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Ultrasound-promoted Catalyst-free Synthesis of α-Aminonitriles in 1-Butyl-3-methylimidazolium Bromide ([Bmim]Br) as a Reusable Neutral Ionic Liquid

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A catalyst-free one-pot three-component methodology for the synthesis of α-aminonitriles under ultrasonic irradiation at room temperature using [Bmim]Br as a neutral reaction medium is described. A broad range of substrates including the aromatic, heteroaromatic and aliphatic aldehydes were condensed with amines (aliphatic and aromatic) and trimethylsilyl cyanide (TMSCN). Using this method, all reactions were completed in short times and the products were obtained in good to excellent yields. The reaction medium could be recycled and reused several times without any loss of efficiency.

Keywords: α-Aminonitrile, 1-Butyl-3-methylimidazolium bromide, Catalyst-free synthesis, Ultrasound-promoted synthesis, Ionic liquid, Green Chemistry

INTRODUCTION

Multi-component reactions are becoming a more and more important class of reactions since they allow combining several starting materials in a single compound and in one-flask operation and have become a significant part of today's arsenal of methods in combinatorial chemistry. Application of MCRs in organic synthesis provides some specific advantages such as enhanced atom economy, straightforward reaction design, and the opportunity to construct target compounds by the introduction of several diversity elements in a single chemical event [1].

Nowadays, activation of organic compounds with ultrasonic irradiation is widely used to promote numerous organic reactions [2]. This is well known that the cavitation phenomenon leading to mass transfer improvement, is the main factor in ultrasound-enhanced synthesis [2]. In comparison with traditional methods, ultrasonic-enhanced synthesis of organic compounds is more convenient and easily controlled and is more appropriate in the consideration of green chemistry concepts [3]. Up to now, this technique has been used for the synthesis of a wide variety of organic materials [4-12]. In all cases, the reactions occurred under mild conditions with good to excellent yields and in a few minutes.

Moreover, ionic liquids (ILs) have recently attracted increasing interest in the context of green organic synthesis. Although ionic liquids were initially introduced as alternative green reaction media because of their unique chemical and physical properties of no volatility, no flammability, thermal stability, and controlled miscibility, today they have marched far beyond this boundary, showing their significant role in controlling reactions as solvent or catalysts [13,14]. Another feature of ionic liquids is their ability to be reused many times. Over the last few years, there have been several reviews published in which ionic liquids have occupied a central theme [15-19].

The first report of a MCR in organic synthesis is the Strecker reaction reported as early as 1850 [20] that gives α-aminonitriles, which are frequently used as intermediates for the synthesis of potentially biological active heterocycles and natural products [21-24] as well as other biological useful compounds such as saframycin A, a

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natural product with anti-tumour activity or phthalascidi, a synthetic analogue, which exhibits even greater potency than saframycin A [25]. However, the most important application of α-aminonitriles is the synthesis of α-amino acids upon hydrolysis of the nitrile group [26-28]. There are several reported methods for the synthesis of α-aminonitriles with the application of various cyanide sources and catalytic systems [29-55]. Many of these methods suffer from some crucial drawbacks such as the use of stoichiometric reagents, low yields of the products, the use of hazardous and often expensive catalysts, harsh reaction conditions, extended reaction times, tedious procedure for the preparation of catalyst, the use of volatile and hazardous organic solvents, no compliance with the green chemistry protocols and also tedious workup leading to the generation of a large amount of toxic waste. Consequently, development of general, more practical and environmentally benign methods for the synthesis of α-aminonitriles is in demand.

As a part of our continuing studies in developing efficient catalyst-free synthetic methodologies in organic preparations [56-60], we found that synthesis of α-aminonitriles *via* a one-pot three-component reaction of aldehydes, amines and TMSCN can be efficiently achieved at room temperature using a neutral ionic liquid ([Bmim]Br) as a promoting media without any catalyst under ultrasonic irradiation.

