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One-pot Synthesis of 1-Amidoalkyl-2-naphthols Catalyzed by Polyphosphoric Acid Supported on Silica-coated NiFe_2O_4 Nanoparticles

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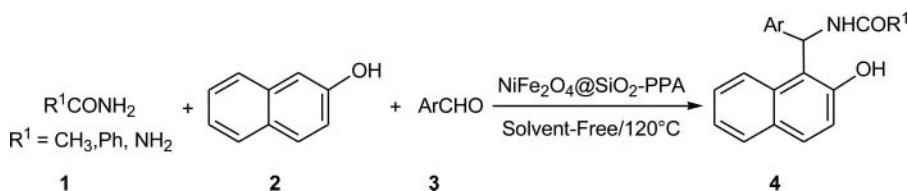
Magnetic nanoparticles (MNPs) have proven to be a valuable asset in organic chemistry. There have been several studies carried out on their biological and technological applications such as drug delivery,^{1,2} magnetic resonance imaging (MRI),³ bio-separation,^{4,5} bimolecular sensors^{6,7} and magneto-thermal therapy.^{8,9} Recent reports show that the use of magnetic nanoparticles can facilitate the isolation and recycling of expensive catalysts from the reaction mixtures.^{10,11}

1-Amidoalkyl-2-naphthols (**4**) have attracted considerable attention due to their presence as a part of some biologically important natural products and as crucial building blocks for a variety of pharmaceuticals, and potent drugs, including a number of nucleoside anti-biotics and HIV protease inhibitors.¹² Furthermore, hydrolysis of these compounds lead to 1-aminoalkyl-2-naphthols¹³ which are useful as hypotensive and bradycardiac agents¹⁴ and may also be converted to 1,3-oxazine derivatives;¹⁵ 1,3-oxazines display various biological action including anti-biotic,¹⁶ anti-tumor,¹⁷ analgesic,¹⁸ anti-convulsant,¹⁹ anti-psychotic,²⁰ anti-malarial,²¹ anti-anginal,²² anti-hypertensive,²³ and anti-rheumatic²⁴ activities. 1-Amidoalkyl-2-naphthols have been obtained *via* the multi-component condensation of 2-naphthol, aldehydes, and acetonitrile or amides in the presence of acid catalysts such as copper perchlorate hexahydrate $[\text{Cu}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}]$,²⁵ tin(II) chloride dihydrate $[\text{SnCl}_2 \cdot 2\text{H}_2\text{O}]$,²⁶ ferric bisulfate $[\text{Fe}(\text{HSO}_4)_3]$,²⁷ strontium(II) triflate $[\text{Sr}(\text{OTf})_2]$,²⁸ molecular iodine (I_2),²⁹ aluminum *tris* (dihydrogenphosphate) $[\text{Al}(\text{H}_2\text{PO}_4)_3]$,³⁰ silicotungstic acid $(\text{H}_4\text{SiW}_{12}\text{O}_{40})$,³¹ *N*-(4-sulfonic acid) butyltriethylammonium bisulfate $([\text{TEBSA}][\text{HSO}_4])$,³² zinc

