

Commercial Zinc Oxide (Zn^{2+}) as an Efficient and Environmentally Benign Catalyst for Homogeneous Benzoylation of Hydroxyl Functional Groups

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The solvent-free *O*-acylation of some alcohols with benzoyl chloride was carried out to the corresponding benzoylated products in good to excellent yields by the mediation of a catalytic amount (5 mol%) of the commercially available and inexpensive zinc oxide in short reaction times. This methodology represents an eco-friendly and simple catalytic alternative for benzoylation of primary, secondary, tertiary, and benzylic alcohols with ZnO. This catalytic system was homogeneous because of high solubility of zinc oxide in the reaction medium. Findings showed that ZnO was dissolved in hydrochloric acid, created *in situ*, after a few minutes. Although, others argued on the catalytic role of solid phase zinc oxide under a heterogeneous condition, it is not surprising to emphasize on the catalytic function of Zn^{2+} in the benzoylation of alcohols under homogeneous reaction conditions. Zinc oxide served as pre-catalyst to form Zn^{2+} , as the catalytically active species.

Keywords acetylation, benzoylation, zinc oxide, catalytic, alcohol

Introduction

Acylation of hydroxyl functional group is an often used protection protocol, because of the ease of formation as well as mild reaction conditions needed for its deprotection.¹ The protection process can be afforded with various anhydrides and/or benzoyl halides in the presence of different Lewis bases such as pyridine, triethyl amine, tributyl phosphine, *etc.*²

Protection of alcohols with Brønsted acids including polymeric Lewis acids, acyl imidazoles and acyl urea is also well known.³⁻⁵ Moreover, metal chlorides, metal triflates, metal perchlorates, distannoxane, ionic liquids, twisted amides, solid supported reagents, lipase, and recently heteropolyacids have been utilized as Lewis acids for the protection of hetero-atoms in a wide range of organic compounds.⁶⁻¹⁵ However, some of the above reported methods have limitations mainly in respect of stability, cost, availability, and reusability of the catalyst.

Although numerous methods are known for the protection of hydroxyl groups, new simpler and more effective protocols still continue to be explored. Therefore the matter of primary interest is enhancing reaction rates as well as improving selectivity via more green or environmentally friendly chemical processes.

Among recent advances, porous metal oxides have attracted the interest of chemists and materials scientists

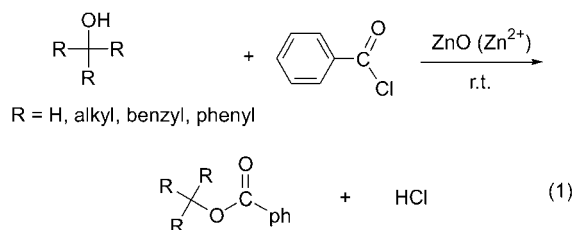
due to commercial attention in their applications in ion exchange, adsorption, and heterogeneous catalysis. The structure of metal oxides usually includes positive metal cations which possess Lewis acid character and negative oxygen anions which act as Brønsted base sites.¹⁶ Nowadays, mineral metallic oxides including alkali, alkali earth, and transition metal oxides having the ability to activate reagents, enhance reaction rate, and increase selectivity in heterogeneous or in homogeneous systems. These compounds are eco-friendly, simple, and act under mild reaction conditions which have been proven as useful catalysts for synthetic purposes in the laboratory and industry.^{17,18}

Commercial zinc oxide as an inexpensive and environmentally benign catalyst has been used in several organic transformations such as oxidation of alcohols,¹⁹ synthesis of cyclic ureas,²⁰ dehydration of aldoximes,²¹ and Friedel-Crafts acylation.²²

In continuation of our research program on evaluation of the efficiency of different metal oxides as catalysts, herein, we report findings on the *O*-acylation of some aliphatic and aromatic alcohols with benzoyl chloride by using a catalytic amount of commercial zinc oxide (as a source of Zn^{2+}) under solvent-free conditions (Eq 1). Although, there is few reports on the use of zinc oxide as heterogeneous catalyst in some organic transformations^{27-29,31} such as formylation, acetylation,

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and acylation of aromatic amines and alcohols, however, we found some new aspects on the catalytic role of zinc oxide in the reaction medium, which was really homogeneous.