MATERIAL AND METHODS

Apparatus and Analysis

Reagents and solvents were purchased from Merck, Fluka or Aldrich. Melting points were determined in capillary tubes in an electro-thermal C14500 apparatus. The progress of the reaction and the purity of compounds were monitored by TLC using analytical silica gel plates (Merck 60 F250). All known compounds were identified by comparison of their melting points and ^{1}H NMR data with those in the authentic samples. The ^{1}H NMR (500 MHz) or (250 MHz) and ^{13}C NMR (125 MHz) or (62.5 MHz) were run on Bruker Avance DPX-500 and Bruker Avance DPX-250 FT-NMR instruments. Chemical shifts are given as δ values against tetramethylsylane as the internal standard and J values are given in Hz. Microanalysis was performed on a Perkin-Elmer 240-B microanalyzer. The ultrasound apparatus was cleaning bath Elmasonic S30H (Germany).

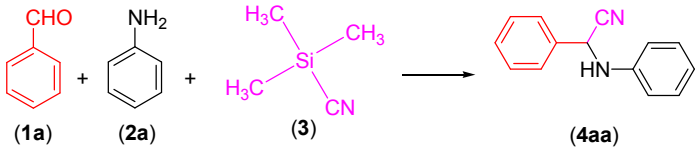
The operating frequency was 50-60 kHz and the output power was 280 W. The reaction flasks were located in the maximum energy area in the water bath, where the surface of reactants (reaction vessel) is slightly lower than the level of the water, and the addition or removal of water controlled the temperature of the water bath. The temperature of the water bath was controlled at 25-30 °C. All experiments performed in this work were repeated three times. The yield reported represents the average of the values obtained for each reaction.

General Procedure for the Synthesis of α-Aminonitriles

Caution: TMSCN is toxic and could produce hydrogen cyanide in contact with water. All operations should be performed with special care under a well-ventilated fume hood.

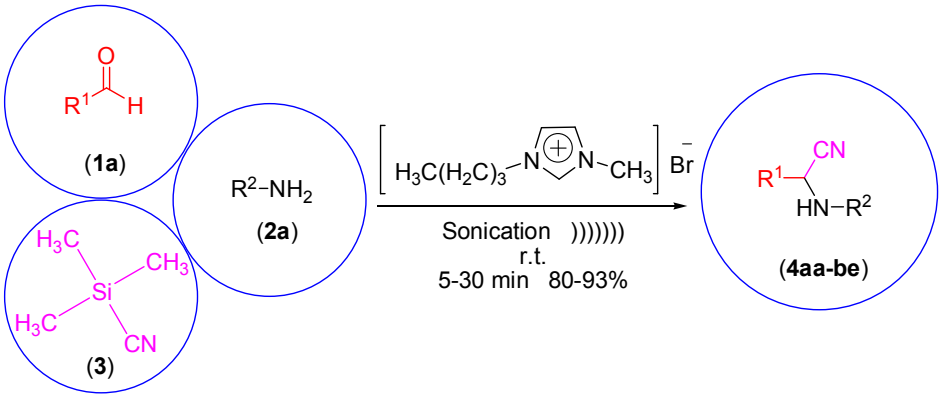
Aldehyde (1 mmol) and amine (1.2 mmol) were added to [Bmim]Br (0.5 g) in a 25 ml Pyrex flask and the resulting mixture was continuously irradiated for 2 min. TMSCN (1.2 mmol) was added and the resulting mixture was continuously irradiated for an appropriate time (Table 2) at room temperature. The reactions were followed by thin layer chromatography (TLC) using hexane/ethyl acetate (3:1) as an eluent. The temperature was controlled and fixed at 25-30 °C by pouring cold water in the bath in the case of any elevation of temperature. After completion of the reaction, the reaction mixture was added to cold water (20 ml) and stirred magnetically for 5 min. Insoluble crude products were filtered and recrystallized from EtOH/H_2O 4:1. In the case of oil products, appropriate amounts of ethyl acetate (5 ml, two times) were added, the organic layer was washed with brine (5 ml), dried over $MgSO_4$, and concentrated. The residue was chromatographed over silica gel (15% ethyl acetate in hexane) to give a pure product. To recover the [Bmim]Br, after the isolation of insoluble products, water was evaporated, and the remaining viscous liquid was washed with ethyl acetate (5 ml) and dried under reduced pressure at 120 °C for 48 h to give [Bmim]Br in 97% yield.

