphenyl ether under ligand-free conditions. It performed more efficiently than other supported CuO catalysts (CuO/SBA-15, CuO/ZrO_2, CuO/CeO_2, CuO/La_2O_3). Reaction conditions were investigated and the yield reached 76% under the optimized terms. As a heterogeneous catalyst, $CuO/\gamma\text{-}Al_2O_3$ could be reused for three cycles with moderate to good yields. According to the results of XRD, TPR and XPS, the catalytic activities of supported CuO catalysts were due to the highly dispersed Cu(II) species and independent of the electronic state of these copper species.

1. Introduction

Diaryl ethers are important structures in numerous biologically active compounds, natural products and polymers [1]. The traditional synthetic method for the synthesis of diaryl ethers was classic Ullmann coupling reaction by using aryl halides and phenols as substrates and copper as catalyst. However, this application was limited by harsh reaction conditions such as high temperature (>200 °C) and stoichiometric amount of copper reagent which consequently led to the problem of waste disposal [2–5].

To overcome these limitations, much attention was paid to the improvement of Ullmann-type coupling reaction and significant progress has been achieved. In recent years, a large number of highly efficient ligands were successfully developed for copper-catalyzed arylations, e.g., 1-naphthoic acid [6], *N*,*N*-dimethylglycine [7], 8-hydroxyquinoline [8], pyrrolidine-2-phosphonic acid phenyl monoester [9] and 2,2,6,6-tetramethylheptane-3,5-dione [10,11]. These elaborate ligands indeed promoted the cross-coupling reaction, but increased the cost. Meanwhile, the procedures proceeded homogeneously were problematic in terms of recovery/recycling of catalyst. From a sustainable point of view, developing of inexpensive, ligand-free and recyclable heterogeneous catalysts is highly desired.

Recently, Cu, CuO and CuI nanoparticles as well as Cu_2O nanocube were explored for the coupling of aryl iodides and aryl bromides with phenols [12–16]. The reactions took place without ligand and the catalysts were reusable. However, the production and the separation of nanomaterials in industrial-scale process still remain problem.

Due to easy separation and efficient recyclability, immobilized catalysts received much attention in industrial applications. Recently, Miao and Wang [17] developed a catalytic system for the preparation of diaryl ethers by using organic–inorganic (silica) hybrid materials-supported copper as catalyst. The drawback of this procedure is the intricacy of the synthesis of silica-supported copper. Lipshutz et al. [18] reported a system based on the use of charcoal as support. To construct diaryl ethers with Cu/C catalyst, they explored the reaction of aryl bromides and phenols in aid of microwave which greatly reduced the reaction time. Nonetheless, ligand was required to achieve high yield. Taillefer et al. [19,20] devised sol–gel immobilized copper catalysts which were applied for the C—O coupling of aryl iodides and aryl bromides with phenols. However, the preparation of this sol–gel immobilized copper catalyst is complicated.

In order to develop an efficient and easy handling catalyst for Ullmann coupling reaction, we investigated supported CuO catalysts for the synthesis of diaryl ether under ligand-free conditions. The support effect and reaction conditions on catalytic performance of supported catalysts were explored. Characterizations of XRD, TPR and XPS were carried out to discuss the active species of supported CuO catalyst for Ullmann coupling reaction.

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2. Experimental

2.1. Catalyst preparation

ZrO_2, La_2O_3, and γ-Al_2O_3 were purchased from Sinopharm Chemical Reagent Co. Ltd. and used without further treatment. SBA-15 was purchased from Shanghai Boyle Chemical Co. Ltd. and used without further treatment. The support of CeO_2 was obtained by calcination of $Ce(NO_3)_3 \cdot 6H_2O$ at 550 °C for 6 h. Preparation of supported CuO catalysts: 1 g support was impregnated with $Cu(NO_3)_2 \cdot 3H_2O$ aqueous solution (0.304 g $Cu(NO_3)_2 \cdot 3H_2O$ in a certain volume of water). The sample was placed at ambient temperature for 12 h, heated in static air at 80 °C for 6 h and calcined at 650 °C for 6 h. CuO catalyst was obtained by calcination of $Cu(NO_3)_2 \cdot 3H_2O$ at 650 °C for 6 h. The copper content in supported CuO catalysts (CuO/SBA-15, CuO/ZrO_2, CuO/CeO_2, CuO/La_2O_3, CuO/γ-Al_2O_3) was calculated as 8 wt%.