Experimental

Materials and methods

Reagents and metal oxides were purchased from commercial resources and were used as received without further purification. All products were characterized by comparison of their spectral and physical data with those of authentic samples. Silica gel 60 (70–230 mesh) was used for column chromatography. GLC analyses were carried out on a Shimadzu GC-17A instrument equipped with a flame ionization detector using 25 m × 0.25 mm CPB 5–20 capillary columns. UV-Vis spectra were recorded on a UV-2550 (Shimadzu) spectrophotometer. Infrared spectra were run on a 8700 Shimadzu Fourier Transform spectrophotometer. ¹H NMR spectra were acquired on a Bruker AC-100 MHz spectrometer at ambient temperature in CDCl₃. Melting point is uncorrected and was obtained by a Kofler Heizbank Rechart type 7841 melting point apparatus.

General experimental procedure for benzylation of alcohols with benzoyl chloride

To a stirred solution of alcohol (10 mmol) and ZnO (dry powder, 0.04 g, 0.5 mmol, 5 mol%) was added benzoyl chloride (10 mmol) at room temperature. The reaction mixture was stirred with a mechanical stirrer for a certain period of time. The progress of the reaction was followed by GC or TLC. After completion of the reaction, 20 mL of CH₂Cl₂ was added, washed successively with 20 mL saturated NaHCO₃ solution, 20 mL of saturated NaCl solution, and the organic phase dried over MgSO₄. After filtration, removal of the organic solvent under reduced pressure gave the corresponding crude product. All of the benzoate esters were known and their identity were established by comparison of their spectral and physical data with those of authentic samples.²⁶

General experimental procedures for acetylation of alcohols with acetyl chloride in the absence of additive

To a solution of alcohol (10 mmol) was added acetyl chloride (10 mmol) with complete stirring at room temperature. Progress of the reaction was followed by GC or TLC. After completion of the reaction, the products

were isolated as described for the benzylation. All of the acetates were known and gave the same spectral and physical data as authentic samples.²⁶

Butyl benzoate²⁶ b.p. 250 °C; ¹H NMR (CDCl₃, 100 MHz) δ: 8.05–7.45 (m, 5H), 4.32 (t, *J*=7.1 Hz, 2H), 1.85–1.69 (m, 2H), 1.65–1.39 (m, 2H), 0.88 (t, *J*=7.1 Hz, 3H); IR (liquid film) ν: 1283 (C–O), 1718 (COPh), 2943 (H-Ph) cm⁻¹.

Phenyl benzoate²⁶ b.p. 298 °C; ¹H NMR (CDCl₃, 100 MHz) δ: 8.04–7.85 (m, 2H), 7.65–7.45 (m, 5H), 7.29–7.26 (m, 3H); IR (liquid film) ν: 1262 (C–O), 1722 (COPh), 3075 (H-Ph) cm⁻¹.

Benzyl acetate²⁶ b.p. 214 °C; ¹H NMR (CDCl₃, 100 MHz) δ: 7.38–7.33 (m, 5H), 5.13 (s, 2H), 2.05 (s, 3H); IR (liquid film) ν: 2890–3090 (C–H), 1740 (COPh), 1230 (C–O) cm⁻¹.

Benzyl benzoate²⁶ b.p. 323 °C; ¹H NMR (CDCl₃, 100 MHz) δ: 8.01–7.45 (m, 5H), 7.42–7.28 (m, *J*=7.7 Hz, 5H), 5.13 (s, 2H); IR (liquid film) ν: 2960–3090 (C–H), 1715 (COPh), 1050–1600 (C–O, strong bands) cm⁻¹.

