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Novel catalytic systems containing *n*-BuSn(O)OH for the transesterification of dimethyl carbonate and phenol

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Abstract

Novel catalytic systems containing *n*-butylstannonic acid (*n*-BuSn(O)OH) were used to synthesize methyl phenyl carbonate (MPC) and diphenyl carbonate (DPC) by the transesterification of dimethyl carbonate (DMC) with phenol in atmospheric conditions. The catalytic system consisting of *n*-BuSn(O)OH and cuprous iodide (CuI) had the highest activity for the transesterification. When the reaction was carried out between 150 and 180 °C, with a molar ratio of *n*-BuSn(O)OH to CuI of 1:1, a catalyst amount 0.005 (molar ratio to DMC), a reaction time 10 h, a molar ratio of DMC to phenol of 2:1, a dropping time of DMC 6 h, the conversion of phenol was 67.2%, and the selectivity of MPC and DPC was 40.2 and 59.6%, respectively. Upon comparison of the activities of Cu_2O, CuX (X = Cl, Br, I) and the catalytic system being composed of *n*-BuSn(O)OH and Cu_2O/CuX, it was found that Cu_2O and CuX were excellent promoters, and the stronger was the anion electronegativity in the promoter, the weaker was the promoting catalysis of the promoter.

Keywords: *n*-Butylstannonic acid; Transesterification; Dimethyl carbonate; Cuprous iodide; Promoting synthesis

1. Introduction

Polycarbonates (PCs) with good mechanical and optical properties are commercially produced by the interfacial polycondensation of biphenol-A with phosgene or the melt transesterification of diphenyl carbonate (DPC) with bisphenol-A [1]. In recent years, the need has arisen for a safe and environmentally benign process for PCs synthesis. The melt transesterification seems to be more practical process. In this process, the synthesis of intermediate DPC is the key.

The synthetic methods of DPC include phosgene processes, oxidative carbonylation of phenol and transesterification of dimethyl carbonate (DMC) with phenol [2,3]. The transesterification of phenol with DMC is considered to the most suitable method for commercial production.

Usually, the catalysts in the transesterification of DMC and phenol include homogeneous and heterogeneous catalysts. Although heterogeneous catalysts are easily separated from the reaction mixture and recovered for reuse, disadvantages of low activities and easy deactivation limit their use in industry [4–7]. Among the homogeneous catalysts, such as Lewis acids, titanium esters, organotin compounds and so on [3,8], titanium esters and organotin compounds are highly active. Titanium esters, however, is sensitive to water and air in the system, and easily deactivated. Organotin compounds can overcome the problems caused by titanium esters, so organotin compounds have received considerable attention [9]. Yao and co-operaters [10] reported that the conversion of phenol was 2.6% in the presence of $Bu_2Sn(OC(O)C_{11}H_{23})_2$, while the total selectivity of methyl phenyl carbonate (MPC) and DPC was 100%. Yoshihisa and Yuichi [11] reviewed that $ClBu_2SnOSnBu_2Cl$ was the best catalyst among $XBu_2SnOSnBu_2Y$ (X, Y = Cl, OH, SCN, OCH_3, $OC(O)CH_3$) catalysts: the conversion of phenol was 13.38% with 99.3% total selectivity toward MPC and DPC. Wang and co-operaters [12] prepared a novel dimeric tetraorganodistannoxane $[Bu_2Sn(OPh)OSnBu_2(OPh)]_2$. The conversion of phenol was up to 23.9%, and the selectivity of MPC and DPC was 100% on it. However, the activities of these single homogeneous catalysts are not high enough for the industrial purpose.

Lee and cooperaters [13] reported the catalytic system consisting of *n*-Bu_2SnO and an alkyl/aryl sulfonic acid was highly efficient catalytic system: the DMC conversion reached 58.5%,

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and the selectivity was 99.3% for MPC and DPC when the mixture of n-Bu_2SnO and CF_3SO_3H was used as the catalyst in the presence of molecular Sieves (4A) under the high-pressure condition.

During the course to improve the catalytic performance of tin compounds, we have found that n-BuSn(O)OH exhibited higher activity than n-Bu_2SnO, producing MPC and DPC in high yield. Sulfonic acids and carboxylic acids are active catalysts for the transesterification of DMC and phenol, and cuprous chloride (CuCl) is the active component for the oxidative carbonylation of methanol to DMC, so we thought that a catalytic system consisting of n-BuSn(O)OH and a sulfonic acid/carboxylic acid/CuCl would also be the active catalysts in this reaction.

