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PPA-SiO₂-Catalyzed Multicomponent Synthesis of Amidoalkyl Naphthols

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PPA-SiO₂-Catalyzed Multicomponent Synthesis of Amidoalkyl Naphthols

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Abstract: Silica gel–supported polyphosphoric acid (PPA-SiO₂) was found to be an efficient catalyst for the multicomponent condensation reaction of aryl aldehydes, 2-naphthol, and urea or amides to afford the corresponding 1-amidoalkyl-2-naphthols in good to excellent yields. This new approach consistently had short reaction times, high conversions, clean reaction profiles, and simple experimental and workup procedures.

Keywords: Amide, aryl aldehyde, multicomponent reaction, 2-naphthol, PPA-SiO₂

INTRODUCTION

Multi component reactions (MCRs) make possible the speedy synthesis of molecular libraries that have a high degree of structural diversity. Combinations of different starting materials can produce a variety of products with facility, which is of great value in the search for new drugs and chemical compounds.^[1]

Compounds bearing 1,3-amino-oxygenated functional groups are ubiquitous to a variety of biologically important natural products and potent drugs, including a number of nucleoside antibiotics and HIV protease inhibitors, such as ritonavir and lipinavir.^[2,3] Our research is an advance in multicomponent synthetic methodology of a class of biologically important molecules; it is noteworthy that 1-amidomethyl-2-naphthols can convert to

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Scheme 1. One-pot preparation of 1-amidoalkyl-2-naphthols using PPA-SiO₂ as catalyst.

important biologically active 1-aminomethyl-2-naphthol derivatives by an amide hydrolysis reaction. The hypotensive and bradycardiac effects of these compounds have been evaluated.^[2–4]

As reported in a previous article,^[5] the reaction of 2-naphthol with aromatic aldehydes in the presence of an acid catalyst is known to give *ortho*-quinone methides (*o*-QMs). The *o*-QMs, generated in situ, have been reacted with 2-naphthol and prepared aryl 14H-dibenzo[a,j]xanthene derivatives. In this research, reaction of *o*-QMs with another nucleophiles such as amides for synthesis of 1-amidoalkyl-2-naphthol derivatives in the presence of PPA-SiO₂ as solid acid heterogeneous catalyst was investigated (Scheme 1).

The preparation of 1-amidoalkyl-2-naphthols can be carried out by condensation of aryl aldehydes, 2-naphthol, and acetonitrile or ethaneamide in the presence of Lewis or Brønsted acid catalysts such as montmorillonite K10 clay,^[6] Ce(SO₄)₂,^[7] iodine,^[8] K₅CoW₁₂O₄₀·3H₂O,^[9] *p*-toluene sulfonic acid (*p*-TSA),^[10] sulfamic acid,^[11] cation-exchanged resins,^[12] silica-sulfuric acid,^[13] SiO₂-HClO₄,^[14] and NaHSO₄·H₂O.^[15] However, some of the reported methods suffer from drawbacks such as unsatisfactory yields,^[7] long reaction times,^[9,10] and environmental pollutants.^[8] Therefore, a great need still exists for versatile, simple, and environmentally friendly processes whereby 1-amidoalkyl-2-naphthols may be formed under simple practical conditions. PPA-SiO₂ has emerged as a promising solid acid catalyst for acid-catalyzed reactions such as conversion of carbonyl compounds into oxathioacetals and dithioacetals^[16] and cyclodehydration of dimedone and aldehydes.^[17]

RESULTS AND DISCUSSION

The one-pot synthesis of amidoalkyl naphthols was achieved by the three-component condensation of aryl aldehydes, 2-naphthol, and amide or urea derivatives in the presence of PPA-SiO₂ as a heterogeneous catalyst (Scheme 1). To find optimum conditions, the reaction of benzaldehyde (1 equiv.), 2-naphthol (1 equiv.), and acetamide (1 equiv.) in the presence of PPA-SiO₂ (0.03 g) as a model was performed under thermal solvent-free conditions at different temperatures in an oil bath. As can be seen from Table 1, the shortest time and best yield were achieved at 120 °C.

Next, to determine the optimum quantity of PPA-SiO₂, the reaction of 2-naphthol, benzaldehyde, and acetamide was carried out under the previously mentioned conditions using different quantities of catalyst at 120 °C. The use of 30 mg of catalyst resulted in the highest yield in 7 min min (Table 1). A slight excess of the acetamide was advantageous; therefore the molar ratio of 2-naphthol, aldehyde, and acetamide was kept at 1:1:1.2, respectively.