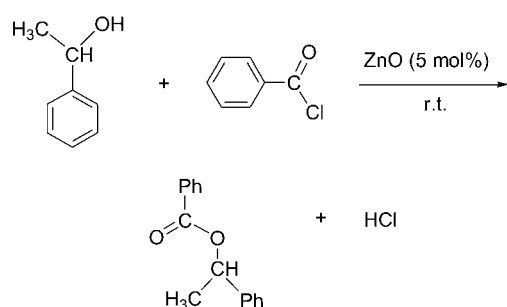
***n*-Butyl acetate**²⁶ b.p. 127 °C; ¹H NMR (CDCl₃, 100 MHz) δ: 4.15 (t, *J*=7.1 Hz, 2H), 2.04 (s, 3H), 1.73–1.38 (m, 4H), 0.87 (t, *J*=7.1 Hz, 3H); IR (liquid film) ν: 2870–2960 (C–H), 1740 (COPh), 1030–1470 (C–O, strong bands) cm⁻¹.

***tert*-Butyl benzoate**²⁶ b.p. 98 °C; ¹H NMR (CDCl₃, 100 MHz) δ: 8.03–7.45 (m, 5H), 1.45 (s, 9H); IR (liquid film) ν: 2930–3090 (C–H), 1712 (COPh), 1250 (C–O) cm⁻¹ and a series of strong bands (1000–1500 cm⁻¹).

Results and discussion

To explore the standard reaction condition, the model reaction was investigated in a variety of solvents including acetonitrile, toluene, dichloromethane, chloroform and 1,2-dichloroethane at room temperature. The reaction conditions were standardized after conducting the *O*-acylation of 1-phenylethanol in the above solvents using 5 mol% ZnO. As shown in Table 1, the best result in terms of reaction time and product yields achieved when the protection reaction was carried out without solvent. Under optimum conditions, 1-phenylethanol was benzylation at room temperature almost quantitatively with benzoyl chloride in the absence of any solvent (Table 1, Entry 6). Under neat reaction conditions, the protection of 1-phenylethanol was faster than those conversions obtained in solution. Therefore, the *O*-acylation reactions were carried out under solvent-free conditions.

The main objective of this protocol was to investigate the catalytic activity of ZnO as catalyst for the *O*-acylation reactions of some alcohols. Our investigation began with 1-phenylethanol as a model substrate. In an initial experiment, when a mixture of the above alcohol (10 mmol), benzoyl chloride (10 mmol), and ZnO (5 mol%) was stirred at room temperature under sol-

Table 1 Effect of different solvents on the *O*-acylation of 1-phenylethanol with benzoyl chloride in the presence of ZnO (5 mol%)^a

Entry	Solvent	GC yield/ %	Time/ min	Selec./ %	TON ^b	TOF ^c / h ⁻¹
1	CH ₂ Cl ₂	100	100	23	20	52
2	CH ₃ CN	100	100	25	20	48
3	PhCH ₃	62	100	43	12.4	17
4	CHCl ₃	100	100	23	20	52
5	(CH ₂ Cl) ₂	100	100	28	20	43
6	neat	100	100	10	20	120

^a Benzoyl chloride (10 mmol) was added to a stirred solution of alcohol (10 mmol) and ZnO (dry powder, 0.04 g, 0.5 mmol, 5 mol%) in 2 mL of solvent (if present) at room temperature.

^b Turnover number (TON) is the number of moles of product per mole of catalyst. ^c Turnover frequency (TOF) was calculated by the expression [product]/([catalyst] × time).

vent-free conditions, the corresponding benzoate formed in 100% yield within 10 min.

