



Contents lists available at ScienceDirect

Ultrasonics Sonochemistry

journal homepage: www.elsevier.com/locate/ultsonch

Cavitation chemistry: A mild and efficient multi-component synthesis of amidoalkyl-2-naphthols using reusable silica chloride as catalyst under sonic conditions

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ARTICLE INFO

Article history:

Received 20 May 2010

Received in revised form 14 September 2010

Accepted 24 September 2010

Available online 8 October 2010

Keywords:

Amidoalkyl-2-naphthols

Silica chloride

Aromatic aldehydes

 β -Naphthol

Acetamide

Benzamide

ABSTRACT

An efficient and direct procedure for the synthesis of amidoalkyl-2-naphthol derivatives has been described. The process employs a three-component cyclocondensation reaction in one-pot using β -naphthol, aromatic aldehyde and acetamide or benzamide in the presence of silica chloride accelerated by ultrasound giving the product in excellent yield in very short duration.

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1. Introduction

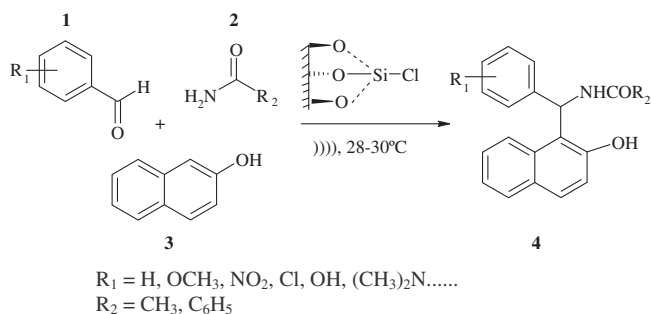
Devising reactions that achieve multi-bond formation in one operation is becoming one of the major challenges in step-economic synthesis. Multi-component reactions (MCRs) in which three or more reactants are combined in a single chemical step to produce products that incorporate portions of all the components comply with many of the stringent requirements for an ideal synthesis [1]. *o*-Quinone methides (*o*-QMs) have emerged as interesting molecules due to their toxicological properties against both normal and cancerous cells and also for their proposed intermediacy in the formation of many biologically important polymers [2]. *o*-QMs also act as intermediates for the synthesis of antitumor agents [3]. Freccero's group [4] and Kresge's group [5] have reported significant findings on the generation of *o*-QMs by photochemical and thermal activation. *o*-QMs mainly involve [4 + 2]-cycloadditions with a wide range of dienophiles and have also been used in many MCRs [6]. We have already reported from our laboratory, the '*in situ*' generation of *o*-QMs which further combine with a molecule of β -naphthol to give dibenzo[*a,j*]xanthenes [7]. One of the tandem reactions involves the '*in situ*' generation of *o*-QMs and its reaction with acetamide or benzamide to give

amidoalkyl-2-naphthols, and a considerable number of protocols for the synthesis of amidoalkyl-2-naphthols have been reported in the literature [8–16]. After consulting the literature, we observed that, the synthesis of amidoalkyl-2-naphthols is generally carried out at very high temperatures, and silica sulfuric acid takes about 2 h at 25 °C. Other disadvantages include use of non-reusable and expensive catalysts, or unsatisfactory yield. Hence, we feel that, there is still scope for further investigation towards milder reaction conditions, shorter reaction durations and better yields, which can possibly be achieved using ultrasound as energy source for this MCR, since ultrasound provides very high temperature and pressure within seconds.

Ultrasonic irradiation was first discovered by Sir John I. Thornycroft and Sydney W. Barnaby in 1894 [17], and the chemical ultrasonics began in 1927, when the acceleration of a conventional reaction was reported by Richards and Loomis [18a]. Ultrasound provides an unusual mechanism for generating high-energy chemistry due to the immense temperature, pressure and the extraordinary heating and cooling rates generated by the cavitation bubble collapse [17]. In some cases, it can also increase the chemical reactivities by nearly a million-folds [17]. Frequencies below 50 kHz are generally preferred for the heterogeneous systems due to the more intense mechanical effects [18b]. Hence, we selected 35 kHz for maximum sonication, and from our laboratory we have already reported the use of ultrasound in various reactions like synthesis of δ -chloroesters [19] and β -iodoesters [20]; reduction

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Scheme 1. Synthesis of amidoalkyl-2-naphthols using silica chloride.

of nitroarenes into arylamines [21] and reduction of aryl nitro compounds to azo arenes or arylamines [22] Li et al. have reported a Biginelli-type reaction to synthesize the 3,4-dihydropyrimidin-2-ones catalyzed by $\text{NH}_2\text{SO}_3\text{H}$ under sonic condition [23].

