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Zeolite Beta nanoparticles assembled Cu catalysts with superior catalytic performances in the synthesis of thioesters by cross-coupling of aldehydes and disulfides

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ABSTRACT

Development of the highly active and stable heterogeneous transition metal catalysts is of great importance in synthetic chemistry. Herein, zeolite Beta nanoparticles assembled Cu catalyst (Cu-Beta-Nano) with high external surface area was directly synthesized, and it exhibits excellent catalytic activity and good recyclability in cross-coupling of aldehydes and disulfides for synthesis of thioesters. The high activity and good recyclability of the Cu-Beta-Nano catalyst should be attributed to the high external surface area of the catalyst and highly dispersed active copper species in the zeolite.

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1. Introduction

Thioesters and its derivatives are important compounds and widespread applications in organic synthesis and biological processes [1–5], which can be synthesized from the condensation of thiol or disulfide with carbonyl compounds, such as acyl halides, anhydrides, aldehydes, and carboxylic acids [6–13] catalyzed by transition-metals such as palladium [14,15], rhodium [16], indium [17], iron [18,19], copper [20–24] and zinc [25–27] as well as ligated metal complexes [28]. Despite these homogeneous metal catalysts being exhibiting high activity, the industrial applications of these catalysts are still limited because of their expensive cost, highly toxic, as well as tedious work-up separation. A number of solid catalysts such as $\text{KF}/\text{Al}_2\text{O}_3$ [29], zinc oxide [30], silica [31,32] have also been investigated for synthesis of the thioesters, nevertheless, the high active starting materials (acyl halides, N-acylphthalimides, thioacids) and unpleasant smelling thiols must be employed due to the low activities of these catalysts. Therefore, developing highly active and stable heterogeneous metal catalysts allowing the use of safe and simple starting materials such as aldehydes and

disulfides for the synthesis of thioesters could be a green and sustainable route.

From a practical point of view, metal catalysts supported on suitable porous materials with good chemical stability are very important for the regeneration of deactivated catalyst by calcination, the fast diffusion of the bulk substrates and the resistance of metal leaching. It is well known, crystalline microporous zeolites, which have high specific surface area, superior thermal and chemical stabilities [33–36], are widely used as metal catalyst supports [37–40]. However, conventional zeolites are ineffective for processing large molecules that cannot diffuse inside the microporous. Therefore, we synthesized Cu-containing zeolite Beta nanoparticles (Cu-Beta-Nano) with high external surface area, and used it as a catalyst with high activity and stability for catalyzing the cross-coupling of aldehydes with disulfides in water. For comparison, the pure zeolite Beta nanoparticles (Beta-Nano) was synthesized by the same procedure in the absence of metal precursor, which was used for preparation of the supported Cu catalysts (Cu-Beta-Nano-IM or Cu-Beta-Nano-IE) by impregnation or ion-exchange of cupric nitrate solution. Meanwhile, the Cu-containing bulky zeolite Beta (Cu-Beta-B) was also synthesized by the similar procedure reported in the literature [41]. Compared with Cu-Beta-B, the Cu-Beta-Nano catalyst exhibits much higher activity in the cross-coupling of aldehydes with disulfides. Importantly, the Cu-Beta-Nano catalyst shows superior reusability and chemical stability than Cu-Beta-Nano-IM and Cu-Beta-Nano-IE catalysts.

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2. Experimental

2.1. Material synthesis

A Cu-containing zeolite Beta nanoparticle (Cu-Beta-Nano) was synthesized from self-assembly of aluminosilicate gel with tetraethylammonium hydroxide (TEAOH) as a template. The molar ratio of compositions was $1\text{Al}_2\text{O}_3/2\text{Na}_2\text{O}/0.36\text{CuO}/6.7\text{SiO}_2/14\text{TEAOH}/667\text{H}_2\text{O}$. Typically, 0.39 g NaOH and 0.65 g NaAlO_2 was dissolved in 30 mL H_2O followed by addition of 26 mL TEAOH, 9.6 g of silica gel and 1.8 g of Beta zeolite seeds. After stirring at room temperature for 2 h, 0.38 g $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ was then introduced and the mixture was stirred for another 1 h, the obtained aluminosilicate gel was transferred into Teflon-coated stainless-steel autoclave for crystallization at 140°C for 6 days. The resultant product was filtered, washed, dried at 120°C overnight and calcined in air at 550°C for 5 h.