Table 2 describes effect of ZnO on the benzoylation of 1-phenylethanol with benzoyl chloride. Results clearly demonstrated that the conversions were increased dramatically with enhancing catalyst concentration. Only 14% of product was obtained in the absence of catalyst after 45 min (Table 2, Entry 1); whereas, 70% of conversion was achieved by the presence of

Table 2 Effect of ZnO (mol%) on the extent of 1-phenylethanol benzoylation with benzoyl chloride^a

Entry	ZnO/ mol%	GC yield/ %	Selec./ %	Time/min	TON	TOF/ h ⁻¹
1	0	14	100	45	—	—
2	1	70	100	20	70	210
3	5	100	100	10	20	120
4	10	100	100	7	10	86
5	20	100	100	0.5 (explosively)	5	600
6	50	100	100	Instant (explosively)	2	—

^a Benzoyl chloride (10 mmol) was added to a stirred solution of alcohol (10 mmol) and ZnO (dry powder, 0.04 g, 0.5 mmol, 5 mol%) at room temperature.

1 mol% of ZnO after 20 min (Table 2, Entry 2). Consequently, efficiency of the catalytic system was improved by enhancing catalyst concentration to 5 mol%. 1-Phenylethanol was converted quantitatively to the corresponding benzoylated product in the presence of 5 mol% zinc oxide after 10 min. Higher amounts of ZnO (> 10 mol%) promoted the benzoylation reaction violently. 1-Phenylethanol directed to complete conversion in the presence of 20 mol% of ZnO with eruption of gas and heat in less than 30 s (Table 2, Entry 5).

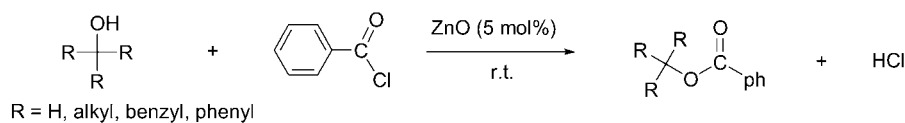
The catalytic potential of some commercial metal oxides (5 mol%) was inspected for the benzoylation of 1-phenylethanol with benzoyl chloride under solvent free conditions (Table 3). As described above, the acylation of 1-phenylethanol with benzoyl chloride led to only 14% of product in the absence of catalyst after 45 min (Table 3, Entry 8). According to the obtained results, TiO₂, Al₂O₃, and Fe₂O₃ led to *ca.* 10% of the corresponding benzoate during 10 min; whereas, BaO, CaO, and ZrO₂ revealed moderate catalytic activity and provided 29%—55% of product at the same time (Table 3, Entries 4—5). Among the examined metal oxides, ZnO was obviously the most efficient catalyst and led to complete conversion of 1-phenylethanol after 10 min (Table 3, Entry 7).

Table 3 Benzoylation of 1-phenylethanol with benzoyl chloride in the presence of some metal oxides (5 mol%)^a

Entry	Metal oxide	GC yield/%	Time/min	TON	TOF
1	Al ₂ O ₃	10	10	2	12
2	Fe ₂ O ₃	9	10	1.8	11
3	TiO ₂	11	10	2.2	13
4	ZrO ₂	29	10	5.8	35
5	BaO	55	10	11	66
6	CaO	45	20	9	27
7	ZnO	100	10	20	120
8	no catalyst	14	45	—	—

^a The reactions were carried out as described below Table 2.

To explore generality of the catalytic system, we studied *O*-acylation of various alcohols under the standard reaction condition. As one can see from Table 4, all alcohols were selectively converted to the corresponding products in good yields without any evidence for the formation of side products. The general efficiency of the protocol was evident from a variety of hydroxyl groups, including primary, secondary, tertiary, and benzylic alcohols which all were acylated (16%—100% yields) in short reaction times by the mediation of 5 mol% of the catalyst. Although the examined alcohols showed different reactivity pattern, the results clearly demonstrated the activating role of zinc oxide on the reaction progress. The acylation protocol was nearly inefficient in the absence of additive and led to 14%—44% of conversions

Table 4 *O*-Acylation of some alcohols with benzoyl chloride catalyzed by ZnO (5 mol%)^a