Here, we reported a highly efficient catalytic system consisting of n-BuSn(O)OH and a cuprous compound for the transesterification of DMC and phenol in the atmosphere pressure.

2. Experimental

2.1. Chemical reagent

n-BuSn(O)OH was obtained from Merck Chemical Co. n-Bu_2SnO was purchased from Aldrich Chemical Co. The other chemicals with A.R. purity were used as received from local manufactures without further purification.

2.2. Reaction procedure

The reaction was carried out in a 100 mL glass flask equipped with a thermometer, a nitrogen inlet, a dropping funnel and a fractionating column connected to a liquid dividing head. Phenol and catalyst were charged into the flask under a nitrogen atmosphere. After the mixture was heated to 176 °C, DMC was added drop-wise, and the reaction was continued for a certain period of time. During the reaction, a distillate of DMC and methanol was collected slowly in a receiver flask attached to the liquid dividing head and for analysis. After the reaction, the mixture was cooled to room temperature. The catalyst was filtrated and then the filtrate was analyzed by gas chromatograph.

2.3. Product analysis

Qualitative and quantitative analysis of reaction products and distillates of DMC and methanol were carried out on a HP 6890/5973 GC–MS spectrometer and a Shimadzu GC-14B gas chromatograph equipped with a SE-30 capillary column and flame ionization detector (FID), respectively.

3. Results and discussion

3.1. Activities of various catalytic systems for the transesterification reaction

In order to make a comparison, the activities of n-Bu_2SnO, n-BuSn(O)OH and other catalytic systems were tested in a refluxing batchwise reactor. The results are listed in Table 1. The activity of the catalyst is evaluated by the conversion of DMC.

As presented in Table 1, n-Bu_2SnO gave the low yields of MPC and DPC (entry 1). However, when equimolar p-$CH_3C_6H_4SO_3H$ or $C_6H_5SO_3H$ was used in conjunction with n-Bu_2SnO, the activity of n-Bu_2SnO increased greatly (entries 1, 2 and 3). The reason for the enhancement of the catalytic activity can be ascribed to the electrophilicity of Sn. During the reaction, $[n\text{-}Bu_2Sn(OH)(O_3SC_6H_4CH_3)(H_2O)]_2$ and $[n\text{-}Bu_2Sn(OH)(O_3SC_6H_5)(H_2O)]_2$ could be in situ synthesized from n-Bu_2SnO and corresponding sulfonic acid. Owing to the electron-withdrawing character of the sulfonate group, the Sn atom becomes more electronphilic in $[n\text{-}Bu_2Sn(OH)(O_3SC_6H_4CH_3)(H_2O)]_2$ or [n-$Bu_2Sn(OH)$

Table 1
Activities of various catalytic systems for the transesterification of DMC and phenol

Entry	Catalyst	DMC conversion (%)	Yield (%)			Total selectivity (%)[c]
			AN[b]	MPC	DPC	
1	n-Bu_2SnO[a]	29.7	–	27.6	2.2	99.9
2	n-Bu_2SnO, $CH_3C_6H_4SO_3H$	34.4	–	28.5	5.8	99.9
3	n-Bu_2SnO, $C_6H_5SO_3H$	42.5	–	35.4	7.1	99.9
4	n-BuSn(O)OH	44.0	–	37.8	6.2	99.9
5	n-BuSn(O)OH, $CH_3C_6H_4SO_3H$	28.5	–	21.5	7.1	99.9
6	n-BuSn(O)OH, $C_6H_4SO_3H$	25.3	–	17.7	7.6	99.9
7	$C_5H_5FeC_5H_4COOH$	0	0	0	0	–
8	$(n\text{-}BuSn(O)O_2CC_5H_4FeC_5H_5)_6$	50.5	–	34.0	16.5	99.9
9	n-BuSn(O)OH, $CpFeC_5H_4COOH$	48.9	–	31.7	17.2	99.9
10	n-BuSn(O)OH, $ClCH_2COOH$	49.9	–	36.4	13.5	99.9
11	n-BuSn(O)OH, Cl_3CCOOH	55.1	–	40.7	14.4	99.9
12	n-BuSn(O)OH, CuCl	51.3	–	37.8	13.5	99.9
13	n-BuSn(O)OH, CuI	57.8	–	39.2	18.6	99.9

Reaction conditions: DMC (0.15 mol), phenol (0.3 mol), reaction time 8 h (including DMC dropping time 3 h), temperature 150–180 °C, n(catalyst):n(DMC) = 0.0226:1, n(Sn):n(other component) = 1:1.