The Cu-containing bulky zeolite Beta (Cu-Beta-B) was synthesized from a starting aluminosilicate gel with a molar ratio of $1\text{Al}_2\text{O}_3/10\text{Na}_2\text{O}/0.36\text{CuO}/40\text{SiO}_2/570\text{H}_2\text{O}$. 0.25 g NaAlO_2 and 0.71 g NaOH was dissolved in 10 mL H_2O followed by addition of 2.5 g of fumed silica and 0.4 g of Beta zeolite seeds. After stirring at room temperature for 4 h, 0.09 g $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ was then introduced and further stirred for another 1 h, the obtained gel was transferred into Teflon-coated stainless-steel autoclave for crystallization at 140°C for 40 h. The resultant product was filtered, washed, dried at 120°C overnight and calcined in air at 550°C for 5 h. For comparison, the zeolite Beta nanoparticles (Beta-Nano) were synthesized by the same procedure except for the absence of metal precursor. The Beta-Nano supported copper catalyst (Cu-Beta-Nano-IM) was prepared by incipient wetness impregnation of the cupric nitrate solution with Beta-Nano. The Beta-Nano supported copper catalyst (Cu-Beta-Nano-IE) was also prepared by ion-exchange with a cupric nitrate solution. After impregnation or ion-exchange, the samples were exposed under ambient conditions for 20 h, and further dried at 120°C for 12 h and calcined in air at 550°C for 5 h. The copper contents in the calcined Cu-Beta-Nano, Cu-Beta-B, Cu-Beta-Nano-IM and Cu-Beta-Nano-IE samples were 2.0, 1.9, 2.1 and 1.9 wt.%, respectively, which was determined by the inductively coupled plasma optical emission spectroscopy (ICP-OES) with a Perkin-Elmer 3300DV emission spectrometer.

2.2. Characterization

X-ray diffraction (XRD) data were collected on a RIGAKU UltimaIV diffractometer using Cu $K\alpha$ radiation. Nitrogen adsorption-desorption isotherms were measured on a Micromeritics ASAP 2020 M apparatus. The sample was degassed for 5 h at 350°C before the measurement. Specific surface area was calculated from the adsorption data, using the Brunauer–Emmett–Teller (BET) equation. Scanning electron microscopy (SEM) image was obtained on a SURPA55 apparatus. Transmission electron microscopy (TEM) experiment was performed on a JEM-2100F microscope with a limited line resolution capacity of $1.4 \text{ \AA}$, under a voltage of 200 kV. Before characterization by TEM, the sample was dispersed in alcohol and dropped on a Cu-grid coated with carbon membrane. X-ray photoelectron spectroscopic (XPS) experiment was performed on an ESCALAB MK II system. UV–vis diffuse reflectance spectra (UV–vis DRS) were recorded in Perkin-Elmer Lambda 25 spectrometer with an integration sphere. BaSO_4 was used as a reference sample to measure the baseline spectrum. The Cu contents and the ratio of Si/Al in the zeolite catalyst were determined by the inductively coupled plasma optical emission spectroscopy (ICP-OES) with a Perkin-Elmer 3300DV emission spectrometer. The acidity of the catalysts was measured using temperature-programmed desorption of ammonia (NH_3 -TPD) on a

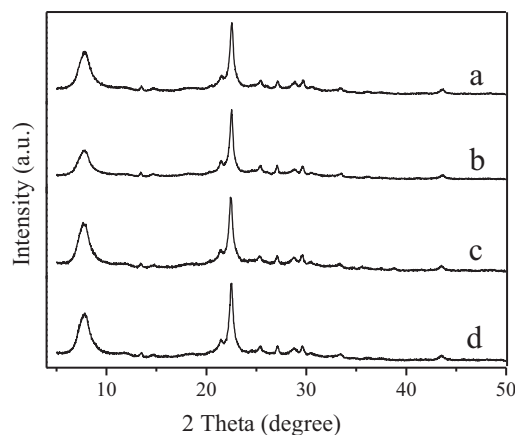


Fig. 1. XRD patterns of the (a) Cu-Beta-Nano, (b) Cu-Beta-B, (c) Beta-Nano and (d) the reused Cu-Beta-Nano samples.