Thus, we prepared a range of amidoalkyl naphthols under the optimized reaction conditions. In all cases, aromatic aldehydes reacted successfully and gave the products in good to excellent yields. Aromatic aldehydes with electron-withdrawing groups, reacted faster than those with electron-releasing groups, as expected (Table 2). Sterically hindered aromatic aldehydes required longer reaction times (Table 2, entry 20). Aliphatic aldehydes such as propionaldehyde were also examined, but the yield of product was low (49%) after 30 minutes at 120 °C (Table 2, entry 26).

A possible mechanism for this transformation is proposed in Scheme 2. As reported in the literature,^[10] reaction of 2-naphthol with

Entry	Amount of SiO ₂ -PPA (g)	Temperature (°C)	Time (min)	Yield (%) ^a
1	0.03	100	17	84
2	0.03	70	85	62
4	0.03	120	7	86
5	0.05	120	6	85
3	0.08	120	6	70
6	0.015	120	10	83

 Table 1. Optimization of the amount of catalyst and temperature in the preparation of N-[phenyl-(2-hydroxynapthalen-1-yl)-methyl]acetamide

^aYield refers to the isolates pure products.

238-239 (234-236)^[11] 200-202 (190-191)^[11] 168-170 (172-174)^[11] 245-246 (241-243)^[7] Mp (%C) (Lit. mp) Yield (%)^a 93 80 95 $(84-86)^{b}$ Time (min) Ś ~ 4 **NHCONHMe NHCONH**2 **NHCOPh** NHCOMe Product(s) ЧQ НŎ НŎ HO. MeHNCONH₂ Amide of urea H₂NCONH₂ CH₃CONH₂ PhCONH₂ Aldehyde СНО CHO CHO CH0 Entry 2 3 4

Table 2. Synthesis of 1-amidoalkyl-2-naphthol derivatives

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Table 2. Continued

ΙЩ	Entry	Aldehyde	Amide of urea	Product(s)	Time (min)	Yield (%) ^a	Mp (%C) (Lit. n
	0	0 ₂ N CHO	MeHNCONH ₂	02N A NHCONHMe	S	96	210–212 (191–192
	-	0,N CHO	H ₂ NCONH ₂	O ₂ N ALCOHI2	Q	95	189–190 (184–186
	7	McCHO	CH ₃ CONH ₂	Me NHCOMe	6	87	222–223 (222–223
-	Ω	Me	PhCONH ₂	Methon	×	77	175–177 (177–178
-	4	Ме	M¢HNCONH ₂	Me h H H H H H H H H H H H H H H H H H H	Ξ	83	178–180 (174–176



Table 2. Continued

	Entry	Aldehyde	Amide of urea	Product(s)	Time (min)	Yield $(\%)^a$	Mp (%C) (Lit. mp)
3382	20	CHO Me	CH ₃ CONH ₂	And the second s	17	76	199–202 (200–202) ^[11]
	21	McO ^{M2} CHO CH,CONH2	CH ₃ CONH ₂	Med	10	87	185–187 (184–186) ^[7]
	22	мео Сно снусоии2	CH ₃ CONH ₂	Meo H NHCOMe	6	71	235–237 (235–236) ^[7]



^aYield refers to the isolated pure products. All known products have been reported previously in the literature and were character-ized by comparison of IR and NMR spectra with authentic samples.^[6–15]

^bYield after recovery of catalyst five times.



Scheme 2. Suggested mechanism for preparation of 1-amidoalkyl-2-naphthols.

aromatic aldehydes in the presence of an acid catalyst is known to give *ortho*-quinone methides (*o*-QMs). The same *o*-QMs, generated in situ, have been reacted with acetamide via conjugate addition to form 1-amidoalkyl-2-naphthol derivatives. Electron-withdrawing groups on the benzaldehyde in the *o*-QMs intermediates increase the rate of the 1,4-nucleophilic addition reaction because the alkene lowest unoccupied molecular orbital (LUMO) is at lower energy in the presence of electron-withdrawing groups compared with electron-donating groups.^[18]

The recyclability of the catalyst in the reaction of benzaldehyde (10 mmol), 2-naphthol (10 mmol), and acetamide (12 mmol) in the presence of PPA-SiO₂ (0.3 g) was checked (Table 2, entry 1). The separated catalyst can be reused after washing with acetone and drying at 100 °C. The catalyst was recovered in excellent yields and used in the reaction five times; it showed the same activity as fresh catalyst without any loss of its activity.