On the other hand, solid supported reactions have been explored way back from 1960's. The core structure of the library molecule resides in use of solution phase technique and use of solid-phase reagents to facilitate the reaction [24]. The solid-phase reagent can be filtered and washed for reuse. Thus, a reliable technique would be the application of ultrasonic irradiation involving use of heterogeneous and reusable catalysts in a suitable medium. When acoustic cavitation occurs near an extended liquid–solid interface, markedly asymmetric bubble collapse occurs, which generates a high-speed jet of liquid directed at the surface. The impingement of this jet and related 'shockwaves' can create localized erosion, causing particle fragmentation and improved mass transport [25]. In continuation of our recent studies to develop mild and environmentally friendly procedures for the synthesis of bio active molecules using green protocols, we herein, report the synthesis of a small library of amidoalkyl-2-naphthols via an ultrasound assisted MCR (Scheme 1).

2. Methods

2.1. Materials and instruments

All starting materials were commercial products, and all were used without further purification except liquid aldehydes, which were distilled before use. Yields refer to yield of the isolated products. Melting points were measured on a Raaga, Chennai, Indian make melting point apparatus. Nuclear magnetic resonance spectra were obtained on a 400 MHz Bruker AMX instrument in DMSO-d_6 using TMS as a standard. LC–Mass spectra were performed on an Agilent Technologies 1200 series instrument and GC–Mass spectra were obtained using a Shimadzu GC–MS QP 5050A spectrometer equipped with a 30 m long and 0.32 mm dia BP-5 column with the column temperature 80–15–250 °C. Infrared spectra were recorded using Shimadzu FT-IR-8400s spectrophotometer as KBr pellets. Silica chloride was obtained according to the method reported in the literature [26]. All the reactions were studied using SIDILU, Indian make sonic bath working at 35 kHz (120 W) maintained at 28–30 °C without mechanical stirring.

2.2. Typical procedure for the synthesis of silica chloride

To a well-stirred silica gel (20 g) in CH_2Cl_2 (50 ml), was added SOCl_2 (20 g) drop wise at 25 °C. Evolution of copious amounts of HCl and SO_2 occurred instantaneously. After stirring for another hour, the solvent was removed to dryness under reduced pressure.

2.3. Typical procedure for the synthesis of amidoalkyl-2-naphthols

A mixture of aromatic aldehydes (5 mmol), β -naphthol (5 mmol), acetamide or benzamide (5 mmol) and silica chloride (0.1 g) was sonicated in a sonic bath working at 35 kHz (constant frequency) maintained at 28–30 °C (by circulating water). For solid aldehydes DCE (1 ml) was added to the mixture. After completion of the reaction (monitored by TLC), product was taken into ethyl acetate (10 ml) and the catalyst was filtered and washed with chloroform for reuse. The organic layer was washed successively with water (5 ml), sat. NaHCO_3 (5 ml), water (5 ml) and then dried over anhydrous sodium sulfate to get the crude compound in almost pure form. The analytical grade of the product was obtained by recrystallization from aq. ethanol.

The spectral data of the novel compounds are

2.3.1. Compound 4m

White solid, IR (KBr, ν_{max}) 3421, 3209, 1650, 1577, 1512, 1438, 1369, 1276, 945, 818 cm^{-1} . $^1\text{H NMR}$ (400 MHz, DMSO-d_6): δ 9.650 (s, 1H, NH); 8.756 (s, 1H, OH); 8.414–8.393 (d, 1H, $J = 8.4$ Hz, ArH); 7.906–7.886 (d, 1H, $J = 8.0$ Hz, ArH); 7.807–7.734 (m, 2H, ArH); 7.390–7.353 (m, 1H, ArH); 7.282–7.199 (m, 2H, ArH); 7.035–7.013 (d, 1H, $J = 8.8$ Hz, ArH); 6.824 (s, 1H, OH); 6.637–6.616 (d, 1H, $J = 8.4$ Hz, CH); 6.531–6.508 (m, 1H, ArH); 3.677 (s, 3H, OCH_3); 1.955 (s, 3H, CH_3) ppm. MS m/z : 337.

