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Silica chloride catalyzed efficient route to novel 1-amidoalkyl-2-naphthylamines under sonic condition in water

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ABSTRACT

A one-pot three-component condensation of an aldehyde, 2-naphthylamine, and acetamide has been achieved by sonication at 35 kHz. The reaction is catalysed by silica chloride in aqueous medium. This protocol afforded corresponding 1-amidoalkyl-2-naphthylamines in shorter reaction durations, and in high yields. The method involves simple work-up procedure, and avoids use of hazardous reagents.

1. Introduction

Multicomponent reactions (MCRs) have drawn high efforts in recent years owing to exceptional synthetic efficiency, intrinsic atom economy, high selectivity, and procedural simplicity [1]. These reactions constitute a valuable approach for the creation of large libraries of structurally related, drug-like compounds, thereby enabling identification and leading to optimization in drug discovery [2]. In a true sense, MCRs represent environment friendly processes by reducing the number of steps, energy consumption and waste production.

The application of ultrasound in organic synthesis has been increasing because of its advantages including shorter reaction times, milder reaction conditions and higher yields in comparison to classical methods [3]. Since in this technique the reaction is carried out normally at lower external temperature relative to the usually thermal methods, the possibility of occurrence of undesired reactions is reduced, and as a result of cleaner reaction the workup is easier [4].

The amide group is an essential building block for numerous natural products, synthetic pharmaceuticals, and a wide variety of biologically active compounds [5]. As a consequence; the development of general methods for the synthesis of amide derivatives has been the subject of considerable synthetic efforts and still requires attention. Amide moiety has been involved in many reactions like Ugi [6] and Passerini reactions [7]. Furthermore, the scope of this pharmacophore has been increased by the identification of these molecules as the precursors of aminoamides which find application in the synthesis of dendrimers [8]. Aminoamides can also be used in the synthesis of metal complexes [9] and could be employed as adjuvents for drugs [10]. Derivatives of aminoamide have also been shown to have antioxidant and anti-inflammatory properties [11]. In view of this we have synthesized some novel amidoalkyl-2-naphthylamine derivatives from aromatic aldehydes, 2-naphthylamine and acetamide.

The need to reduce the amount of toxic waste and byproduct arising from chemical process requires increasing emphasis on the use of less toxic and environmentally compatible materials in the design of new synthetic methods [12]. One of the most promising approaches is use of water as a reaction medium. Breslow, who showed that hydrophobic effects and polarity effect could strongly enhance the rate of several organic reactions, rediscovered the use of water as a solvent in organic chemistry in the 1980s [13]. There has been growing recognition that water is an attractive medium for many organic reactions [14] and many MCRs in aqueous medium have been reported [15]. However, to the best of our knowledge, we are the first ones to report the present synthesis of amidoalkyl-2-naphthylamine derivatives under aqueous sonic condition. As a consequence of our interest in the aqueous medium organic synthesis [16a] and our continual work on the synthesis of biologically important molecules [16b,c]. We investigated a first three-component one-pot synthesis of 1-amidoalkyl-2-naphthylamines from 2-naphthylamine in the presence of silica-chloride

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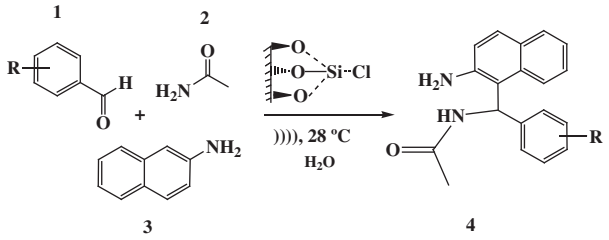


Scheme 1. Synthesis of 1-amidoalkyl-2-naphthylamines.

as a catalyst under aqueous sonic condition as shown in the Scheme 1.

2. Methods

2.1. Materials and instruments

All starting materials were commercial products, and 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, 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. HRMS analyses were carried out using ESI-Q TOF instrument. Infrared spectra were recorded using Shimadzu FT-IR-8400s spectrophotometer as KBr pellets. All the reactions were studied using SIDILU, Indian make sonic bath working at 35 kHz (constant frequency, 120 W) maintained at 28 °C without mechanical stirring. Silica chloride was prepared using the reported method [17].

