Preparation of different amides via Ritter reaction from alcohols and nitriles in the presence of silica-bonded N- propyl sulphamic acid (SBNPSA) under solvent-free conditions

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Abstract. A number of methods have been proposed for the modification of the Ritter reaction. However, many of these methods involve the use of strongly acidic conditions, stoichiometric amounts of reagents, harsh reaction conditions and extended reaction times. Therefore, the development of mild, efficient, convenient and benign reagents for the Ritter reaction is desirable. In this research, we have developed a clean and environmentally friendly protocol for the synthesis of amides by using different benzylic or tertiary alcohols and different nitriles in the presence of silica-bonded N- propyl sulphamic acid (SBNPSA) as catalyst under solvent-free conditions in high yields.

Keywords. Ritter reaction; SBNPSA; alcohol; nitrile; amide.

1. Introduction

The conversion of nitriles to amides by reaction with alcohols or alkenes in the presence of sulphuric acid is named Ritter reaction. Acidification of the appropriate alcohol or alkene generates a carbenium ion which reacts with nitrile. While the successful course of the reaction certainly depends on the reactivity of nitrile, a major factor is the stability and reactivity of the carbocationic intermediates.[1] Ritter reaction is a well-known reaction in the synthetic organic processes which provides versatile synthetic method for preparation of a variety of amides.[2] It is also used in industrial processes as it can be effectively scaled up from laboratory experiments to large-scale applications while maintaining high yield. Real world applications include Merck's industrial-scale synthesis of anti-HIV drug crixivan (indinavir),[3] the production of the falcipain-2 inhibitor PK 11195, the synthesis of the alkaloid aristotelone,[4] and synthesis of Amantadine, an antiviral and antiparkinsonian drug.[5] Other applications of the Ritter reaction include synthesis of dopamine receptor ligands[4] and production of amphetamine from allylbenzene.[6] A problem with the Ritter reaction is the need for an extremely strong acid catalyst in order to produce the carbocation. This poses an environmental risk, as the acids are extremely corrosive and also cannot

be reused. In recent years, the search for environmentally benign chemical processes or methodologies has received much attention. Heterogenization of homogeneous catalysts has been an interesting area of research from an industrial point of view; this combines the advantages of homogeneous catalysts (high activity, selectivity, etc.) with the engineering advantages of heterogeneous catalysts (easy catalyst separation, long catalytic life, easy catalyst regeneration, thermal stability, and recyclability). Application of solid acids in organic transformations is important because the solid acids have many advantages such as simplicity in handling, decreased reactor and plant corrosion problems, and more environmentally safe disposal.[7–30] Green chemistry not only requires the use of environmentally benign reagents and solvents, but also requires the use of recyclable catalysts. One way to overcome the problem of recyclability of the traditional acid catalysts is to chemically anchor their reactive centre onto an inorganic solid carrier with a large surface area to create a new organic–inorganic hybrid catalyst.[11–16] The reactive centres in these solid-supported catalysts are highly mobile similar to homogeneous catalysts and at the same time have the advantage of recyclability like heterogeneous catalysts. In this regard, various organic and inorganic materials have been investigated; one of the most popular of these materials is silica gel with a surface area equal to $200 \, \text{m}^2/\text{g}$ that has high thermal and chemical stability (with the exception of a few

number of small nucleophiles such as OH^- and F^-) and the surface area could be extended up to $600 \, m^2/g$ or higher. Also the size of surface pores could be changed between 5–500 Å. The surface of silica gel has two functional groups that include siloxane (Si–O–Si) and silanol (Si–OH). Hence, silica gel is modified and its structure is changed via two different nucleophile substitutions on Si, or direct reaction with hydroxyl group in silanol. The second method (hydroxyl group reaction) for modified silica gel is often selected as the main approach. In view of this, several types of solid sulphonic-acid-functionalized silica (both amorphous and ordered) have been synthesized and applied as an alternative to traditional sulphonic acid resins and homogeneous acids in catalyzing chemical transformations.[11–14,17,18] As a result, a variety of acidic reagent such as: H_2SO_4,[31] TfOH,[32] Zeolits,[33,34] $MnO_2.SiO_2$,[35] $BF_3.OEt_2$,[36] $TiCl_4$,[37] $Bi(OTf)_3$,[38] $Ca(HSO_4)_2$,[39] DNBSA,[40] Fe^{3+}-K10 Montmorillonite,[41] $P_2O_5.SiO_2$,[42] $H_2PW_{12}O_{40}$[43] and $PMA.SiO_2$[44] have been reported for the synthesis of amides by using Ritter reaction. Therefore, the development of mild, efficient, convenient and benign reagents for the Ritter reaction is desirable. In this research, we have developed a clean and environmentally friendly protocol for the synthesis of amides by using different benzylic or tertiary alcohols and different nitriles in the presence of silica-bonded N-propyl sulphamic acid (SBNPSA) under solvent-free conditions with good to excellent yields. It is interesting to note that SBNPSA was first prepared by the authors of this study in 2009.[45]

