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Short Communication

Ultrasound-promoted regio and chemoselective synthesis of pyridazinones and phthalazinones catalyzed by ionic liquid [bmim]Br/$AlCl_3$

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ABSTRACT

The first ultrasound-promoted multicomponent synthesis of pyridazinones and phthalazinones from arenes, cyclic anhydrides and $ArNHNH_2$ in the presence of an efficient recyclable catalyst, [bmim]Br/$AlCl_3$, in high yield and short reaction time is reported.

1. Introduction

Pyridazinones are useful compounds with a broad array of biologically activities. They possess notable hypertensive [1], platelet aggregation inhibition [2], phosphodiesterase [3], antiasthmatic [4], antisecretory and antiulcer [5], antidepressant [6], antibacterial [7], antifongic [8], α-adrenoceptor antagonists [9], analgesic [10], antiinflammatory [11], antianemic [12], nephrotropic [13], cardiotonic [14], anticancer [15], pesticidal and herbicidal [16] properties.

Previously reported synthetic routes to synthesis of pyridazinones include reaction of γ-ketoacids and their derivatives with alkylhydrazines or phenyl-hydrazines [17], condensation of Wittig reagents with arylhydrazones or condensation of α-ketoesters with hydrazinocarbonyl-acetic acid esters [18], catalytic reactions of alkynes with arylhydrazines (hydrohydrazination) [19], condensation of hydrazine with appropriate substituted lactones [20].

Green chemistry has become a major driving force for organic chemists to develop environmentally benign routes for the preparation of organic compounds. For example, the possibility of performing reactions under ultrasound irradiation to enhance the reaction efficiency from both economic and ecological points of view has given to this kind of procedures a remarkable synthetic value and received great attention [21,22].

In recent years, ionic liquids have shown great promise as an attractive alternative to conventional catalysts and solvents for synthesizing organic chemicals [23–26]. The increased interest for their investigations is mainly due to their green characteristics, such as chemical and thermal stability, no measurable vapor pressure, non-flammability, non-coordinating and solvation properties [27]. They can be readily recycled, have profound effect on the activity and selectivity in reactions and in some cases, facilitate the isolation of products. Therefore, ionic liquids are considered valuable substitute for volatile organic solvents. To the best of knowledge, this is the first report on the ultrasound-promoted multicomponent synthesis of pyridazinones and phthalazinones from arenes, cyclic anhydrides and $ArNHNH_2$ in the presence of the efficient recyclable catalyst, [bmim]Br/$AlCl_3$.

2. Materials and methods

2.1. Apparatus and analysis

For the ultrasound reactions, the ultrasound apparatus Astra 3D (9.5 dm^3, 45 kHz frequency, input power with heating, 305 W, number of transducers, 2) from TECNO-GAZ was used. Chemicals were purchased from Merck and Fluka and used as purchased.

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Melting points were measured on an Electro-thermal 9100 apparatus and are uncorrected. ^{1}H NMR spectra were obtained on a Bruker DRX 500, 250 and those of ^{13}C NMR spectra on a Bruker DRX 125 Avance spectrometer in $CDCl_3$ as solvent and with TMS as internal standard. FT-IR spectra were recorded on a Shimadzu FT-IR-8400S spectrometer. Elemental analyzes were recorded on a Carlo-Erba EA1110CNNO-S analyzer.

2.2. General procedure for the synthesis of pyridazinones and phthalazinones

A mixture of anhydride (10 mmol), arenes (10 mmol) and [bmim]Br/$AlCl_3$ (20 mmol) were placed into Pyrex-glass open vessel and irradiated in a water bath under silent condition by ultrasound (45 kHz) at 60 °C for the required reaction times (4–5 h). The progress of the reaction was monitored by TLC (EtOAc: petroleum ether 1:4). Then the reaction mixture was cooled to room temperature, and induced into two liquid phases (organic phase and ionic liquid phase) by extracting with $CHCl_3$. The ionic liquid could be reused after the organic phase was extracted out with $CHCl_3$. The products **1–9** was purified by column chromatography (EtOAc: petroleum ether 1:4) to furnish the desired pyridazinones and phthalazinones. The pure products were collected in 65–75% yields.