2.2. General procedure for the synthesis of 1-amidoalkyl-2-naphthylamines

Aromatic aldehyde (5 mmol), 2-naphthylamine (5 mmol), acetamide (5 mmol) and silica chloride (100 mg) was added to water (5 mL) in a 25 mL RB flask and irradiated in an ultrasonic bath. After completion of the reaction ethyl acetate (10 mL) was added and the catalyst was filtered, washed with warm ethanol and dried for reuse. The filtrate after distilling the solvent was dried under vacuum to afford the crude product. The pure product was obtained after silica gel column chromatography (30% EtOAc). All the products were characterized by the IR, ^{1}H NMR, ^{13}C NMR and HRMS analysis.

The spectral data of the products:

(1) *N-[(2'-aminonaphthalen-1-yl)(phenyl)methyl]acetamide, 4a: IR (KBr) v*: 3409, 3372, 3172, 2887, 2796, 2646, 2312, 1585, 1419, 1340, 1274, 1147, 1093, 983, 813, 746, 698 cm^{-1}
^{1}H NMR (DMSO–d_6, 400 MHz) δ: 2.07 (s, 3H); 5.83 (s, 1H); 6.42 (d, *J* = 8 Hz, 5H); 6.55 (t, *J* = 8 Hz, 1H); 6.67 (d, *J* = 8 Hz, 1H); 6.94 (d, *J* = 8 Hz, 1H); 7.12 (s, 1H); 7.37–7.41 (m, 2H); 8.42 (s, 2H), 9.83 (s, 1H) ppm;
^{13}C NMR (DMSO–d_6, 100 MHz) δ: 22.45, 47.43, 118.44, 118.95, 122.32, 123.23, 126.00, 126.24, 128.17, 128.41, 129.23, 132.25, 132.29, 134.37, 134.56, 140.47, 142.57, 153.11, 169.48 ppm;
HRMS (M + Na)*:* 314.1399.

(2) *N-[(2'-aminonaphthalen-1-yl)(4-methoxyphenyl)methyl]acetamide, 4b: IR (KBr) v:* 3330, 3212, 2993, 2960, 2937, 2738, 2322, 1608, 1573, 1444, 1307, 1253, 1168, 1027, 941, 865, 813, 752, 613 cm^{-1};
^{1}H NMR (DMSO–d_6, 400 MHz) δ: 1.94 (s, 3H); 3.67 (s, 3H); 6.28 (s, 1H); 6.80 (s, 1H); 7.05 (d, *J* = 8 Hz, 3H); 7.18–7.26 (m, 2H); 7.34 (t, *J* = 8 Hz, 1H); 7.73 (d, *J* = 8 Hz, 1H); 7.78 (d, *J* = 8 Hz, 1H); 7.83 (d, *J* = 8 Hz, 1H); 8.38 (s, 2H); 9.92 (s, 1H) ppm;
^{13}C NMR (DMSO–d_6, 100 MHz) δ: 22.65, 47.42, 54.95, 118.55, 118.95, 122.29, 122.56, 125.92, 126.03, 126.18, 127.20, 127.47, 128.41, 128.45, 129.02, 153.00, 155.23, 157.63, 168.99 ppm;
HRMS (M + Na)*:* 344.1504.

(3) *N-[(2'-aminonaphthalen-1-yl)(4-hydroxy-3-methoxyphenyl)-methyl]acetamide, 4c:*IR (KBr) *v*: 3502, 3423, 3199, 1643, 1517, 1438, 1371, 1033, 987, 945, 850, 813, 750 cm^{-1};
^{1}H NMR (DMSO–d_6, 400 MHz) δ: 2.00 (s, 3H); 3.28 (s, 3H); 3.60 (s, 1H); 6.314 (s, 1H); 7.16 (d, *J* = 8 Hz, 1H); 7.20 (d, *J* = 8 Hz, 1H); 7.28 (t, *J* = 8 Hz, 1H); 7.40 (t, *J* = 8 Hz, 1H); 7.52–7.58 (m, 2H); 7.80 (t, *J* = 8 Hz, 2H); 7.85 (d, *J* = 8 Hz, 1H); 7.99 (s, 1H); 8.02–8.05 (m, 1H); 8.58 (s, 1H) ppm;
^{13}C NMR (DMSO–d_6, 100 MHz) δ: 22.66, 47.81, 55.66, 118.56, 118.88, 119.13, 122.30, 123.57, 127.48, 127.69, 128.47, 128.91, 129.23, 132.31, 134.25, 152.97, 153.02, 155.24, 157.65, 168.89 ppm;
HRMS (M + Na)*:* 360.1453.