2.2. Catalyst characterizations

The Brunauer–Emmett–Teller (BET) specific surface area, pore volume and average pore size were determined by physical adsorption of N_2 at −196 °C in an automatic volumetric system using Quantachrome NOVA instrument.

X-ray diffraction (XRD) spectra of catalysts were recorded in a RINT2000 vertical goniometer, using Cu-Kα radiation. Spectra were registered between 20° and 80° (2θ) with an increment of 0.02° and a scan-speed of 6°/min.

Temperature programmed reduction experiments were carried out with H_2 (H_2-TPR) in a Micromeritics device. 50 mg of catalyst was heated at 10 °C/min from 30 °C to 600 °C under a 5% H_2/N_2 flow (45 ml/min), and the H_2 consumption was monitored with a thermal conductivity detector (TCD).

X-ray photoelectron spectrometer (XPS) characterization was carried out in a Thermo ESCALAB 250 electron spectrometer using Al-Kα (1486.6 eV) radiation source. To obtain the XPS spectra, the pressure of the analysis chamber was maintained at 5×10^{-10} mbar. The binding energy (B.E.) scale was adjusted by setting the C 1s transition at 285.0 eV.

2.3. Catalytic activity measurement

A mixture of iodobenzene (3 mmol), *m*-cresol (2 mmol), catalyst (0.032 g, 0.04 mmol Cu), K_3PO_4 (4 mmol) and dimethyl sulfoxide (DMSO) (4 ml) in a sealed tube was stirred at 150 °C under N_2 atmosphere for 10 h. Subsequently, the reaction was allowed to cool to room temperature and the catalyst were filtered off and washed with ethyl acetate (EtOAc). The excess EtOAc was evaporated followed by adding tridecane (0.6 mmol) as internal standard. All samples were analyzed by gas chromatography with flame ionization detector (FID).

2.4. Recycle of catalyst

After the completion of coupling reaction of iodobenzene with *m*-cresol, the reaction mixture was filtered. The residue was washed with EtOAc, ethanol and water. The solid was dried at 80 °C for 6 h and calcined at 650 °C for 2 h. The obtained catalyst was used for the next reaction of iodobenzene with *m*-cresol.

3. Results and discussion

3.1. Catalyst activity

CuO catalysts with different supports were tested with the coupling reaction of iodobenzene and *m*-cresol to give 3-methyldiphenyl ether and the results are summarized in Table 1. Under heterogeneous conditions, steric hindrance of methyl on *m*-cresol was thought to be a disadvantage for Ullmann coupling reaction [13]. In our experiment, no product was detected in absence of copper catalyst (Table 1, Entry 1). With pure CuO as catalyst, reaction afforded low yield of 28% (Table 1, Entry 2). When SBA-15 and ZrO_2 were employed as supports, lower yields of 8% and 19% were obtained and no improvement was achieved compared with pure CuO (Table 1, Entries 3 and 4). The reaction yields increased dramatically when CeO_2, La_2O_3 and γ-Al_2O_3 were used as supports (Table 1, Entries 5–7). Amongst the catalysts screened, CuO/γ-Al_2O_3 gave the highest yield of 76%.

The structural characteristics (BET surface area, pore volume and average pore size) of supported CuO catalysts were measured (listed in Table 1). The results showed that there was no correlation between these properties and catalytic activity.

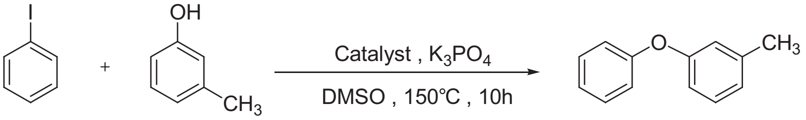
To optimize the reaction conditions, CuO/γ-Al_2O_3 was used as catalyst and the effect of base, solvent, substrates mole ratio and reaction time were tested (see Table 2). Obviously, base showed a significant influence on the coupling reaction. The use of carbonates (K_2CO_3, Cs_2CO_3, Na_2CO_3) did not lead to high yield of 3-methyldiphenyl ether (Table 2, Entries 1–3). Cs_2CO_3 as a strong base, though it was remarkable in homogeneous reaction [6,7,9–11], only afforded 19% yield in heterogeneous reaction. K_3PO_4, on the contrary, exhibited its effectiveness to give 76% yield of product in combination with CuO/γ-Al_2O_3 heterogeneous catalyst (Table 2, Entry 4), which was consistent with previous reports [21–24]. In order to investigate the effect of solvent on the coupling reaction, DMSO and *N*,*N*-dimethyl formamide (DMF) were taken into account (Table 2, Entries 4 and 5). It can be seen that a lower yield (15%) was obtained in DMF than in DMSO (76%). This might be due to the unstability of DMF under high temperature and strong basic circumstance. As to the mole ratio of iodobenzene to *m*-cresol, 61% yield was obtained with stoichiometric ratio (Table 2, Entry 8). Increasing *m*-cresol to the mole ratio of iodobenzene to *m*-cresol of 1:1.5, improvement of yield presented only 4% (Table 2, Entry 9). The further increasing *m*-cresol to the mole ratio of iodobenzene to *m*-cresol of 1:2 resulted in the slight decrease of the yield (Table 2, Entry 10). However, yield reached 76% when iodobenzene increased to the mole ratio of 1.5:1 and remained unchanged with the mole ratio of 2:1 (Table 2, Entries 4 and 7). But further changing of the ratio to 3:1, a lower yield of 72% was obtained (Table 2, Entry 6).