Entry	Substrate	In the absence of ZnO		In the presence of ZnO	
		GC yield/%	Time/min	GC yield/%	Time/min
1		14	45	100	10
2		44	30	80	20
3		4	45	16	40
4		16	5	25	5
5		22	30	30	5
6		35	25	73	25
7		30	10	90	30

^a Benzoyl chloride (10 mmol) was added to a stirred solution of alcohol (10 mmol) and ZnO (dry powder, 0.04 g, 0.5 mmol, 5 mol%) at room temperature and the homogeneous reaction mixture was stirred with a mechanical stirrer for a certain period of time. Progress of the reactions was followed by GC or TLC. All of the benzoylated products were known compounds and were characterized by comparison with authentic samples by means of spectral and physical data.²⁶

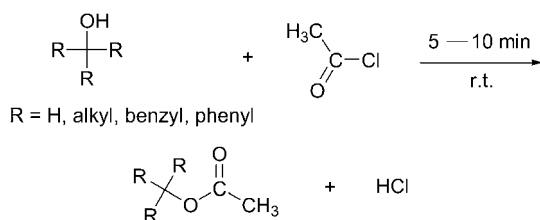
in longer reaction times. Menthol and benzyl alcohol revealed low activity toward benzoylation and led to 16% and 25% of their corresponding products after 40 and 5 min, respectively. Phenol was also benzoylated and generated the corresponding benzoate in 30% yield in the presence of 5 mol% catalyst after 5 min (Table 4, Entry 5); whereas, 22% of conversion was achieved in the absence of catalyst after 30 min. It seems that both electronic and steric demands around the hydroxyl functional groups affected the reactivity of alcohol toward the benzoylation reaction.

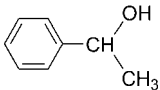
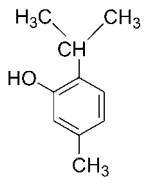
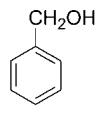
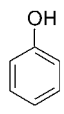
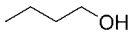

The general experiments were carried out using 10 mmol of alcohol. The reactions can be scaled up to 100 mmol or more for industrial purposes.

There are some reports on the use of metal oxides, especially ZnO, in the acetylation of alcohols with acetyl chloride under aerobic reaction conditions.^{27–29} It is worth mentioning, acetylation of familiar alcohols with acetyl chloride, which is more reactive than benzoyl

chloride, needed no additional catalyst under the experimental reaction conditions reported here at room temperature. Results in Table 5 proved that acetyl chloride is sufficiently reactive to promote acetylation of alcohols in the absence of catalyst and under neat reaction conditions as described in the experimental section.^{27–29} The reactions were fast and led to high conversions (70%–90%) in less than 10 min.

The present catalytic system for the benzoylation of alcohols was unexpectedly homogeneous. It means that zinc oxide should be solved in the reaction medium afterwards addition of substrate and during progress of the reaction. Controlled experiments were carried out to explore the reason for high solubility of zinc oxide in the reaction medium. Findings showed that ZnO was dissolved in hydrochloric acid, created *in situ*, after a few minutes. Presence of Zn²⁺ was confirmed qualitatively in the reaction medium by formation of an intensively colored complex with dithizone, as specific indi-

Table 5 Acetylation of some alcohols with acetyl chloride in the absence of catalyst under neat reaction conditions^a

Entry	Substrate	GC yield/%	Time/min
1		70	10
2		78	5
3		90	10
4		82	5
5		90	5
6		80	5

^a To a solution of alcohol (10 mmol) was added acetyl chloride (10 mmol) and the resulting homogeneous mixture was stirred completely at room temperature. Progress of the reactions was followed by GC or TLC.

cator of zinc ion. Therefore, in contrast to the previous protocols using similar reaction conditions,²⁷⁻²⁹ the reaction system was surprisingly homogeneous. Accordingly, it is not surprising to emphasize on the catalytic activating role of Zn^{2+} species in the introduced homogeneous system.³⁰