(4) *N-[(2'-aminonaphthalen-1-yl)(3-nitrophenyl)methyl]acetamide, 4d:* IR (KBr) *v*: 3374, 3226, 1647, 1533, 1436, 1350, 1110, 991, 923, 804, 734, 713, 675, 622, 584, 491 cm^{-1};
^{1}H NMR (DMSO–d_6, 400 MHz) δ: 1.97 (s, 3H); 6.53 (s, 1H); 7.10–7.16 (m, 2H); 7.21 (t, *J* = 8 Hz, 2H); 7.26–7.29 (m, 2H), 7.31–7.36 (m, 1H); 7.75 (d, *J* = 8 Hz, 1H); 7.79 (dd, *J* = 8 Hz, 8 Hz, 1H); 7.83 (d, *J* = 8 Hz, 1H); 8.40 (s, 2H); 9.6 (s, 1H) ppm;
^{13}C NMR (DMSO–d_6, 100 MHz) δ: 22.50, 47.58, 117.74, 118.42, 120.37, 121.18, 122.55, 122.71, 126.71, 128.34, 128.64, 129.52, 129.83, 132.11, 132.80, 145.37, 147.69, 153.35, 169.62 ppm;
HRMS (M + Na)*:* 359.1250.

(5) *N-[(2'-aminonaphthalen-1-yl)(4-nitrophenyl)methyl]acetamide, 4e:* IR (KBr) *v*: 3307, 3210, 2850, 2451, 1967, 1622, 1596, 1510, 1467, 1394, 1342, 1294, 1209, 1161, 1070, 985, 823 cm^{-1};
^{1}H NMR (DMSO–d_6, 400 MHz) δ: 2.07 (s, 3H); 6.61 (s, 1H); 7.17 (d, *J* = 8 Hz, 1H); 7.21 (d, *J* = 8 Hz, 1H); 7.26–7.30 (m, 1H); 7.40 (t, *J* = 8 Hz, 1H); 7.54 (m, 2H); 7.80 (m, 2H); 7.86 (d, *J* = 8 Hz, 1H); 8.00–8.05 (m, 1H); 8.59 (s, 2H); 9.32 (s, 1H) ppm;
^{13}C NMR (DMSO–d_6, 100 MHz) δ: 22.50, 47.58, 118.42, 118.74, 123.37, 124.48, 125.55, 126.33, 126.71, 127.77, 128.73, 129.12, 129.85, 141.81, 143.50, 148.85, 168.81 ppm;
HRMS (M + Na)*:* 359.1244.

(6) *N-[(2'-aminonaphthalen-1-yl)(thiophen-2-yl)methyl]acetamide, 4f:* IR (KBr) *v*: 3292, 3161, 2923, 2513, 2110, 1170, 1108, 1045, 945, 813 cm^{-1};
^{1}H NMR (DMSO–d_6, 400 MHz) δ: 1.73 (s, 3H); 5.71 (s, 1H); 6.41 (s, 2H); 6.70 (d, *J* = 8 Hz, 1H); 7.10–7.30 (m, H); 7.67 (dd, *J* = 8 Hz, 8 Hz, 1H); 8.12 (s, 2H); 9.76 (s, 1H) ppm;
^{13}C NMR (DMSO–d_6, 100 MHz) δ: 22.85, 45.18, 119.26, 120.76, 122.54, 123.95, 124.16, 124.97, 126.19, 126.40, 128.76, 128.93, 129.13, 134.00, 148.95, 152.97, 172.11 ppm;
HRMS (M + Na)*:* 320.0963.

(7) *N-[(2'-aminonaphthalen-1-yl)(pyridin-2-yl)methyl]acetamide, 4g:* IR (KBr) *v*: 3425, 3306, 1649, 1519, 1442, 1375, 1338, 1284, 1263, 1033, 987, 939, 852, 821 cm^{-1};
^{1}H NMR (DMSO–d_6, 400 MHz) δ: 1.94 (s, 3H); 5.72 (s, 1H); 6.41 (s, 2H); 7.04 (d, *J* = 8 Hz, 1H); 7.14 (t, *J* = 8 Hz, 2H); 7.22 (t, *J* = 8 Hz, 1H); 7.35 (d, *J* = 8 Hz, 2H); 7.66–7.76 (m, 3H); 7.95 (d, *J* = 8 Hz, 1H); 8.38 (s, 2H); 8.50 (t, *J* = 8 Hz, 1H); 10.05 (s, 1H) ppm;

^{13}C NMR (DMSO–d_6, 100 MHz) δ: 23.01, 50.52, 118.07, 118.96, 119.40, 121.25, 121.92, 122.71, 123.64, 126.63, 128.79, 129.58, 132.98, 136.83, 148.56, 153.58, 161.81, 169.70 ppm; *HRMS* (M + Na)*:* 315.1351.