[a] n-Bu_2SnO (5.43 mmol).

[b] AN: anisole.

[c] Total selectivity is the sum of the selectivity of MPC and DPC.

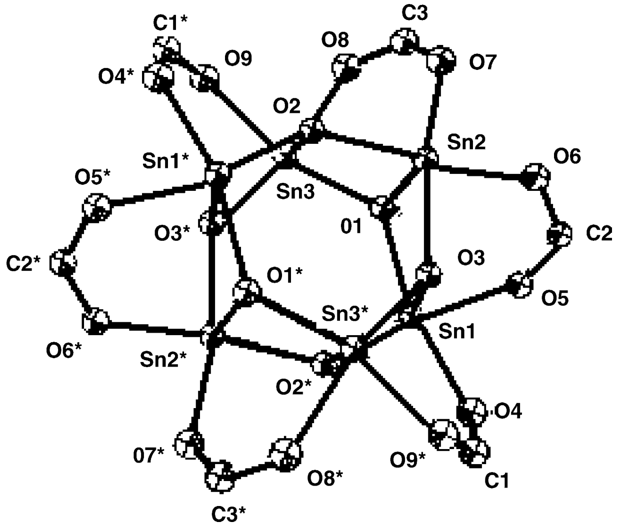


Fig. 1. ORTEP diagram of $[n\text{-BuSn(O)}O_2CC_5H_4FeC_5H_5]_6$ showing the central stannoxane core along with the connecting carboxylate unite.

$(O_3SC_6H_5)(H_2O)]_2$ than in n-Bu_2SnO, thereby coordination of DMC to electrodeficient Sn center can take place more easily, further activating the carbonyl group of DMC toward the nucleophilic attack by phenol.

As was clear from Table 1, n-BuSn(O)OH exhibited higher activity than n-Bu_2SnO (entries 1 and 4). However, as distinguished from the performance of the catalytic system of n-Bu_2SnO and sulfonic acid being superior to that of n-Bu_2SnO, the catalytic system of n-BuSn(O)OH and sulfonic acid gave the lower activity than n-BuSn(O)OH (entries4, 5 and 6). The previous literature [14] reported that a dodecanuclear organotin cluster $\{(BuSn)_{12}(\mu_3\text{-O})_{14}(\mu_2\text{-OH})_6\}^{2+}$ $(O_3SC_6H_4CH_3\text{-}p)_2{}^-$ could be prepared from n-BuSn(O)OH and p-$CH_3C_6H_4SO_3H$. The cation $\{(BuSn)_{12}(\mu_3\text{-O})_{14}(\mu_2\text{-OH})_6\}^{2+}$ of the cluster is like a *football* covered by butyl groups; the anion (p-$CH_3C_6H_4SO_3{}^-$) is not bonded to the Sn atom, but the bridging hydroxyl groups of the dication, only acting as the charge balance. Compared with n-BuSn(O)OH, the electrophilicity of Sn atom in this cluster is not strengthened, but the steric hindrance arising from the butyl group is far higher. So the activity of the catalyst decreased with in situ synthesis of the cluster during the reaction.

Interestingly, ferrocene monocarboxylic acid had no activity (entry 7), but the hexameric drum compound $[n\text{-BuSn(O)}O_2CFc]_6$ [15], which was synthesized from n-BuSn(O)OH and ferrocene monocarboxylic acid, showed higher activity than n-BuSn(O)OH (entries 4 and 8). The higher activity might be ascribed to its unique structure (as shown in Fig. 1): the basic framework of the drum compound is made up of two hexameric Sn_3O_3 rings which are joined to each other to afford six Sn_2O_2 distannoxane units as its side face, and the center is a chunnel. The structure is of benefit to DMC's inset and activation. In addition, the ferrocene monocarboxylate moiety is involved in binding to two alternate Sn atoms of a distannoxane unit at the side face, and the various drum compounds can be prepared from stannonic acids and carboxylic acids [16–19]. That offers a method of adjusting the electronphilicity of Sn by changing the substituent on the carboxylic acid. In the entries 9, 10 and 11, the trichloromethyl group is the most electron-withdrawing group, so the activity was the highest (the conversion of DMC reached 55.1%, the yields of MPC and DPC being 40.7 and 14.4%, respectively).

