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A Facile, Direct Synthesis of Styrene Carbonate from Styrene and CO_2 Catalyzed by $Au/Fe(OH)_3–ZnBr_2/Bu_4NBr$ System

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Abstract A three-component catalyst composed of $Au/Fe(OH)_3$, $ZnBr_2$, and Bu_4NBr was shown to be very efficient for direct oxidative carboxylation of styrene to styrene carbonate. An enhanced styrene carbonate yield (53%) was obtained under mild reaction conditions of 80 °C, 10 h, and 4 MPa CO_2 pressure. In this reaction, CO_2 acts as both a reagent and a solvent. The influence of various reaction parameters, such as catalyst pretreatment, CO_2 pressure, and reaction time has been investigated carefully. Furthermore, the active sites in supported Au catalyst are studied. The results obtained suggest that synergistic effect of gold ions and ferric hydroxide over $Au/Fe(OH)_3$ catalyst might be important for its better catalytic performance in epoxidation of styrene as compared with supported gold catalyst on oxides.

Keywords Styrene carbonate · Styrene · Carbon dioxide · Supported Au catalyst

1 Introduction

Carbon dioxide is one of the most abundant wastes produced by human activities and industrial emission. In order to prevent risky reinforcement of the greenhouse effect, the accumulation of CO_2 in the atmosphere should be controlled by removing it from industrial effluents. On the other hand, CO_2 is recognized as a safe and cheap C1 building block. Under these circumstances, the conversion of carbon dioxide to industrially useful compounds has recently attracted much attention in view of the so-called "sustainable society" [1] and "green chemistry" [2] concepts. Up to date, one of the most effective methodologies is the synthesis of five-membered cyclic carbonate via the coupling of CO_2 with epoxides and, in this process, one carbon atom and two oxygen atoms can be incorporated without forming any byproducts with high atom efficiency [3]. Moreover, cyclic carbonates have found extensive use as excellent aprotic polar solvents, precursors for polycarbonates and other polymeric materials, electrolytic element of lithium secondary batteries and chemical ingredients for pharmaceutical/fine chemicals like dialkyl carbonates, glycols, carbomates, pyrimidines, purines, etc. [4–7].

After Inoue et al. [8, 9] first reported the cyclic carbonate could be prepared by cycloaddition of CO_2 to epoxides in 1969, numerous catalysts such as alkali metal halides [10–13], organic bases [14–16], metal oxides [17–20], zeolites [21, 22], smectites [23, 24], and metal complexes [25–28] have been investigated. However, activity, stability, and recovery of the catalysts still remain to be improved. Besides the direct coupling of CO_2 with epoxides to cyclic carbonate, alternative synthetic route from CO_2 is oxidative carboxylation of olefin (Scheme 1), which couples two sequential reactions of epoxidation of olefin and CO_2 cycloaddition to epoxides formed. Synthesis of cyclic carbonate from olefin and CO_2 gives a simple route that avoids the preliminary synthesis and isolation of epoxides. Furthermore, easily available and low-priced chemicals of olefins may be used to produce valuable chemicals as compared with epoxides as substrates. This direct synthesis route for cyclic carbonate from olefin is not

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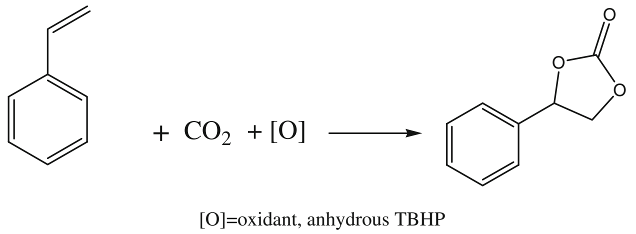


Scheme 1 One-pot synthesis styrene carbonate using styrene and CO_2 as substrates

only energy saving but also simple and economical. Despite the usefulness of this reaction, little efforts have been given to it so far, in contrast to extensive study on the coupling reaction of CO_2 and epoxides. Aresta [29–31] first reported one-pot synthesis of styrene carbonate from styrene, CO_2 and O_2 with homogeneous rhodium complex and metal oxides as catalysts, however, the process is known for its very low yields for styrene carbonate and large amounts of by-products such as benzaldehyde and benzoic acid. Srivastava et al. [32] developed a two-step synthesis of styrene carbonate in a single reactor from styrene using titanosilicate catalysts and organic base as co-catalyst. Although the carbonate yields can be improved to 13% under optimized reaction conditions, the long reaction time and toxic organic solvents are necessary. Therefore, the development of highly active catalysts for this reaction is still of great importance.