3. Results and discussion

Initially we conducted a catalyst-free reaction of benzaldehyde (**1**), 2-naphthylamine (**2**) and acetamide (**3**) in water at ambient temperature and at reflux for 3 h and found that, no condensation product was formed. Subsequently we tested the reaction of **1**, **2** and **3** in water under sonic condition in presence of various heterogeneous catalysts. The results of this study are summarized in Table 1. It was found that, when the reaction was carried out without any additives only trace amount of product was detected (Table 1, entry 1). Only silica gel could not catalyze this reaction (Table 1, entry 2). Some other catalysts such as Amberlyst-120, Dowex-50 and acidic alumina were employed and were found to catalyze this reaction with moderate yields (Table 1, entries 3–5). The best result was obtained when silica chloride was used, which is evident from the yield and the reaction duration (Table 1, entry 8). As the catalysts other than Si–Cl are protic catalysts, removal of –OH group for the creation of Si^+ from Si–OH is probably relatively difficult when compared to Si–Cl cleavage; hence, there is difference in the reactivity between silica chloride and other catalysts.

We also evaluated the amount of silica chloride required for the present reaction. It was found that, increasing the amount of the silica chloride from 50 to 100, 150 and 200 mg resulted in 86–94%, 93%, and 91% yield, respectively (Table 1, entries 6–9). As can be seen 100 mg of silica chloride in water under sonic condition is sufficient to push this reaction forward, and more amounts of the silica chloride did not improve the yields. Hence, all the other reactions were carried out using 100 mg silica chloride. Finally, we studied the reaction at different durations and the results of this study are also presented in Table 1. It can be seen from the Table 1 that, the reaction was complete within 20 min (Table 1, entries 7, 10, 11) to give the product in very high yield.

The choice of an appropriate reaction medium is crucial for a successful synthesis. We examined the same reaction under similar conditions in different solvents as shown in Table 2, and water was found to be the best solvent in terms of reaction time and yield of the product. As cavitation in water is most effective compared to other solvents, the ultrasonic intensity in aqueous solutions is higher because of the low sound absorption coefficient [18], hence, the effect of nature of solvent on the reactivity is predominant in the present reaction. We also conducted the reaction of liquid aldehydes under solvent-free condition but the product was formed in low yield.

Table 1
Effect of catalyst on the synthesis of 1-amidoalkyl-2-naphthylamine.[a]

Entry	Catalyst	Quantity (mg)	Time (min)	Yield (%)[b]
1	None	20	90	Trace
2	Silica gel	100	20	15
3	Amberlyst-120	100	20	53
4	Dowex-50	100	20	69
5	Acidic alumina	100	60	36
6	Silica chloride	50	20	86
7	Silica chloride	100	18	96
8	Silica chloride	150	20	93
9	Silica chloride	200	20	91
10	Silica chloride	100	30	93
11	Silica chloride	100	10	82

[a] Reaction condition: Benzaldehyde (5 mmol), 2-naphthylamine (5 mmol), acetamide (5 mmol) and water (10 mL) under sonic condition (35 kHz).
[b] Isolated yield.

Table 2
Screening of solvents.

Entry	Solvent	With sonication time (min)/yield[a] (%)	Without sonication time (min)/yield[a] (%)
1	Ethanol	20/55	60/50
2	CH_3CN	/50	60/55
3	$CHCl_3$	20/44	60/42
4	DCM	20/60	60/30
5	DCE	20/80	60/36
6	Water	18/96	60/70
7	Solvent-free condition	60/65	60/ND

ND, not detected.
[a] Isolated yield.

Table 3
Synthesis of novel 1-amidoalkyl-2-naphthylamines from aldehydes, 2-naphthylamine and acetamide under sonic condition.