It was generally reported that it would take a long time to accomplish Ullmann coupling reaction in homogeneous conditions [7,11,21–24]. But, in our experiments, yield reached 65% within 4 h and 74% at 8 h, which is much shorter than those in homogeneous conditions (Table 2, Entries 11 and 12). After that, 76% yield was obtained at 10 h and no increase could be observed at extended time (Table 2, Entries 4 and 13).

3.2. Structure and surface characterization

XRD patterns of the supported CuO catalysts are shown in Fig. 1. The diffraction peaks corresponding to copper-containing phases could not be observed for CuO/γ-Al_2O_3 and CuO/La_2O_3 catalysts, while the intense reflections corresponding to CuO at 35.5° and 38.8° were clearly presented for the catalysts of CuO/ZrO_2 and CuO/SBA-15. For CuO/CeO_2 catalyst, the CuO diffraction peaks were relatively weak. The absence of CuO diffraction peaks on CuO/γ-Al_2O_3 and CuO/La_2O_3 catalysts indicated the formation of highly dispersed copper species on the supports surfaces, which were not detectable by XRD. The diffraction peaks of CuO on CuO/ZrO_2 and CuO/SBA-15 manifested the formation of bulk CuO on these catalysts surfaces. As to CuO/CeO_2 catalyst, the relatively weak diffraction peaks could attribute to

Table 1
Catalyst screening for Ullmann coupling reaction of iodobenzene with *m*-cresol.[a] .



Entry	Catalysts	BET surface area ($m^2\,g^{-1}$)	Pore volume ($cm^3\,g^{-1}$)	Pore size (nm)	Yield (%)[b]
1	–	–	–	–	0
2	CuO	–	–	–	28
3	CuO/SBA-15	278.3	0.359	3.5	8
4	CuO/ZrO_2	3.3	0.019	2.5	19
5	CuO/CeO_2	45.5	0.145	7.8	64
6	CuO/La_2O_3	15.9	0.048	12.0	71
7	**$CuO/\gamma\text{-}Al_2O_3$**	**133.7**	**0.424**	**7.9**	**76**

Entries in bold are the best item (highest yield, optimized reaction conditions and high recyclability).

[a] Reaction conditions: PhI (3 mmol), *m*-cresol (2 mmol), K_3PO_4 (4 mmol), catalysts (0.032 g, 0.04 mmol Cu), in DMSO (4 ml) at 150 °C under N_2 for 10 h.

[b] GC yield determined with tridecane as internal standard.

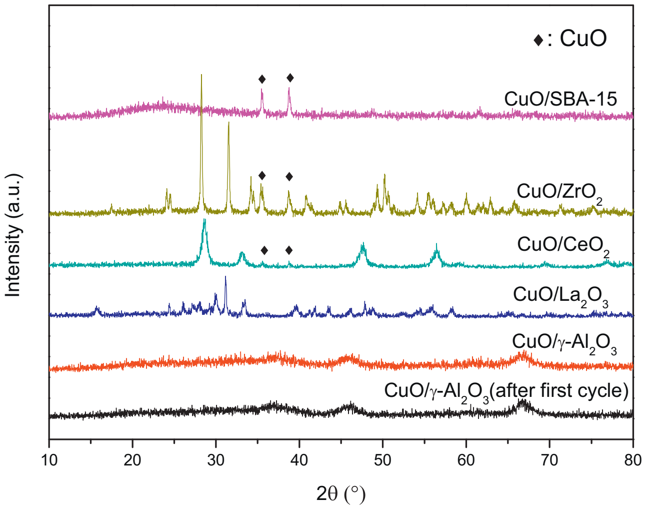


Fig. 1. XRD patterns of supported CuO catalysts.