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benzenesulfonate (ZBS),³³ montmorillonite K10,³⁴ molten 4-(1-imidazolium)butanesulfonate,³⁵ perchloric acid supported on alumina ($\text{Al}_2\text{O}_3\text{-HClO}_4$),³⁶ silica gel-supported polyphosphoric acid (PPA-SiO_2),³⁷ alum $[\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$,³⁸ phosphorus pentoxide (P_2O_5),³⁹ potassium dodecatungstocobaltate trihydrate ($\text{K}_5\text{CoW}_{12}\text{O}_{40} \cdot 3\text{H}_2\text{O}$),⁴⁰ sulfamic acid/ultrasound,⁴¹ silica-supported perchloric acid ($\text{HClO}_4\text{-SiO}_2$),⁴² nanosilica phosphoric acid ($\text{H}_3\text{PO}_4\text{-SiO}_2$),⁴³ 3-methyl-1-sulfonic acid imidazolium chloride $[\text{Msim}][\text{Cl}]$,⁴⁴ silica sulfuric acid (SSA),⁴⁵ nano-S ($\text{S}_8\text{-NP}$),⁴⁶ $\text{NiFe}_2\text{O}_4@ \text{MCM-41-SO}_3\text{H}$,⁴⁷ zirconyl(IV) chloride (ZrOCl_2),⁴⁸ oxalic acid,⁴⁹ magnesium perchlorate $[\text{Mg}(\text{ClO}_4)_2]$,⁵⁰ dodecylphosphonic acid (DPA),⁵¹ wet cyanuric chloride,⁵² cerium(IV) sulfate $[\text{Ce}(\text{SO}_4)_2]$,⁵³ silica-supported sodium bisulfate ($\text{NaHSO}_4\text{-SiO}_2$),⁵⁴ $\text{Fe}_3\text{O}_4@ \text{SiO}_2\text{-Imid-H}_3\text{PMo}_{12}\text{O}_{40}$,⁵⁵ nano- Fe_3O_4 modified carbon nanotubes using microwave irradiation,⁵⁶ MCM-41-*N*-propylsulfamic acid,⁵⁷ and polyphosphate ester (PPE).⁵⁸ However, some of these catalysts suffer from drawbacks such as prolonged reaction times, use of toxic and corrosive solvent, non-re-usable and/or expensive catalysts, unsatisfactory yields, and the need to use microwave or ultrasonic irradiation in some cases. Therefore, the development of new procedures, utilizing eco-friendly and effective catalysts that can be easily recycled at the end of the reaction, would be a worthy goal. To the best of our knowledge, there is no report on the use of polyphosphoric acid supported on silica-coated NiFe_2O_4 nanoparticle ($\text{NiFe}_2\text{O}_4@ \text{SiO}_2\text{-PPA}$) for the preparation of 1-amidoalkyl-2-naphthols. In continuation of our previous studies on the development of green and new catalysts,⁵⁹⁻⁷⁴ we describe here a simple, mild and efficient procedure for the one-pot three-component synthesis of 1-amidoalkyl-2-naphthols from 2-naphthol, various aldehydes, and amides (benzamide, acetamide or urea) using $\text{NiFe}_2\text{O}_4@ \text{SiO}_2\text{-PPA}$ as the catalyst under solvent-free conditions (Scheme 1).

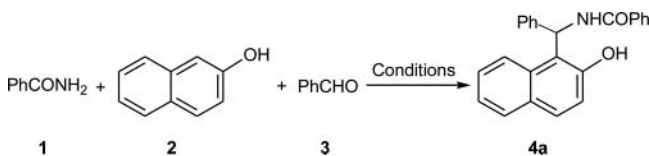


Scheme 1

To determine the optimum conditions, the reaction of benzaldehyde (0.106 g, 1 mmol), 2-naphthol (0.144 g, 1 mmol), and benzamide (0.145 g, 1.2 mmol) in the presence of $\text{NiFe}_2\text{O}_4@ \text{SiO}_2\text{-PPA}$ (0.04 g), prepared according to the literature procedure,⁶⁸ was performed under thermal solvent-free conditions at different temperatures in an oil bath (Table 1, Entries 1–3). Table 1 shows that the shortest time and best yields were achieved at 120°C (Entry 1) and the optimal amount $\text{NiFe}_2\text{O}_4@ \text{SiO}_2\text{-PPA}$ (0.04 g, Entries 4 and 5) to catalyze the reaction smoothly. Further increase the amount of $\text{NiFe}_2\text{O}_4@ \text{SiO}_2\text{-PPA}$ did not lead to appreciable increases in yields; no conversion to product was obtained in the absence of catalyst even after 1 h (Entry 6).

The use of these optimized conditions allowed us to explore the scope and efficiency of this procedure for the synthesis of a wide variety of substituted corresponding 1-amidoalkyl-2-naphthol derivatives. The results summarized in Table 2 shows that the reaction works well with a variety of aldehydes including those bearing electron-withdrawing and electron-donating groups. On the other hand, only traces of corresponding products were produced from the reaction with α,β -unsaturated and aliphatic aldehydes such as

Table 1

 Optimization of the Amount of $\text{NiFe}_2\text{O}_4@\text{SiO}_2\text{-PPA}$ and Temperature in the Synthesis of *N*-[phenyl-(2-hydroxynaphthalen-1-yl)-methyl]benzamide (**4a**)


Entry	Amount of $\text{NiFe}_2\text{O}_4@\text{SiO}_2\text{-PPA}$ (g)	Temperature ($^{\circ}\text{C}$)	Time (min)	Yield (%)
1	0.04	120	5	92
2	0.04	110	5	79
3	0.04	100	10	78
4	0.03	120	5	83
5	0.05	120	4	85
6	— ^a	120	60	— ^b

^aIn the absence of $\text{NiFe}_2\text{O}_4@\text{SiO}_2\text{-PPA}$.