As mentioned above, a catalyst based on appropriate multi-component mixture exhibits a better performance than single compound. When we used CuCl or CuI as an additive, we delightedly found CuCl and CuI could promote catalytic performance of n-BuSn(O)OH. Especially, the catalytic system consisting of n-BuSn(O)OH and CuI gave the highest activity: the conversion of DMC was up to 57.8%, and the total selectivity of MPC and DPC was over 99%. Therefore, the effect of molar ratio of CuI to n-BuSn(O)OH and effect of various factors on the transesterification of DMC with phenol were discussed in detail, respectively, in the following text.

3.2. Effect of the molar ratio of CuI to n-BuSn(O)OH on the transesterification reaction

Apparently, as shown in Table 1 (entries 4, 13), the catalytic activity of n-BuSn(O)OH was highly improved by the CuI additive. So, the effect of the molar ratio of CuI to n-BuSn(O)OH on the catalytic activity was studied under atmospheric condition. The results are summarized in Table 2.

Compared with no CuI added, the conversion of DMC and yield of DPC were promoted remarkably when a trance amount of CuI (the molar ratio of CuI to n-BuSn(O)OH is 0.063:1) was added to the reaction system, especially the yield of DPC increased sharply from 6.2 to 15.6%. With CuI:n-BuSn(O)OH molar ratio's rise from 0.25:1 to 1:1, the conversion of DMC was gradually improved. Above 1:1, however, DMC conversion and yields of MPC and DPC remained approximately constant. The proper molar ratio of CuI to n-BuSn(O)OH was 1:1.

3.3. Effect of catalyst amount on the transesterification reaction

The conversion of DMC and yields of MPC and DPC changing with the molar ratio of n-BuSn(O)OH (n(CuI):n(n-BuSn(O)OH) = 1:1) to DMC in the transesterification are shown in Fig. 2.

When catalyst amount (molar ratio to DMC) was only 0.0005, DMC conversion was up to 43.0% with nearly 100% selectivity of MPC and DPC. Thereafter, with a rise of catalyst amount from 0.001 to 0.005, DMC conversion increased slightly from 50.7 to 57.1%. Simultaneously, the yields of MPC and DPC were improved from 36.3 and 14.4 to 37.3 and 19.8%, respectively. Above 0.005, however, DMC conversion and yields of MPC and DPC remained almost unchanged. Throughout various catalyst amount, no anisole was obtained. So optimal catalyst amount should be 0.005. As shown in Fig. 1, the high yields of MPC and DPC can be obtained at low catalyst amount using the catalytic system consisting of n-BuSn(O)OH and CuI, while a large amount of conventional catalysts including titanium esters and n-Bu_2SnO is required in order to reach the maximum catalytic activity (usually the catalyst amount is 0.01–0.05 of phenol or

Table 2
Effect of the molar ratio of CuI to *n*-BuSn(O)OH on the transesterification

Entry	Ratio *n*(CuI):*n*(BuSnOOH)	DMC conversion (%)	Yield (%)			Total selectivity (%)
			AN	MPC	DPC	
1	2:1	58.0	–	36.5	21.5	99.9
2	1.5:1	57.0	–	36.2	20.8	99.9
3	1:1	57.8	–	39.2	18.6	99.9
4	0.5:1	55.7	–	33.7	22.0	99.9
5	0.25:1	54.2	–	33.5	20.7	99.9
6	0.063:1	52.6	–	36.0	15.6	99.9
7	0:1	44.0	–	37.8	6.2	99.9

Reaction conditions: DMC (0.15 mol), phenol (0.3 mol), *n*-BuSn(O)OH (3.39 mmol), reaction time 8 h (including dropping time 3 h), temperature 150–180 °C.

