Preparation and Characterization of Chloroacetylated Polystyrene-Supported Zinc Complexes and Evaluation of Their Catalytic Activities for the Synthesis of 1,1-Diacetates

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ABSTRACT: Zinc chloride was immobilized onto chloroacetylated polystyrene via different linkages to afford polymer-supported Lewis acid catalysts. The functionalized beads and the immobilized catalysts were characterized by elemental analysis, Fourier transform infrared spectroscopy, X-ray photoelectron spectroscopy, and inductively coupled plasma and thermal analysis. The reaction of aldehydes with anhydride in the presence of the supported catalysts was investigated. The effects of various parameters, including the solvent, amount of catalyst, and temperature, on the reaction were also studied. We found that the reaction proceeded smoothly under solvent-free conditions at room temperature and afforded the corresponding acylals in good yields. Moreover, the catalysts could be recovered easily and reused several times without a significant loss in activity. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 112: 2087–2093, 2009

Key words: functionalization of polymers; metal–polymer complexes

INTRODUCTION

In catalytic reactions, the easy handling of the catalyst together with its straightforward recovery and possible reuse remain an important topic.[1] A widespread solution for reaching these goals is the application of immobilized catalysts. Crosslinked polymers with specific properties are widely used as catalyst supports as they are inert, nontoxic, and nonvolatile and offer the advantageous features of heterogeneous catalysis, such as thermal stability, selectivity, and recyclability.[2] Recently, the use of polymer-supported catalysts in organic transformations has been receiving extraordinary attention.[3] Nevertheless, one serious drawback often encountered is the leakage of metal from the support to the solution, which results in the decreased activity of the catalyst. This problem can, however, often be eliminated by the use of chelating resins.[4]

The most common method for introducing chelating groups is the functionalization of some active resins, such as Merrifield resin, with chelating ligands. Although a large number of resins containing functional ligands, such as porphyrin,[5,6] Schiff bases,[7,8] diphosphine,[9,10] and diketones,[11,12] have been reported widely, less work has been focused on the support itself. In the literature, most chelating resins have been modified on Merrifield resin. However, the preparation of Merrifield resin has sometimes been problematic with respect to the utilization of carcinogenic chloromethyl methyl ether, polysubstitution, and secondary crosslinking. An advancement was achieved by Elman and Moberg,[13] who prepared chloroacetylated polystyrene resin, which has the same function as Merrifield resin, whereas the preparation process is green and the structure is simple. Also, the chloroacetyl group is stable and unhydrolyzable.[13] Liu et al.[14] optimized the reaction conditions for the preparation of chloroacetylated polystyrene and controlled the loading capacity of the chloroacetyl group, making it applicable for industry processes. However, such a new polymer has not received enough attention, and most polymer modification processes still rest on the Merrifield resin.

The selective protection and deprotection of carbonyl groups are essential steps in modern organic chemistry.[15] 1,1-Diacetates (acylals) are one useful carbonyl-protecting group because of their stability under both neutral and basic media and under acidic conditions.[16] Moreover, the acylal functionality can be converted into other functional groups by reaction with appropriate nucleophiles.[17,18] Some of the reported reagents for the preparation of 1,1-diacetates from aldehydes and acetic anhydride include sulfuric acid,[19] triflic acid,[20] PCl_3,[21] $FeCl_3$,[16] n-bromosuccinimide (NBS),[22] β-zeolite,[23] $LiBF_4$,[24] H_2NSO_3H,[25] $H_3PW_{12}O_{40}/MCM-41$,[26] [Hmim] HSO_4^-,[27] P_2O_5/Al_2O_3,[28] and $Bi(NO_3)_2·5H_2O$.[29] Although some of these methods have convenient protocols with good to high

yields, the majority of these methods suffer from at least one of the following disadvantages: reaction under oxidizing conditions, high temperature, or long reaction times; moisture sensitivity of the used reagent; high cost and high toxicity; tedious workup; and environmental pollution.

So, as a part of our program aiming at the development of novel functionalized polymer-supported Lewis acid catalysts, here, we describe the preparation of novel functionalized resins and their zinc complexes based on chloroacetylated polystyrene, and we report a mild and efficient method for the synthesis of 1,1-diacetates.