2.3.2. Compound 4v

White solid, IR (KBr, ν_{max}) 3500, 3406, 3121, 2944, 1627, 1571, 1512, 1436, 1348, 1274, 1035, 935, 850, 819, 754 cm^{-1} . $^1\text{H NMR}$ (400 MHz, DMSO-d_6): δ 10.298 (s, 1H, OH); 9.029 (d, 1H, $J = 8.8$ Hz, NH); 8.841 (s, 1H, ArH); 8.116 (d, 1H, $J = 8.8$ Hz, ArH); 7.859–7.776 (m, 4H, ArH); 7.570–7.454 (m, 4H, ArH); 7.333–7.296 (d, 1H, $J = 7.2$ Hz, CH); 7.296–7.196 (m, 2H); 6.981 (s, 1H, OH); 6.673–6.628 (t, 2H, $J = 9.0$ Hz, ArH); 3.664 (s, 3H, OCH_3) ppm. MS m/z : 399.

3. Results and discussion

Silica chloride ($\text{SiO}_2\text{-Cl}$) is an inexpensive heterogeneous reagent, which can be prepared easily by treating thionyl chloride with silica gel [26]. Ease and safety in handling, rate enhancement, high yields, easy work up procedures for reuse, and economy are the properties which made us to use this interesting reagent as a catalyst. To the best of our knowledge there are no reports on the applicability of silica chloride for the synthesis of amidoalkyl-2-naphthols in the literature.

In an initial endeavor, a mixture of same equivalents of benzaldehyde, β -naphthol and acetamide was taken and sonicated at 28–30 °C for 9 min (TLC) in an ultrasonic bath [27]. As expected, high yield of the product was obtained. When we extended the reaction to *m*-nitrobenzaldehyde, to our surprise only 65% of the product was obtained even after sonicating for 1 h. To improve the yield of the target product, we carried out the reaction in presence of various solvents and the results are presented in Table 1. As can be seen from this table, dichloroethane (DCE) accelerated the reaction and therefore high yields were obtained for all solid aromatic aldehydes including *m*-nitrobenzaldehyde (DCE has the cavity intensity of 39% compared to that of water (std. 100%) [18b]). We wanted to optimize the amount of catalyst used for this cyclocondensation, hence, we carried out the reaction with different amounts of catalyst, but it was observed that, the best result could be obtained with 0.1 g of the catalyst. The same reaction was carried out three times consequently to check the reproducibility and it was found that, all the three times, it gave same yield of the product ($\pm 1\%$).

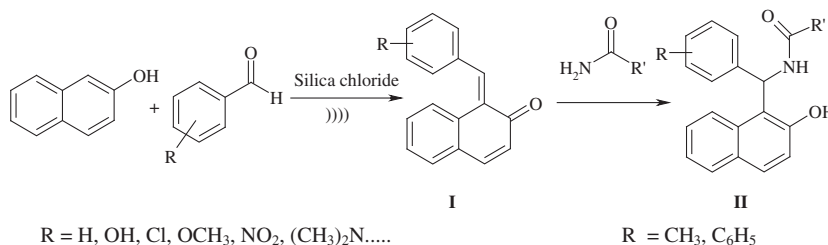
Table 1

Reaction of *m*-nitrobenzaldehyde (2 mmol), β -naphthol (2 mmol) and acetamide (2 mmol) to obtain amidoalkyl-2-naphthols in the presence of silica chloride in different solvents and under solvent-free condition.

Entry	Solvent	Yield (%) ^a	Time (min)
1	Ethanol	55	20
2	Methanol	50	20
3	CH ₃ CN	44	20
4	CHCl ₃	60	20
5	DCM	80	20
6	DCE	91	18
7	Solvent-free condition	65	60

^a Isolated yield.

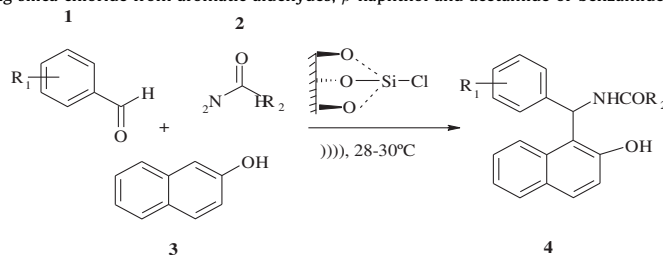
Possible mechanism of this one pot reaction under sonic condition is expected to include the 'in situ' formation of intermediate *o*-QM in the presence of solid silica chloride. It is assumed that, due to the collapse of the cavitation bubbles near the surface of the catalyst, the oxygen of the carbonyl group in step 1 may easily replace the chlorine atom present on the catalyst silica chloride to give the activated aldehyde which may attack β -naphthol to give *o*-QM (I). Formation of the intermediate (I) has been suggested by others under silent conditions in the recent past [8–10]. Further, the *o*-QM intermediate (I) may undergo a nucleophilic conjugate addition with iminol form of the amide to give amidoalkyl naphthol (II) as shown in Scheme 2. The first step is the rate determining step wherein the activated aldehyde reacts with a molecule of β -naphthol to give the intermediate *o*-QM (I).