The stability of the solid-supported catalyst was examined to evaluate if the PPA from PPA-SiO₂ could be leached out by solvent. In a control experiment, the catalyst was removed by simple filtration after washing with acetone at half of the reaction time. The filtrate solution was evaporated, and the reaction was continued using the resulting mixture. The reaction stopped in the absence of the solid heterogeneous catalyst (Table 2, entry 1). Thus, the absence of free PPA species in the reaction medium was confirmed. When the separated catalyst was transferred to the reaction medium, the conversion of benzaldehyde to N-[phenyl-(2-hydroxy-napthalen-1-yl)-methyl]-acetamide progressed and completed. To reconfirm that there were no active catalyst species in solution, the PPA-SiO₂ catalyst was subjected to vigorous stripping in acetone for 1h. The catalyst was filtered, and the filtrate solution was evaporated. No residue and species of PPA was observed by IR spectroscopy. This phenomenon confirmed that PPA bonded on the silicagel surface and that this bond is stable in reaction media. This evidence showed that the process is heterogeneous and that no leaching occurs. In addition, the weight of the recovered catalyst is the same as the amount of the fresh catalyst that was used the first time in the reaction.

Amidoalkyl Naphthol Synthesis

To show the merit of the present work in comparison with reported results in the literature, we compared reactions of PPA-SiO₂ with montmorillonite K10 clay,^[6] Ce(SO₄)₂,^[7] iodine,^[8] K₅CoW₁₂O₄₀·3H₂O,^[9] *p*-TSA,^[10] sulfamic acid,^[11] cation-exchanged resins,^[12] silica-sulfuric acid,^[13] and SiO₂-HClO₄^[14] in the synthesis of 1-amidomethyl-2naphthol derivatives. As shown in Table 3, PPA-SiO₂ is a better catalyst with respect to reaction times and yields of the obtained products. Thus, the present protocol with PPA-SiO₂ catalyst is convincingly superior to the recently reported catalytic methods.

CONCLUSION

In conclusion, a reliable, rapid, and environmentally benign method for synthesizing 1-amidoalkyl-2-naphthols has been developed, which involves the use of PPA-SiO₂. In addition to the purity of the products, the short reaction times and ease of workup make the method advantageous.

EXPERIMENTAL

All reagents were purchased from Merck and Aldrich and used without further purification. All yields refer to isolated products after purification. Products were characterized by comparison with authentic samples and by spectroscopy data (IR, ¹H NMR spectra). The NMR spectra were recorded on a Bruker Avance DPX 500-MHz instrument. The spectra were measured in dimethyl sulfoxide-d₆ (DMSO-d₆) relative to tetramethylsilane (TMS, 0.00 ppm). Mass spectra were recorded on an Agilent Technologies 5973 network mass selective detector (MSD) operating at an ionization potential of 70 eV. Elemental analyses for C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer. Melting points were determined in open capillaries with a Buchi 510 melting-point apparatus. Thin-layer chromatography (TLC) was performed on silicagel polygram SIL G/UV 254 plates.

Preparation of PPA-SiO₂

PPA (2.1 g) was charged in a round-bottom flask, and CHCl₃ (100 mL) was added. After the mixture was stirred at 50 °C for 1 h, SiO₂ [Merck, (100–200 mesh), 4.91 g] was added to the solution, and the mixture was stirred for another 1 h. The CHCl₃ was removed with a rotary

Entry	Catalyst	Catalyst and conditions	Time	Yield (%)
1	Ce(SO ₄) ₂	100 mol%; under reflux	36 h	72
2	I_2	5 mol%; solvent-free, 125°C	$5.5\mathrm{h}$	85
Э	Montmorillonite K10	0.1 g; solvent-free, 125 °C	1.5 h	89
	Clay			
4	$K_5CoW_{12}O_{40}.3H_2O$	0.01 (1 mol%); solvent-free, 125 °C	$2 \mathrm{h}$	90
5	Cation-exchanged resins	0.25 g; solvent-free, 110 °C	20 min	81
9	p-TSA	10 mol%; solvent-free, 125 °C	5 h	88
7	Sulfamic acid	50 mol%; solvent-free, ultrasonic; 28-30 °C	15 min	89
8	Silica sulfuric acid	0.02 g; solvent-free, rt	2 h	85
6	SiO ₂ -HClO ₄	0.006 g (0.6 mol%); solvent-free, 125 °C	40 min	89
10	$PPA-SiO_2$	$0.03 \text{ g} (1.5 \text{ mol}\%)$; solvent-free, 120°C	7 min	86

Table 3. Comparison results of PPA-SiO₂ with other catalysts reported in the literature^a

"Based on 2-naphthol (1 equiv.), benzaldehyde (1 equiv.) and acetamide (1.2 equiv.).

