

N-Propylsulfamic acid supported onto magnetic Fe_3O_4 nanoparticles (MNPs-PSA) as a green and reusable heterogeneous nanocatalyst for the chemoselective preparation and deprotection of acylals

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Abstract *N*-propylsulfamic acid supported onto magnetic Fe_3O_4 nanoparticles (MNPs-PSA) was simply synthesized and used as a highly efficient, environmentally friendly, and chemoselective catalyst for the synthesis of 1,1-diacetates (acylals) from the one-pot condensation reaction of various aromatic aldehydes with acetic anhydride, in high yield of products (86–96%) and short reaction time (20–60 min) under solvent-free conditions at room temperature. In addition to these results, we further studied the possibility of deprotection of the resulting acylals into benzaldehyde derivatives in this catalytic system by the addition of water. More importantly, noteworthy advantages of this study are non-use of toxic organic solvents and catalysts, simple work-up procedure, short reaction time, high yield of products, and recovery and reusability of MNPs-PSA by an external magnet.

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Graphical Abstract A simple and highly efficient procedure for the protection of various aldehydes with acetic anhydride in the presence of *N*-propylsulfamic acid supported onto magnetic Fe_3O_4 nanoparticles (MNPs-PSA) is reported. We further studied the possibility of deprotection of the resulting acylals into benzaldehyde derivatives in this catalytic system by the addition of water as a green solvent. The catalyst was reused several times without loss of its catalytic activity.



Keywords Nanocatalyst \cdot Reusable catalyst \cdot Magnetic nanoparticle \cdot *N*-Propylsulfamic acid \cdot Acylals \cdot Solvent-free

Introduction

The selective protection and deprotection of carbonyl groups are one of the most important and most versatile modern organic reactions. The protection of aldehyde compounds such as oxathioacetals, dithioacetals, acetals, and acylals are one of the most important reactions in multi-step syntheses. Among different methods for the protection of aldehyde, geminal diacetates (acylals) are appropriate candidates for this aim [1]. Also, geminal diacetate (acylals) from various aldehydes are essential for the synthesis of other compounds: for instance, as starting materials for Diels–Alder reactions, as crosslinking reagents for cellulose in cotton, and as useful intermediates in industries. Moreover, they can be converted to some appropriate functional groups using appropriate nucleophiles [2–5].

The simple and convenient separation and reusability of the catalysts are the important and unique factors in the selection of catalytic processes and green chemistry [6-10]. Application of nanoparticles (NPs), especially iron oxides, have received special attention in academic research and industry due to their simple and convenient preparation, their high surface area, low toxicity, abundance, and unique magnetic properties. In addition, they have characteristics which are useful in various fields such as magnetic resonance imaging [11], magnetic fluids [12], data

storage [13], biology and medical applications [14], and environmental remediation [15]. Also, solvent-free reactions and catalysis research are an appropriate direction for the future of organic synthesis, in particular from the point of view of green chemistry and industry [16–21].

In recent years, several catalysts or reagents such as silica-supported boron sulfonic acid (SBSA) [22], PEG-SO₃H [23], silica sulfate [24], titanium-modified MCM-41 [25], LiOTf [26], polymer-supported gadolinium triflate [27], P₂O₅/ Al₂O₃ [28], In(OTf)₃ [29], solid silica sulfuric acid [30], Fe(NO₃)₃·9H₂O [31], keggin hetero poly acid [32], ZSM-5-SO₃H [33], sulfuric acid [3-(3-silicapropyl)sulfanyl]propyl ester [34], selectfluorTM [35], Ni²⁺@hydroxyapatite-core@shell γ -Fe₂O₃ nanoparticles [36], 1-methyl-3-(2-(sulfooxy)ethyl)-1H-imidazol-3-ium chloride [37], and $Fe_3O_4@SiO_2-SO_3H$ [38] have been reported for the synthesis of geminal diacetates (acylals) from the reaction between various aldehydes and acetic anhydride under different reaction conditions. Although some of these methods are quite useful and satisfactory for the synthesis of geminal diacetates (acylals), most of them suffer from disadvantages such as the use of strong acids, reactions under oxidizing conditions, high toxicity of the reagents, prolonged reaction time, low yield of products, use of toxic and harmful solvents, requiringing high temperatures, moisture sensitivity, and the use of unrecyclable catalysts. Therefore, any effort to find a simple, inexpensive, and green procedure for the promotion of the acetylation of aldehydes with better yield might be of high potential and interest. A non-toxic, expensive, and non-recyclable catalyst (Lewis and Brønsted acid) is one of most important advantages of this method; therefore, replacement of these catalysts with magnetic nanoparticles as a reusable heterogeneous, non-corrosive nature, and eco-friendly catalysts is an area of current interest. Thus, magnetic nanoparticles can play a significant role in the development of clean technologies.