EXPERIMENTAL

Materials and equipment

Chloroacetylated polystyrene (7% divinylbenzene, 14.6% chlorine, grain size range = 240–280 μm, surface area = 32 m^2/g, pore diameter = 80–100 Å, total pore volume = 0.11 cc/g) was obtained from Nanjing Microspheres Co., Ltd. (Nanjing, China). Diethanolamine (DEA), tetraethylenepentamine, L-phenylalanine, L-proline, aminomethyl phosphoric acid (APA), triethylbenzylammonium chloride (TEBA), anhydrous zinc chloride, and other reagents were purchased from Meryer (Shanghai, China) and were used without further purification.

IR spectra were recorded in KBr disks with a Bomem (Quebec, Canada) MB154S Fourier transform infrared spectrometer. Metal content was measured by inductively coupled plasma (ICP) analysis on a Varian (Palo Alto, CA) AA240 analyzer after the samples were digested in concentrated HNO$_3$. X-ray photoelectron spectroscopy (XPS) analyses were obtained with a PHI-5702/electron spectroscopy for chemical analysis/scanning auger microscopy (SAM) instrument equipped with an MgKα (1253.6 eV) X-ray source. The binding energy (BE) of the C$_{1s}$ peak at 284.8 eV was taken as an internal standard. Thermogravimetric analyses were carried out on a Shimadzu (Kyoto, Japan) DT-30 instrument at a heating rate of 20°C/min under an atmosphere of nitrogen. ^{1}H-NMR spectra were recorded with a Bruker (Rheinstetten, Germany) Advance RX300 analyzer. Elemental analyses were performed on a Yanagimoto (Kyoto, Japan) MT3CHN recorder. All acylals were known compounds and were identified by comparison of their physical and spectral data with those of authentic samples.

Amination of the chloroacetylated polystyrenes (1a–1b)

Prewashed chloroacetylated polystyrene beads (1.0 g, 4.1 mmol of Cl) were swollen in 30 mL of dioxane overnight; then, 16.4 mmol of amine (DEA, tetrae-

thylenepentamine) was added, and the mixture was stirred and refluxed for 12 h in the presence of pyridine (1.30 g, 16.4 mmol). Then, the beads were filtered and washed thoroughly with water and methanol and dried at 80°C *in vacuo* overnight.

Preparation of the APA (1c) and amino acid (1d–1e) functionalized chloroacetylated polystyrenes

Prewashed chloroacetylated polystyrene beads (1.0 g, 4.1 mmol of Cl) were swollen in 30 mL of 1,2-dichloroethane for 12 h; then, 10 mmol of amino acid or APA, 0.2 g of TEBA, and 10 mL of 25% sodium hydroxide solution were added subsequently, and the mixture was stirred for 8 h at 90°C. After the mixture was cooled to room temperature (RT), the pH was adjusted to 5.0 with a 5% HCl solution, and the color of the beads changed to yellow. Then, the beads were filtered and washed thoroughly with water, a 5% HCl solution, and methanol and dried at 80°C *in vacuo* overnight.

Preparation of the polymer-supported zinc complexes

The functionalized polymer beads (1.0 g) obtained previously were kept in contact with methanol (10 mL) for 2 h. A methanolic solution (30 mL) of zinc chloride (4 mmol) was added to the polymer suspension. The mixture was heated under reflux and stirred for 12 h. Then, the resulting beads were filtered off, washed with water (20 mL × 3) and methanol (20 mL × 3) and dried at 80°C *in vacuo* overnight.

General procedure for the conversion of aldehydes to 1,1-diacetates

A mixture of aldehyde (1 mmol), acetic anhydride (5 mmol), and catalyst (5 mol %) was stirred at RT for the appropriate time indicated in Table IV. After the completion of the reaction (monitored by thin-layer chromatography, ethyl acetate (EtOAc)*n*-hexane = 1:9), the mixture was diluted with ether, and the catalyst was removed by filtration. The organic layer was washed with a 10% NaHCO$_3$ solution, dried over anhydrous Na$_2$SO$_4$, and concentrated under reduced pressure to give the corresponding pure products. The spectral data of the corresponding 1,1-diacetates are listed next.

1,1-Diacetoxy-1-phenylmethane

^{1}H-NMR (300 MHz, CDCl$_3$, δ): 7.6 (s, 1H), 7.4–7.2 (m, 5H), 1.92 (s, 6H). IR (KBr, cm^{-1}): 3060, 1755, 1605, 1470, 1375, 1245, 1205, 1065, 1010, 760, 700.