Micromeritics ASAP2920 instrument. 200 mg of sample was placed in a quartz tube and pretreated in a helium stream at 500°C for 3 h. After the sample was cooled to 120°C , NH_3 –He mixed gas (10 vol% NH_3) was passed over the sample for 40 min. After removal of the physically adsorbed NH_3 by flowing helium for 3 h at 120°C , the sample was treated from 120 to 800°C at a rate of $10^\circ\text{C min}^{-1}$.

2.3. Catalytic testing

In a typical run, a mixture of diphenyl disulfide (0.2 mmol), p-tolualdehyde (2.0 mmol), K_2CO_3 (0.4 mmol), tert-butyl hydroperoxide (TBHP) (0.6 mmol), catalyst (40 mg), H_2O (2.0 mL) was stirred at desired temperature for 2 h. After the reaction was completed, the mixture was cooled down to room temperature and then extracted with ethyl acetate. The organic phase was analyzed on an Agilent 7890A GC equipped with a FID detector and mass spectrometer. The product was purified by column chromatography on silica gel (200–300 mesh) (eluent: petroleum ether and ethyl acetate). The obtained product was identified by NMR spectra using a Bruker 500 MHz spectrometer instrument. The ^1H NMR (500 MHz) and ^{13}C NMR (125 MHz) were recorded with spectrometers at 20°C using CDCl_3 as the solvent. Chemical shifts are given in parts per million relative to TMS as the internal standard at room temperature. The spectra data were consistent with authentic compounds.

3. Results and discussion

Fig. 1 shows the XRD patterns of the Cu-Beta-Nano, Cu-Beta-B, Beta-Nano and reused Cu-Beta-Nano samples, exhibiting typical peaks associated with zeolite Beta structure [42]. The ICP analysis results show that the copper content in the Cu-Beta-Nano and Cu-Beta-B is 2.0 and 1.9 wt.%, respectively, suggesting that the copper species has been successfully introduced into the zeolite.

Fig. 2 shows the SEM and TEM images of Cu-Beta-Nano and Cu-Beta-B samples. Clearly, the Cu-Beta-Nano sample shows a globule morphology with particle sizes in the range of 200–250 nm (Fig. 2a). In contrast, the Cu-Beta-B sample shows large particle with sizes about $1 \mu\text{m}$ (Fig. 2c). It is worth noting that the copper particles in the Cu-Beta-Nano and used Cu-Beta-Nano samples were not observed in the TEM images (Fig. 2b and d), while the Cu content in the two samples is 2.0 and 1.9 wt.% determined from ICP analysis, which indicates that the Cu species could be highly dispersed into the zeolite structure. Sample textual parameters are presented in Table 1.

To investigate the state of Cu species in the zeolite catalysts, the UV–vis diffuse reflectance spectroscopy (UV–vis DRS)