Based on our previous research in magnetic nanoparticles and green chemistry, our group prepared *N*-propylsulfamic acid supported onto magnetic Fe_3O_4 nanoparticles (MNPs-PSA) (Fig. 1) [39] as a new and heterogeneous magnetic Brønsted acid catalyst in our laboratory, and reported on the chemoselective preparation of the gem-diacetates (acylals) from a variety of aldehydes with acetic anhydride under solvent-free conditions at room temperature, and deprotection of the corresponding acylals in water using catalytic amounts of MNPs-PSA under water (Scheme 1).

Fig. 1 *N*-propylsulfamic acid supported onto magnetic Fe₃O₄ nanoparticles (MNPs-PSA)



Ar-CHO +
$$AC_2O$$

MNPs-PSA (25 mg), solvent free, r.t.
MNPs-PSA (25 mg), H₂O, r.t. Ar-CH(OAc)₂

Scheme 1 Synthesis of gem-diacetates and their deprotection using catalyst MNPs-SA

Materials and methods

All chemicals were commercially available and used without further purification. The ¹H NMR (400 MHz) and ¹³C NMR (100 MHz) were run on a Bruker Avance DPX-400 FT-NMR spectrometer (δ in ppm). Melting points were obtained in open capillary tubes and were measured on an electrothermal 9200 apparatus. Monitoring of the reactions and the purity check of the final products were carried out by thin layer chromatography (TLC) using silica gel-precoated aluminium sheets (60–120 mesh; Merck) and visualization with ultraviolet light at 365 and 254 nm. The products were characterized by comparing the physical data with those of known samples or by their spectral data. All yields refer to isolated yields.

Preparation of *N*-propylsulfamic acid supported onto magnetic Fe₃O₄ nanoparticles (MNPs-PSA)

In this research, *N*-propylsulfamic acid supported onto magnetic Fe_3O_4 nanoparticles (MNPs-PSA) was easily prepared via the condensation of MNPs powder with (3-aminopropyl)-trimethoxysilane (APTMS), and then its acidification using chlorosulfuric acid, according to the literature procedure. The mean size and surface morphology of the MNPs-SA were characterized by transmission electron microscopy (TEM), scanning electron microscopy (SEM), vibrating-sample magnetometry (VSM), X-ray diffraction (XRD) analysis, and Fourier-transform infrared (FT-IR) spectroscopy techniques [39, 40] (Scheme 2).

General procedure for the preparation of 1,1-diacetates

To a mixture of a variety of aldehydes (1 mmol) and freshly distilled Ac_2O (1.5 mmol) was added MNPs-SA (25 mg), and the whole mixture was stirred in a round-bottomed flask at room temperature for the appropriate time (Table 3). The completion of the reaction was monitored by TLC (hexane/ethyl acetate, 5:1). Then, EtOAc (20 mL) was added to the reaction mixture, and the resulting mixture was filtered and the extracts concentrated under vacuum. The pure product was isolated following silica gel column chromatography to afford pure acylals. All the products were fully characterized by ¹H NMR, ¹³C NMR, IR spectroscopy and melting points, which were consistent with the literature data.



Scheme 2 Synthesis of N-propylsulfamic acid supported onto magnetic Fe_3O_4 nanoparticles (MNPs-PSA)

General procedure for deprotection of 1,1-diacetates

To a stirred mixture of aldehyde (1 mmol) and acetic anhydride (1.5 mmol), MNPs-SA (25 mg) was added, and the mixture was stirred at room temperature for the appropriate time (Table 3). After completion of the protection reaction, water (3 mL) was added to the reaction mixture to perform the deprotection reaction. After completion of the cleavage reaction, Et_2O (20 mL) was added and the reaction mixture was filtered to separate the catalyst. The solvent was evaporated under reduced pressure to afford the corresponding aldehydes.