1,1-Diacetoxy-1-(4-methylphenyl)methane

^{1}H-NMR (300 MHz, CDCl$_3$, δ): 7.62 (s, 1H), 7.42 (d, J = 8.4 Hz, 2H), 7.22 (d, J = 8.4 Hz, 2H), 2.4 (s, 3H),
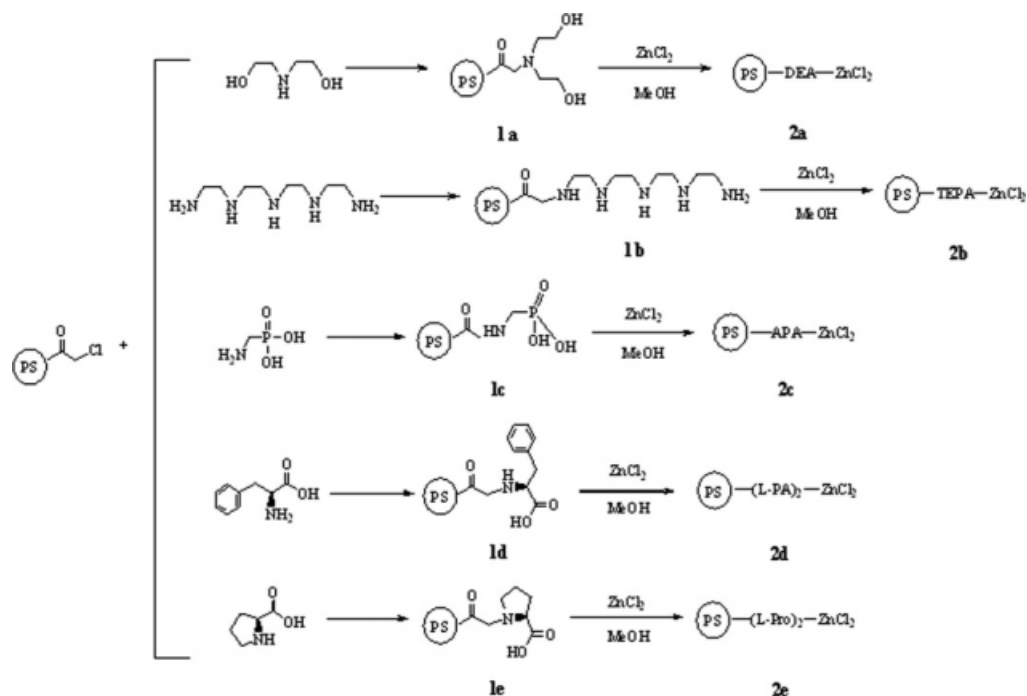
Scheme 1 Preparation of the functionalized chelating resins and their zinc complexes (PS = polystyrene).

2.15 (s, 6H). IR (KBr, cm^{-1}): 3033, 2360, 1771, 1367, 1241, 1206, 1068, 1006, 959, 816.

1,1-Diacetoxy-1-(4-methoxyphenyl)methane

^{1}H-NMR (300 MHz, CDCl$_3$, δ): 7.40 (s, 1H), 7.35 (d, $J = 9$ Hz, 2H), 6.75 (d, $J = 9$ Hz, 2H), 3.80 (s, 3H), 2.10 (s, 6H). IR (KBr, cm^{-1}): 3033, 1763, 1614, 1519, 1372, 1241.

1,1-Diacetoxy-1-(4-nitrophenyl)methane

^{1}H-NMR (300 MHz, CDCl$_3$, δ): 8.1 (d, $J = 9$ Hz, 2H), 7.55 (d, $J = 9$ Hz, 2H), 7.45 (s, 1H), 2.10 (s, 6H). IR

(KBr, cm^{-1}): 3095, 1765, 1610, 1530, 1345, 1210, 1200, 1090, 1000, 960, 850.

1,1-Diacetoxy-1-(4-chlorophenyl)methane

^{1}H-NMR (300 MHz, CDCl$_3$, δ): 7.55 (s, 1H), 7.3–7.2 (s, 4H), 2.1 (s, 6H). IR (KBr, cm^{-1}): 3060, 3020, 1760 (s), 1605, 1480, 1210, 1010, 780, 620.