Table 1. Catalyst-free One-pot Three-component Reaction between Benzaldehyde (1a, 1 mmol), Aniline (2a, 1.2 mmol) and TMSCN (3, 1.2 mmol) in Several Reaction Medium under Ultrasonic Irradiation at Room Temperature



Entry	Reaction medium	Time (min)	Yield (%)[a]
1	H_2O (0.5 ml)	60	Trace
2	EtOH (0.5 ml)	60	36
3	CH_3CN (0.5 ml)	60	30
4	$CHCl_3$ (0.5 ml)	60	30
5	PEG 400 (0.5 ml)	60	51
6[b]	SiO_2 (0.5 g)	60	68
7	[Bmim]Br (0.5 g)	10	91

[a]Isolated yield. [b]Reaction conditions: substrates were truly mixed with SiO_2 and the obtained mixture was irradiated under the neat condition.



Scheme 1. The ultrasound-promoted one-pot three-component synthesis of α-aminonitriles in [Bmim]Br at room temperature.

Selected Spectral Data

2-(4-(1*H*-tetrazol-5-yl)phenyl)-2-(phenylamino) acetonitrile (4bc). White solid, m.p.: 138-140 °C. υ_{max} (KBr): 3355, 3010, 2970, 2920, 2880, 2870, 2855, 2220 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$): δ (ppm) 3.78 (s, 1H), 6.57 (s, 1H), 6.78 (d, J = 7.5 Hz, 2H), 6.84 (t, J = 7.5 Hz,

1H), 7.15 (t, J = 7.5 Hz, 2H), 7.49 (d, J = 7.2 Hz, 2H), 8.31 (d, J = 7.2 Hz, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$): δ (ppm) 45.5, 115.7, 117.3, 119.2, 123.5, 125.0, 128.4, 129.0, 134.3, 145.5, 156.0. Anal. Calcd. for $C_{15}H_{12}N_6$: C, 65.21; H, 4.38; N, 30.42%; Found: C, 65.46; H, 4.03; N, 30.44%.

2-(4-(1H-tetrazol-5-yl)phenyl)-2-(*p*-tolylamino) acetonitrile (4bd). White solid, m.p.: 123-125 °C. υ_{max} (KBr): 3340, 3017, 2984, 2925, 2890, 2880, 2840, 2225 cm^{-1}. ^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) 2.30 (s, 3H), 3.84 (s, 1H), 6.62 (s, 1H), 6.68 (d, J = 7.5 Hz, 2H), 6.84 (d, J = 7.5 Hz, 2H), 7.53 (d, J = 7.2 Hz, 2H), 8.33 (d, J = 7.2 Hz, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$): δ (ppm) 21.0, 45.5, 115.0, 117.8, 123.4, 124.3, 128.3, 129.8, 130.1, 134.0, 142.9, 155.5. Anal. Calcd. for $C_{16}H_{14}N_6$: C, 66.19; H, 4.86; N, 28.95%; Found: C, 65.88; H, 4.93; N, 28.77%.

2-(4-(1H-tetrazol-5-yl)phenyl)-2-(benzylamino) acetonitrile (4be). White solid, m.p.: 133-135 °C. υ_{max} (KBr): 3350, 3012, 2990, 2960, 2870, 2860, 2830, 2310 cm^{-1}. ^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) 2.95 (s, 1H), 3.50 (distorted AB system, 2H), 5.83 (s, 1H), 67.20-7.53 (m, 8H), 8.31 (d, J = 7.2 Hz, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$): δ (ppm) 48.5, 48.7, 117.9, 122.5, 125.0, 126.8, 128.0, 128.3, 128.5, 135.0, 139.5, 155.8. Anal. Calcd. for $C_{16}H_{14}N_6$: C, 66.19; H, 4.86; N, 28.95 %; Found: C, 65.99; H, 4.72; N, 29.17 %.