2. Experimental

2.1 *General remarks*

Chemicals were purchased from Merk and Fluka Companies. IR spectra were run on a Shmadzu IR-435 FTIR spectrophotometer. ^{1}H NMR was run on Bruker Avence (DRX 500 MHz) and Bruker UltraShield (400 MHz). Melting points were recorded on a Melting Point SMP1 apparatus in open capillary tubes. The progress of reaction was followed with TLC, using silica gel SILG/UV 254 plates. All the products are known and were characterized by comparison of their spectra (IR, ^{1}H NMR), TLC and physical data with those reported in the literature.[46]

2.2 *General procedure*

Diphenyl methanol (1.0 mmol, 0.18 g) and cyclopropanecarbonitrile (1.0 mmol, 0.07 mL) were mixed with SBNPSA (0.1 g) under solvent free condition and the mixture was heated to 80°C (see table 1, entry 3i). After completion of the reaction as indicated by TLC, the reaction mixture was diluted with EtOAc (10.0 mL). The catalyst was separated from the reaction mixture by simple filtration. Then, the resulting product was diluted (EtOAc-n-hexane, 2:8) to prepare the pure amide in crystalline form. Compounds **3a**, **3b**,[38] **3c**, **3d**, **3m**, **3n**,[44] **3g**, **3p**,[47] **3q**, **3r**, **3s**, **3t**[39] are reported in the literature. Spectroscopic data for selected compounds are presented below:

2.2a *N- (t-phenyl methano)-2- methoxy ethyl carboxamide: (3f)*: White solid; m.p. = 160–161°C; IR (KBr): ν_{max}: 3500, 3250, 3000, 1640, 1520, 1480, 1430, 1120 cm^{-1}. ^{1}H NMR (CDCl$_3$, 500 MHz): δ (ppm) 2.55 (t, 2H, J = 5.5 Hz), 3.41 (s, 3H), 3.71 (t, 2H, J = 5.5 Hz), 7.26 (m, 7H), 7.29 (m, 2H), 7.33 (m, 6H), 7.74 (s, 1H). ^{13}C NMR (CDCl$_3$, 125 MHz): δ (ppm) 38.6, 59.24, 69.30, 70.72, 127.33, 128.31, 129.1, 145.3, 170.8.

2.2b *N- (d-phenyl methano)-2- methoxy ethyl carboxamide: (3h)*: white solid; m.p. = 96–97°C; IR (KBr): ν_{max}: 3500, 1640, 1540, 1120. ^{1}H NMR (CDCl$_3$, 500 MHz): δ (ppm) 2.57 (t, 2H, J = 5.5 Hz), 3.40 (s, 3H), 3.71 (t, 2H, J = 5.5 Hz), 6.29 (d, 1H, J = 10 Hz), 6.93 (d, 1H, J = 10 Hz), 7.26 (d, 4H, J = 7.8 Hz), 7.29 (m, 2H), 7.35 (m, 4H). ^{13}C NMR (CDCl$_3$, 125 MHz): δ (ppm) 37.59, 57.16, 59.20, 69.20, 127.75, 127.77, 129.1, 142.21, 170.98.

2.2c *N- (d-phenyl methano)-cyclo propan carboxamide: (3i)*: white solid; m.p. = 173–175°C; IR (KBr): ν_{max}: 3500, 3300, 3050, 1620, 1540, 1480. ^{1}H NMR (CDCl$_3$, 500 MHz): δ (ppm) 0.77 (m, 2H), 1.03 (m, 2H), 1.45 (m, 1H), 6.30 (d, 1H, J = 5.6 Hz), 6.31 (d, 1H, J = 5 Hz) 7.3 (m, 6H), 7.36 (m, 4H). ^{13}C NMR (CDCl$_3$, 125 MHz): δ (ppm) 7.78, 15.24, 57.51, 127.83, 127.86, 129.1, 142.16, 173.1.

