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ZnO and ZnO-nanoparticles: Efficient and reusable heterogeneous catalysts for one-pot synthesis of *N*-acylsulfonamides and sulfonate esters

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ABSTRACT

Commercially available and preparative ZnO nanoparticles are reported as efficient and reusable catalysts for the chemoselective synthesis of *N*-acylsulfonamides and sulfonate esters. A one-pot sequential sulfonylation and acylation of amines took place to afford the *N*-acylsulfonamides in excellent yields under solvent-free conditions. The ZnO catalyst can be reused for without significant loss of catalytic activity. © 2011 Elsevier B.V. All rights reserved.

1. Introduction

Sulfonylation and acylation of heteroatoms are valuable transformations which resulted in the imide, sulfonimide, amide, sulfonamide, ester and sulfonate ester moieties as building blocks of important biologically active and polyfunctional molecules [2–5]. Sulfonate esters are well-known alkylating agents and cell proliferation inhibitors [6], while sulfonamide derivatives are clinically used as antibacterial and antibiotic medicines [7–11]. Moreover, a number of enzyme inhibitors [12], new therapeutic agents for Alzheimer's disease [13], and hepatitis C virus NS protease inhibitors [14] are derived from *N*-acylsulfonamides.

As a result of wide range of activity and importance, there are several available procedures for the preparation of these compounds. *N*-acylsulfonamides can be prepared by either base-catalyzed acylation of sulfonamides [7–12] or sulfonylation of amides [10]. Due to the less nucleophilicity of amide nitrogen and sensitivity of imide bond, acylation of sulfonamides is often preferred to sulfonylation of amides. Similarly, sulfonate esters

rezasabeti97@yahoo.com (M.R. Sabeti), jafari@yazduni.ac.ir (A.A. Jafari), ftamaddon@yazduni.ac.ir (F. Tirgir), ftamaddon@yazduni.ac.ir (E. Keshavarz). and sulfonamides have been prepared through the sulfonylation of alcohols and amines [1–6,15–24] in the presence of basic catalysts like pyridine, triethyl amine and aqueous metal hydroxides. Some of these reactions are together with the formation of undesired side products, use of toxic or corrosive reagents, and tedious processes for purification of products. A good option of catalyst be able to enhance the rate of sulfonylation and acylation reactions via dual activation of S=0, C=0 and NH groups. Therefore, searching for one-pot catalytic procedures with less reaction steps is still of interest.

Recently, metal oxides have been used as efficient heterogeneous catalysts in various organic transformations [25-28]. Although metal oxide surfaces exhibit both Lewis acid and base properties, nature of metal cation and surface area of metal oxides have extensively manipulated to their catalytic properties. Zinc oxide is a low-priced metal oxide which as both industrial and nano type has been used as a professional catalyst in various organic transformations [29-33]. In our recent reports [33-36], we have pointed to the raising of carbon-hetero atom bonds activities by coordination to accessible zinc cation of heterogeneous catalysts [33,34]. Accordingly, it was supposed that coordination of S=O and NH bonds to ZnO and nanoZnO could activate these groups, and facilitate their reactions. In the present paper, we report our results on the use of ZnO and ZnO nanoparticles as efficient and reusable catalysts in the one-pot preparation of N-acylsulfonamides, sulfonamides and sulfonate esters (Scheme 1).

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Scheme 1. ZnO-catalyzed sulfonylation and acylation.

2. Experimental

2.1. Preparation of nanocatalysts

2.1.1. Preparation of nanoZnO I [30]

Zinc acetate dihydrate (5.5 g) was dissolved in 50 mL of deionized water and then solid NaOH (16 g) was added slowly into the solution under magnetic stirring at room temperature. A transparent Zn(OH)₄ solution was formed. Then 2 mL of ionic liquid 1-butyl-3-methylimidazolium bis (trifluoromethylsulfonyl) imide ([bmim][NTf₂]) was added to 3 mL of the above solution. The suspension was put into a domestic microwave oven (850 W) in air, 30% of the output power of the microwave was used to irradiate the mixture for 5 min (on for 10 s, off for 5 s). The white precipitate was collected by centrifugation, washed with deionized water and ethanol several times, and dried in vacuum oven at 40 °C for 10 h. The mean particle size of these nanoparticles was 47–37 nm [30].