RESULTS AND DISCUSSION

In the first step, to find an appropriate reaction medium for the catalyst-free synthesis of the titled compounds, the one-pot three-component condensation of benzaldehyde (1a, 1 mmol), aniline (2a, 1.2 mmol) and TMSCN (3, 1.2 mmol) was selected as a model reaction and examined in several reaction mediums. The yield and reaction times were monitored in the presence of ultrasonic irradiation at room temperature. The obtained results are summarized in Table 1.

As it is clear form Table 1, the best results were obtained in the presence of 1-butyl-3-methylimidazolium bromide ([Bmim]Br) as a reaction medium. In order to examine the scope and efficiency of [Bmim]Br as a neutral promoting reaction medium for the catalyst-free synthesis of α-aminonitriles, various aliphatic as well as aromatic aldehydes and amines were condensed with TMSCN (Scheme 1) and the results are summarized in Table 2. From the obtained results listed in Table 2, it can be concluded that all reactions proceeded efficiently and the desired products were produced in good to excellent yields in short reaction times without the formation of any detectable by-products. The presence of electron withdrawing groups on the aromatic ring of aldehydes (Table 2, entries 4an, 4ao and 4at) caused the enhancement on the reaction rates compared with those that substituted with electron releasing groups (Table 2, entries 4aq, 4ar and 4as). Besides, desired products were successfully obtained in the case of acid and base-sensitive heteroaromatic aldehydes (Table 2, entries 4au, 4av and 4aw) as well as aldehydes bearing a tetrazole moiety in their structures (Table 2, entries 4bc, 4bd and 4be). Surprisingly, no decrease in the reaction rate was observed in the case of aliphatic amines (Table 2, entries 4ae, 4af, 4aq, 4ah, 4ai, 4ar, 4au, 4av and 4aw), whereas a substantial decrease in the rate of the reaction has been observed in our previously reported method for the synthesis of titled compounds with the use of Fe_3O_4-BF_3 magnetic nanoparticles [51]. As it has been reported, the unstable nature of aliphatic imines in the presence of water and acid (Brönsted or Lewis) is the main reason for the decrease in the reaction rate of the synthesis of α-aminonitriles with aliphatic amines [51]. We think that the neutral conditions of the present method leads to the more stability of imines, and consequently lack of any decrease in the reaction rate of the aliphatic amines. Moreover, the accelerated reaction of H_2O, which was prepared during imine formation with TMSCN may also cause to the lack of decrease in the reaction rates in the case of aliphatic amines.

To assess the capability and efficiency of our methodology with respect to the reported procedures for the synthesis of α-aminonitrile derivatives, the results of the application of these methods are tabulated in Table 3. As it is clear from Table 3, our presented methodology was more efficient.

Many recent studies have established that hydrogen bonding can occur between the solute and the cationic or anionic component of ILs [61-63]. Based on these facts, Deb and Bhuyan have suggested the synthesis of bis(indolyl)methanes *via* condensation of indoles with aldehydes that hydrogen bond formation between a carbonyl group and solvent leads to activation of aldehydes [64].

Table 2. The Catalyst-free One-pot Three Component Synthesis of α-Aminonitriles in the Presence of [Bmim]Br under Ultrasonic Irradiation at Room Temperature