Table 6 describes *O*-acylation of 1-phenylethanol, *n*-butanol, and *tert*-butanol with benzoyl chloride in the presence of 5 mol% zinc chloride, zinc nitrate, and zinc oxide under the standard reaction conditions. Findings revealed that two first Zn^{2+} salts behaved like zinc oxide and all of them created almost the same conversions under similar reaction conditions. $ZnCl_2$, $Zn(NO_3)_2$, and ZnO led to complete conversion of 1-phenylethanol after 10 min. These three catalysts formed 71%–73% *n*-butyl benzoate and 79%–81% *tert*-butyl benzoate in the protection of the corresponding alcohols, respectively, after 20–25 min. Results in Table 6 convinced

us to the mediation of Zn^{2+} as a common catalytically active species in the above three systems.

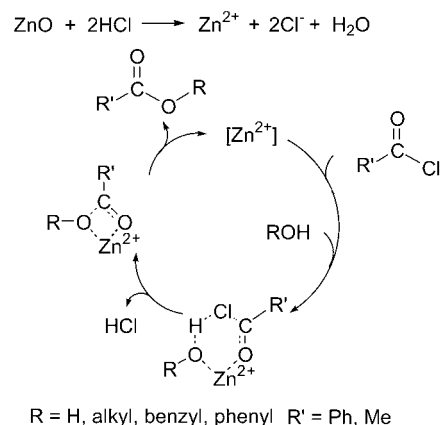
Table 6 Benzoylation of some alcohols with benzoyl chloride in the presence of (5 mol%) Zn^{2+} salts^a

Entry	Zinc salt	Alcohol	GC yield/%	Time/min	TOF/h ⁻¹
1	$ZnCl_2 \cdot 6H_2O$	1-Phylethanol	100	10	120
		<i>n</i> -Butanol	71	25	34
		<i>t</i> -BuOH	79	20	47
2	$Zn(NO_3)_2$	1-Phylethanol	100	10	120
		<i>n</i> -Butanol	72	25	34
		<i>t</i> -BuOH	81	20	49
3	ZnO	1-Phylethanol	100	10	120
		<i>n</i> -Butanol	73	25	35
		<i>t</i> -BuOH	80	20	48
4	no catalyst	1-Phylethanol	14	45	—
		<i>n</i> -Butanol	35	25	—
		<i>t</i> -BuOH	44	30	—

^a The reactions were carried out as described below Table 2.

Moreover, catalytic efficiency of the present system was independent to the air moisture and water molecules created as a by-product in the plausible reaction of hydrochloric acid with zinc oxide. These water molecules along with moisture of the reaction medium could hydrolyze benzoyl chloride to benzoic acid. Benzoic acid may be reacted with Zn^{2+} , and produce zinc benzoate. This species would served as another catalytically active species as reported previously for other zinc carboxylates.³²

Based on the above observations, Scheme 1 would be proposed for the protection of alcohols with acetyl/benzoyl chloride. In the first step, approach of alcohol and protecting agent to Zn^{2+} led to formation of a six-membered species, which was changed to a four-membered metallacycle by removing one HCl

Scheme 2 Proposed reaction pathway for the protection of alcohols with acetyl/benzoyl chloride

molecule from it in the second step. Finally, one ester molecule was eliminated from the metallacycle, and the primarily zinc ion was recovered.

In summary, we have demonstrated the catalytic efficacy of commercial ZnO towards *O*-acylation of alcohols with benzoyl chloride under very mild reaction conditions. Some important features of the present methodology were simple reaction procedure, good yields of products in short reaction times, using inexpensive and nonpoisonous metal oxide, and moisture compatibility of the protocol. Thus, this methodology represents an eco-friendly alternative to many existing procedures and is suitable for industrial purposes. Our new important findings proved that Zn²⁺ was the catalytically active species in homogeneous benzylation of alcohols. However, the catalytic efficiency of commercial zinc oxide in homogeneous benzylation of alcohols was indeed superior to some of the previously reported protocols using other mineral oxides.^{23–29}

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