1,1-Diacetoxy-1-(4-acetoxyphenyl)methane

^{1}H-NMR (300 MHz, CDCl$_3$, δ): 7.5 (s, 1H), 7.4 (d, $J = 8$ Hz, 2H), 6.9 (d, $J = 8$ Hz, 2H), 2.2 (s, 3H), 2.0 (s, 6H). IR (KBr, cm^{-1}): 2960, 1755, 1735, 1010, 780.

TABLE I
IR Spectral Data and Analytical Data of the Polymer-Supported Ligands

Sample	IR (cm^{-1})	Element found (%) N	Conversion (%)	Loading (mmol/g)
1a	3450 (s), 2929 (m), 1665 (s), 1620 (s), 1366 (m), 1254 (w), 1068 (m), 689 (w)	3.49	90.0	3.0
1b	3437 (s), 2942 (m), 1654 (s), 1358 (m), 1226 (w), 1057 (w), 701 (w)	13.31	62.1	1.9
1c	3421 (s), 3110 (m), 2920 (m), 1659 (s), 1642 (m), 1456 (m), 1227 (m), 1079 (m),, 705 (w)	3.36	71.3	2.4
1d	3446 (s), 2923 (m), 1683 (s), 1658 (s), 1479 (m), 1352 (m), 1076 (m), 702 (w)	3.51	87.0	2.5
1e	3440 (s), 2925 (m), 1668 (s), 1647 (s), 1450 (m), 1356 (m), 1082 (m), 707 (w)	3.50	75.9	2.5

1,1-Diacetoxybutane

^{1}H-NMR (300 MHz, CDCl$_3$, δ): 6.50 (m, 1H), 2.4 (brm, 2H), 2.0 (s, 6H), 1.90–1.30 (brm, 2H), 1.00 (t, J = 7 Hz, 3H). IR (KBr, cm^{-1}): 2960, 1765, 1374, 1244, 1208, 1010.

1,1-Diacetoxy-1-(2-furyl)methane

^{1}H-NMR (300 MHz, CDCl$_3$, δ): 7.72 (s, 1H), 7.45 (t, 1H), 6.55 (d, 1H), 6.40 (d, 1H), 2.17 (s, 6H). IR (KBr, cm^{-1}): 3100, 1750, 1500, 1240, 1200, 1060, 935, 830, 750.

RESULTS AND DISCUSSION

Characterization of the functionalized polystyrenes and their zinc complexes

The functionalized resins were readily prepared by the direct reaction of amines, APA, and amino acids with chloroacetylated polystyrenes, as shown in Scheme 1. Earlier methods for anchoring amines or amino acids to Merrifield resin have usually involved a NaI/base system, but the yield is low, and a long time is required.[30] In this study, the amination of chloroacetylated polystyrene was conducted in the presence of pyridine, and the products were obtained in moderate to good yields (62.1 and 90.0%, respectively). However, such a system did not work when APA and amino acids were used as substrates. Preliminary studies showed that the conversion of chloroacetylated polystyrene was increased by the use of NaOH as a base and TEBA as a phase-transfer catalyst. The elemental analysis and IR spectra data of the functionalized resins are given in Table I.

As shown in Table I, the elemental analysis data of nitrogen indicated the attachment of ligands onto the support. In the spectra of all resins, strong bands at 3400–3500 cm^{-1} were assigned to —NH (or —OH) vibrations. Medium-intensity peaks due to C—N stretching appeared at about 1080 cm^{-1}. For amino acids, the characteristic absorptions due to V_{asym} (asymmetric vibration) COO$^-$ and V_{sym} (symmetric vibration) COO$^-$ of carboxylic groups appear at about 1600 and 1400 cm^{-1}. All these changes indicated that
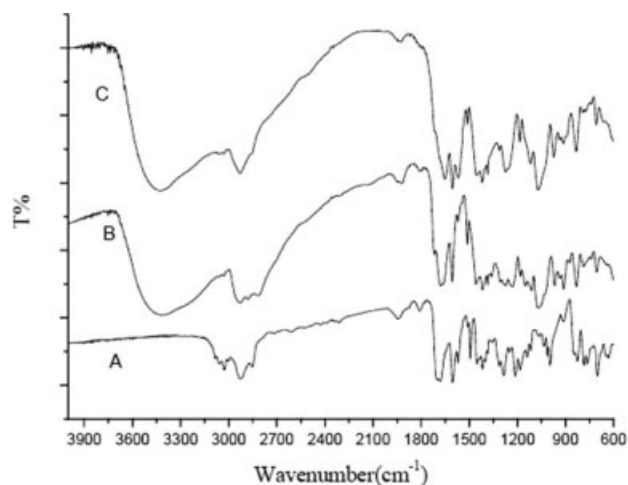