Entry	R^1	R^2	Time (min)	Yield (%)[a]	M.P. (°C)	
					Found	Rep. [Ref.]
4aa	C_6H_5	C_6H_5	10	91	73-75	71-72 [50]
4ab	C_6H_5	3,4-CH_3-C_6H_3	15	91	94-96	93-95 [50]
4ac	C_6H_5	4-Br-C_6H_4	10	90	90-92	91-93 [49]
4ad	C_6H_5	4-CH_3-C_6H_4	10	92	105-106	102-104 [49]
4ae	C_6H_5	-$CH_2CH_2OCH_2CH_2$-	15	91	68-71	69-71 [49
4af	C_6H_5	C_6H_5-CH_2	10	92	Oil	Oil [34]
4ag	C_6H_5	$CH_3CH_2CH_2CH_2$	20	90	Oil	Oil [34]
4ah	C_6H_5	$(CH_3)_2CH$	20	80	Oil	Oil [34]
4ai	C_6H_5	-$CH_2CH_2CH_2CH_2$-	20	90	Oil	Oil [34]
4aj	C_6H_5	4-Cl-C_6H_4	15	90	111-113	110-112 [50]
4ak	C_6H_5	1-Naphthyl	25	90	127-129	125-127 [50]
4al	4-Cl-C_6H_4	4-CH_3-C_6H_4	10	93	83-84	85-87 [49]
4am	4-Cl-C_6H_4	C_6H_5	10	91	109-11	111-112 [34]
4an	4-F-C_6H_4	C_6H_5	15	90	102-105	99-101 [49]
4ao	4-F-C_6H_4	4-CH_3-C_6H_4	15	93	100-102	101-103 [49]
4ap	2,4-Cl-C_6H_3	4-CH_3-C_6H_4	30	90	114-116	116-118 [50]
4aq	4-OCH_3-C_6H_4	C_6H_5	35	90	96-98	95-97 [49]
4ar	3-OCH_3-C_6H_4	$CH_3CH_2CH_2CH_2$	20	83	Oil	Oil [32]
4as	3-OCH_3-C_6H_4	C_6H_5	30	90	63-64	60-61 [32]
4at	3-NO_2-C_6H_4	C_6H_5	5	94	88-93	89-92 [49]
4au	2-Thienyl	C_6H_5-CH_2	15	91	Oil	Oil [32]
4av	2-Furyl	C_6H_5-CH_2	15	90	Oil	Oil [32]
4aw	3-Pyridyl	$CH_3CH_2CH_2CH_2$	50	80	Oil	Oil [50]
4ax	4-CH_3-C_6H_4	C_6H_5	25	92	74-76	77-78 [47]
4ay	1-Naphthyl	C_6H_5	40	82	92-93	94-97 [40]

[a]Yields are referred to isolated yields of pure products.

Table 3. Comparison of Results of the Condensation Reaction of Benzaldehyde (1a) and Aniline (2a) with TMSCN (3) Using the Present Work with those of Previously Reported Methods