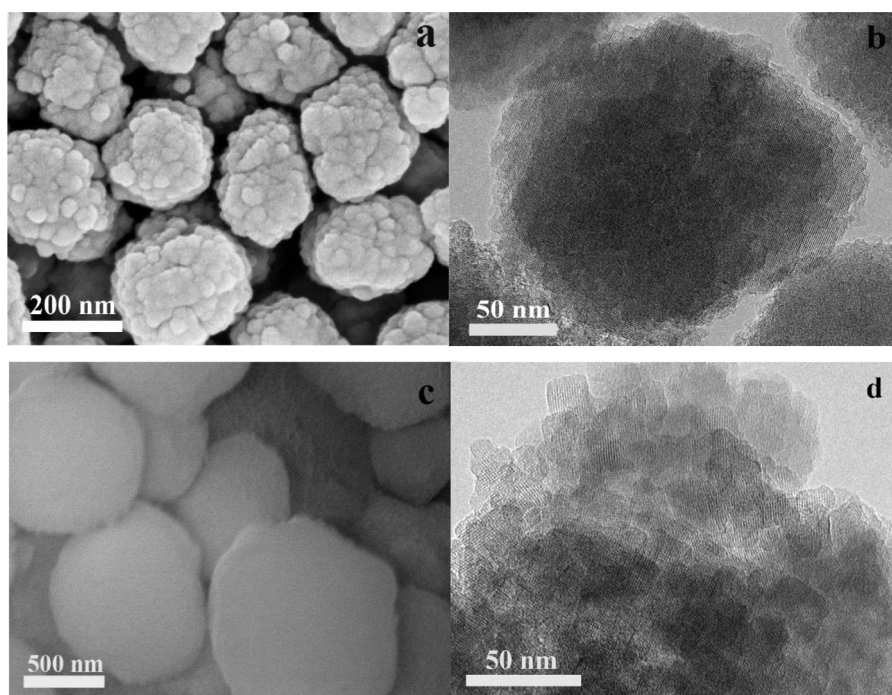


Fig. 2. (a) SEM image of the calcined Cu-Beta-Nano sample. (b) TEM image of the Cu-Beta-Nano sample. (c) SEM image of the Cu-Beta-B sample. (d) TEM image of the used Cu-Beta-Nano sample.

analysis and X-ray photoelectron spectroscopy (XPS) were performed. Fig. 3a shows the UV–vis spectra of the Cu-Beta-Nano and Cu-Beta-Nano-IE samples. The catalysts display one adsorption band at 207 nm, which could be assigned to the charge-transfer in the O-to-metal related to the Cu^+ or Cu^{2+} species stabilized in the zeolite framework. The broad band centered at 280 nm for the Cu-Beta-Nano-IE sample is ascribed to Cu^{2+} ion [43–45]. These results indicate that the Cu species in the Beta zeolites are mainly existed in the Cu^{2+} ion. Fig. 3b shows that the binding energy of $\text{Cu}2p_{3/2}$ for the Cu-Beta-Nano, Cu-Beta-Nano-IE, Cu-Beta-Nano-IM and reused Cu-Beta-Nano samples is 933.4, 933.6, 933.7 and 933.3 eV, which is well-ascribed to Cu^{2+} ion [46,47]. Compared with Cu-Beta-Nano, the binding energy of $\text{Cu}2p_{3/2}$ for the reused Cu-Beta-Nano sample did not change, which indicated that the surface structure of the catalyst was maintained after the reaction.

Screening of reaction conditions for the synthesis of thioesters was initiated with the cross-coupling of p-tolualdehyde and diphenyl disulfide as model substrates (Table 2). Firstly, the blank experiment showed that only 12% yield was obtained when the reaction was carried out in the absence of catalyst (entry 1). Delightfully, introducing Cu catalysts in the reaction mixture, high product yields were obtained under using water as solvent. Although

Cu-Beta-B catalyst with lower Si/Al ratio presents a relative strong acidity (Fig. 4, Table 1), its catalytic activity is much lower than zeolite Beta nanoparticles assembled Cu catalysts (entries 2–5, Table 2). The result indicates that the high catalytic performance of the zeolite Beta nanoparticles assembled Cu catalysts was not induced by the acidity of the catalyst. In our case, the cross-coupling reaction with large molecule dimension of reactants and products could mainly occur on the outer surface of the Cu-containing catalysts. And the mass transfer could be strongly existed in the micropore of the catalyst. The zeolite Beta nanoparticles assembled Cu catalysts show high external surface area ($148\text{--}163\text{ m}^2/\text{g}$), but the bulky Cu-Beta-B catalyst has lower external surface area ($27\text{ m}^2/\text{g}$, Table 1). Therefore, zeolite Beta nanoparticles assembled Cu catalysts can provide large amount of accessible active sites for reactant, as compared to Cu-Beta-B catalyst. In addition, several organic solvents were also screened and lower yields were obtained (entries 7–10). The use of the water as solvent is environmental friendly for green and sustainable production process in industry.

The reusability of Cu-Beta-Nano, Cu-Beta-Nano-IM and Cu-Beta-Nano-IE catalysts was also examined (Table 3). At the end of the reaction, each catalyst was separated by filtration and subsequently washed with ethyl acetate and water, and then dried

Table 1
Texture parameters of series catalyst samples.