Recently, we have found that direct oxidative carboxylation of styrene to styrene carbonate is feasible in the presence of ionic liquids [33–35], the carbonate yields being much higher than those reported previously. However, the homogeneous nature of ionic liquid, tetrabutylammonium bromide (Bu_4NBr), is the main drawback limiting its use. Later on, we have also shown that an Au/SiO_2–$ZnBr_2$/Bu_4NBr catalyst is active for the direct synthesis of styrene carbonate (SC) from styrene [36]. In this system, supported metallic Au is active for the epoxidation of styrene, and $ZnBr_2$–Bu_4NBr cooperatively catalyze the subsequent CO_2 cycloaddition to epoxides. Moreover, the efficiency of the SC one-pot synthesis would be determined mostly by the catalytic performance of the gold catalyst. Hence, if more active and selective catalysts for the epoxidation reaction were to be explored, more effective catalyst system for the one-pot synthesis SC would be realized.

Herein, we synthesized highly effective ferric hydroxide supported gold catalyst $Au/Fe(OH)_3$, which was very active for the epoxidation of styrene, and then supported gold catalyst was coupled with $ZnBr_2$/Bu_4NBr which cooperatively catalyzed CO_2 cycloaddition reaction. It was shown the three components**,** $Au/Fe(OH)_3$, $ZnBr_2$, Bu_4NBr efficiently catalyzed the direct oxidative carboxylation of styrene to styrene carbonate. The influence of factors such as catalyst pretreatments, Au loadings, kinds of oxidants, CO_2 pressure, and reaction time are investigated in detail. An improved styrene carbonate yield (53%) was obtained under mild reaction conditions of 80 °C, 10 h, and 4 MPa CO_2 pressure. Furthermore, the active sites in supported Au catalysts are studied, and a synergistic effect of gold ions and ferric hydroxide possibly plays an important role for the high catalytic performance in epoxidation of styrene.

2 Experimental

2.1 Preparation of Supported Gold-based Catalysts

The catalysts with various Au loadings were prepared by co-precipitation method [37]. The aqueous of $HAuCl_4$ (0.0074 g mL^{-1}) and $Fe(NO_3)_3$ (1 mol L^{-1}) were added dropwise into Na_2CO_3 (1 mol L^{-1}) solution with vigorous stirring and the pH value of the solution was controlled to 8. The suspension was further aged at room temperature for 4 h while the pH was kept a constant. Then the precipitate obtained was filtrated and washed thoroughly with distilled water until it became free of chloride ions. The resulting precipitate was dried at room temperature for overnight to form ferric hydroxide supported Au catalyst (referred to as Au/Fe-U). Some samples were then calcined in air at 300 or 550 °C for 4 h to form two different supported Au catalysts denoted as Au/Fe-3 and Au/Fe-5, respectively.

For comparison, the catalyst reduced by flowing H_2 at 400 °C for 2 h was also prepared according to a method described in the literature [38].

A sample of gold ion supported on iron oxide, denoted as Au^{3+}/Fe_2O_3, was prepared in the same manner as reported previously [39]. Briefly, iron oxide in the form of haematite was contacted with an aqueous solution of $HAuCl_4$ and subsequently dried at room temperature.

$Au^0/Fe(OH)_3$ was prepared by deposition–precipitation method in accordance with the literature [36, 40]. At first, $HAuCl_4$ was reduced by quantitative $NaBH_4$ solution at pH 8 for 2 h, then iron hydroxide support, which was prepared with ferric nitrate poured into an aqueous solution of Na_2CO_3 (1 mol L^{-1}) and maintained at 80 °C under vigorous stirring, was added to the above reduced solution. The slurry was maintained at 70 °C for 2 h with vigorous stirring. Then the sample was filtered, washed with distilled water until chloride was eliminated, and dried under vacuum for 1 day.