Figure 1 IR spectra of the (A) chloroacetylated polystyrene, (B) DEA-functionalized resin, and (C) supported catalyst.

the ligands were attached to the chloroacetylated polystyrene successfully. The physical and analytical data of the supported catalysts are summarized in Table II. It is obvious that the ligands had a strong influence on the loading capacity and the ratios of metal to ligand. The amino resins showed good affinities to zinc chloride, whereas relatively lower metal loadings and metal/ligand ratios were obtained when APA and amino acids resins were used as supports.

The success of the attachment of ligands onto the support and the formation of the metal complexes was also confirmed by Fourier transform infrared spectroscopy and XPS analysis, with resin **1a** and catalyst **2a** used as examples (Fig. 1 and Table III).

As shown in Figure 1, the C—Cl peaks at 1285 and 700 cm^{-1} in the starting polymer were practically absent or weakened after the introduction of diethanol amine on the support. Strong and broad bands at 3450 cm^{-1} in curves B and C were assigned to —OH vibration, and the sharp peaks at 1100–1200 cm^{-1} were attributed to the vibration of the bond of C—N. Moreover, sharp peaks at about 1650–1700 cm^{-1} in the three curves were attributed to the vibration of C=O. Although it was difficult to tell whether the peak of v(—OH) changed after the introduction of zinc

TABLE II
Physical and Analytical Data of Supported Catalysts 2a–2e

Catalyst	Color	Loading (mmol/g)^a	Metal/ligand ratio (mol/mol)^b
2a	Pink	1.52	0.65
2b	Brown	2.12	1.52
2c	Brown	1.47	0.76
2d	Yellow	0.98	0.47
2e	Yellow	1.23	0.59

^{a} Determined by ICP.
^{b} Caculated according to ICP and elemental analysis.

TABLE III
XPS Data of ZnCl$_2$ and Resin 1a and Its Zinc Complex

	BE (eV)			
XPS peak	ZnCl$_2$–ligand complex			ΔE_b (eV)
Zn$_{2p3/2}$	1023.1	—	1022.0	−1.1
N$_{1s}$	—	399.1	399.8	+0.7
O1$_{1s}$	—	533.3	533.7	+0.4
O2$_{1S}$	—	531.8	532.2	+0.4
O3$_{1S}$	—	528.4	528.4	—

The BE referred to is C$_{1s}$ = 284.8 eV. ΔE_b, chemical shift of binding energy.

Figure 2 TGA curves of supported catalysts **2a–2e**.

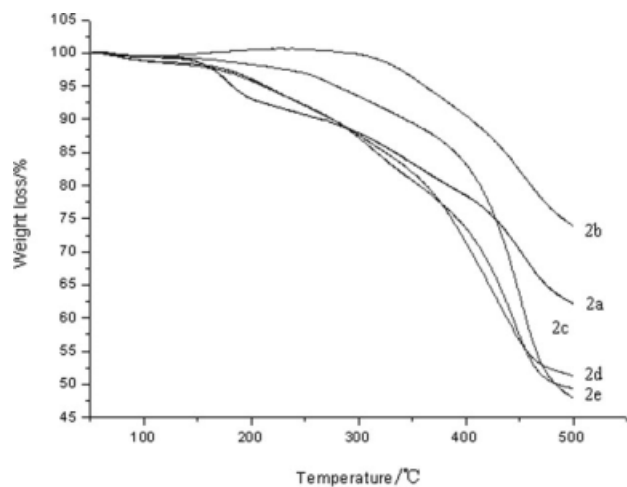**Figure 3** Reusability of supported catalysts **2a–2e**.