2.1.2. Preparation of nanoZnO II [31]

The ZnO nanoparticles type II was prepared according to the previously reported procedure [31]. In a typical procedure, 0.22 g (1 mmol) of $Zn(CH_3CO_2)_2 \cdot 2H_2O$ was suspended in 120 mL of 2-propanol under vigorous stirring at 50 °C. A NaOH alcoholic solution was prepared by adding 0.08 g (2 mmol) NaOH to 30 mL of 2-propanol under vigorous stirring at 50 °C. The flasks containing $Zn(CH_3CO_2)_2 \cdot 2H_2O$ and NaOH alcoholic solution were cooled in an ice-water bath. The NaOH solution was then added to $Zn(CH_3CO_2)_2 \cdot 2H_2O$ solution under vigorous stirring to give a total volume of 150 mL. Final solution was heated up to 80 °C by microwave irradiation. After 5 min, the transparent solution was obtained. The centrifugation of transparent solution yields white solid which was then calcined at 600 °C for 1 h. The mean particle size of ZnO nanoparticles was reported as 30 nm [31].

2.2. General procedure for the one-pot synthesis of *N*-acylsulfonamides

Amine (2 mmol) was added to a stirred mixture of ZnO (0.5 mmol) and sulfonylating agent (2 mmol) and the mixture was stirred at ambient temperature for the given times (Table 1). After completion of the sulfonylation reaction (TLC monitoring), acylating agent (2 mmol) was added and the reaction was monitored by TLC again. Then, EtOAc (2×10 mL) was added and the precipitated ZnO was filtered off. The resulting organic solution was washed with 10% NaHCO₃ and brine, dried over anhydrous Na₂SO₄, and. evaporated to give the desired *N*-acylsulfonamide. The structure of products was assigned by analysis of their IR, ¹H NMR, ¹³C NMR spectra and comparison to authentic samples or elemental analysis. Known products showed physical states; melting points and spectroscopic data in agreement with authentic samples.

2.3. General procedure for sulfonylation of amines and alcohols

Substrate (amine or alcohol) (2 mmol) was added to a stirred mixture of ZnO (0.2 mmol, 20 mol% or nanoZnO type I) and sulfonylating agent (2 mmol). Then, the reaction mixture was stirred at ambient temperature for the given times (Table 2). After completion of the reaction (TLC monitoring), EtOAc (2×10 mL) was added and the precipitated ZnO was filtered off. The resulting organic solution was washed with NaHCO₃ (10%) and dried over anhydrous Na₂SO₄. Finally, the solvent was removed to give the sulfonylated product in 60–95% yields. No further purification was required for sulfonamides, while sulfonate esters were typically purified by short column chromatography (Hexane, EtOAc).

2.4. Reusability of catalyst

ZnO, nanoZnO type I and nanoZnO type II was regenerated by simple washing with EtOAc and drying under microwave irradiation. Using the recycled catalysts for three consecutive times in both sulfonylation and acylation reactions furnished the product with no significant decreasing in reaction yield.

2.5. Selected spectral data

2.5.1. N-phenyl-N-tosyl acetamide (Table 1, entry 12, compound (j))

Colorless needles (EtOH), Mp = 154–156 °C (Lit. 149–150 °C [8]). FT-IR (KBr) ν_{max} : 1700, 1598, 1370, 1267, 1170 cm⁻¹. ¹H NMR (500 MHz, CDCl₃) δ : 1.90 (s, 3H, COCH₃), 2.48 (s, 3H, CH₃), 7.26–7.30 (m, 2H, H_{meta} Ph-N), 7.35 (d, 2H, *J* = 7.8 Hz, H_{ortho} Ph-CH₃), 7.46–7.50 (m, 3H, H_{ortho,para} N-Ph), 7.95 ppm(d, 2H, *J* = 7.8 Hz, H_{meta} Ph-CH₃). ¹³C NMR (125 MHz, CDCl₃) δ : 21.50, 25.20, 129.10, 129.30, 130.00, 130.50, 136.20, 140.00, 145.00, 170.30 ppm.