Entry	Aldehyde (R)	Product (min)	Time (min)	Yield[a] (%)	Melting point (°C)
1	H	**4a**	18	96	218–220
2	4-OCH_3	**4b**	27	91	208–210
3	3-OCH_3, 4-OH	**4c**	36	90	>300
4	3-NO_2	**4d**	9	95	220–222
5	4-NO_2	**4e**	12	97	178–180
6	Pyridine	**4f**	20	95	234–236
7	Thiophene	**4g**	30	92	200–202

[a] Isolated yield.

Subsequently, this study was extended to a one-pot three-component synthesis of diverse 1-amidoalkyl-2-naphthylamines by condensing aromatic aldehydes, 2-naphthylamine, acetamide, in water as a solvent in a sonic bath (35 kHz) maintained at 28 °C by continuously circulating water. We demonstrate that this protocol accepts different aromatic aldehydes and produces respective 1-amidoalkyl-2-naphthylamines in excellent yields and the results are presented in Table 3. As shown in Table 3, aromatic aldehydes with electron-donating or electron-withdrawing groups reacted successfully and gave the products in high yields. It was also observed that, the aromatic aldehydes with electron-withdrawing groups react faster than the aromatic aldehyde with electron-donating groups. Heteroaromatic aldehydes also underwent the transformation conveniently (Table 3, entries 6 and 7). All the prepared products were characterized by the IR, ^{1}HNMR, ^{13}CNMR and HRMS analysis.

Next, we investigated the reusability and recycling of silica chloride. At first, we took a mixture of benzaldehyde (5 mmol), 2-naphthylamine (5 mmol), acetamide (5 mmol), water (10 mL)

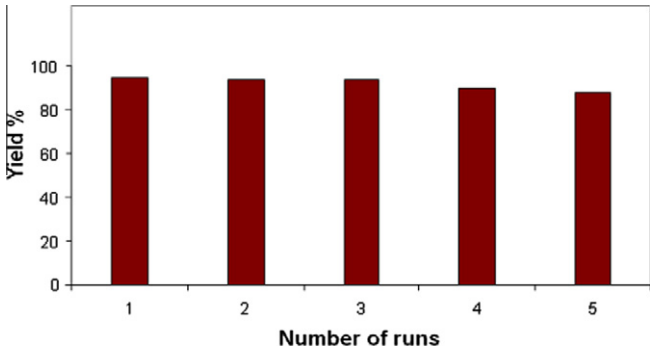


Fig. 1. Reusability of silica chloride.

Table 4
Comparative study on the synthesis of 1-amidoalkyl-2-naphthylamines under different conditions.

Entry	R (4)	Product	Time/yield (min)/(%)[a]	Time/yield (min)/(%)[b]
1	H	**4a**	20/65	18/96
2	4-OCH_3	**4b**	30/50	27/91
3	3-NO_2	**4c**	20/75	18/95