2.2 Characterization of Catalysts

Au contents were analyzed by inductively coupled plasma (ICP) technique with a Perkin–Elmer 2400 elemental analyzer. X-ray powder diffraction (XRD) data were collected on a Siemens D5005 diffractometer with Cu Kα radiation ($\lambda = 1.5418$ Å). Fe Mössbauer measurements were made on Oxford MS-500 instrument at room temperature. X-ray photoelectron spectra (XPS) were recorded on a VG ESCA LAB MKII instrument using Al source for excitation.

2.3 Catalytic Reaction

The one-pot synthesis of styrene carbonate from styrene was conducted in a 50 mL stainless steel autoclave. At first, catalysts containing supported Au (0.1 g), $ZnBr_2$ (0.44 mmol), Bu_4NBr (0.88 mmol), substrates comprised of styrene (17.3 mmol) and anhydrous tetrabutyl hydroperoxide (TBHP 25.4 mmol), were fed into the reactor, and heated to 80 °C, then liquid CO_2 was charged with a high-pressure liquid pump to the desired pressure. The reaction was conducted for a certain time with stirring. After the reaction, the reactor was cooled to room temperature and depressurized slowly to atmospheric pressure. The catalysts were filtered off from the reaction mixture, then the organic phase was analyzed on a gas chromatograph (Agilent GC 6890N) with a capillary column HP-5. The quantities of styrene consumed and products formed were determined from the results obtained from authentic standards. The structures of the products were further confirmed by GC-MS (Shimadzu QP5050A) by comparing retention times and fragmentation patterns with authentic samples.

The used Au/Fe(OH)$_3$ catalyst was removed from the reaction system by filtration, washed with acetone, dried at room temperature, and then reused for subsequent one-pot synthesis styrene carbonate with a fresh charge of $ZnBr_2$ and Bu_4NBr for testing the supported Au catalyst reusability.

3 Results and Discussion

3.1 Characterization of Supported Gold Catalysts

Figure 1 shows XRD patterns of various gold-based catalysts. As-synthesized Au/Fe-U did not give crystalline gold diffraction peaks from 35 to 70° (Fig. 1a), which indicated that gold particles could be highly dispersed under the support Fe(OH)$_3$; with the calcination temperature increased to 300 °C, the metallic gold peaks appeared (Fig. 1b); when calcination temperature was further raised to 550 °C, the peaks associated with Au^0 phase became stronger (Fig. 1c); Fig. 1d showed the peaks of metallic gold; the peaks attributed to Fe_2O_3 were shown in Fig. 1e, suggesting that gold ions were highly dispersed on Fe_2O_3 support. After reduction by H_2, the sample displayed the peaks associated with Fe_3O_4, Fe, and metallic Au phases (Fig. 1f).