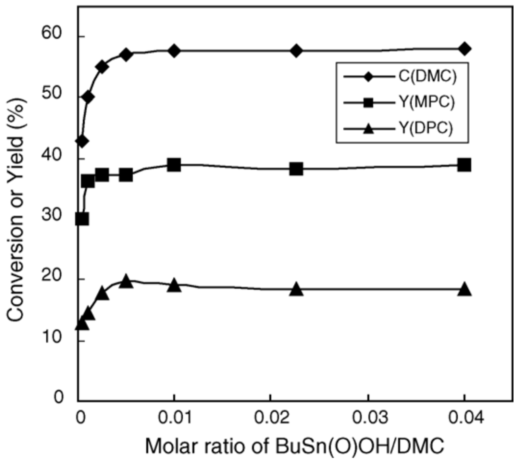


Fig. 2. Effect of molar ratio of BuSn(O)OH/DMC on the transesterification. Reaction conditions: DMC (0.15 mol), phenol (0.3 mol), *n*(*n*-BuSn(O)OH):*n*(CuI) = 1:1, reaction time 8 h (including dropping time 3 h), temperature 150–180 °C.

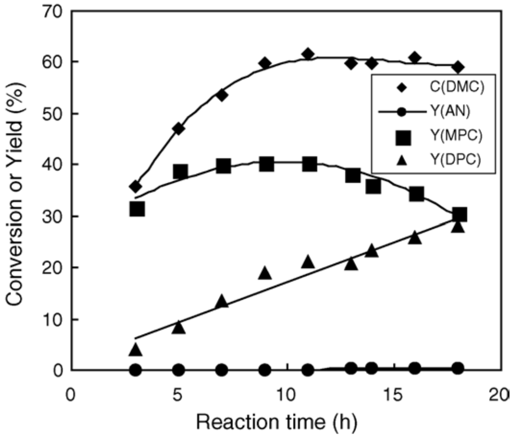


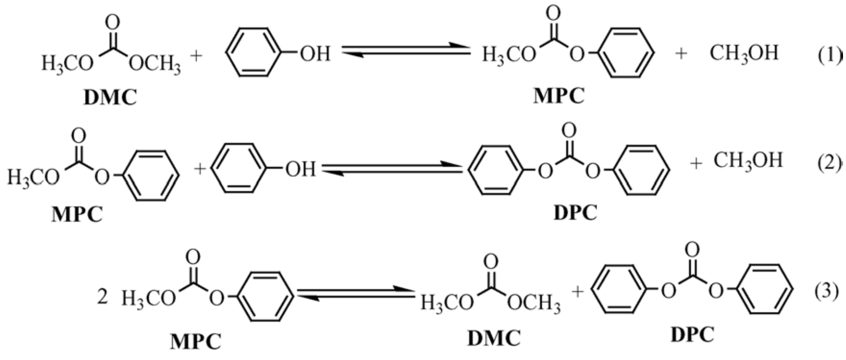
Fig. 3. Effect of reaction time on the transesterification. Reaction conditions: DMC (0.15 mol), phenol (0.3 mol), *n*-BuSn(O)OH (0.75 mmol), CuI (0.75 mmol), dropping time 3 h, temperature 150–180 °C.

DMC) [8,10,13]. So the catalytic system is economic for the transesterification.

3.4. *Effect of reaction time on the transesterification reaction*

The conversion of DMC and the yields of MPC and DPC were also monitored with reaction time using the catalytic system consisting of *n*-BuSn(O)OH and CuI. The results are given in Fig. 3.

In general, the yield of DPC was enhanced in straight line; the yield of MPC increased up to 40.6% with the reaction time and then decreased slightly; the conversion of DMC was improved gradually to 61.5% with time and then remained approximately constant. The transesterification of DMC and phenol includes three equilibrium reactions (as shown in Scheme 1). Originally, the reaction mixture had the high concentration of DMC and low concentrations of MPC and DPC. Methanol produced during the reaction could be removed in time by the binary azeotrope of DMC and methanol via the fractionating column, so the reaction 1 and 2 were very fast, that is, the conversion of DMC and the yields of MPC and DPC were continually enhanced with time.



Scheme 1. Transesterification of DMC with phenol to DPC.

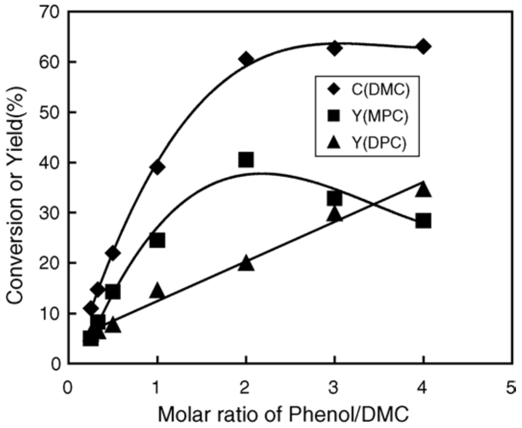


Fig. 4. Effect of molar ratio of phenol to DMC on the transesterification. Reaction conditions: DMC (0.15 mol), *n*-BuSn(O)OH (0.75 mmol), CuI (0.75 mmol), reaction time 10 h (including dropping time 3 h), temperature 150–180 °C.