2.2d *N- (d-phenyl methano)-buthyl carboxamide (3j)*: white solid; m.p. = 122–124°C; IR (KBr): ν_{max}: 3800, 3400, 2800, 1670, 1550, 1480, 700. ^{1}H NMR (CDCl$_3$, 500 MHz): δ (ppm) o.95 (t, 3H, J = 7.5 Hz), 1.38(m, 2H), 1.68 (m, 2H), 2.29(t, 2H, J = 7.5), 6.11 (d, 1H, J = 5 Hz), 6.3 (d, 1H, J = 5 Hz), 7.26 (d, 4H, J = 7.3 Hz), 7.3 (d of d, 2H, J$_1$ = 8.5 Hz, J$_2$ = 5 Hz), 7.36 (t, 4H, J = 7.1 Hz). ^{13}C NMR (CDCl$_3$, 125 MHz): δ (ppm) 14.22, 22.86, 28.24, 36.98, 57.22, 127.84, 129.07, 142.1, 172.56.

Table 1. SBNPSA catalysed amidation of alcohols with nitriles (the reactions were carried out at 80°C under solvent-free conditions).

Entry	Alcohol (1)	Nitrile (2)	Amide (3)[a]	Time (min)	Yield[b] (%)	M.P(°C)
3a[38]	Me—C(Me)(Me)—OH	C₆H₅-CN	(Me)₃-C-NH-C(=O)-C₆H₅	45	98	133–135
3b[38]	Me—C(Me)(Me)—OH	▷—CN	(Me)₃-C-NH-C(=O)-◁	80	96	140–141
3c[44]	C₆H₅-CH(OH)-Me	Cl-CH₂-CN	C₆H₅-CH(Me)-NH-C(=O)-CH₂-Cl	70	94	77–78
3d[44]	C₆H₅-CH(OH)-Me	C₆H₅-CN	C₆H₅-CH(Me)-NH-C(=O)-C₆H₅	40	92	114–116
3e	(C₆H₅)₃C-OH	CH₃CN	(C₆H₅)₃C-NH-C(=O)-CH₃	40	94	199–200
3f	(C₆H₅)₃C-OH	CH₃O-CH₂-CH₂-CN	(C₆H₅)₃C-NH-C(=O)-CH₂-CH₂-OCH₃	170	91	160–161

2.2e N-(d-phenyl methano)-3-choloro benzo carboxamide: (3k): white solid; m.p. = 153–155°C; IR (KBr): ν_{max}: 3500, 1640, 1520, 1480, 700. ¹H NMR (CDCl₃, 500 MHz): δ (ppm) 6.47 (d, 1H, J = 5 Hz), 6.66 (d, 1H, J = 5 Hz), 7.32 (m, 6H), 7.40 (m, 5H), 7.51 (d, 1H, J = 7.8 Hz), 7.72 (d, 1H, J = 7.5 Hz), 7.83 (s, 1H). ¹³C NMR (CDCl₃, 125 MHz): δ (ppm) 58.04, 125.59, 127.79, 127.90, 128.13, 129.24, 130.41, 132.16, 135.27, 136.45, 141.56.

2.2f N-(d-phenyl methano)-cyclo hexane carboxamide (3o): white solid; m.p. = 184–186°C; IR (KBr): ν_{max}: 3500, 3200, 2900, 1640, 1540, 1480, 1440. ¹H NMR (CDCl₃, 500 MHz): δ (ppm) 1.28 (m, 3H), 1.52 (m, 2H), 1.81 (m,1H), 1.84 (m, 2H), 1.94 (m, 2H), 2.20 (m, 1H), 6.08 (d, 1H, J = 5 Hz), 6.28 (d, 1H, J = 5 Hz), 7.24 (d, 4H, J = 7.2 Hz), 7.29 (t, 2H, J = 5 Hz), 7.35 (t, 4H, J = 5.6 Hz). ¹³C NMR (CDCl₃, 125 MHz): δ (ppm) 26.14, 30.13, 45.95, 56.94, 127.81, 129.1, 142.18, 175.41.