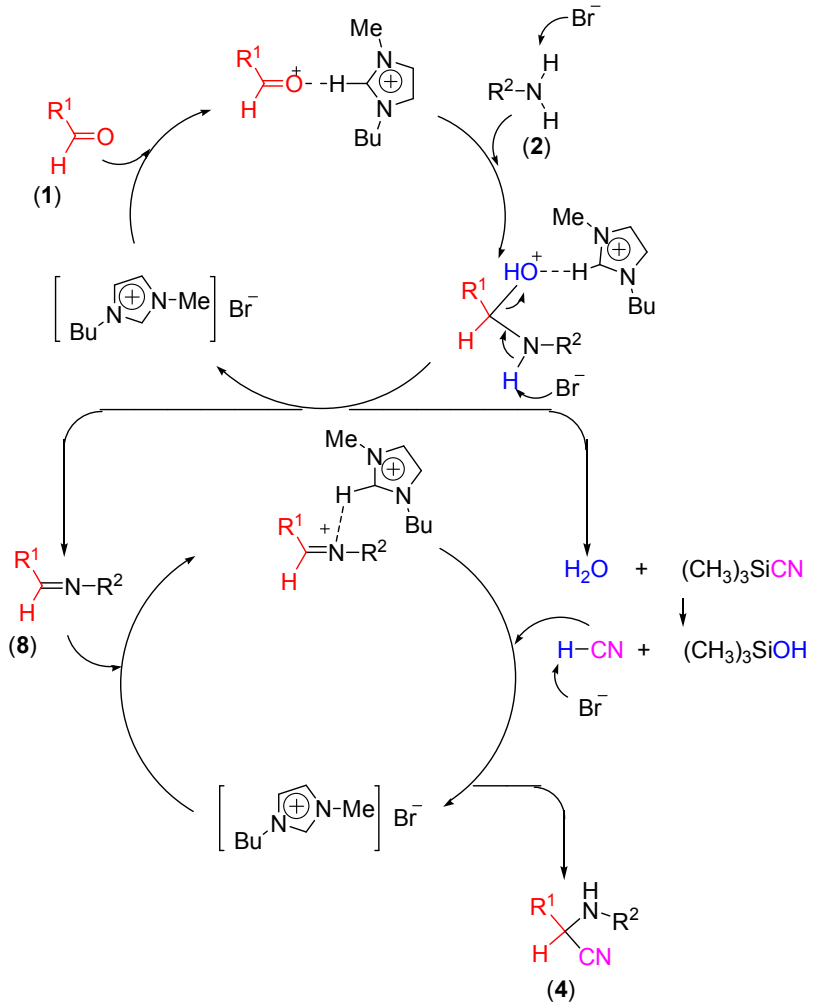
Entry	Conditions	Time (min)	Yield (%)	Ref.
1	$InCl_3$ (20 mol%), Dry THF, r.t.	360	75	[32]
2	$BiCl_3$ (10 mol%), CH_3CN, r.t.	600	84	[33]
3	Montmorillonite KSF clay, 1.0 g, CH_2Cl_2, r.t.	210	90	[34]
4	Silica-based scandium(III) (3 mol%), CH_2Cl_2, r.t.	840	94	[35]
5	SO_4^{2-}/ZrO_2 (10 mol%), Dry THF, N_2 atmosphere, r.t.	90	93	[36]
6	Guanidine hydrochloride (3 mol%), EtOH, 40 °C	60	94	[37]
7	Xanthane sulfuric acid (6 mol%), Dry CH_3CN, r.t.	65	97	[38]
8	Silica sulfuric acid (25 mol%), CH_2Cl_2, r.t.	360	88	[39]
9	Silica bonded S-solfonic acid (6.6 mol%), EtOH, r.t.	30	94	[41]
10	Catalyst-free, [Bmim]Br (0.5 g), Ultrasonic irradiation, r.t.	10	91	This work

Table 4. Catalyst-free One-pot Three-component Reaction between Benzaldehyde (1a) (1 mmol), Aniline (2a) and TMSCN (3) (1.2 mmol) in the Presence of [Bmim]Cl, [Bmim]Br and [Bmim]I under Ultrasonic Irradiation at Room Temperature

Entry	Reaction medium	Time (min)	Yield (%)[a]
1	[Bmim]Cl (0.5 g)	25	88
2	[Bmim]Br (0.5 g)	10	91
3	[Bmim]I (0.5 g)	20	90

[a]Isolated yield.

Moreover, Crowhurts *et al.* demonstrated that imidazolium ILs are able to act as strong hydrogen bond acids as well as hydrogen bond bases at the same time [63]. Recently, we studied the effect of counter-anion in the total yield and reaction time of the catalyst-free one-pot four-component synthesis of 2H-indazolo[2,1-*b*]phthalazine-triones in the case of buthyl methylimidazolium based ILs ([Bmim]X, $X = Cl^-$, Br^- and I^-) and this has been established that the



Scheme 2. The proposed mechanism for the one-pot three-component synthesis of α-aminonitriles in [Bmim]Br

best results are accessible when X = Br^- [57]. Based on those observations, we expected that [Bmim]Br must be the best reaction medium for the synthesis of titled compounds due to the moderate interaction between cationic component (buthyl methylimidazolium) and the anionic component (Br^-) that leads to the sufficient hydrogen bond acidity and hydrogen bond basicity of the IL. In order to examine our theory, the model reaction was studied in the presence of three ILs ([Bmim]Cl, [Bmim]Br and [Bmim]I) and the obtained results are summarized in Table 4. As it is clear from Table 4, the best results were obtained in the presence of [Bmim]Br as we expected.