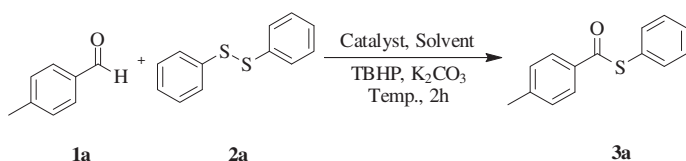
Samples	S_{BET}^a (m^2/g)	S_{Ext}^b (m^2/g)	V_{micro}^c (m^3/g)	Cu content (wt.%)	Si/Al
Cu-Beta-Nano	679	163	0.21	2.0 (1.9) ^d	17.0
Cu-Beta-B	661	27	0.25	1.9	10.0
Beta-Nano	640	157	0.19	0	17.5
Cu-Beta-Nano-IM	633	156	0.19	2.1 (1.7) ^d	
Cu-Beta-Nano-IE	610	153	0.19	1.9 (1.7) ^d	
Reused Cu-Beta-Nano-IM	615	148	0.19	1.5	16.5
Reused Cu-Beta-Nano-IE	603	150	0.18	1.3	16.8
Reused Cu-Beta-Nano	675	165	0.22	2.0	16.7

^a BET surface area.

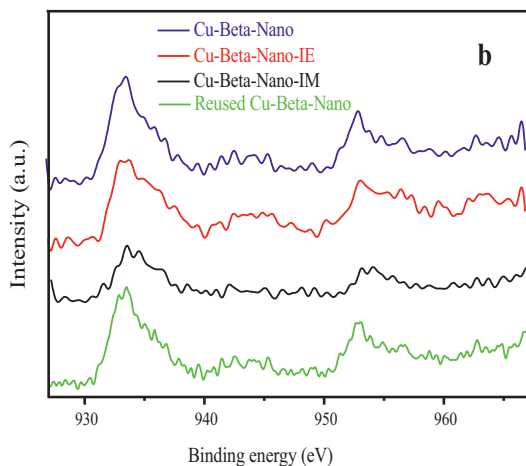
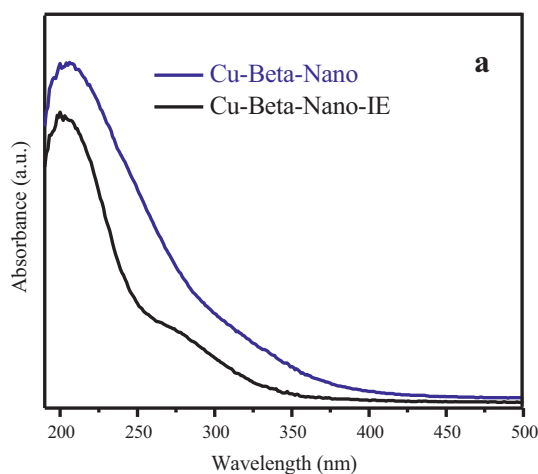
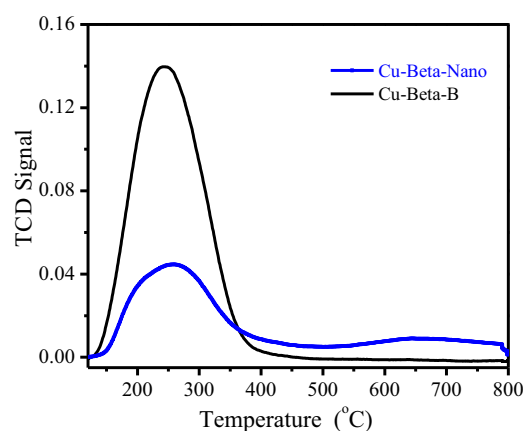
^b External surface area that including mesoporous surface area.

^c Microporous volume.

^d The value in parenthesis is analyzed by X-ray fluorescence technique.

Table 2
Cross-coupling of aldehydes with disulfides over series catalysts in different conditions.^a

Entry	Catalyst	Solvent	T (°C)	Yield (%) ^b
1	–	H ₂ O	80	12
2	Cu-Beta-Nano	H ₂ O	80	75 (70) ^c
3	Cu-Beta-Nano-IM	H ₂ O	80	76 (70) ^c
4	Cu-Beta-Nano-IE	H ₂ O	80	74 (67) ^c
5	Cu-Beta-B	H ₂ O	80	60
6	Cu-Beta-Nano	H ₂ O	100	99 (93) ^c
7	Cu-Beta-Nano	DMF	80	6
8	Cu-Beta-Nano	DMSO	80	58
9	Cu-Beta-Nano	NMP	80	0
10	Cu-Beta-Nano	1,4-dioxane	80	64