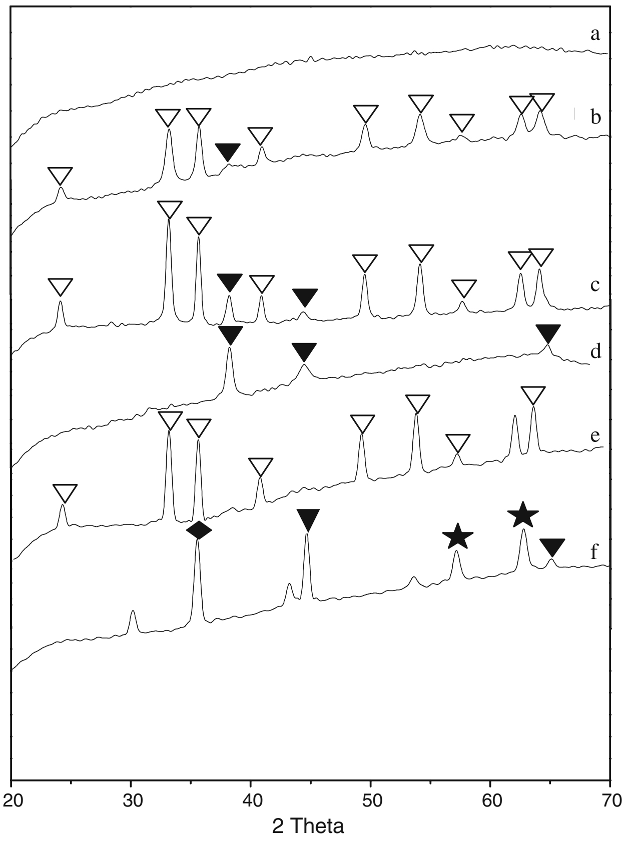


Fig. 1 XRD patterns of *a* as-synthesized sample; *b* sample calcined at 300 °C; *c* sample calcined at 550 °C; *d* Au^0/Fe(OH)$_3$ sample; *e* Au^{3+}/Fe_2O_3 sample; *f* sample reduced by H_2. Au loadings are 6.67 wt%. “▽” Fe_2O_3; “▼” Au^0; “★” Fe_3O_4;“◆” Fe

XPS measurements of various samples (Fig. 2) indicate the presence of gold species with different oxidation states. As-synthesized sample showed three peaks at 83.8, 86.5, and 90.4 eV (Fig. 2a), which were ascribed to the most cationic gold Au^{3+} and a small amount of Au^0 [41]. Calcinations at 300 and 550 °C resulted in the formation of metallic Au^0, giving $4f_{7/2}$ and $4f_{5/2}$ signals at 83.9 and 87.6 eV, respectively [42], which were well consistent with XRD characterization (Fig. 1b, c).

The parameters of Fe Mössbauer spectra are listed in Table 1. The results indicate that ferric species of as-synthesized and calcined (550 °C) samples were Fe(OH)$_3$ [43] and Fe_2O_3 [44], respectively (Fig. 3a, b). After reduction by H_2, ferric hydroxide was reduced to Fe_3O_4 and partially Fe [45] (Fig. 3c).

3.2 Effect of Catalyst Pretreatment Conditions

Table 2 shows the influence of the pretreatments for Au/Fe catalysts on the one-pot synthesis SC. For as-synthesized Au/Fe-U catalyst, the styrene conversion and desired

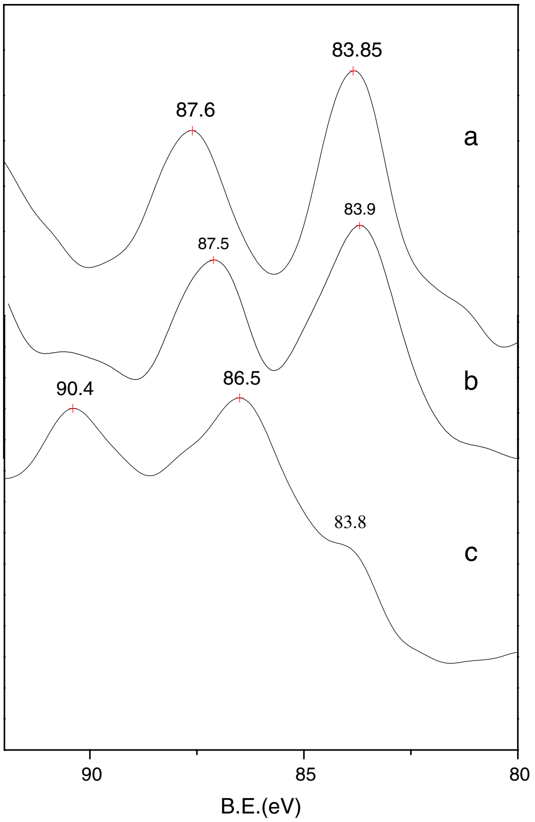


Fig. 2 Au (4f) XPS spectra of *a* as-synthesized sample; *b* sample calcined at 300 °C; *c* sample calcined at 550 °C. Au loadings are 6.67 wt%