According to these observations, we suggest a mechanism for this reaction in which the ILs serves two catalytic functions. First, electrophilically activation of carbonyl groups through hydrogen-bonding to the carbonyl oxygen, and second, the enhancement of the nucleophilicity of amines through interaction with polar hydrogens of the N-H bond, as shown in Scheme 3. Our proposed mechanism contains two steps. Initially, the imine (8) forms by nucleophilic addition of amine (2) to carbonyl compound (1) followed by dehydration. In the second step, addition of cyanide anion (prepared by hydrolysis of TMSCN in the presence of water) to imine leads to the formation of desired

Table 5. The catalyst-free One-pot Three-component Synthesis of α-Aminonitriles in the Presence of [Bmim]Br under Stirring without Ultrasound (Silent Reactions) Conditions at Room Temperature

Entry	R^1	R^2	R^3	Time (min)	Yield (%)[a]
4aa	C_6H_5	H	C_6H_5	360	20
4ai	C_6H_5	H	$-CH_2CH_2CH_2CH_2-$	360	No reaction
4aj	C_6H_5	H	4-Cl-C_6H_4	360	20
4am	4-Cl-C_6H_4	H	C_6H_5	360	15
4aq	4-OCH_3-C_6H_4	H	C_6H_5	360	No reaction
4as	3-OCH_3-C_6H_4	H	C_6H_5	360	Trace
4at	3-NO_2-C_6H_4	H	C_6H_5	360	25
4au	2-Thienyl	H	C_6H_5-CH_2	360	15
4av	2-Furyl	H	C_6H_5-CH_2	360	15
4aw	3-Pyridyl	H	$CH_3CH_2CH_2CH_2$	360	Trace
4ay	1-Naphthyl	H	C_6H_5	360	Trace
4bb	$(CH_3)_2CH$	H	$-CH_2CH_2CH_2CH_2-$	360	No reaction

[a]Yields refer to isolated pure products.

product (4).

As shown in Scheme 2, our proposed mechanism for the one-pot three-components synthesis of α-aminonitriles in the presence of ultrasonic irradiation in [Bmim]Br consisted of several reactions that all of them have negative activation volumes owing to the condensation of the molecules into a reactive intermediate. In this regard, it is well-known that reactions with negative activation volumes are accelerated with pressure [65]. On the other hand, ultrasound irradiation as well as solvophobic interactions of ionic liquids generates a microscopic internal pressure in the solvent cavity [66]. So, owning to the ultrasonic cavitations, microscopic internal high pressures and high temperatures have been generated in reaction media [67]. Moreover, it is well known that applied ionic liquid ([Bmim]Br) is probably the most viscous solvent examined, so, we think ultrasound helps to the diffusion of species. Accordingly, it is reasonable to assume that these effects should accelerate this type of three-component condensation reactions in the presence of ultrasonic irradiation.

To investigate the promoting role of ultrasonic irradiation for the synthesis of titled compounds in [Bmim]Br as a neutral reaction medium, the reactions were carried out using the same amount of [Bmim]Br at room temperature without ultrasonic irradiation and obtained results are summarized in Table 5. It is clear that without ultrasonic irradiation, the reactions yields are very low and in some cases the reaction was not proceeded even after a long time. This observation establishes the effective and important role of ultrasonic irradiation on the proceeding of the synthesis of titled compounds in [Bmim]Br as a neutral reaction medium without a catalyst.

CONCLUSIONS

In summary, a highly efficient and practical method for

one-pot three-component synthesis of α-aminonitriles using [Bmim]Br as an neutral and reusable reaction medium under ultrasonic irradiation has been introduced. This method not only offers substantial improvements in the reaction rates and yields, but also avoids the use of hazardous catalysts or solvents. The promising points for the presented methodology are efficiency, generality, high yield, short reaction time, cleaner reaction profile, ease of product isolation, simplicity, potential for recycling of the reaction medium and finally agreement with some green chemistry protocols, which all make it a useful and attractive process for the synthesis of α-aminonitrile derivatives. Application of ultrasonic irradiation as a more efficient energy source is another promissing point that highlights this work in the case of green chemistry point of view. Moreover, the neutral reaction conditions causes the lack of decrease in the reaction rate of aliphatic amine (as observed in most previousely reported methods) in one-pot condensation of amines, aldehydes and TMSCN.

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