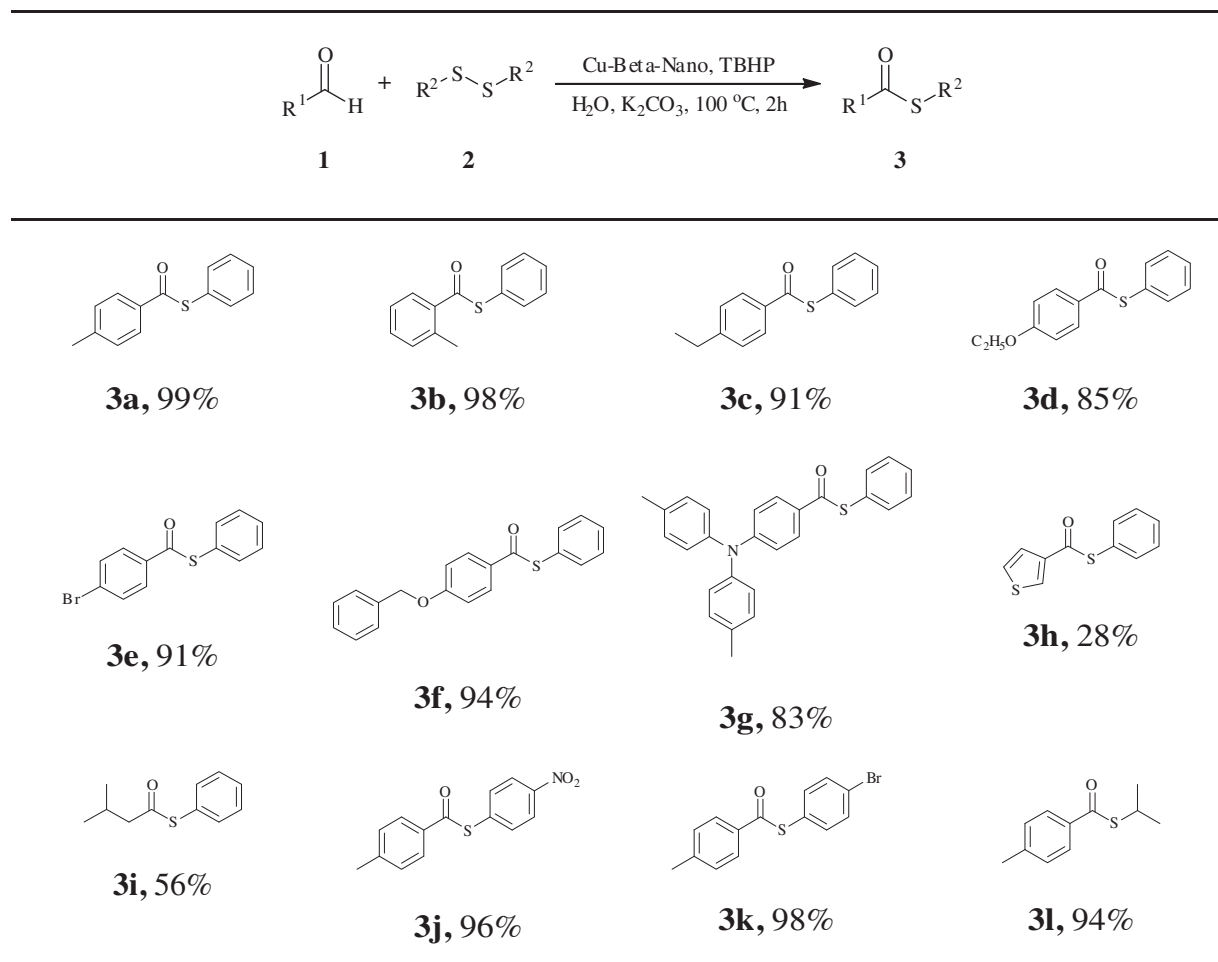
Conditions:^a Unless otherwise noted, the reaction were carried out using diphenyl disulfide (0.2 mmol), p-tolualdehyde (2.0 mmol), catalyst (40 mg), K₂CO₃ (0.4 mmol), TBHP (0.6 mmol), solvent (2.0 mL), 2 h.^b GC yield.^c The value in parenthesis is isolated yield.**Fig. 3.** (a) UV–vis DR spectra of Cu-Beta-Nano and Cu-Beta-Nano-IE catalysts. (b) XPS spectra of Cu-Beta-Nano, Cu-Beta-Nano-IE, Cu-Beta-Nano-IM and the Reused Cu-Beta-Nano catalysts.**Fig. 4.** NH₃-TPD profiles of the Cu-Beta-B and Cu-Beta-Nano catalysts.

at 120 °C for 12 h for the next run. The Cu-Beta-Nano catalyst can recycle six runs without any activity loss, whereas the product yield on the Cu-Beta-Nano-IM and Cu-Beta-Nano-IE catalysts decreased from 99 and 100% to 70 and 71%, respectively (Table 3). These results demonstrate that the Cu-Beta-Nano catalyst has a good reusability, which is one of the key features in the practical application in industry.