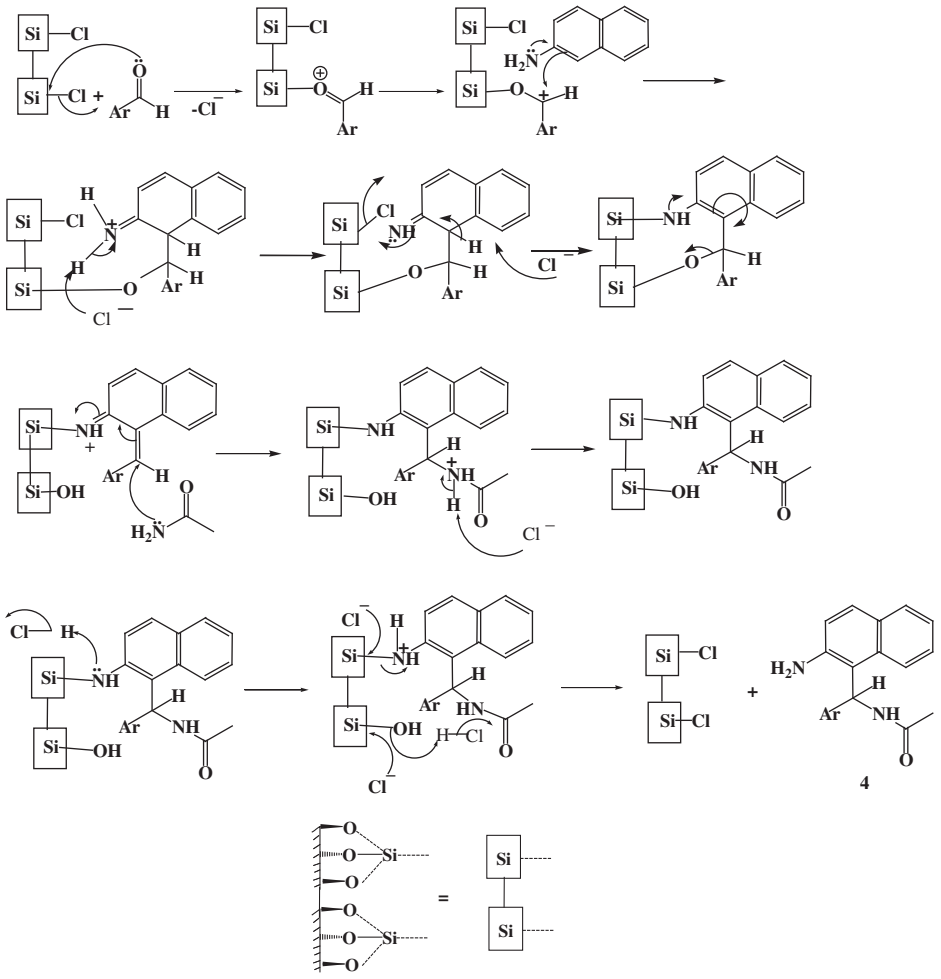
[a] At reflux [reaction condition: aromatic aldehyde (5 mmol), 2-naphthylamine (5 mmol), acetamide (5 mmol), silica chloride (100 mg) and water (10 mL) as solvent].
[b] Under sonication [35 kHz, reaction condition: aromatic aldehyde (5 mmol), 2-naphthylamine (5 mmol), acetamide (5 mmol), silica chloride (100 mg) and water (10 mL) as solvent].

and 100 mg of silica chloride in a 25 mL RB flask; the mixture was irradiated in a sonic bath for 18 min. When the reaction was complete, silica chloride was separated by filtration after adding ethyl acetate (10 mL). The recovered silica chloride was washed with warm ethanol, dried and reused in subsequent reactions without significant decrease in activity till fourth run (Fig. 1). In fifth run the catalytic activity reduced considerably. The reduction of the observed catalytic activity may be due to catalyst damage by the shock waves and high speed jets.

To verify the effect of ultrasound on this reaction the synthesis of 1-amidoalkyl-2-naphthylamines was carried out in water in the presence of silica chloride under ultrasonication and at reflux and the results of this study are presented in Table 4. It is clear from this table that, the desired product could form only in moderate yield at reflux temperature of the solvent.

A plausible mechanism for the formation of 1-amidoalkyl-2-naphthylamines is envisaged. Due to high speed jets and shock waves and in the presence of an aldehyde the Cl of the silica chloride may get more easily and selectively displaced as Cl^- to give a cationic center. 2-Naphthylamine may then react with this activated aromatic aldehyde in the next step, and in one of the subsequent steps the addition of acetamide can take place to give the final product with the release of silica chloride as shown in Scheme 2.

In all the reactions it was found that, use of ultrasound leads to faster reaction and higher yields. Symmetrical cavitation and symmetrical collapse of bubbles in the liquid medium is very common; if cavitation bubbles are formed at or near a solid surface the bubble collapse will no longer be symmetrical. The solid surface hinders liquid movement from that side and the major liquid flow into the collapsing bubble will be from the opposite side. As a result of this a strong high energy liquid jet will be formed which is targeted at the surface. These jets can induce mechanical deaggregation and dispersion of loosely held clusters, and improve mass transfer to the surface. In the present reaction, we feel that the shock waves and high speed jets are responsible for the enhancement in the rate and yield of the target molecules under sonic condition.



Scheme 2. A plausible mechanism.

4. Conclusions

In conclusion, we have described an ultrasound assisted, efficient, one-pot three-component synthesis of novel 1-amidoalkyl-2-naphthylamines from a variety of aromatic aldehydes, 2-naphthylamine and acetamide in the presence of catalytic amounts of silica chloride in water. This new method has the advantages of higher yields, mild reaction conditions, and shorter reaction duration; and is a convenient, energy efficient, environment friendly procedure.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.ultsonch.2012.06.008.

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