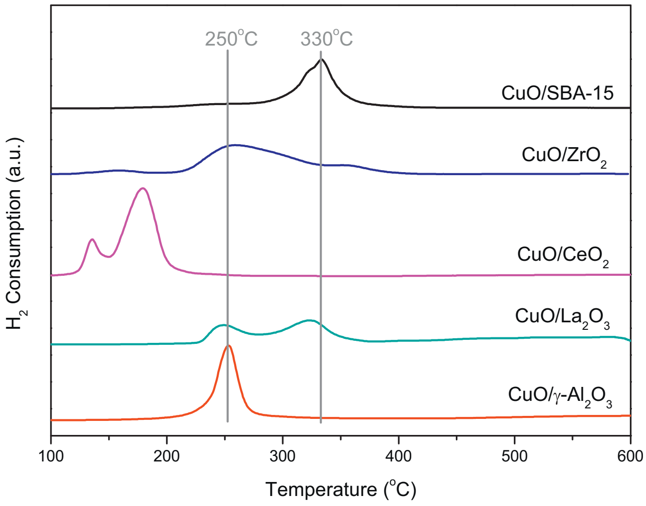


Fig. 2. H_2-TPR profiles of supported CuO catalysts.

small CuO particles on CeO_2 surface. Consequently, the dispersion of Cu species on different supports decreased in order of $CuO/\gamma\text{-}Al_2O_3 \approx CuO/La_2O_3 > CuO/CeO_2 > CuO/ZrO_2 \approx$ CuO/SBA-15, which agreed with the change of catalyst activities. Therefore, it can be assumed that highly dispersed copper species are more active than bulk ones for Ullmann coupling reaction.

TPR experiments were conducted to study the reducibility of CuO catalysts and the profiles are presented in Fig. 2. The profile of CuO/SBA-15 catalyst showed one peak centered at 330 °C, which was attributed to the reduction of bulk CuO [25]. Compared with CuO/SBA-15 catalyst, the reduction of CuO/ZrO_2 appeared at lower temperature (260 °C), which was definitely not caused by the dispersion according to the result of XRD. The reason for enhanced reducibility of Cu^{2+} species might be caused by the redox property of ZrO_2 and the metal-support interaction between CuO and ZrO_2 [26]. On the contrary, the lower reduction peak of $CuO/\gamma\text{-}Al_2O_3$ at 250 °C should be caused by the dispersion of copper

Table 2
Effect of reaction conditions on Ullmann coupling reaction of iodobenzene with *m*-cresol using $CuO/\gamma\text{-}Al_2O_3$ as catalyst.

Entry	Base	Solvent	Mole ratio of iodobenzene to *m*-cresol	Reaction time (h)	Yield (%)[a]
1	K_2CO_3	DMSO	1.5:1	10	35
2	Cs_2CO_3	DMSO	1.5:1	10	19
3	Na_2CO_3	DMSO	1.5:1	10	11
4	**K_3PO_4**	**DMSO**	**1.5:1**	**10**	**76**
5	K_3PO_4	DMF	1.5:1	10	15
6	K_3PO_4	DMSO	3:1	10	72
7	K_3PO_4	DMSO	2:1	10	76
8	K_3PO_4	DMSO	1:1	10	61
9	K_3PO_4	DMSO	1:1.5	10	65
10	K_3PO_4	DMSO	1:2	10	62
11	K_3PO_4	DMSO	1.5:1	4	65
12	K_3PO_4	DMSO	1.5:1	8	74
13	K_3PO_4	DMSO	1.5:1	24	76

Entries in bold are the best item (highest yield, optimized reaction conditions and high recyclability).

[a] GC yield determined with tridecane as internal standard.

Table 3
Binding energy and relative ratios of surface Cu species derived from XPS results.