3. Results and discussion

In this paper, an efficient, complete and high yielding Ritter reaction of alcohols with nitriles in the presence of Silica-bonded N-propyl sulphamic acid has been examined as a catalyst under solvent-free conditions (figure 1). Silica-bonded N-propyl sulphamic
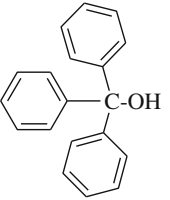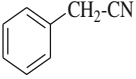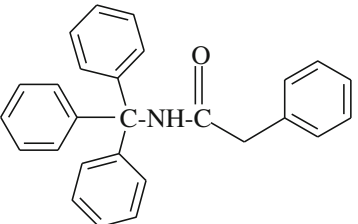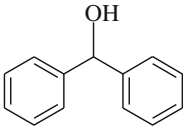
Table 1. *(continued)*
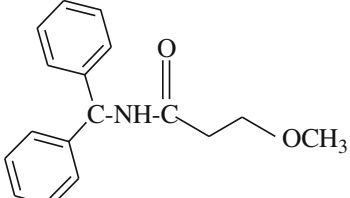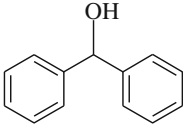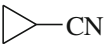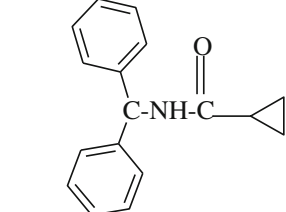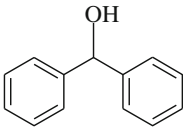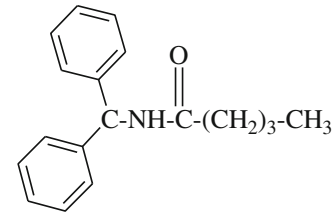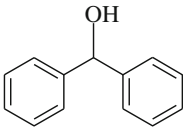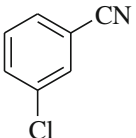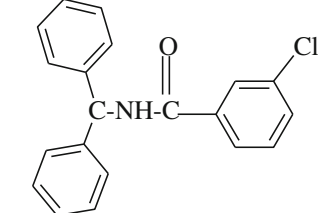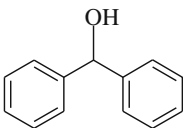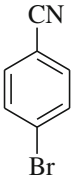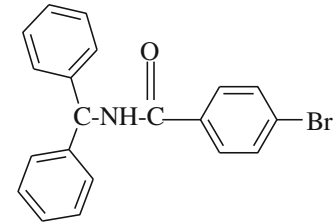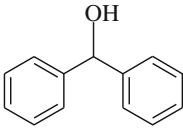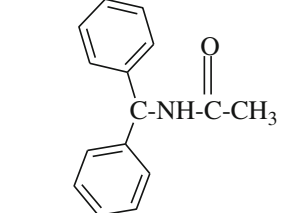
Entry	Alcohol	Nitrile	Amide	Time (min)	Yield (%)	M.P
3g [47]	(triphenylmethanol) C-OH	(benzyl) CH$_2$-CN	(triphenylmethyl) C-NH-C(=O)-CH$_2$-phenyl	180	93	173–174
3h	(diphenylmethanol) OH	CH$_3$O-CH$_2$-CH$_2$-CN	C-NH-C(=O)-CH$_2$-CH$_2$-OCH$_3$	60	91	96–97
3i	(diphenylmethanol) OH	(cyclopropyl) CN	C-NH-C(=O)-cyclopropyl	25	94	173–175
3j	(diphenylmethanol) OH	CH$_3$-(CH$_2$)$_3$-CN	C-NH-C(=O)-(CH$_2$)$_3$-CH$_3$	45	96	122–124
3k	(diphenylmethanol) OH	3-chlorobenzonitrile CN, Cl	C-NH-C(=O)-(3-Cl-phenyl)	25	95	153–155
3l	(diphenylmethanol) OH	4-bromobenzonitrile CN, Br	C-NH-C(=O)-(4-Br-phenyl)	20	94	146–148
3m [44]	(diphenylmethanol) OH	CH$_3$CN	C-NH-C(=O)-CH$_3$	15	98	143–144