Results and discussion

Recently, Rostami and et al. reported *N*-propylsulfamic acid supported onto magnetic Fe_3O_4 nanoparticles (MNPs-PSA) as a nanocatalyst for the chemoselective oxidation of sulfides to sulfoxides in the absence of a solvent at ambient temperature [34]. Similarly, our group prepared *N*-propylsulfamic acid supported onto magnetic Fe_3O_4 nanoparticles (MNPs-PSA) as a nanocatalyst and its applications in another reaction. Here, we wish to report a new, rapid, and eco-friendly method for the synthesis of 1,1-diacetates (acylals) from the one-pot condensation reaction of various aromatic aldehydes with acetic anhydride in the presence of catalytic amounts of *N*-propylsulfamic acid supported onto magnetic Fe_3O_4 nanoparticles (MNPs-PSA) as a new, green and reusable nanocatalyst under solvent-free conditions at room temperature.

In the first step, to study the effect of catalyst loading, the reaction between benzaldehyde (1 mmol) and acetic anhydride (1.5 mmol) in the presence of various amounts of MNPs-PSA in the absence of a solvent at room temperature was chosen for the modeling reaction, and the results are summarized in Table 1. In the absence

Table 1 The effect of catalyst loading on the synthesis of phenylmethylene diacetate (3a)	Entry	Catalyst loading (mg)	Time (min)	Yield (%) ^a
	1	_	24 h	Trace
	2	5	75	68
	3	10	50	79
	4	15	35	88
	5	20	35	90
	6	25	20	96
^a Isolated vield	7	30	20	92

of the MNPs-PSA, the reaction was slow, boring, and no product was obtained even after 24 h (Table 1, entry 1). By increasing the amount of the MNPs-PSA, acceptable reaction times and yields were obtained (Table 1, entries 2–4), but the acylation of benzaldehyde was incomplete. An excellent yield and short reaction time was obtained when we used 25 mg of the MNPs-PSA in the same reaction (Table 2, entry 6). The yield of the compound **3a** decreased when 30 mg of the MNPs-PSA was used (Table 1, entry 7).

In the second step, to study the effect of various solvents, the reaction between benzaldehyde (1 mmol) and acetic anhydride (1.5 mmol) in the presence of 25 mg of MNPs-PSA in the various solvents at room temperature was chosen for the modeling reaction. The results are summarized in Table 2, form which it can be seen that solvent-free conditions gave the best yield and short reaction time rather than a solvent.

Hence, under solvent-free conditions at room temperature and 25 mg of MNPs-PSA, a study to explore the scope of this catalytic system was initiated (Table 3, entries 1–18). It is noteworthy that the reactions proceeded efficiently in excellent yields and short reaction times for all varieties of aldehydes (electron-donating and electron-withdrawing). In this procedure, 1-naphthol gave the corresponding 1,1diacetate in a short reaction time and excellent yield (Table 3, entry 9). This procedure is also efficient for the acetylation of heterocyclic compounds such as furanaldehyde (Table 3, entry 10) and thiophenealdehyde (Table 3, entry 11).

In the next study, in order to show the chemoselectivity of protection of aldehydes in the presence of ketones, we investigated the mixture of benzaldehyde with acetophenone in the presence of MNPs-PSA as a heterogeneous magnetic Brønsted acid catalyst under solvent-free conditions at room temperature, which

Table 2 The effect of various solvent on the synthesis of phenylmethylene diacetate (3a)	Entry	Solvent	Time (min)	Yield (%) ^a
	1	CH ₃ COOC ₂ H ₅	120	57
	2	CH ₃ CN	120	51
	3	<i>n</i> -Hexane	120	40
	4	CH_2Cl_2	90	56
^a Isolated yield	5	$C_2H_5OC_2H_5$	90	52