Entry	Samples	B.E. (eV)			Ratio (%) of Cu^0/Cu^{1+}:CuO:Cu^{2+}
		Cu^0/Cu^{1+}	CuO	Cu^{2+}	
1	CuO	–	933.9	–	0:100:0
2	CuO/SBA-15	–	933.8	936.1	0:69:31
3	CuO/ZrO_2	932.5	933.7	934.9	2:23:75
4	CuO/CeO_2	932.5	933.7	935.1	9:30:61
5	CuO/La_2O_3	932.2	933.7	–	21:79:0
6	$CuO/\gamma\text{-}Al_2O_3$	932.5	933.7	935.2	2:20:78
7	$CuO/\gamma\text{-}Al_2O_3$ (after first cycle)	932.5	933.7	935.0	4:32:64

species in accordance with XRD result, since $\gamma\text{-}Al_2O_3$ does not show redox property. The profile of CuO/La_2O_3 catalyst presented two reduction peaks (250 °C and 325 °C), indicating that CuO species were partially aggregated. However, the aggregated CuO was not big enough to exhibit CuO reflection peaks with XRD. Similar to CuO/La_2O_3, CuO on CuO/CeO_2 was partially aggregated with bigger crystal size that exhibited weak CuO diffraction peaks with XRD. The lower reduction peaks (136 °C and 179 °C) were due to the strong metal-support interaction and redox property of CeO_2 via electron transfer between the oxides [27]. Due to the different properties of supports, the copper species showed different redox capabilities. Obviously, there is no direct relationship between the redox property and catalytic performance for supported CuO catalysts.

Commonly, Cu^+ was believed to be active in homogeneous synthesis of diaryl ethers. However, Cu^0, Cu^+ and Cu^{2+} showed capabilities to catalyze Ullmann coupling reaction in heterogeneity without specific explanation. Here, XPS was performed to explore the electronic state of catalytic centers of supported CuO catalysts.

Fig. 3 shows the Cu 2p3/2 spectra of supported CuO catalysts. The binding energy and relative ratios of surface Cu species derived from XPS results are listed in Table 3. As reported [28–30], the peak at 935 eV indicated the appearance of Cu^{2+} species strongly interacting with support, while the peak at 933.7 eV was assigned to CuO species and the one at 932.5 eV was related to low valence copper species (Cu^+/Cu^0). For most supported CuO catalysts, Cu^+/Cu^0 were the minor species (lower than 10%), suggesting that high valence copper species played a crucial role in Ullmann coupling reaction. As to catalysts with low catalytic activity, CuO/ZrO_2 contained Cu^{2+} as the major species, while CuO dominated on CuO/SBA-15. On the other hand, CuO/CeO_2, CuO/La_2O_3 and $CuO/\gamma\text{-}Al_2O_3$ presented relatively high catalytic activity. For CuO/CeO_2 and $CuO/\gamma\text{-}Al_2O_3$, Cu^{2+} was presented as the dominant copper species. Nonetheless, XPS of CuO/La_2O_3 showed that CuO was the main species on the catalyst. In combination with the catalytic activity results, it can be figured out that both Cu^{2+} strongly interacted with support and CuO could catalyze Ullmann coupling reaction under heterogeneous conditions, and electronic state of copper species was not the key to the catalytic performance of supported CuO catalysts.

3.3. *Recycle of catalyst*

As a heterogeneous catalyst, the recyclability of $CuO/\gamma\text{-}Al_2O_3$ catalyst in the coupling reaction of iodobenzene with *m*-cresol was examined and the results were listed in Table 4. The yield for the second cycle (61%) was lower than the first one (Table 4, Entry 3). For the third cycle, nearly no loss of activity was observed (Table 4, Entry 3). Compared with fresh $CuO/\gamma\text{-}Al_2O_3$ catalyst, XRD of used $CuO/\gamma\text{-}Al_2O_3$ did not exhibit CuO diffraction peaks yet (see Fig. 1). The XPS spectra indicated that $CuO/\gamma\text{-}Al_2O_3$ catalyst contained Cu^{2+} as the main copper species before and after the reaction (see Fig. 3c). The XRD and XPS results suggested that the structure and