2.5.2. N-butyl-N-tosyl acetamide (Table 1, entry 15, compound (m))

Thick oil. FT-IR (neat) ν_{max} : 1704, 1596, 1358, 1250, 1168 cm⁻¹. ¹H NMR (500 MHz, CDCl₃) (: 0.90 (t, *J* = 7.5 Hz, 3H, CH₂CH₃), 1.35 (m, 2H, CH₂CH₃), 1.65 (m, 2H, NCH₂CH₂), 2.28 (s, 3H, CH₃Ph), 2.38 (s, 3H, CH₃CO), 3.80 (t, *J* = 7.5 Hz, 2H, NCH₂CH₂), 7.30 (d, *J* = Hz, 2H_{arom}), 7.75 ppm (d, *J* = 7.8 Hz, 2H_{arom}).

2.5.3. 1-Octyl tosylate (Table 2, entry 2b)

Thick oil. FT-IR (neat) ν_{max} : 2927, 2857, 1448, 1364, 1188, 1097, 951, 826, 754, 688 cm⁻¹. ¹H NMR (500 MHz, CDCl₃) (: 0.90 (t, *J* = 7.5 Hz, 3H, CH₂CH₃), 1.25–1.50 (m, 10H, (CH₂)₅), 1.60–1.80 (m, 2H, CH₂), 3.00 (s, 3H, CH₃SO₂), 4.20 ppm (t, *J* = 7.5 Hz, 3H, CH₂O).

2.5.4. Pyridin-2-yl-N-tosylmethanamine (Table 2, entry 10)

White needles (H₂O:EtOH), Mp = 92–94 °C. FT-IR (KBr) ν_{max} : 3210, 3060, 1598, 1441, 1327, 1161, 1111, 1090, 817, 762, 660 cm⁻¹. ¹H NMR (500 MHz, DMSO-d₆) δ : 2.37 (s, 3H, CH₃Ph), 4.50 (d, *J* = 6.30 Hz, CH₂), 7.24 (br t, 1H, NHSO₂), 7.35 (m, 3H, 2H_{ortho} to CH₃ and H₃ *pyridyl*, 7.68 (d, *J* = 6.3 Hz, 2H, H_{ortho} SO₂), 7.26 (td, *J* = 6.3, 1.5 Hz, H₄ *pyridyl*), 8.16 (t, *J* = 6.3 Hz, H₅ pyridyl), 8.43 (d, *J* = 4.20 Hz, H₆ *pyridyl*) ppm. ¹³C NMR (125 MHz, DMSO-d₆) δ : 21.82, 48.78, 122.48, 123.23, 127.43, 130.43, 137.58, 138.55, 143.50, 149.56, 158.04. Anal. Calcd (%) for C₁₃H₁₄N₂O₂S: C, 59.52; H, 5.38; N, 10.68; O, 12.20; S, 12.22; Found C, 59.60; H, 5.48; N, 10.88.

2.5.5. N-glycyl 4-methylbenzenesulfonamide (Table 2, entry 13b)

White needles (EtOH), Mp = $170 \degree$ C. IR (KBr): 3356, 1317, 1149, 918 cm⁻¹. ¹H NMR (500 MHz, DMSO-d₆) δ : 2.42 (s, 3H, CH₃), 3.58 (d, *J* = 6 Hz, 2H, CH₂), 6.71 (br s, 1H, NH), 7.26–7.37 (m, 2H, H_{arom}), 7.45–7.73 (m, 2H, H_{arom}), 8.03 (br s, 1H, COOH) ppm.

Table 1

ZnO-catalyzed one-pot synthesis of N-acylsulfonamides.