^bNo reaction

Table 2

 One-pot Synthesis of 1-Amidoalkyl-2-naphthol in the Presence of $\text{NiFe}_2\text{O}_4@\text{SiO}_2\text{-PPA}$ under Solvent-Free Conditions

Product (4)	Ar	R^1	Time (min)	Yield (%)	mp ($^{\circ}\text{C}$)	
					Found	Lit.
4a	C_6H_5	Ph	5	92	236–238	234–236 ⁴⁴
4b	4- ClC_6H_4	Ph	7	91	188–190	187–188 ⁴⁴
4c	2- ClC_6H_4	Ph	8	82	284–285	284–285 ³⁹
4d	4- $\text{NO}_2\text{C}_6\text{H}_4$	Ph	7	96	228–229	228–229 ³⁹
4e	3- $\text{NO}_2\text{C}_6\text{H}_4$	Ph	7	93	210–212	214–216 ⁴⁴
4f	4- $\text{CH}_3\text{C}_6\text{H}_4$	Ph	10	94	214–216	216–217 ⁵²
4g	4- BrC_6H_4	Ph	8	90	225–227	228–230 ²⁷
4h	4- $\text{CH}_3\text{OC}_6\text{H}_4$	Ph	10	90	204–206	208–209 ³⁹
4i	C_6H_5	CH_3	7	86	239–240	241–243 ⁵²
4j	4- $\text{NO}_2\text{C}_6\text{H}_4$	CH_3	7	92	245–247	247–249 ⁴²
4k	3- $\text{NO}_2\text{C}_6\text{H}_4$	CH_3	7	85	238–240	235–237 ⁴²
4l	4- BrC_6H_4	CH_3	10	95	230–232	228–230 ⁵²
4m	2- ClC_6H_4	CH_3	10	80	213–215	213–215 ³⁶
4n	4- ClC_6H_4	CH_3	10	88	224–227	223–225 ³⁹
4o	4- $\text{CH}_3\text{C}_6\text{H}_4$	CH_3	15	80	220–221	220–222 ⁴²
4p	C_6H_5	NH_2	7	90	169–171	172–174 ⁵²
4q	4- ClC_6H_4	NH_2	10	90	174–176	170–172 ⁵²
4r	3- $\text{NO}_2\text{C}_6\text{H}_4$	NH_2	7	95	192–194	189–190 ⁵²

Table 3
Comparison of Methods for the Synthesis of 1-Amidoalkyl-2-naphthols

Entry	Conditions	Time (min)	Yield (%)
4a	Present work	5	92
	Cu(ClO ₄) ₂ .6H ₂ O/ultrasound/rt ²⁵	45	89
	SnCl ₂ .2H ₂ O/solvent-free/80°C ²⁶	18	96
	Zinc benzenesulfonate (ZBS)/solvent-free/80°C ³³	15	92
	KAl(SO ₄) ₂ .12H ₂ O/solvent-free/85°C ³⁸	15	91
	Nano silica phosphoric acid/solvent-free/80°C ⁴³	50	95
	3-Methyl-1-sulfonic acid imidazolium chloride [Msim][Cl]/solvent-free/120°C ⁴⁴	6	86
	NiFe ₂ O ₄ @MCM-41-SO ₃ H/solvent-free/100°C ⁴⁷	240	92
	Fe ₃ O ₄ @SiO ₂ -Imid-H ₃ PMo ₁₂ O ₄₀ /solvent-free/microwave irradiation (360 w) ⁵⁵	8	93
	Nano-Fe ₃ O ₄ modified carbon nanotubes/solvent-free/microwave irradiation ⁵⁶	5	90
	Polyphosphate ester (PPE)/solvent-free/80°C ⁵⁸	10	90
	Present work	6	93
	Cu(ClO ₄) ₂ .6H ₂ O/ultrasound/rt ²⁵	30	90
	SnCl ₂ .2H ₂ O/solvent-free/80°C ²⁶	120	92
4f	KAl(SO ₄) ₂ .12H ₂ O/solvent-free/85°C ³⁸	15	93
	Nano silica phosphoric acid/solvent-free/80°C ⁴³	45	91
	NiFe ₂ O ₄ @MCM-41-SO ₃ H/solvent-free/100°C ⁴⁷	300	80
	Fe ₃ O ₄ @SiO ₂ -Imid-H ₃ PMo ₁₂ O ₄₀ /solvent-free/microwave irradiation (360 w) ⁵⁵	15	86
	Present work	5	95
	Fe(HSO ₄) ₃ /solvent-free/85°C ²⁷	45	89
	Cu(ClO ₄) ₂ .6H ₂ O/ultrasound/rt ²⁵	60	90
	KAl(SO ₄) ₂ .12H ₂ O/solvent-free/85°C ³⁸	10	91
4l	Wet cyanuric chloride/100°C ⁵²	10	92
	Fe ₃ O ₄ @SiO ₂ -Imid-H ₃ PMo ₁₂ O ₄₀ /solvent-free/microwave irradiation (360 w) ⁵⁵	12	87
	MCM-41- <i>N</i> -propylsulfamic acid/solvent-free/130°C ⁵⁷	190	87

crotonaldehyde, cinnamaldehyde, formaldehyde, acetaldehyde, butyraldehyde and isobutyraldehyde.