chloride because of its broad peak, the vibration of C—O shifted to a higher wave number (~ 44 cm^{-1}) in the spectra of the supported catalyst, which indicated that there was a coordination between —OH and the zinc. The vibration of C=O shifted toward a lower frequency (~ 24 cm^{-1}) from 1676 to 1652 cm^{-1}, which suggested the coordination of C=O and zinc. There was also a slight shift (~ 7 cm^{-1}) to lower wave number of the C—N absorption peak, which indicated the coordination of N and the metal. XPS analysis was also used to investigate the conformation of the complex. The results are complied in Table III. The $Zn_{2p3/2}$ BE of the metal complex decreased by 1.1 eV compared with $ZnCl_2$, which indicated an increase in its electron density. The N_{1s} BEs of the ligand and complex were 399.1 and 399.8 eV, respectively, which

means that there was coordination between the nitrogen and zinc. The XPS for oxygen atoms of the complex were divided into three peaks, which may have corresponded to C=O, —OH and other types of oxygen. The BEs of $O1_{1s}$ and $O2_{1s}$ were both increased by 0.4 eV, whereas the BE of $O3_{1s}$ remained the same, which belonged to the uncoordinated oxygen. All these results indicate that the polymer-supported zinc complex was formed.

Thermal analysis of the supported catalysts was carried out to investigate the stability of the catalysts. As shown in Figure 2, all the catalysts were stable up to 180°C. At less than 150°C, there appeared to be a small decrease in weight, which was the result of the loss of loosely bound volatiles on the surface of the catalysts. The catalyst **2b,** however, was more stable, and it did not begin to decompose until 300°C. All these suggest that the supported catalysts were very stable and could be used within a broad temperature scale.

Catalytic acetylation of the aldehydes

The catalytic activities of the supported catalysts were then investigated with the reaction of benzaldehyde

TABLE IV
Screening of the Reaction Parameters for the Conversion of Benzaldehyde to Its Corresponding Diacetate

Entry	Catalyst (mol %)	Solvent[a]	Time (h)	Yield (%)[b]
1	**2a** (5)	Dichloromethane	2	65
2	**2a** (5)	Acetonitrile	2	76
3	**2a** (5)	Diethyl ether	4	50
4	**2a** (5)	Cyclohexane	4	61
5	**2a** (5)	Solvent free	1	87
6	—	Solvent free	12	0
7[c]	—	Solvent free	12	13
8	$ZnCl_2$ (5)	Solvent free	1	83
9	**2a** (10)	Solvent free	0.5	76
10	**2a** (10)	Solvent free	1	89
11	**2a** (15)	Solvent free	1	89
12	**2b** (5)	Solvent free	1	85
13	**2c** (5)	Solvent free	1	79
14	**2d** (5)	Solvent free	1	72
15	**2e** (5)	Solvent free	1	75

[a] The reaction was carried out in 1 mL of solvent at RT.
[b] Isolated yields.
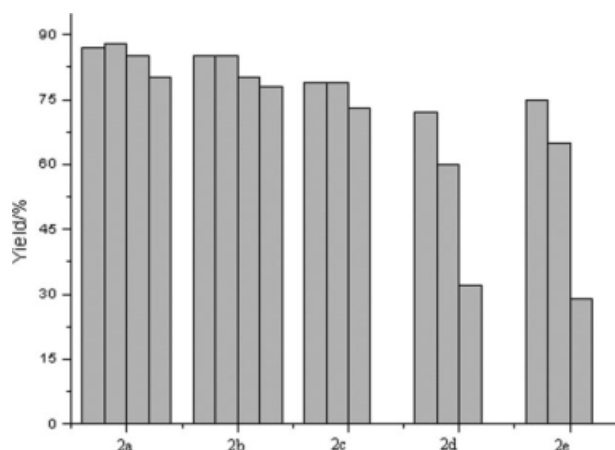[c] The reaction was carried out under reflux conditions.

TABLE V
Syntheses of Acylals Catalyzed by 2a

Entry	Aldehyde	Time (h)	Yield (%)[a]	mp (°C) Found	mp (°C) Reported
1	Benzaldehyde	1	87	44–46	44–45[25]
2	4-Me—C_6H_4CHO	1	95	80–82	81–82[25]
3	4-MeO—C_6H_4CHO	1	92	65–66	64–65[25]
4	4-NO_2—C_6H_4CHO	2	85	124–126	125–127[25]
5	4-Cl—C_6H_4CHO	2	87	80–82	82–83[25]
6	4-OH—C_6H_4CHO	2	84[b]	91–92	89–90[31]
7	Butyraldehyde	1	82	Oil	Oil[32]
8	Furfural	1	83	52–53	52–53[25]

[a] Isolated yields.
[b] Yield of triacetate.