Above 11 h, however, with the sharp decrease of concentration of DMC, not all methanol formed in reaction could be eliminated from the system by the binary azeotrope. The reaction 1 and 2 were slow, while the reaction 3, the disproportionation of MPC, was dominant. So the yield of MPC decreased, the yield of DPC ceaselessly increasing, while the conversion of DMC remained approximately constant. Unfortunately, after 11 h of reaction, anisole was detected in the level of 0.2–0.4%. These results indicate that the optimal reaction time for the transesterification is 9–11 h.

3.5. Effect of molar ratio of phenol to DMC on the transesterification reaction

The effect of molar ratio of phenol/DMC on the transesterification was examined in the range 0.25–4 in the presence of the catalytic system containing *n*-BuSn(O)OH and CuI. The results are shown in Fig. 4 (the yield is based on DMC) and Fig. 5 (the yield is based on phenol).

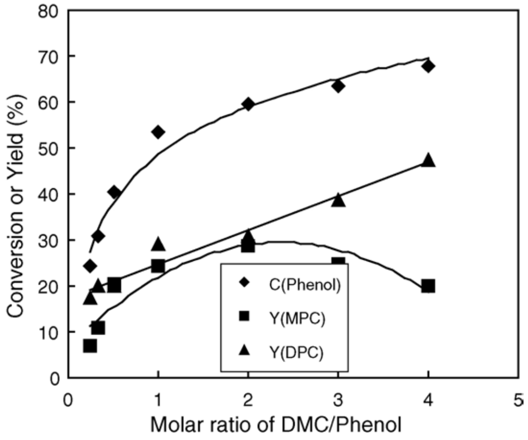


Fig. 5. Effect of molar ratio of phenol to DMC on the transesterification. Reaction conditions: DMC (0.15 mol), *n*-BuSn(O)OH (0.75 mmol), CuI (0.75 mmol), reaction time 10 h (including dropping time 3 h), temperature 150–180 °C. The yield is based on phenol.

It could be seen from Fig. 4 that the conversion of DMC and yields of MPC and DPC were fairly low when the molar ratio of phenol/DMC was below 1:1. This may be ascribed to the evaporation of DMC with methanol under the reaction conditions. When the ratio of phenol/DMC was increased from 1:1 to 2:1, the DMC conversion increased greatly to 60.6%, but thereafter did not change much. In addition, with the rise of the molar ratio of phenol/DMC, MPC yield was up to the maximum value (40.6%) at the molar ratio of phenol/DMC = 2:1, then decreased, whereas the yield of DPC kept increasing. This demonstrates that the excess phenol is in favor of the formation of DPC.

Due to the high boiling point of phenol (181.6 °C), it was difficult for phenol to separate from MPC and DPC, thereof the effect of molar ratio of DMC/phenol on the transesterification was studied on the basis of phenol (as shown in Fig. 5). From Fig. 5, the tendency shown in Fig. 5 was similar to that shown in Fig. 4. Considering the utilization of DMC, the proper molar ratio of DMC to phenol was 2:1.

3.6. Effect of the dropping time of DMC on the transesterification reaction

In the transesterification, DMC possesses dual functions: the reactant and the azeotrope. However, the excess DMC in the reaction mixture will result in the low reaction temperature and low conversion of phenol. So the effect of dropping time of DMC on the transesterification was studied. The results are summarized in Fig. 6. It was evident from Fig. 6 that the conversion of phenol increased 5.3% when dropping time varied from 3 to 4 h. But the phenol conversion only increased 2.5% when dropping time was prolonged from 4 to 6 h, so 6 h dropping time was feasible. Under this condition, the phenol conversion reached 67.2%, and the yields of MPC and DPC were 27.1 and 40.0%, respectively.