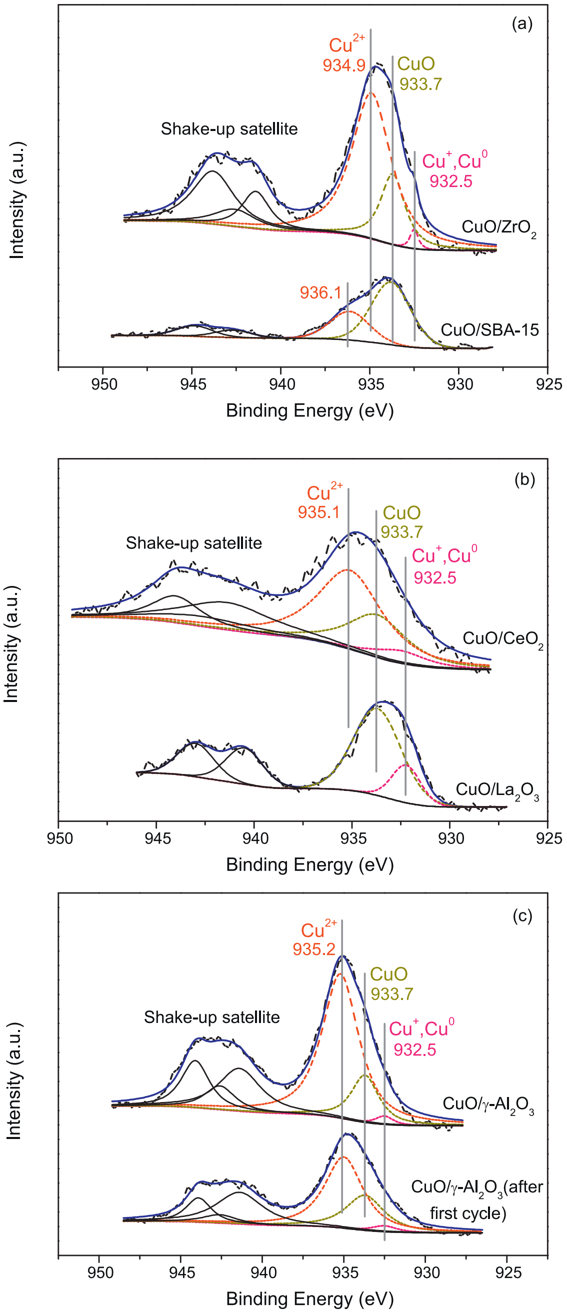


Fig. 3. Cu 2p3/2 XPS spectra of supported CuO catalysts: (a) CuO/ZrO_2 and CuO/SBA-15; (b) CuO/CeO_2 and CuO/La_2O_3; (c) fresh and used $CuO/\gamma\text{-}Al_2O_3$.

Table 4
Recycle of catalyst.[a]

Entry	Catalyst	Yield (%)[b]			Cu leaching after first cycle (%)[c]
		Run-1	Run-2	Run-3	
1	CuO/CeO_2	68	58	39	19
2	CuO/La_2O_3	71	56	37	26
3	**CuO/γ-Al_2O_3**	**76**	**61**	**60**	**20**

Entries in bold are the best item (highest yield, optimized reaction conditions and high recyclability).

[a] Reaction conditions: PhI (3 mmol), *m*-cresol (2 mmol), K_3PO_4 (4 mmol), catalysts (0.04 mmol Cu), in DMSO (4 ml) at 150 °C under N_2 for 10 h.

[b] GC yield determined with tridecane as internal standard.

[c] The content of copper leaching was determined by ICP-AES.

electronic state of surface copper species did not change in the reaction. Therefore, the loss of activity for used CuO/γ-Al_2O_3 catalyst should be caused by the copper leaching (20% of total copper content determined by inductively coupled plasma atomic emission spectroscopy (ICP-AES)) in the first catalytic reaction. In addition, the copper leaching in the second catalytic reaction was lower than 5%, which could explain the reason for the catalytic activity of CuO/γ-Al_2O_3 catalyst remained after the second run. The recyclability of CuO/CeO_2 and CuO/La_2O_3 catalysts was also examined. The yields of CuO/CeO_2 and CuO/La_2O_3 catalysts for third run were 39% and 37%, which were much lower than that of CuO/γ-Al_2O_3 (Table 4, Entries 1 and 2).

4. Conclusions

In summary, we developed an easy handling catalytic system for the synthesis of diaryl ether by using supported CuO catalysts under ligand-free conditions. The supported Cu-catalysts have a potential to be used in coupling reactions. CuO/γ-Al_2O_3 was proved to be the most effective catalyst for the coupling reaction and it could be reused for three cycles providing the product in moderate to good yields. Highly dispersed copper species on the supports showed higher catalytic activity than bulk CuO. Meanwhile, electronic state of Cu(II) did not affect catalytic performance of heterogeneous catalysts. Namely, Cu^{2+} or CuO was contained as the main active copper species of supported CuO catalysts in heterogeneous catalytic system. In addition, the redox property of supported CuO catalysts did not affect the catalyst activity directly. Further investigations on optimization of the supported CuO catalyst and reaction conditions are under study in our laboratory.

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