In order to examine the recyclability of NiFe₂O₄@SiO₂-PPA, the major portion of the catalyst was recovered by placing a magnet (1.4 T field strength) on the side of the reaction vessel⁷⁵ and decantation of the clear solution containing the product. After washing with acetone and drying at 100°C for 2h, the recovered catalyst was re-used six times in the reaction of 2-naphthol and benzaldehyde with benzamide, to afford 92%, 90%, 89%, 88%, 88 and 87% yields, respectively.

We also compared our results using NiFe₂O₄@SiO₂-PPA with data reported in the literature. *Table 3* clearly demonstrates NiFe₂O₄@SiO₂-PPA to be a simple and effective catalyst with respect to reaction times, simplicity of procedure, facile work-up and yields comparable to those obtained using some of the other catalysts.

Although the yields utilizing $\text{NiFe}_2\text{O}_4@\text{SiO}_2\text{-PPA}$ are not better than some of those using other methods, comparison with homogeneous polyphosphoric acid (PPA) shows its effective-ness. Polyphosphoric acid (PPA) suffers from drawbacks such as **a**) non-reusability and difficult separation, **b**) necessity to neutralize the reaction mixtures prior to product isolation **c**) low stability, **d**) higher cost, **e**) higher toxicity and **f**) sensitivity to moisture and air. $\text{NiFe}_2\text{O}_4@\text{SiO}_2\text{-PPA}$ is easily separated using a magnet⁶⁸ and may be re-used without loss of activity. It is very stable, highly efficient due to its greater surface area and inexpensive because of the re-usability. These properties make $\text{NiFe}_2\text{O}_4@\text{SiO}_2\text{-PPA}$ attractive as novel magnetically separable catalyst for the synthesis of aminoalkyl-2-naphthols.

Experimental Section

Chemicals were obtained from Merck and Fluka. IR spectra were recorded on a Shimadzu 435-U-04 spectrophotometer (KBr pellets). ^1H NMR spectra were obtained using Jeol FT NMR 90 MHz and spectrometer in CDCl_3 using TMS as an internal reference. Melting points were determined in open capillary tubes in a Stuart BI Branstead Electrothermal Cat No:IA9200 apparatus and uncorrected. A neodymium magnet (also known as NdFeB, NIB or Neo magnet) with field strength of 1.4T was purchased from Ningbo Tongchuang Strong Magnet Material Co., Ltd (China).

General Procedure for the Synthesis of 1-Amidoalkyl-2-naphthols (4a-r)

To mixture of aldehyde (1 mmol), 2-naphthol (0.144 g, 1 mmol), the amide (benzamide or acetamide or urea) [0.145 g or 0.072 g or 0.0708 g, 1.2 mmol] was added $\text{NiFe}_2\text{O}_4@\text{SiO}_2\text{-PPA}$ (0.04 g) and the mixture was heated on an oil bath at 120°C for the time show in Table 2. After completion of the reaction (monitored by TLC, *n*-hexane-ethyl acetate, 4:1), the reaction mixture was diluted with hot EtOH (96%, 2 ml) and partitioned as follows. The nanomagnetic catalyst was separated from the reaction mixture by employing an external magnet [the catalyst that was suspended in the ethanolic solution could be made to adhere to the side of reaction vessel with the aid of the external magnet (1.4 T field strength)] and the thus separated catalyst was then loosened, washed with acetone and dried at 100°C for 2 h and re-used]. The clear solution containing the crude product that had been decanted to another vessel was poured onto crushed ice. The solid product which precipitated, was collected and recrystallized from 96% ethanol (2 ml) to afford the 1-amidoalkyl-2-naphthols.

Larger Scale Preparation of 4a

To mixture of benzaldehyde (5.3 g, 50 mmol), 2-naphthol (7.2 g, 50 mmol), and benzamide (7.26 g, 60 mmol) was added $\text{NiFe}_2\text{O}_4@\text{SiO}_2\text{-PPA}$ (2 g) and the mixture was heated on an oil bath at 120°C for the time 10 min. After completion of the reaction (monitored by TLC, *n*-hexane:ethyl acetate, 4:1) the reaction mixture was diluted with hot EtOH (96%, 5 ml) and the nanomagnetic catalyst was separated from the reaction mixture as described above and the product isolated as described above and recrystallized from 96% ethanol (15 ml) to afford 15.18 g (86%) of *N*-[phenyl-(2-hydroxynaphthalen-1-yl)-methyl]benzamide (**4a**).

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