TABLE VI
Comparison of Catalyst 2a with Other Catalysts for the Acetylation of Benzaldehyde

Entry	Catalyst	Solvent	Time (min)	Temperature (°C)	Yield (%)[a]
1	Catalyst **2a** (5 mol %)	Solvent free	60	RT	87
2	$FeCl_3$ (3 mol %)	Solvent free	20	RT	79[16]
3	NBS (10 mol %)	Solvent free	360	RT	95[22]
4	β-Zeolite (50 wt %)	Solvent free	120	60	92[23]
5	$H_3PW_{12}O_{40}$/MCM-41(20 wt %)	Solvent free	120	60	84[26]
6	[Hmim] HSO_4 (3.8 mol %)	Solvent free	25	RT	90[27]
7	P_2O_5/Al_2O_3 (15 mol %)	Solvent free	45	RT	86[28]
8	$Bi(NO_3)_2 \cdot 5H_2O$ (10 mol %)	CH_3CN	90	RT	87[29]

[a] Isolated yields.

and acetic anhydride as a model reaction. Initially, to optimize the reaction conditions, we tried to convert benzaldehyde to 1,1-diacetoxy-1-phenylmethane in the presence of 5 mol % of catalyst **2a** and acetic anhydride in various solvents and also under solvent-free conditions at RT. As shown in Table IV, in comparison to conventional methods in solvent, the yield under solvent-free conditions was higher, and the reaction time was shorter. With these good results at hand, we then examined the effect of catalyst loading on the reaction. It was obvious that the reaction did not proceed in the absence of catalyst. Elevating the temperature and prolonging the reaction time did not have any effect (Table IV, entry 7). However, an 83% yield was obtained when 5 mol % zinc chloride was used as a catalyst, which indicated that the zinc chloride was the real active site in the supported catalyst. When increasing the catalyst **2a** to 10 mol % or even 15 mol %, the rate of the reaction was obviously accelerated; however, the results clearly indicate that 5 mol % of catalyst was sufficient. Next, the catalytic activities of catalysts **2b** ~ **2e** were tested with the previous conditions. The results suggest that all the supported catalysts promoted the reaction smoothly; however, catalysts **2c** ~ **2e** were less effective than catalysts **2a** and **2b** (Table IV, entries 5 and 12–15). To continue our work, the reusability of the supported catalysts was also investigated. As shown from Figure 3, catalysts **2a** and **2b** preserved their activities for four runs, and then, their activities decreased. Catalyst **2c** maintained its activity for three runs. Even though catalysts **2d** and **2e** showed good activities in the first run, their activities decreased suddenly in the third run.

The generality of this procedure was examined with 5 mol % **2a** as a catalyst under the previous conditions. The results are summarized in Table V. Both aromatic aldehydes and aliphatic aldehydes reacted smoothly with acetic anhydride to afford the corresponding diacetates in good to excellent yields. Deactivated aldehydes, such as *p*-nitrobenzaldehyde, also produced good yields but need a longer time (Table V, entry 4). In the case of aliphatic aldehyde, a high yield was also observed. Furthermore, acid-sensitive aldehydes, such as furfural, reacted well without any decomposition or polymerization under such reaction conditions.

To show the merit of this work in comparison with reported protocols, we compared the results of the acylal synthesis from benzaldehyde in the presence of $H_3PW_{12}O_{40}$/MCM-41, [Hmim] HSO_4, $FeCl_3$, P_2O_5/Al_2O_3, β-zeolite, $Bi(NO_3)_2 \cdot 5H_2O$, and NBS (Table VI). The results show that our catalyst was competitive with other catalysts with respect to the amount of catalyst, reaction times, reaction conditions, and yields. Moreover, this protocol offers advantages over current methodologies in terms of safety, efficiency, mild conditions, and reusability of the catalyst, which thus makes them environmentally more acceptable.

CONCLUSIONS

In conclusion, a series of new functionalized resins and their zinc complexes were prepared based on chloroacetylated polystyrene by a simple method. The supported Lewis catalysts showed high catalytic activities for the synthesis of diacetates under solvent-free conditions at RT. The significant advantages of this methodology are mild reaction conditions, high yields, short reaction times, simple workup procedures, and low cost. Most importantly, the supported catalysts could be recovered easily and reused without an obvious loss of activity.

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