Entry	Substrate	Products ^a	Time (min) ^b	Yield (%) ^c	$M.p./^{\circ}C^{d}$	
					Found	Reported
1	O H	OAc OAc H	20	95	41–43	44-45 [35]
2	O H	OAc OAc H	40	92	55–57	57–58 [35]
3	O H	OAc H	30	96	97–99	99–100 [22]
4		Cl OAc H	35	90	88 –90	88-89 [28]
5	CI O H	OAc H	25	91	81–83	80-82 [35]
6	CI V	OAc OAc H	30	93	89–91	91–92 [35]
7	NO ₂	NO ₂ OAc H	25	96	125–127	126–128 [34]
8		OAc OAc H	30	91	92–94	90–91 [28]
9	O H	OAc H OAc	30	91	103–105	101–102 [35]
10	K H	OAc OAc	25	92	53–54	50-52 [35]
11	С s H	$\langle S \rangle$ (Ac)	20	92	68–70	65–67 [38]
12	О НО ОМе	OAc OAc HO OAc	60	88	92–94	89–91 [24]

Table 3 Acetylation of various aldehydes using $\mbox{Ac}_2\mbox{O}$ in the presence of MNPs-PSA at room temperature

Entry	Substrate	Products ^a	Time (min) ^b	Yield (%) ^c	M.p./°C ^d	
					Found	Reported
13	O H	OAc OAc H	40	89	101–103	102–104 [22]
14	ОН	OAc OAc H	25	89	88 –90	91–92 [28]
15	O ₂ N OH	O ₂ N OH OAc H	25	86	99 –101	100–102 [28]
16	NC O H	NC O H	25	95	61–62	63-64 [35]
17	CH30	CH ₃ O OAc H	20	90	81–83	80-82 [35]
18	H ₃ C O H	H ₃ C OAc H	25	91	92–94	93–94 [28]
19	Br OAc HO	Br OAc H	20	93	92–94	90–91 [28]

Table 3 continued

^a All the products were characterized by ¹H NMR and ¹³C NMR spectroscopy

^b Reactions were run till the completion as indicated by TLC

c Isolated yield

^d Melting points are uncorrected and compared with those reported in the literature

gave only aldehyde diacetyl. In this comparison, phenylmethylene diacetate was obtained in a short reaction time and excellent yield (Scheme 3).

The recyclability of the MNPs-PSA is a valuable advantage in green chemistry and catalysis research, and is also very important from commercial and economical points of view. In this respect, the recovery and reusability of the MNPs-PSA was investigated in the synthesis of phenylmethylene diacetate via the reaction between benzaldehyde (1 mmol) and acetic anhydride (1.5 mmol) with 25 mg of MNPs-PSA under solvent-free conditions at room temperature (Fig. 2; Table 4). After completion of the reaction, the MNPs-PSA as a catalyst was filtered by an external magnet field (Fig. 3), and then washed with ethyl acetate and tested for its activity



Scheme 3 Chemoslective acetylation of aldehydes in the presence of ketones

Fig. 2 Catalyst reusability



 Table 4
 Calculator of turnover frequency (TOF) and turnover number (TON) for the recyclability of the MNPs-PSA

	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6
TON ^a	105.5	104.4	103.3	103.3	102.2	100
TOF ^b	5.27	4.74	4.22	3.69	3.40	2.85

^a Turnover number

^b Turnover frequency

in subsequent runs. This process was repeated for 6 cycles, and phenylmethylene diacetate was obtained in excellent yields after 15 min, without significant loss of its catalytic activity.

On the other hand, the acidic content of MNPs-PSA was characterized by the determination of its H^+ of the catalyst, through titration with NaOH (0.01 mol/L). For this purpose, MNPs-PSA (25 mg) was titrated by 0.9 ml of NaOH (0.01 mol/L) and then the mol % of catalyst was calculated. Turnover frequency (TOF) and



Fig. 3 Magnetic separation of catalyst by external magnet

turnover number (TON) values for MNPs-PSA were calculated for the recyclability of the MNPs-PSA in each of the 6 runs. The TOF and TON values are summarized in Table 4.

In another study, we studied the application of MNPs-PSA for the deprotection of 1,1-diacetoxy-1-phenyl methane (Table 3, entry 1) to their corresponding benzaldehyde by the addition of water at room temperature. In this method, benzaldehyde was obtained from the related 1,1-diacetoxy-1-phenyl methane under mild reaction conditions in a short reaction time and excellent yield (Scheme 4).

As can be seen in Scheme 5, a plausible mechanism for the synthesis of geminal diacetates (acylals) promoted by *N*-propylsulfamic acid supported onto magnetic Fe_3O_4 nanoparticles (MNPs-PSA) as a Brønsted acidic ionic liquid catalyst under solvent-free conditions at room temperature can be selected as the most probable one.