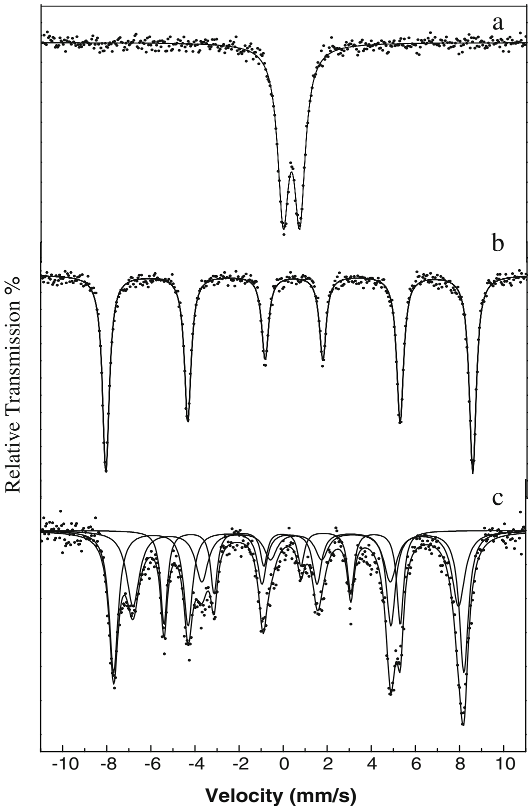


Fig. 3 Fe Mössbauer spectra of *a* as-synthesized sample; *b* sample calcined at 550 °C; *c* sample reduced by H_2. Au loadings are 6.67 wt%

Table 1 The parameters of Mössbauer spectra over various Au/Fe samples

Sample (mm/s)	Au/Fe-U	Used Au/Fe-U sample	Au/Fe-5	Reduction of Au/Fe-U by H_2		
				I	II	III
IS	0.37	0.37	0.38	0.01	0.32	0.62
QS	0.74	0.75	0.21	0.01	0.03	0.01

The Au loadings of Au/Fe samples are 6.67 wt%

IS isomer shifts, *QS* quadrupole splitting

product of SC yield were 88.3 and 39.5%, respectively. When the catalyst was calcined at 300 °C, the conversion and SC yield decreased slightly. With further increase of calcination temperature to 550 °C, the conversion and SC yield decreased markedly to 78.9 and 33.0%, respectively (entries 2, 3). These results indicate that the one-pot synthesis of styrene carbonate was sensitive to heat treatment of catalyst, and calcination at elevated temperatures was detrimental to the catalytic performance. The great decrease in SC yield was obtained when the catalyst was reduced with H_2 (entry 4). The differences in the activity of various pretreatments may be ascribed to the changes in the chemical states of gold and support.

XPS and Mössbauer spectra of Au/Fe catalysts by various pretreatments showed significant differences in Au and Fe species on the catalyst surface. Cationic gold (Au^{3+}) and partially Au^0 combined with $Fe(OH)_3$ support existed in the as-synthesized sample; at the calcination temperature of 550 °C, metallic gold (Au^0) and Fe_2O_3 were formed; Au^0 and Fe_3O_4/Fe appeared in the reduced sample by H_2. Obviously, the different chemical states of Au and Fe species over various catalysts were responsible for the change in catalytic activity for direct synthesis of SC from styrene. Notably, the activity of Au^{3+} was better than Au^0, which contradicts with our earlier report [36] that metallic Au^0 was more active than cationic Au^{3+} in Au/SiO_2

Table 2 Influence of pretreatments of Au/Fe catalysts on the activity in one-pot synthesis of styrene carbonate

Entry	Pretreatment sample	Conversion (%)	Yield (%)			
			SC	SO	BA	Others
1	Au/Fe-U	88.3	39.5	14.2	25.3	9.3
2	Au/Fe-3	85.4	39.0	14.1	20.9	11.5
3	Au/Fe-5	78.9	33.0	12.1	24.6	9.2
4	Reduced Au/Fe-U by H_2	77.0	30.0	6.7	18.3	22.0

Reaction conditions: 6.67 wt% Au/Fe, 0.1 g; $ZnBr_2$, 0.44 mmol; Bu_4NBr, 0.88 mmol; styrene, 17.3 mmol; anhydrous TBHP, 25.4 mmol; CO_2 pressure, 4 MPa; 80 °C; 4 h

SC styrene carbonate; *SO* styrene epoxide; *BA* benzaldehyde; others, some oligomers

catalyst for epoxidation of styrene. The difference of active sites between the two catalysts may be related to the support effect. It was accepted that the different support materials could be playing a substantial role in perturbing the surface chemistry in the two catalysts [46].