2.2.1. 2,6-Diphenyl-4,5-dihydropyridazin-3(2H)-one 1

Off white solid, mp 92–94 °C, IR (KBr, cm^{-1}) ν_{max} 1680, 1600, 1541, 1330, 1490; ^{1}H NMR (500 MHz, $CDCl_3$): 2.84 (2H, t, J = 8.4 Hz), 3.15 (2H, t, J = 8.5 Hz), 7.30–7.33 (1H, m), 7.45–7.48 (5H, m), 7.64–7.66 (2H, d, J = 7.5 Hz), 7.85–7.86 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 23.33, 28.47, 125.31, 126.51, 127.00, 129.07, 130.42, 135.91, 141.68, 151.93, 165.69. Anal. Calcd. for $C_{16}H_{14}N_2O$: C, 76.78; H, 5.64; N, 11.19. Found: C, 76.62; H, 5.77; N, 11.10.

2.2.2. 6-(4-Methylphenyl)-4,5-dihydropyridazin-3(2H)-one 2

Red oil, IR (KBr, cm^{-1}) ν_{max} 1680, 1595, 1492, 1326, ^{1}H NMR (500 MHz, $CDCl_3$): 2.4 (3H, s), 2.78 (2H, t, J = 8.5 Hz), 3.06 (2H, t, J = 8.5 Hz), 7.27–7.30 (2H, d, J = 8.1 Hz), 7.32–7.35 (2H, t, J = 7.4 Hz), 7.47–7.50 (2H, t, J = 7.6 Hz), 7.67–7.69 (2H, d, J = 8.3 Hz), 7.75–7.77 (2H, d, J = 8.3 Hz); 13C NMR (125 MHz, $CDCl_3$): 21.86, 23.21, 125.33, 126.53, 126.96, 128.48, 128.97, 129.01, 129.82, 133.12, 140.71, 152.16, 165.87; Anal. Calcd. for $C_{17}H_{16}N_2O$: C, 77.25; H, 6.10; N, 10.60. Found: C, 77.26; H, 6.33; N, 10.58.

2.2.3. 6-(2,4-Dimethylphenyl)-2-phenyl-4,5-dihydropyridazin-3(2H)-one 3

Red oil, IR (KBr, cm^{-1}) ν_{max} 1677, 1600, 1494, 1326; ^{1}H NMR (500 MHz, $CDCl_3$): 2.4 (3H, s), 2.5 (3H, s), 2.8 (2H, t, J = 8.4 Hz), 3.0 (2H, t, J = 8.5 Hz), 7.10 (s, 1H), 7.12 (1H, s), 7.28–7.33 (m, 2H), 7.43–7.46 (2H, t, J = 8.2 Hz), 7.61–7.62 (2H, d, J = 7.6 Hz); ^{13}C NMR (125 MHz, $CDCl_3$): 21.60, 21.61, 26.95, 28.74, 125.30, 126.93, 127.12, 128.93, 132.51, 134.02, 136.22, 139.66, 141.49, 155.60, 165.75; Anal. Calcd. for $C_{18}H_{18}N_2O$: C, 77.67; H, 6.52. N, 10.06. Found: C, 77.56; H, 6.23; N, 10.18.

2.2.4. 6-(4-Chlorophenyl)-2-phenyl-4,5-dihydropyridazin-3(2H)-one 4

Red oil, IR (KBr, cm^{-1}) ν_{max} 1683, 1595, 1490, 1326, 1012, ^{1}H NMR (500 MHz, $CDCl_3$): 2.8 (2H, t, J = 8.5 Hz), 3.06–3.10 (2H, t, J = 8.5 Hz), 7.32–7.34 (1H, t, J = 7.3 Hz), 7.41–7.42 (2H, d, J = 8.6 Hz), 7.46–7.49 (2H, t, J = 8.2 Hz), 7.62–7.63 (2H, d, J = 7.4 Hz), 7.76–7.79 (2H, d, J = 8.62 Hz); ^{13}C NMR (125 MHz, $CDCl_3$): 23.17, 28.31, 125.26, 125.39, 127.14, 127.91, 129.00, 129.29, 136.44, 141.59, 150.68, 165.53; Anal. Calcd. for $C_{16}H_{13}ClN_2O$: C, 67.49; H, 4.60; N, 9.84. Found: C, 67.21; H, 4.58; N, 9.81.