Table 1. *(continued)*

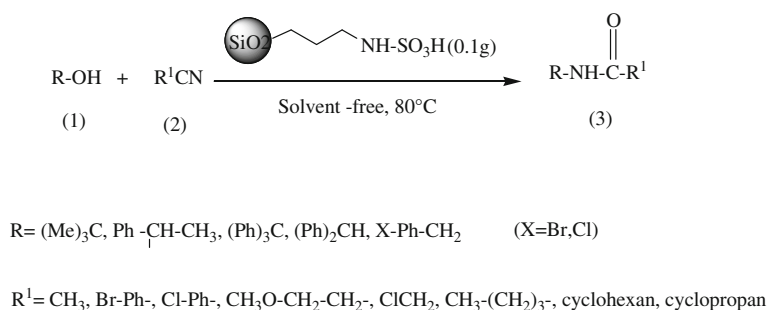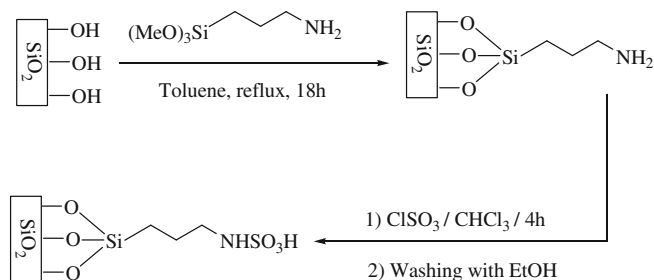| Entry	Alcohol	Nitrile	Amide	Time (min)	Yield (%)	M.P
3n [44]	(diphenylmethanol)	Cl-CH₂-CN	(amide structure)	20	96	123–125
3o	(diphenylmethanol)	CN (cyclohexanecarbonitrile)	(amide structure)	20	94	184–186
3p [47]	(4-chlorobenzyl alcohol)	CN (benzonitrile)	(amide structure)	50	96	133–135
3q [39]	(4-chlorobenzyl alcohol)	CH₃CN	(amide structure)	150	92	93–95
3r [39]	(4-chlorobenzyl alcohol)	Cl-CH₂-CN	(amide structure)	120	93	110–111
3s [39]	(4-bromobenzyl alcohol)	CH₃CN	(amide structure)	60	93	101–102
3t [39]	(4-bromobenzyl alcohol)	Cl-CH₂-CN	(amide structure)	90	91	115–116

[a]All products were characterized by IR, NMR spectra.
[b]Yield refers to pure products after re-crystallization with EtOH/*n*-hexane(2:8).

acid (SBNPSA) preparation is described as illustrated in figure 2.[45]

In order to optimize the reaction conditions, we have studied the reaction of diphenyl methanol with acetonitrile (table 1, entry **3 m**) in the presence of SBNPSA at different amounts of catalyst (table 2, entry 2) and various solvents (table 3, entry 5) and different temperatures (table 4, entry 2).

The optimized condition with respect to the ratio of nitrile/alcohol/cat./temperature was 1.0 mmol/ 1.0 mmol/0.1 g/80°C. Accordingly, treatment of diphenyl methanol with acetonitrile in the presence of opti-

R= (Me)₃C, Ph -CH-CH₃, (Ph)₃C, (Ph)₂CH, X-Ph-CH₂   (X=Br,Cl)

R¹= CH₃, Br-Ph-, Cl-Ph-, CH₃O-CH₂-CH₂-, ClCH₂, CH₃-(CH₂)₃-, cyclohexan, cyclopropan

Figure 1. General scheme of Ritter reaction.

Figure 2. Preparation of silica bonded N-propyl sulphamic acid (SBNPSA).

Table 2. Optimization of catalyst's amount.

Entry	Catalysis (g)	Time (min)	Yield (%)
1	0.05	90	98
2	**0.1**	**15**	**98**
3	0.2	15	93
4	0.3	15	93

Table 3. Selection of the appropriate solvent.

Entry	Solvent	Time (h)	Yield (%)
1	*n*-hexane (reflux)	24	<10
2	CH₂Cl₂ (reflux)	24	20
3	Water (80°C)	24	10>
4	EtOAc (reflux)	24	10>
5	**Solvent-free (80°C)**	**15 min**	**98**
6	CHCl₃(reflux)	24	20

mized condition gave the corresponding N-(diphenyl methano)-2-methyl carboxamide with 98% yield. Then, we applied this ratio for the reaction of other nitriles with alcohols. All the reactions were completed and proceeded well in high yields (91–98%) as summarized in table 1.

Table 4. Optimization of temperature.