3.3 The Effect of Reaction Parameters on Direct Synthesis SC over Au/Fe-U/$ZnBr_2$/Bu_4NBr Catalysts

Table 3 summarizes the influences of Au loadings and oxidants on the results of one-pot synthesis SC over the three-component catalysts. It was expected that the conversion of styrene and SC yield increased with Au loadings. When Au loading reached 4.67%, the conversion and SC yield increased to 90.5 and 43.3%, respectively. However, with further increase of Au loading over 4.67%, SC yield and conversion decreased slightly. One possible reason for the reduced activity with the higher loading is the lower dispersion of gold species on the support surface. The presence of an optimal amount of Au loading was also observed in the Au/TiO_2 photocatalysed reduction of disulfide [47]. The influence of oxidant is obvious in the direct synthesis SC from styrene (entry 3). When the oxidant was changed to H_2O_2, the SC yield dropped drastically and much more byproduct benzaldehyde was produced. The poor activity with H_2O_2 is attributed to two possible reasons. One is the poor miscibility of reactant styrene with the oxidant; the other important factor is the hydrolysis of epoxide to phenyl glycol [48], which would decrease the quantity of the starting material for the second step of SC synthesis.

Table 3 Influence of Au loadings and oxidants on the activity in one-pot synthesis of styrene carbonate

Entry	Au loadings (wt%)	Conversion (%)	Yield (%)			
			SC	SO	BA	Others
1	1.20	77.3	36.4	9.9	17.8	13.2
2	4.67	90.5	43.3	8.8	21.2	17.3
3[a]	4.67	74.4	14.2	0	31.6	28.6
4	6.67	88.3	39.5	14.1	25.3	9.5
5[b]	6.67	88.1	38.7	10.8	26.1	12.6
6	11.0	87.0	36.4	12.9	25.5	12.2
7	16.0	84.6	32.6	11.9	17.2	22.9

Reaction conditions: Au/Fe-U, 0.1 g; $ZnBr_2$, 0.44 mmol; Bu_4NBr, 0.88 mmol; styrene, 17.3 mmol; anhydrous TBHP, 25.4 mmol; CO_2 pressure, 4 MPa; 80 °C; 4 h

[a] With H_2O_2 (30%) (25.4 mmol) as the oxidant, the other conditions are the same to the above

[b] With the used Au/Fe-U and fresh $ZnBr_2$ and Bu_4NBr

The influence of CO_2 pressure on the styrene conversion and product yields of styrene carbonate (SC), styrene epoxide (SO), and benzaldehyde (BA) is shown in Fig. 4. The catalyst system could operate very efficiently at low CO_2 pressures. The styrene conversion and SC yield increased with pressure from 1 to 4 MPa. The highest catalytic activity occured at 4 MPa. Increasing the pressure beyond the optimal level resulted in a decrease in activity, and styrene conversion and SC yields showed the minima at 15 MPa. Such a decrease in the activity with increasing pressure of carbon dioxide was also reported previously [26, 49, 50]. It can be inferred that the introduced CO_2 dissolves in styrene or “liquefies” through the formation of a CO_2-styrene complex [51]. Too high CO_2 pressure may retard the interaction between styrene and the catalyst, resulting in a low conversion. Visual observations of the reaction mixture [33] also indicated that three phases of CO_2-rich gas, liquid (styrene, TBHP, $ZnBr_2$/Bu_4NBr), and solid (Au/Fe-U) existed at lower pressures, where it