The deactivation of the Cu-Beta-Nano-IM and Cu-Beta-Nano-IE catalysts should be attribute to the Cu species are leached from the zeolite support under the reaction conditions. This suggestion can be supported by the element analysis results. The Cu content in the Cu-Beta-Nano catalyst was not decreased after the reaction.

Table 3
Catalyst recycles in the condensation of p-tolualdehyde with diphenyl disulfide.

Recycle	Yield (%)		
	Cu-Beta-Nano	Cu-Beta-Nano-IM	Cu-Beta-Nano-IE
Run 1	99	99	100
Run 2	99	97	94
Run 3	98	93	90
Run 4	99	87	86
Run 5	100	79	80
Run 6	99	70	71

Table 4
Cu-Beta-Nano catalyzed the cross-coupling of aldehydes with diphenyl disulfides.^a

^a Unless otherwise noted, the reaction were carried out using diphenyl disulfides (0.2 mmol), aldehydes (2.0 mmol), Cu-Beta-Nano (40 mg), K₂CO₃ (0.4 mmol), TBHP (0.6 mmol), H₂O (2.0 mL), 2 h.

However, the Cu content in the as synthesized Cu-Beta-Nano-IM and Cu-Beta-Nano-IE catalysts decreased from 2.1 and 1.9 wt.% to 1.5 and 1.3 wt.% after recycled six runs. The Si/Al ratio for reused and as synthesized Cu-Beta-Nano is 16.7 and 17.0, respectively. These results indicate that the directly synthesized Cu-containing zeolite Beta nanoparticles present good chemical stability in the reaction process, which is very important for industrial applications. In addition, the intensity of the XRD diffraction peaks for the reused Cu-Beta-Nano is similar to the as synthesized catalyst (Fig. 1d and a), and the textual structure of the reused Cu-Beta-Nano almost has no changed (Table 1), which demonstrates that the Cu-Beta-Nano catalyst has good mechanical stability. The good mechanical stability of the catalyst is also important parameter for industrial applications in the future.

Encouraged by the above results, the generality of the protocol was investigated for both substituted aldehyde and disulfide (Table 4). At first, the Cu-Beta-Nano catalyst gave high yields for the aldehyde not only with electron-donating group as –CH₃, –CH₂CH₃ and –OC₂H₅ (**3a**, **3b**, **3c**, **3d**), but also with electron-withdrawing group as halogens (**3e**). To further extend the utility of this thioesterification, we next turn our attention to the bulky molecule aldehydes. It is clear that the bulky molecule aldehydes can also transform into thioesters (**3f**, **3g**) in high yields. In addition, aldehydes bearing heteroaryl and aliphatic groups are also readily tolerated in this coupling protocol (**3h**, **3i**). Finally, disulfides with different substituted groups (aryl and aliphatic) over Cu-Beta-Nano

catalyst proceeded readily and gave the corresponding products in high yields (**3j**, **3k**, **3l**). These results indicate that the zeolite Beta nanoparticles assembled Cu catalysts present good generality for aldehydes or disulfides.

4. Conclusions

In conclusion, the Cu-Beta-Nano catalyst with high external surface area was synthesized and used for catalytic synthesis of thioesters by cross-coupling of aldehydes with disulfides. A variety of functional groups on both the aldehyde and disulfide substrates are well tolerated. Compared with bulky zeolite Beta (Cu-Beta-B), and Cu-Beta-Nano-IM and Cu-Beta-Nano-IE catalysts, the Cu-Beta-Nano catalyst shows high activity and good reusability in the cross-coupling reaction. This is attributed to the fact that the highly dispersed Cu²⁺ in zeolite crystals present high activity and the high external surface of the Cu-Beta-Nano catalyst provides more accessible active sites, improving its catalytic performance.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.cattod.2015.07.010>

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