2.2.5. 2,6-Bis(4-methoxyphenyl)-4,5-dihydropyridazin-3(2H)-one 5

Red oil, IR (KBr, cm^{-1}) ν_{max} 1683, 1595, 1490, 1326, 1H NMR (500 MHz, $CDCl_3$): 2.72–2.75 (2H, t, J = 8.5 Hz), 3.01–3.04 (2H, t, J = 7.64 Hz), 3.81 (3H, s), 3.83 (3H, s), 6.90–6.94 (4H, m), 7.46–7.48 (2H, d, J = 8.9 Hz), 7.72–7.74 (2H, d, J = 8.86 Hz); ^{13}C NMR (125 MHz, $CDCl_3$): 23.23, 28.37, 55.81, 55.92, 114.24, 126.83, 128.07, 128.42, 134.95, 151.61, 158.50, 161.53, 165.86; Anal. Calcd. for $C_{18}H_{18}N_2O_3$: C, 69.66; H, 5.85; N, 9.03. Found: C, 69.51; H, 5.69; N, 9.45.

2.2.6. 2,4-Diphenylphthalazin-1(2H)-one 6

Light brown solid, mp 161–163 °C, IR (KBr, cm^{-1}) ν_{max} 1656, 1580, 1487, 1325. ^{1}H NMR (500 MHz, $CDCl_3$): 7.40–7.43 (1H, t, J = 7.3 Hz), 7.51–7.57 (5H, m), 7.69–7.70 (2H, d, J = 5.5 Hz), 7.78–7.79 (2H, d, J = 7.6 Hz), 7.84–7.87 (3H, m), 8.66–8.67 (1H, d, J = 7.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$): 126.18, 127.18, 128.08, 128.20, 129.11, 129.40, 129.87, 130.06, 131.98, 132.18, 133.59, 135.47, 142.43, 148.05, 159.37; Anal. Calcd. for $C_{20}H_{14}N_2O$: C, 80.52; H, 4.73; N, 9.39. Found: C, 80.25; H, 4.51; N, 9.27.

2.2.7. 2-Phenyl-4-o-tolyl-2H phthalazin-1-one **7**

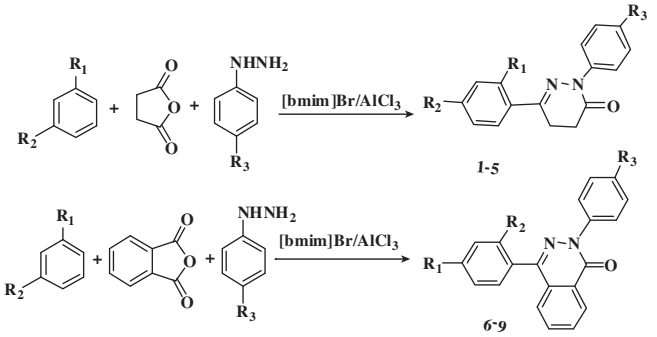
Brown solid, mp 124–126 °C, IR (KBr, cm^{-1}) ν_{max} 1662, 1595, 1490, 1330, ^{1}H NMR (500 MHz, $CDCl_3$): 2.5 (3H, s), 7.37–7.42 (3H, m), 7.51–7.54 (2H, t, J = 8.0 Hz), 7.58–7.60 (2H, d, J = 7.9 Hz), 7.79–7.80 (2H, d, J = 7.7 Hz), 7.82–7.86 (m, 3H), 8.65–8.67 (1H, d, J = 7.6 Hz); ^{13}C NMR (125 MHz, $CDCl_3$): 21.80, 126.26, 127.35, 128.00, 128.15, 129.11, 129.41, 129.65, 129.74, 129.88, 131.99, 132.62, 133.51, 139.67, 142.50, 148.07, 159.37; Anal. Calcd for $C_{21}H_{16}N_2O$: C, 80.75; H, 5.16; N, 8.97. Found: C, 80.35; H, 5.87; N, 8.43.