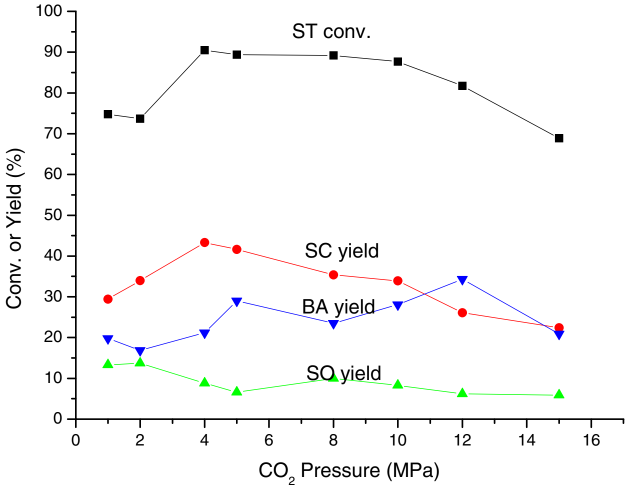


Fig. 4 Effect of CO_2 pressure on the conversion and product yields. Reaction conditions: styrene, 17.3 mmol; anhydrous TBHP, 25.4 mmol; 4.67 wt% Au/Fe-U, 0.1 g; $ZnBr_2$, 0.44 mmol; Bu_4NBr, 0.88 mmol; 80 °C; 4 h

became two phases (fluid phase and catalyst solid phase) at a high pressure of 15 MPa. This phase change would cause an increase in the volume where the reaction proceeds, and hence the concentration of the substrate would be low at this elevated pressure, resulting in lower styrene conversion and SC yield. It was also found that much oligomer was produced at such a high pressure [24].

Reaction time versus styrene conversion and product yields is plotted in Fig. 5. It can be seen that conversion increased with the time up to 4 h, a further increase of reaction time did not improve the conversion as much. And the yield of desired product SC increased to maximum of 53% at 10 h, whereas a further increase of reaction time led to slight decrease in SC yield because of decomposition of SC to SO with long time [19]. Moreover, as described in the preliminary paper [36], the cycloaddition of CO_2 to styrene epoxide proceeded much faster over $ZnBr_2$/Bu_4NBr than the epoxidation catalyzed by Au/Fe-U. For the Au/Fe-U, epoxidation of styrene gave maximum epoxide yield of 61% at 10 h (confirmed by separate epoxidation of styrene catalyzed by Au/Fe-U catalyst at identical reaction conditions), which was close to the total values of SC and SO yields. These results confirmed that the first reaction, epoxidation of styrene, was a rate-determing step in one-pot synthesis SC. Hence, the efficiency of the SC one-pot synthesis would be determined mostly by the catalytic performance of the supported gold catalyst.

The reusability of Au/Fe-U catalyst was examined. After one run, the solid catalyst was removed from the reaction system by filtration, washed with acetone, dried at room temperature, and then subjected to the next catalytic run, in which fresh $ZnBr_2$ and Bu_4NBr were added. The results with the used catalyst were similar to those obtained with fresh catalyst (Table 3, entry 5). In order to check if the reaction is effectively heterogeneous, the Au/Fe-U catalyst was filtered off after catalytic reaction, then the resultant final reaction solutions was further reacted for another run. It was shown that no reaction occurred with the remaining solution, indicating that Au/Fe-U was recyclable without any loss of activity.