				COR	23		
$R^1 - NH_2 +$	$R^2 SO_2 Cl$ Z	$\xrightarrow{\text{nO}}$ RNR ¹ SO ₂	$R^2 - \frac{R^3 CO}{R^3}$	$X \to R^1 - N$	SO_2R^2		
	Т	1	T ₂				
Entry	R ¹	R ²	R ³	Х	Product	Time (h) T_1/T_2	Yield (%) ^{a,b}
1	Н	CH ₃	CH ₃	OCOCH ₃	MeCONH–SO ₂ Me (\mathbf{a})	1/0.5	93, 95 ^c
2	Н	CH₃	CH ₃	Cl	$MeCONH-SO_2Me(\mathbf{a})$	1/0.5	95
3	Н	CH₃	Ph	Cl	PhCONH $-SO_2Me(\mathbf{b})$	1/0.5	92
4	Н	CH₃	Н	OH	$HCO-NH-SO_2Me(\mathbf{c})$	1/3	_d
5	Н	Ph	CH₃	OCOCH ₃	$CH_3CONH-SO_2Ph(\mathbf{d})$	1/0.75	91, 93 ^c
6	Н	Ph	Ph	Cl	PhCONH–SO ₂ Ph (\mathbf{e})	1/1	90, 91°
8	Н	$p-MeC_6H_4$	CH ₃	Cl	MeCO-NH-Ts (f)	1/0.5	92
9	Н	p-MeC ₆ H ₄	Ph	Cl	PhCO-NH-Ts (g)	1/0.5	90
		1 0 .			COPh	,	
					Ph-N<		
10	Dh	CU	DP	CI	SO_2Me (h)	1/0.75	90.010
10	PII		PII	CI	COM-	1/0.75	89,91
					Ph - N		
					SO_2Ph (i)		
11	Ph	Ph	CH3	Cl	(1)	1/0.75	93, 93 ^c
					COMe		
					$Ph - N - T_{e}$		
12	Ph	p-MeC ₆ H₄	CH₃	Cl	15 (j)	1/0.5	93.91 ^c
13	Bu	n-MeC ₆ H₄	CH ₂	Cl	COMe	1/0.5	92
14	Bu	p-MeC ₆ H₄	Ph	Cl	Bu - N	1/1	93
		P			Du N Ts	-/-	
					(K)		
					COPh		
					Bu-N_		
					1s (m)		
					Ts Ts		
					$N - (CH_2)_2 - N$		
					Ac Ac		
15	$H_2N-(CH_2)_2$	$p-MeC_6H_4$	CH ₃	Cl	(n)	1/0.5	90

^a The substrate was stirred with the corresponding sulfonyl chloride using 0.5 mmol ZnO at the given times followed by addition of acyl chloride or anhydride (1.1 mmol) at ~50 or 70 °C and extra stirring.

^b Isolated yield.

^c 0.3 mmol (30 mol%) of prepared ZnO nanoparticles (type I) was used and both T_1 and T_2 were reduced 1.5 times.

^d No product was obtained.

2.5.6. *N*-methyl-*N*-nosyl-aniline (Scheme 2)

Pale brown solid (EtOH:H₂O), Mp = 133 °C (Lit. 130–132 °C [37]. IR (KBr): 3082, 2975, 1604, 1520, 1355, 1125, 918 cm⁻¹. ¹H NMR (500 MHz, DMSO-d₆) δ : 3.20 (s, 3H, CH₃), 7.00–7.10 (m, 2H, H_{arom}), 7.34–7.38 (m, 3H, H_{arom}), 7.75–7.80 (m, 2H, H_{arom}), 8.28–8.35 (m, 2H, H_{arom}) ppm. ¹³C NMR (DMSO-d₆): 38.52; 124.14; 126.68; 128.32; 129.00; 129.38; 140.61; 143.04; 150.53 ppm.

3. Results and discussion

In the preliminary experiments the catalytic behaviors of two types of prepared ZnO nanoparticles I [30 mL] and II [31] were compared with commercial ZnO in the acetylation and tosylation of benzamide with acetyl chloride, Ac₂O, and tosyl choride (TsCl) at various conditions. The ZnO-catalyzed reaction of benzamide with Ac₂O and TsCl were not successful anyway. While, reaction of acetyl chloride with benzamide in the presence of ZnO (20 mol%) or each of nanoZnO (10 mol%) at ~50 °C and solvent-free conditions yielded the desired unsymmetrical imide in 70%. Reaction time was clearly reduced by 1.5 times using two types of ZnO nanoparticles but yields were the same (Scheme 2).