Entry	Temperature (°C)	Time (min)	Yield (%)
1	50	120	80
2	**80**	**15**	**98**
3	100	10	93
4	r.t	24 h	20
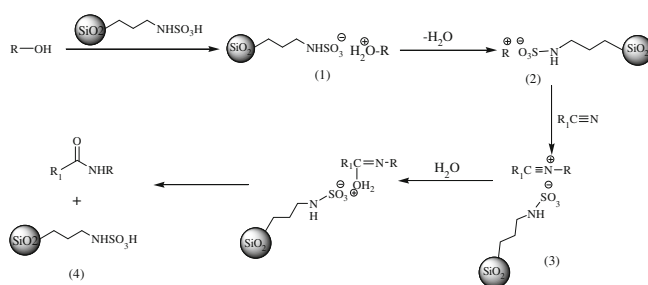
Figure 3. Proposed mechanism for the conversion of alcohols and nitriles into the corresponding amides by using SBNPSA.

Table 5. Recyclability of SBNPSA (0.1 g).

Entry	Time (min)	Yield (%)
1	15	98
2	15	98
3	30	95
4	60	90
5	360	70

Based on the presented results in table 1, it can be concluded that:

(i) The entire reactions in the presence of SBNPSA are completed.

(ii) Generally, amidation of alcohols with nitriles in the presence of an SBNPSA catalyst could be done in a short time.

(iii) Diphenyl alcohols perform amidation reaction directly and in a short time compared to tertiary, triphenyl and benzyl alcohols.

Table 6. A comparative study for the Ritter reaction using solid acids.

Entry	Alcohol	Nitrile	Time /yield (%)				
			I	II	III	IV	V
1	H₃C–C(CH₃)(CH₃)–OH	CN (benzonitrile)	45 min/98	2 h/91	17 h/85	10 h/99	6.5 h/95
2	(diphenylmethanol) OH	CH_3CN	15 min/98	–	–	–	6.5 h/87
3	(1-phenylethanol) OH CH₃	$ClCH_2CN$	70 min/94	2 h/85	–	–	6.5 h/87

I= SiO_2-CH_2-CH_2-CH_2-NH-SO_3H; II= $Ca(HSO_4)_2$; III= $Bi(OTf)_3$ (20 mol%); IV= $H_3PW_{12}O_{40}$; V= PMA/SiO_2.

(iv) This catalyst selectively performs the amidation reaction of tertiary alcohols, benzyl, diphenyl and triphenyl alcohols.

(v) The SBNPSA is a clean catalyst and can be separated simply from the reaction mixture.

The Ritter reaction proceeds by the electrophilic addition of either the carbenium ion or covalent species[48,49] to the nitrile. The resulting nitrilium ion is hydrolyzed by water to the desired amide.

Based on the obtained results, the proposed mechanism could be explained as follows (figure 3). Initially, alcohol reacts with the catalyst (SBNPSA) (1). Then, alcohol taking a proton and releasing water constructs carbocation intermediate (2). Nitrile reacts with carbocation intermediate and results in N-alkyl nitronium intermediate (3). Finally, by hydrolysis of N-alkyl nitronium intermediate and tautomery (proton transfer), the product will be produced (4).

The possibility of recycling the catalyst was examined using the reaction of diphenyl methanol and acetonitrile under the optimized conditions. On completion, the reaction mixture was washed with EtOAc (10 mL).The catalyst was separated by filtration. The recovered catalyst was washed with ethanol, dried and reused for subsequent runs. The recycled catalyst could be reused four times without any additional treatments (table 5).

The results obtained from SBNPSA catalyst was compared with the results of other catalysts reported by other researchers (table 6). For instance, the reaction of *t*-butanol with benzonitrile (entry 1) in the presence of SBNPSA for 45 min was performed with 98% yield. The same reactions in the presence of Ca (HSO₄)₂ for 2 h, Bi (OTf)₃ for 17 h, H₃PW₁₂O₄₀ for 10 h and PMA/SiO₂ for 6.5 h were resulted in 91%, 85%, 99% and 95% yield, respectively. So, the achieved yield with the application of SBNPSA was superior to other catalysts. On the other hand, the time of reaction for the implemented catalyst of this study was shorter than the other ones and in the other cases, the reported times for finishing the reaction were longer. Hence, high speed reaction with a higher yield was the strength and advantage for the applied catalyst (SBNPSA).

4. Conclusions

Based on the findings of this study, we introduced SBNPSA as a solid acid catalyst to increase the speed of synthesis of amides. The proposed catalysis showed a good ability to speed up these reactions. The applied method with SBNPSA had several advantages such as high yields, taking less reaction time, simple separation and purification.

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