Scheme 2. Formation of amidoalkyl-2-naphthols via *o*-QM intermediate.

Table 2

Synthesis of amidoalkyl-2-naphthols (4) using silica chloride from aromatic aldehydes, β -naphthol and acetamide or benzamide.



Entry	R ₁	R ₂	Product (4) ^a	Time (min)	Yield (%) ^b	Mp [Lit. Mp] (°C)
1	H	CH ₃	4a	9	95	240[241–243] [13] ^c
2	4-OCH ₃	CH ₃	4b	20	91	184[184–186] [13] ^c
3	3-NO ₂	CH ₃	4c	18	98	182[182–184] [13] ^c
4	4-OH	CH ₃	4d	30	92	207 [10] ^c
5	3-OCH ₃	CH ₃	4e	27	90	202[201–204] [15]
6	4-NO ₂	CH	4f	18	94	248[247–249] [13]
7	2,4-Cl ₂	CH ₃	4g	24	93	99[198–199] [13]
8	3,4-(OCH ₃) ₂	CH ₃	4h	36	90	235[235–236] [13]
9	3,4,5-(OCH ₃) ₃	CH ₃	4i	30	92	191[190–193] [13]
10	4-Cl	CH ₃	4j	9	96	223[224–227] [13]
11	2-Cl	CH ₃	4k	18	92	193[194–196] [13]
12	4-(CH ₃) ₂ N	CH ₃	4l	30	93	123[123–125] [13]
13	3-CH ₃ ,4-OH	CH ₃	4m	27	95	210 ^{c,d}
14	H	C ₆ H ₅	4n	9	98	233[234–236] [13]
15	4-OCH ₃	C ₆ H ₅	4o	18	94	208[208–209] [13]
16	4-NO ₂	C ₆ H ₅	4p	15	97	227[228–229] [16]
17	3-NO ₂	C ₆ H ₅	4q	9	98	215[216–217] [13] ^c
18	4-(CH ₃) ₂ N	C ₆ H ₅	4r	20	92	219[220–221] [16]
19	4-Cl	C ₆ H ₅	4s	9	96	177[177–178] [10]
20	3,4,5-(OCH ₃) ₃	C ₆ H ₅	4t	18	90	231[231–233] [13]
21	3-OCH ₃	C ₆ H ₅	4u	15	93	213[214–216] [11]
22	3-OCH ₃ -4-OH	C ₆ H ₅	4v	17	95	219 ^{c,d}

^a 4a–4v were also characterized by IR spectral analysis and by comparing the melting points with samples prepared by the reported methods and by comparison on TLC.

^b Isolated yields.

^c 4a, 4b, 4c, 4d, 4m, 4q and 4v were characterized by GC–mass/LC–mass spectral analysis and by ¹H NMR spectral analysis.

^d Novel compound.

After optimizing the reaction conditions, and understanding the mechanism, we subsequently extended the scope of the present method using silica chloride as a catalyst with a variety of aromatic aldehydes to prepare a small library of amidoalkyl-2-naphthols. Various aromatic aldehydes containing electron-donating and electron-withdrawing substituents were found to react successfully with β -naphthol to give high to excellent yield of the desired products as shown in Table 2. From this table, it is clear that, aldehydes containing electron-withdrawing substituents such as NO₂, Cl (Table 2, entries 3,6,10,11,16,17,19) react faster and give higher yields of the respective products when compared to aldehydes bearing electron-donating substituents like OCH₃, OH, (CH₃)₂N (Table 2, entries 2,4,5,8,10,12,13,15,18,20,21,22). As reported by Shaterian, the lower energy of the LUMO of the alkene containing electron-withdrawing groups (a carbonyl group as in *o*-QM intermediate) as compared to that of alkene containing electron-donating groups may be responsible for making the present reaction faster [13]. We feel that, the electron-donating groups present on the arene nucleus destabilize the *o*-QM intermediate towards the

1,4-nucleophilic addition reaction and hence, the reaction is slower and gives low yield of the desired products. Satisfied with the above results, we extended the reaction of β -naphthol and aromatic aldehydes with benzamide instead of acetamide to get the respective products in high yield in the presence of catalytic amounts of silica chloride under sonic condition, and the results of this study are also presented in Table 2 (entries 16–22).