2.2.8. 4-(2,4-Dimethylphenyl)-2-phenyl-2H phthalazin-1-one **8**

Brown solid, mp 156–158 °C, IR (KBr, cm^{-1}) ν_{max} 1662, 1583, 1492, 1330, ^{1}H NMR (500 MHz, $CDCl_3$): 2.25 (3H, s), 2.45 (3H, s), 7.17–7.19 (1H, d, J = 7.6 Hz), 7.21(1H, s), 7.30–7.31 (1H, d, J = 7.5 Hz), 7.38–7.43 (2H, m), 7.50–7.53 (2H, t, J = 7.7 Hz), 7.76–7.79 (3H, d, J = 7.8 Hz), 7.83–7.86 (1H, t, J = 7.3 Hz), 8.63–8.65 (1H, d, J = 7.8 Hz); ^{13}C NMR (125 MHz, $CDCl_3$): 20.28, 21.70, 126.17, 126.35, 127.07, 127.28, 127.98, 129.14, 130.30, 131.78, 131.98, 133.54, 133.74, 137.30, 139.54, 142.41, 148.42, 159.48; Anal. Calcd. for $C_{22}H_{18}N_2O$: C, 80.96; H, 5.56; N, 8.58. Found: C, 80.80; H, 5.57; N, 8.39.

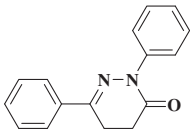
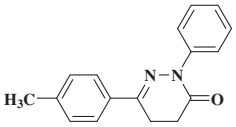
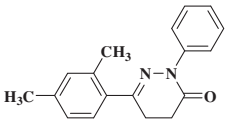
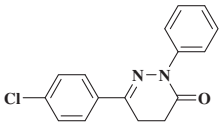
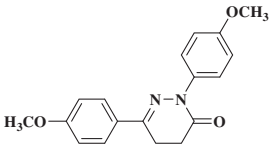
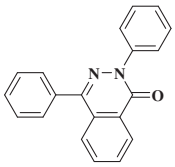
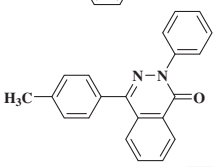
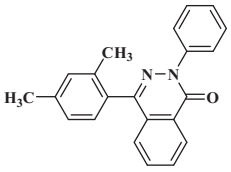
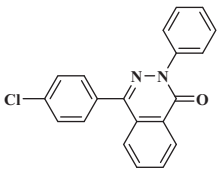
2.2.9. 4-(2-Chlorophenyl)-2-phenyl-2H phthalazin-1-one **9**

Brown solid, mp 164–165 °C, IR (KBr, cm^{-1}) ν_{max} 1662, 1593, 1569, 1490, 1323, 1139, ^{1}H NMR (500 MHz, $CDCl_3$): 7.40–7.43



Scheme 1. Multicomponent synthesis of pyridazinones and phthalazinones.

Table 1
Multicomponent synthesis of pyridazinones and phthalazinones.

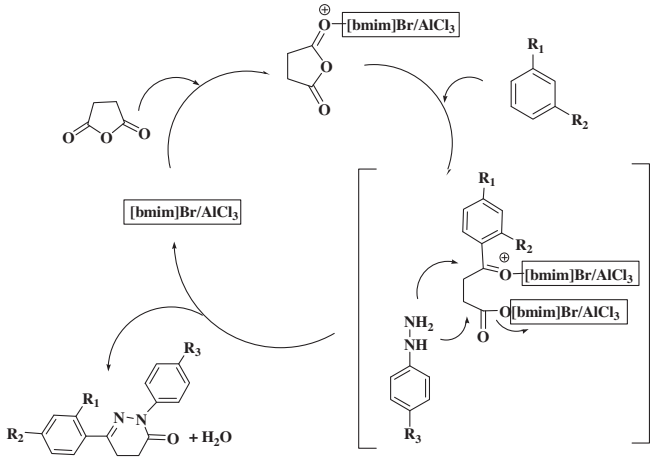
Entry	Product[b]	Reflux		Ultrasound		mp (°C)
		Time (h)	Yield[a](%)	Time (h)	Yield[a](%)	
1		11	52	5	65	92–94
2		10	57	4	75	Oil
3		11.5	63	4.5	72	Oil
4		12	54	5	65	Oil
5		10.5	65	4	72	Oil
6		12	45	5	65	161–163
7		10.5	68	4	75	124–126
8		11	62	4.5	71	156–158
9		12.5	58	5	69	164–166

[a] Isolated yield.
[b] Identified by spectroscopic analysis (IR, ^{1}H NMR, ^{13}C NMR and elemental analysis).