3.7. Role of CuI in the transesterification reaction

In order to understand the role of CuI in the transesterification of DMC with phenol, we studied the effect of Cu_2O,

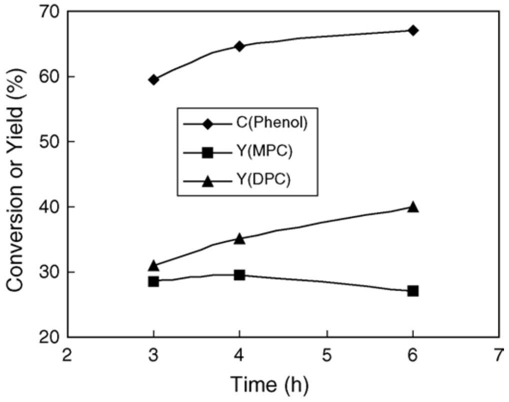


Fig. 6. Effect of dropping time on the transesterification. Conditions: DMC (0.20 mol), phenol (0.10 mol), *n*-BuSn(O)OH (1.00 mmol), CuI (1.00 mmol), reaction time 10 h, temperature 150–180 °C. The yield is based on phenol.

Table 3
Effect of different catalysts on the transesterification of DMC and phenol to DPC

Entry	Catalyst		DMC conversion (%)	Yield (%)[a]			Total selectivity (%)
				AN	MPC	DPC	
1	Cu_2O	–	0	0	0	0	–
2	CuCl	–	0	0	0	0	–
3	CuBr	–	0	0	0	0	–
4	CuI	–	0	0	0	0	–
5	BuSn(O)OH	Cu_2O	50.8	–	34.0	16.8	99.9
6	BuSn(O)OH	CuCl	51.3	–	37.8	13.5	99.9
7	BuSn(O)OH	CuBr	52.8	Trance	38.2	14.3	99.4
8	BuSn(O)OH	CuI	57.8	–	39.2	18.6	99.9

Reaction conditions: DMC (0.15 mol), phenol (0.3 mol), *n*-BuSn(O)OH (3.39 mmol), promoter(3.39 mmol), reaction time 8 h (including dropping time 3 h), temperature 150–180 °C.

[a] The yield is based on DMC.

CuX (X = Cl, Br, I) and the catalytic system consisting of *n*-BuSn(O)OH and Cu_2O/CuX on the transesterification, respectively (as listed in Table 3). Cu_2O and CuX had no activity, *n*-BuSn(O)OH only giving 44.0% conversion of DMC. However, when Cu_2O or CuX was used in conjunction with *n*-BuSn(O)OH, the activity of *n*-BuSn(O)OH increased greatly: the conversion of DMC was over 50%, and the total selectivity of MPC and DPC was close to 100%. In combination with these results, we are led to a conclusion that Cu_2O and CuX are excellent promoters in the presence of *n*-BuSn(O)OH.

The promoting catalysis of Cu_2O and CuX, as shown in Table 3, increased in the following order: $Cu_2O \sim CuCl < CuBr < CuI$, while the electronegative order of oxygen and haloid atoms in the promoters is $I < Br < Cl < O$. The result implies that the anion electronegativity in the promoter is relative to its promoting catalysis: the stronger is the anion electronegativity in the promoter, the weaker is its promoting catalysis.

4. Conclusions

Upon comparison of activities of *n*-Bu_2SnO, *n*-BuSn(O)OH and other catalytic systems and total selectivity toward MPC and DPC in the transesterification of DMC with phenol, it was found that the catalytic system consisting of *n*-BuSn(O)OH and CuI was the best. When the reaction was carried out between 150 and 180 °C under atmospheric condition, with a molar ratio of *n*-BuSn(O)OH to CuI of 1:1, a catalyst amount 0.005 (molar ratio to DMC), a reaction time 10 h, a molar ratio of DMC to phenol of 2:1, a dropping time of DMC 6 h, the conversion of phenol was 67.2%, and the yields of MPC and DPC were 27.1 and 40.0%, respectively. The formation of the by-product anisole could be suppressed to a great extent.

Two facts, the inactivities of Cu_2O and CuX (X = Cl, Br, I) and the high performance of the catalytic system consisting of *n*-BuSn(O)OH and Cu_2O/CuX, proved that Cu_2O and CuX were excellent promoters. Combining the order of their promotional catalysis ($Cu_2O \sim CuCl < CuBr < CuI$) with the anion electronegativity in promoters, it was pointed out that the stronger was the anion electronegativity in the promoter, the weaker was the promoting catalysis of the corresponding cuprous compound. Further study for the detail of the novel catalytic systems is now in progress and will be reported separately.

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