The possibility of recycling the catalyst was then examined. As a model reaction, benzaldehyde (2 mmol), β -naphthol (2 mmol) and acetamide (2 mmol) were sonicated in the presence of silica chloride. After completion of the reaction (9 min), ethyl acetate (5 ml) was added to the reaction mixture. The catalyst was filtered and washed with chloroform and then recycled for four times after giving washings every time. From the Fig. 1 it can be seen that in the first three runs the activity was more or less maintained but after three runs it starts decreasing, which may be due to degradation of the catalyst under sonic condition. The yields for the five runs were found to be 95%, 94%, 94%, 90% and 88%, respectively.

Further, to investigate the role of ultrasonic irradiation on this reaction, the reactions were also carried out in the presence of the same amount of silica chloride at 28–30 °C under mechanical stirring. Equivalent quantity of aromatic aldehyde, β -naphthol and acetamide were mechanically stirred in a magnetic stirrer in DCE (10 ml) as solvent in presence of silica chloride (0.1 g). The results of this study are summarized in Table 3. It is clear from this table that, reactions under the influence of ultrasound give relatively higher yields of the products in relatively shorter reaction durations due to inrush of liquid from one side of the surface of the catalyst due to the collapse of the cavitation bubbles as mentioned above. This high pressure jet of the liquid is expected to activate the surface of the solid catalyst and hence increase the rate of the reaction [18].

To show the merits of our protocol, the reaction between benzaldehyde, β -naphthol and acetamide was selected for comparison. The yield of the product formed in the presence of silica chloride under ultrasonic condition was compared with previously reported



Fig. 1. Reusability of silica chloride (0.1 g) in the synthesis of amidoalkyl-2-naphthols for 9 min under sonic condition.

Table 3

Synthesis of amidoalkyl-2-naphthols catalyzed by silica chloride under mechanically stirring at room temperature and under sonication.

Entry	R ₁	R ₂	Product (4)	Time/yield (min)/(%) ^a	Time/yield (min)/(%) ^b
1	H	CH ₃	4a	9/65	9/95
2	4-OCH ₃	CH ₃	4b	20/50	20/91
3	3-NO ₂	CH ₃	4c	20/75	18/98
4	4-OH	CH ₃	4d	30/67	30/92
5	3-OCH ₃	CH ₃	4e	30/70	27/90
6	4-NO ₂	CH ₃	4f	20/69	18/94

Reaction condition: Aromatic aldehyde (2 mmol), β -naphthol (2 mmol), acetamide(2 mmol), silica chloride (0.1 g) and dichloroethane (10 ml).

^a Mechanical stirring, the reaction was carried out on a magnetic stirrer (1 l capacity) and the reaction mixture was stirred at its maximum speed.

^b Under sonication.

Table 4

Comparative study on the present method with the reported methods.

Entry	Catalyst/reagent	Conditions	Time	Yield (%)
1	Ce(SO ₄) ₂ [9]	Under reflux	36 h	72
2	I ₂ [10a]	Solvent-free, 125 °C	5.5 h	85
3	Montmorillonite K10 [8]	Solvent-free, 125 °C	1.5 h	89
4	Sulfamic acid [14b]	Solvent-free, ultrasonic 28–30 °C	15 min	89
5	<i>p</i> -TSA [12]	Solvent-free, 125 °C	5 h	88
6	SiO ₂ -HClO ₄ [16a]	Solvent-free, 125 °C	40 min	89
7	PPA-SiO ₂ [13]	Solvent-free, 120 °C	7 min	86
8	Oxalic acid [16d]	Solvent-free, 125 °C	8 min	96
9	SiO ₂ -Cl	Solvent-free, ultrasound, 28–30 °C ^a	9 min	95

Reaction condition: Benzaldehyde (2 mmol), β -naphthol (2 mmol), silica-chloride (0.1 mg) and acetamide (2 mmol).

^a Present method.

methods and the comparison is shown in Table 4. As can be seen from Table 4, silica chloride can serve as an efficient catalyst in the formation of amidoalkyl-2-naphthols with high yields in shorter reaction times, and the method is as efficient as oxalic acid at 125 °C under thermal condition [16d].

4. Conclusion

In conclusion, we have developed a highly efficient sonochemical method for the synthesis of amidoalkyl-2-naphthols from aromatic aldehydes, β -naphthol and acetamide or benzamide. This method has the advantages derived from multi-component reactions (MCRs) accompanied with the application of a heterogeneous and reusable catalyst – silica chloride. We feel that, this economically viable procedure will prove to be a better, an alternative and green protocol for the synthesis of amidoalkyl-2-naphthols as compared to the existing methods.

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