Comparing the catalytic potentiality of *N*-propylsulfamic acid supported onto magnetic Fe_3O_4 nanoparticles (MNPs-PSA) in the synthesis of 1,1-diacetoxy-1-phenyl methane from benzaldehyde with acetic anhydride in the presence of various catalysts with respect to the amounts of catalyst, reaction time, and the yield of the products is presented in Table 5. As can be seen, in some methods, toxic solvents



Scheme 4 Cleavage of acylal to benzaldehyde in water catalyzed by MNPs-PSA



Scheme 5 The probable mechanism for the synthesis of geminal diacetates (acylals) N-propylsulfamic acid supported onto magnetic Fe₃O₄ nanoparticles (MNPs-PSA) as a MNPs catalyst

like CH₂Cl₂ and *n*-hexane were used (Table 5, entries 9–12), and on the other hand, in some methods, reflux conditions or high temperature were required (Table 5, entries 7–11). In other methods, using sulphated zirconia (Table 5, entry 8), TBBDA and PBBS (Table 5, entries 9 and 10), and Cu(OTf)₂ (Table 5, entry 11), the reaction time was longer and boring. Thus, the present procedure with *N*-propylsulfamic acid supported onto magnetic Fe₃O₄ nanoparticles (MNPs-PSA) as a catalyst is more convincing in proportion to the above methods in terms of reaction time and yield of the desired product and conditions of the reaction like solvent, temperature and catalyst loading.

Entry	Entry Catalyst Conditions		Time (min)	Yield (%) ^a	References
1	SBSA ^b	Solvent-free, r.t.	4	97	[22]
2	P ₂ O ₅ /Al ₂ O ₃	Neat, r.t.	45	86	[28]
3	Selectfluor TM	Solvent-free, r.t.	70	86	[35]
4	Selectfluor TM	Solvent-free, M.W.	5	99	[35]
5	γ-Fe ₂ O ₃ @HAp-Ni ^{2+c}	Solvent-free, r.t.	10	99	[36]
6	[Msei]Cl ^d	Solvent-free, r.t.	10	99	[37]
7	Si-[SbSipim][PF ₆]	Solvent-free, 30 °C	3 ^g	90	[41]
8	Sulphated zirconia	Solvent-free, 0 °C	5 ^g	99	[42]
9	TBBDA ^e	CH ₂ Cl ₂ , reflux	8 ^g	85	[43]
10	PBBS ^f	CH ₂ Cl ₂ , reflux	5 ^g	85	[43]
11	Cu(OTf) ₂	CH ₂ Cl ₂ , r.t.	4 ^g	94	[44]
12	Ruthenium(III) Chloride	<i>n</i> -hexane, r.t.	10	76	[45]
13	Saccharin sulfonic acid	Solvent-free, 90 °C	0.8 ^g	90	[46]
14	Phospho sulfonic acid	Solvent-free, r.t.	5	98	[47]
15	MNPs-PSA	Solvent-free, r.t.	20	96	-

 Table 5
 Comparison of different catalysts

^a Isolated yield

^b Silica boron sulfonic acid

^c Ni²⁺@hydroxyapatite-core@shellγ-Fe₂O₃ nanoparticles

^d 1-methyl-3-(2-(sulfooxy)ethyl)-1H-imidazol-3-ium chloride

^e N,N,N0,N0-tetrabromobenzene-1,3-disulfonamide

^f Poly(*N*,*N*0-dibromo-*N*-ethyl-benzene-1,3-disulfonamide)

g In hour

Conclusion

In summary, an effective, green, and rapid procedure has been developed for the chemoselective preparation of the geminal diacetates (acylals) from a variety of aldehydes with acetic anhydride under solvent-free conditions at room temperature and deperotection of the corresponding acylals in water using catalytic amounts of MNPs-PSA in water. This method is very selective for the preparation of 1,1-diacetates (acylals) from a variety of aldehydes in the presence of ketones (acetophenone). The simple work-up procedure, non-toxic organic solvents and catalysts, excellent catalytic capacity, chemoselectivity, short reaction times, high yield of products, and recovery and reusability of MNPs-PSA by an external magnet are advantages of this method.

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