Amidoalkyl Naphthol Synthesis

evaporator, and the resulting solid was dried in vacuum at room temperature for 3 h (50 mg of PPA-SiO₂, 0.025 mmol).^[16]

General Procedure

PPA-SiO₂ (30 mg) was added to a mixture of 2-naphthol (1 mmol), aldehydes (1 mmol), and acetamide or urea (1.2 mmol). The mixture was stirred at 120 °C in an oil bath, and the reaction was followed by TLC. After completion of the reaction, the mass was cooled to 25 °C, the solid residue was dissolved in acetone, and the mixture was stirred for 5 min. The catalyst was filtered and washed with acetone, then dried in an oven at 100 °C. The solvent of the filtrate solution was evaporated, and the remaining solid product was recrystallized in aqueous EtOH (15%).

All of the desired pure product(s) were characterized by comparison of their physical data with those of known compounds.^[6–15]

Spectral Data of Two Representative Amidoalkyl Naphthols

N-[(4-Methylphenyl)-(2-hydroxynapthalen-1-yl)-methyl]-acetamide (Table 2, Entry 12)

Mp: 222–223 °C]; ¹H NMR (500 MHz, DMSO-d₆): $\delta = 1.96$ (s, 3H), 2.21 (s, 3H), 7.08–7.03 (m, 5H), 7.19 (d, J = 8.8 Hz, 1H), 7.24 (t, J = 7.1 Hz, 1H), 7.34 (m, 1H), 7.74 (d, J = 8.8 Hz, 1H), 7.78 (d, J = 7.9 Hz, 1H), 7.82 (brd, 1H), 8.36 (d, J = 8.1 Hz, 1H), 9.91 (s, 1H) ppm; ¹³C NMR (125 MHz, DMSO-d₆): 20.4, 22.6, 47.6, 118.4, 118.9, 122.2, 123.1, 125.9, 126.1, 128.3, 128.4, 128.9, 132.2, 134.9, 139.4, 143.3, 152.9, 168.9 ppm; IR (KBr, cm⁻¹): 3419, 3316, 3070, 1621, 1595, 1561, 1514, 1466, 1392, 1283, 1202, 1141, 1051, 939, 884, 784, 745, 712; MS (EI, 70 eV): m/z = 305 (M⁺, 21.00%), 246 (29.16%), 245 (50.55%), 231 (100.00%), 232 (31.20%), 202 (16.12%), 115 (10.04%). Anal. calcd. for C₂₀H₁₉NO₂: C, 78.66; H, 6.27; N, 4.59%. Found: C, 78.72; H, 6.21; N, 4.63%.

N-[(4-Nitrophenyl)-(2-hydroxynapthalen-1-yl)-methyl]-acetamide (Table 2, Entry 15)

Mp: 247–249 °C); ¹H NMR (500 MHz, DMSO-d₆): $\delta = 2.02$ (s, 3H), 7.19 (d, J = 8.0, 1H), 7.22 (d, J = 8.8, 1H), 7.28 (t, J = 7.47 Hz, 1H), 7.41 (t, J = 7.34, 1H), 7.52–7.58 (m, 2H), 7.81 (t, J = 9.38 Hz, 2H), 7.89 (brd, 1H), 8.05–8.03 (m, 2H), 8.60 (d, J = 8.0 Hz, 1H), 10.11 (s, 1H)

ppm; ¹³C NMR (125 MHz, DMSO-d₆): 22.5, 47.6, 117.7, 118.4, 120.3, 121.1, 122.5, 126.7, 128.6, 129.4, 129.8, 132.1, 132.7, 145.3, 147.7, 153.2, 169.5 ppm; IR (KBr, cm⁻¹): 3391, 3267, 2593, 1648, 1603, 1522, 1438, 1063, 825, 739, 447; MS (EI, 70 eV): m/z = 336 (M⁺, 26.66%), 319 (75.99%), 276 (52.02%), 260 (54.15%), 231 (63.80%), 202 (45.11%), 230 (100.00%), 115 (18.05%). Anal. calcd. for C₁₉H₁₆N₂O₄: C, 67.85; H, 4.79; N, 8.33%. Found: C, 67.91; H, 4.81; N, 8.24%.

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