In order to obtain a symmetrical imide, benzamide was treated with benzoyl chloride at \sim 70 °C in the presence of nano ZnO but *N*-benzoylbenzamide was isolated in only 32% yield after 12 h.

The difficulties associated with the ZnO-catalyzed tosylation of benzamide and more suitable acidity of sulfonamide NH ($pK_a = 4-5$) than amide NH led us to prefer the synthesis of *N*-acylsulfonamides via acylation of sulfonamides. This would be possible by one-pot

sequential sulfonylation and acylation of amines in the presence of ZnO.

Therefore, reactions of ammonia and aniline with PhSO₂Cl were tested under various conditions. The excellent yields of desired sulfonamides were obtained under solvent-free conditions in the presence of 0.5 equiv. ZnO. Further acetylation of the isolated products with both acetyl chloride and Ac_2O in the presence of ZnO was successful, although acetylation with acetyl chloride was much superior to Ac_2O (for example, 93% of *N*-acetyl benzenesulfonamide versus 80% yield).

A study was made on the effect of catalyst on % yield of *N*-acetyl benzenesulfonamide by reaction of PhSO₂NH₂ with AcCl in the presence of 20 mol% of ZnO, two types of prepared ZnO nanoparticles and recovered catalyst (Fig. 1). As figure shows, the recovered catalyst acted as the same as fresh ZnO and the maximum yield of product is belong to the use of nanoZnO type I, which may be due to the better diffusion of this kind of nanoZnO in the reaction mixture.

In order to combining the two sulfonylation and acylation steps and eliminating of the isolation process of sulfonamide intermediate, our synthetic propose turned to the one-pot preparation of



Scheme 2. ZnO-catalyzed N-acylation of benzamide.

Table 2



Entry	R ¹ XH	R ²	Time (h)	Yield (%) ^a
 1a	1-Butanol	Me	2	93
1h	r Butanor	Ph	2	92
10		n-MeC ₆ H ₄	3	92
1d		p-O2NCeH4	1.5	95
2a	1-Octanol	Me	4	93
2b		n-MeC ₆ H ₄	4	91
20		p	•	01
3	ОН СОН	$p-MeC_6H_4$	1.5	95
4	2-Octanol	Ph	4	52 ^b
5a	(–) Menthol	Ph	8	65 ^b
5b		p-MeC ₆ H ₄	10	48 ^b
6	PhCH ₂ NH ₂	Ph	1	96
7a	PhNH ₂	Ph	1	95
7b	2	$p-MeC_6H_4$	1	94
7c		$p-O_2NC_6H_4$	0.75	97
8	$4-O_2NC_6H_4NH_2$	Ph	12	62
9	$4-MeOC_6H_4NH_2$	$p-O_2NC_6H_4$	0.5	95
10	NH2	p-MeC ₆ H ₄	1	94
11a		Ph	1	92
11b	NH	$p-MeC_6H_4$	1.5	92
12a		Ph	1	90
12b	O NH	$p-MeC_6H_4$	1.5	93
13a		Ph	2	95
13b	HO_2C NH ₂	$p-MeC_6H_4$	2	93
14a	- 2	Ph	1.5	94
14b		$p-MeC_6H_4$	1.5	91
15a	HU ₂ NH ₂	Ph	1	97
15b	H ₂ N NH ₂	p-MeC ₆ H ₄	1	95

^a Isolated yield.

^b The corresponding carbonyl compound was formed as competitive product which was identified by 2.4-dinitrophenyl hydrazine.