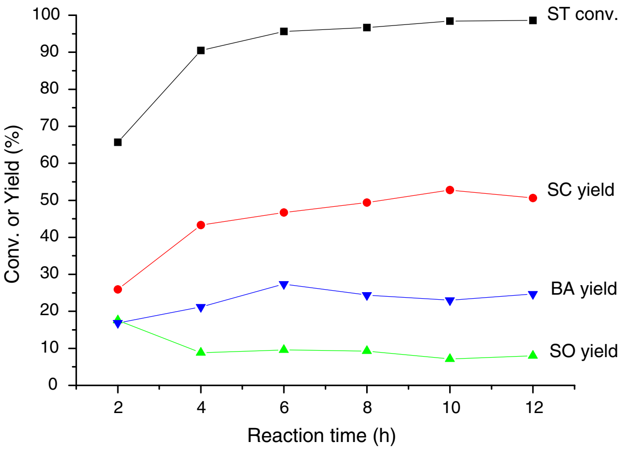
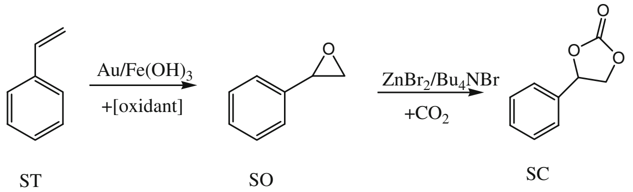


Fig. 5 Reaction time versus styrene conversion and product distributions. Reaction conditions: styrene, 17.3 mmol; anhydrous TBHP, 25.4 mmol; 4.67 wt% Au/Fe-U, 0.1 g; $ZnBr_2$, 0.44 mmol; Bu_4NBr, 0.88 mmol; 80 °C



Scheme 2 Reaction pathways for one-pot synthesis of styrene carbonate (*SC*) from styrene (*ST*) and CO_2

3.4 Consideration of Active Sites in Supported Au/Fe-U Catalyst

As reported in our previous study [36] (Scheme 2), in three-component catalyst system, supported gold was active for the epoxidation (first step), $ZnBr_2$/Bu_4NBr cooperatively catalyzed the CO_2 cycloaddition to epoxide (second step). In order to obtain more information on active sites of Au/Fe-U catalyst, various catalysts with different chemical states of Au and Fe were checked for the epoxidation of styrene, and obtained results are presented in Table 4.

With the same support of $Fe(OH)_3$ but different oxidation states of Au, the activity decreased by 20%, while the selectivity to SO almost unchanged (entries 1, 2), however, with the same cationic gold but different supports (entries 1, 3), both the activity and selectivity decreased markedly over Fe_2O_3 support, compared with those over $Fe(OH)_3$ support. Obviously, both Au and Fe chemical states strongly influenced styrene conversion and SO selectivity (entry 4). These results suggest that the synergistic effect of

Table 4 Catalytic results of styrene epoxidation over different catalysts

Entry	Sample	Conversion (%)	Selectivity (%)			
			SO	BA	PA	BC
1	Au/Fe-U	83.3	66.6	30.9	1.0	1.5
2	$Au^0/Fe(OH)_3$	63.2	68.9	22.7	5.5	2.9
3	Au^{3+}/Fe_2O_3	67.6	49.0	35.0	4.7	11.4
4	Reduced Au/Fe-U by H_2	32.9	37.8	41.6	9.7	10.9

Reaction conditions: catalyst (Au 4.67 wt%), 0.1 g; styrene, 10 mmol; anhydrous TBHP, 15 mmol; 80 °C; 3 h

SO styrene epoxide; *BA* benzaldehyde; *PA* phenylacetaldehyde; *BC* benzenecarboxylic acid cationic Au^{3+} and $Fe(OH)_3$ support might be important for catalytic epoxidation of styrene. Possibly, the synergistic effect was resulted from an effective interaction between Au species and OH groups or defects in the amorphous $Fe(OH)_3$ surface [52].

4 Conclusion

We have shown that the catalyst with Au^{3+} ions supported on ferric hydroxide is very active for styrene epoxidation. When the above gold catalyst is combined with $ZnBr_2$/Bu_4NBr, the one-pot synthesis of styrene carbonate from styrene could be efficiently proceeded. This process disclosed here represents a simple, cost-effective synthesis route for styrene carbonate. In this reaction, CO_2 acts as both a reagent and a solvent. Since the chemical composition and state of Au/Fe-U are completely different from that of Au/Fe-3, Au/Fe-5, and its reduction sample by H_2, it could be conjectured that direct synthesis styrene carbonate was highly sensitive to such changes. Possibly, synergistic effect of supported gold ions and ferric hydroxide support over Au/Fe-U is important for its better catalytic performance for styrene epoxidation.

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