(1H, t, J = 7.4 Hz), 7.51–7.59 (m, 5H), 7.68–7.73 (2H, d, J = 7.2 Hz), 7.78–7.80 (2H, d, J = 7.6 Hz), 7.83–7.88 (2H, m), 8.66–8.67 (1H, d, J = 7.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$): 126.27, 127.30, 128.08, 128.18, 129.07, 129.15, 129.40, 129.55, 129.66, 129.98, 132.08, 133.59, 135.48, 142.44, 148.04, 159.37; Anal. Calcd. for $C_{20}H_{13}ClN_2O$: C, 72.18; H, 3.94; N, 8.42. Found: C, 72.52; H, 3.57; N, 8.27.

3. Results and discussion

The remarkable catalytic activity together with easy availability, operational simplicity of [bmim]Br/$AlCl_3$ and our continued interests for the development of efficient and environmentally friendly procedures for the synthesis of heterocyclic compounds [28–30] triggered us to synthesize pyridazinones and



Scheme 2. Proposed mechanism for the multicomponent synthesis of pyridazinones and phthalazinones.

Table 2
Studies on the reuse of [bmim]Br/$AlCl_3$ in the preparation of 1.

Round	1	2	3	4	5	6	7
Yield	65	65	64	62	63	62	60

phthalazinones in a multicomponent reaction in the presence of [bmim]Br/$AlCl_3$ (Scheme 1). This class of reactions is of particular interest in combinatorial chemistry because it allows the production of vast arrays of molecules in an efficient mode.

As shown in Table 1, this multicomponent reaction (entry **1–9**) in the absence of ionic liquid/$AlCl_3$ lead to no product after 24 h. A combination of ionic liquid/$AlCl_3$ and ultrasound irradiation gave products in shorter reaction time and higher yields in comparison with the thermal reaction using catalyst ionic liquid/$AlCl_3$. The aryls containing electron releasing substituents such as toluene, m-xylene and anisole react better than benzene or chlorobenzene, because the electron releasing substituent improves acylation in the reaction mechanism.

The mechanism of reaction goes through Friedel–Crafts acylation between arenes and cyclic anhydride over an efficient acidic catalyst to prepare keto-carboxylic acids. Intermolecular hydrazone formation followed by intramolecular cyclization led to the formation of pyridazinones **1–5** and phthalazinones **6–9** (Scheme 2). For these reasons, we used [bmim]Br/$AlCl_3$ as a catalyst, because of its environmental compatibility, reusability, operational simplicity, no toxicity, noncorrosiveness, low cost and ease of isolation.

All of the products were fully characterized by spectroscopic methods (IR and ^{1}H NMR and ^{13}C NMR). The results are summarized in Table 1.

In order to investigate the performance of [bmim]Br/$AlCl_3$ as a reusable catalyst, a recycling experiment was conducted. After extracting the reaction mixture with $CHCl_3$, the reaction mixture became two liquid phases, namely, the organic phase (unreaction reactants and products phase) and the [bmim]Br/$AlCl_3$ ionic liquid phase. The ionic liquid [bmim]Br/$AlCl_3$ was reused as a catalyst after extracting out the organic phase with ether and vacuum drying at 80–100 °C for 30 min. The reaction results using the recycled [bmim]Br/$AlCl_3$ are summarized in Table 2.

To the best of our knowledge, this is the first report of the one pot synthesis of pyridazinones and phthalazinones, which has several advantages, such as virtue of their convergence, productivity, ease of execution and work-up, the small amount of waste, short reaction time, reusability of catalyst coupled with replacement of any volatile organic solvent, since the ionic liquid plays dual roles of Lewis acid catalyst and solvent.

4. Conclusion

In conclusion, a convenient one pot [bmim]Br/$AlCl_3$-catalyzed synthesis of pyridazinones and phthalazinones from arenes, anhydrides and $ArNHNH_2$ in high yield and short reaction time was developed.

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