N-acylsulfonamides. Thus, sulfonylation of aniline with PhSO₂Cl at solvent-free conditions (~1 h) was followed by subsequent addition of acetyl chloride in the presence of various amounts of ZnO. Although reaction preceded well using 0.1, 0.2 and 0.5 equiv. of catalyst, final one was selected to catalyze sulfonylation of amines and in situ acylation of the obtained sulfonamide. The optimized conditions for the one-pot reaction of aniline were as follow; addition of 1 mmol of sulfonyl chloride and 0.5 mmol of ZnO (0.04g) at ~50 °C and stirring (~1 h), further addition of 1.1 mmol of AcCl and stirring for additional time. This method was also useful for the tosylation/benzoylation of ammonia with PhCOCl and *N*,*N*-diphenyl benzenesulfonamide was isolated in 90% yield at ~70 °C and solvent-free conditions. No product was obtained from a control experiment without ZnO.



Fig. 1. The effect of catalyst on the reaction yield of benzensulfonamide with AcCl.

The versatile application of the one-pot synthesis of *N*-acylsulfonamides catalyzed by ZnO was confirmed by sulfonylation and subsequent acylation of various aliphatic and aromatic amines and diamines in good to excellent yields (Table 1).

Alternatively, the *O*- and *N*-sulfonylation of 1° and 2° alcohols and amines were carried out at optimal conditions using 20 mol% ZnO or 10 mol% of ZnO nanoparticles I under solvent-free conditions. The results are summarized in Table 2 and confirmed the efficiency of nanoZnO for the simple preparation of various sulfonate esters and sulfonamides.

ZnO-catalyzed *N*-acylation of a number of isolated sulfonamides produced again the corresponding *N*-acylsulfonamides in high yields.

With in hand results, we encouraged to test two consequent sulfonylation of amines. Thus, tosylation of 2-amino-benzothiazol and subsequent tosylation of the product at the optimized reaction conditions was attempted in the presence of nanoZnO I and (*E*)-*N*,3-ditosylbenzo[d]thiazol-2(3H)-imine was obtained in excellent yield (Scheme 3).

Alkylation of sulfonamides is a key process in the preparation of amines. In this reaction acidity of sulfonamide NH is important







Scheme 4. ZnO-catalyzed N-methylation of sulfonamide.



Scheme 5. Proposed mechanism for the ZnO-catalyzed synthesis of Nacylsulfonamides and sulfonamides.



Scheme 6. Chemoselectivity of ZnO-catalyzed sulfonylation and acylation.

and presence of an electron with-drawing group is helpful. Therefore, aniline (1 mmol) was treated with 4-nitrobenzene-1-sulfonyl chloride (nosyl chloride) and ZnO at 60°C under solvent-free conditions. After completion of the reaction, subsequent methylation of the in situ generated sulfonamide with diazomethane in chloroform at room temperature afforded the N-methyl-N-phenyl-4-nitrobenzene sulfonamide in 95% yield in less than 10 min (Scheme 4).

Due to the both Lewis acid and base properties of ZnO, we proposed the following mechanism for the sulfonylation and subsequent acylation of amines. ZnO activates the S=O or C=O groups by its Lewis acid property, while its basic property assists to fast absorption of the released HCl and the nucleophilic attack of sulfonamide NH. This dual activation accelerates attack of NH to the activated C=O group and results in the formation of Nacylsulfonamides (Scheme 5).

High surface area and better dispersion of nanoparticles in the reaction mixture are reasons for better activities of nanoZnO type I and II. This size-dependent property made difficulties for separation of catalyst by centrifugation during the work-up.

Finally, a number of competitive reactions were examined to confirm the chemoselectivity of the method. The results showed that 1° sulfonamides react with AcCl much faster than 2° sulfonamides, while 1° amines were sulfonylated versus 2° amines and alcohols (Scheme 6).

4. Conclusion

In conclusion, we have demonstrated an efficient one-pot synthesis of N-acylsulfonamides, sulfonamides and sulfonate esters from amines and alcohols using commercial ZnO or nanoZnO as eco- and environmental-friendly catalysts under solvent-free conditions. The simplicity, chemoselectivity, fewer steps of synthesis, easy workup, as well as safety and reusability of catalyst are advantages of this procedure over the previous reported ones.

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