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Expeditious one-pot multicomponent microwave-assisted green synthesis of substituted 2-phenyl Quinoxaline and 7-bromo-3-(4-ethylphenyl) pyrido[2,3-b]pyrazine in water–PEG and water–ethanol

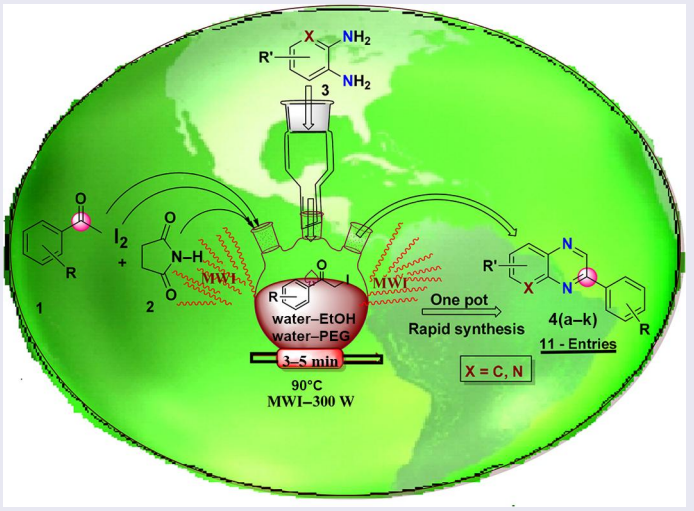
Santosh A. Jadhav[a], Aniket P. Sarkate[b], Mahesh G. Shioorkar[a], and Devanand B. Shinde[c]

[a]Department of Chemistry, Vivekanand Arts, Sardar Dalipsingh Commerce and Science College, Aurangabad, Maharashtra, India; [b]Department of Chemical Technology, Dr. Babasaheb Ambedkar Marathwada University, Aurangabad, Maharashtra, India; [c]Shivaji University, Vidya Nagar, Kolhapur, Maharashtra, India

ABSTRACT

An eco-friendly, expeditious one-pot multicomponent synthesis of substituted 2-phenyl quinoxaline and 7-bromo-3-(4-ethylphenyl) pyrido [2,3-*b*]pyrazine **4a–k** in water–ethanol from easily available starting materials as acetophenone **1**, succinamide **2**, aromatic amine **3**, *in situ*-generated α-iodo acetophenone from acetophenone, succinamide and catalyzed by silver iodide in combination with green solvent polyethylene glycol-400 and water (2:1) under microwave irradiation. The newly developed protocol with excellent yield of products in very short time of reaction by avoiding the use of lacrimatic α-chloro and α-bromocarbonyl compounds, volatile, toxic organic hazardous solvents, and reagents is the advantage of this research work. The final products were confirmed by their characterization data such as FTIR, ^{1}H NMR, ^{13}C NMR, Mass, HRMS and were compared with its reported method.

GRAPHICAL ABSTRACT



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KEYWORDS

Acetophenone; one-pot green synthesis; polyethylene glycol-400; quinoxalines; water

CONTACT Santosh A. Jadhav profsantoshjadhav@gmail.com Department of Chemistry, Vivekanand Arts, Sardar Dalipsingh Commerce and Science College, Aurangabad, Maharashtra 431001, India

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Introduction

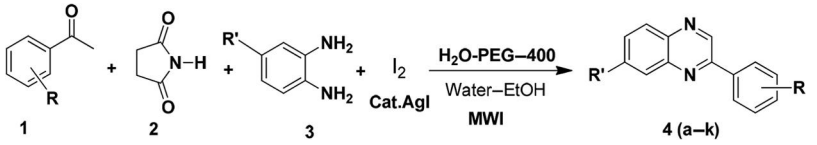
More than one nitrogen-containing fused heterocycles specially quinoxalines are common structural motifs in pharmacologically important molecules that possess a broad spectrum of biological activities including antibacterial, antiviral, anti-inflammatory, anticancer, and kinase inhibitors.[1] Evaluated as anthelmintic agents, semiconductors, dyes etc.,[2,3] used in the agricultural field as fungicides, herbicides, and insecticides.[4]

The use of microwave energy is one of the green protocol in organic synthesis[5,6] which may fascinate many researchers and have several advantages such as less time of reaction, cleaner reaction profile, no side products, and high yield in addition to use of green solvent like polyethylene glycol (PEG) and/or water.[7] Therefore, the use of green solvent in microwave reaction for the synthesis of reported organic molecules is considered a part of green approach protocols.

In past, a variety of synthetic strategies have been developed for the preparation of substituted quinoxalines and/or functionalized quinoxalines, which can be achieved by the reaction of 1,2-phenylenediamine with two-carbon synthones such as α-dicarbonyls,[8,9] α-halogeno carbonyls, α-hydroxycarbonyls, α-azocarbonyls, epoxides, and α, β-dihalides.[10–17] Condensation of an aryl-1,2-diamine with 1,2-diketone compounds in refluxing ethanol or acetic acid[18–23] or using different catalysts and reaction conditions.[24–30] The reactions of phenacyl halides with phenyl 1,2-diamines through condensation–oxidation process in different catalysts and/or medium as sodium hexafluoro phosphate-Amberlite,[31] KF-alumina,[32] $CeCl_3.7H_2O$,[33] PEG-400,[34] cetyltrimethyl ammonium bromide,[35] sodium tetrachloroaurate(III) dehydrate,[36] trimethylsilyl chloride (TMSCl)–water,[37] β-cyclodextrine–water,[38] tetrabutyl ammonium bromide in basic media,[39] DMSO in solvent-free conditions,[40,41] microwave irradiation,[42] $HClO_4$–SiO_2,[43] by pyridine catalyzed.[44] It involved the use of expensive starting reactants, reagents, and harmful organic reagents, catalyst, and solvents having longer reaction times, high temperatures and frequently offers poor yield. There is no any efficient greener approach, rapid synthetic protocol for the synthesis of 2-phenyl quinoxaline and 7-bromo-3-(4-ethylphenyl) pyrido[2,3-*b*]pyrazine. So we wish to report a simple, an efficient and expeditious one-pot multicomponent green synthesis of quinoxaline derivatives in water–PEG and water–ethanol as green solvent from the starting as substituted acetophenones, ortho-phenyl diamine/1,2-diamino benzene, and succinamide in the presence of iodine under microwave irradiation method (Scheme 1).

Results and discussion

In continuation of our earlier research work to investigate some green approaches, synthetic methodologies,[45] recently *in situ*-prepared phenacyl iodide (succinamide



Scheme 1. Synthesis of compound **4**.

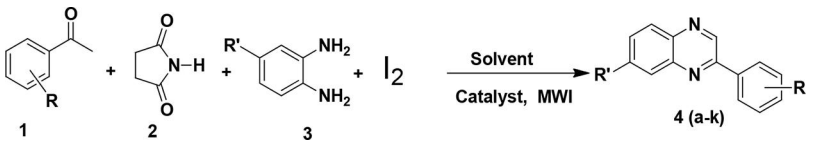
Table 1. Screening of solvent for the synthesis of compound **4a**.

Entry	Solvent	Time (min)	Yield[a] (%)
1	Solvent free	15	00
2	H_2O	07	52
3	MeOH	07	56
4	CH_3CN	07	49
5	EtOH	07	60
6	PEG	7, 8, 9	58, 63, 63
7	H_2O–EtOH (1:1)	2, 3, 5	80, 97, 97
8	H_2O–EtOH (2:1)	3, 5, 7	70, 70, 70
9	H_2O–EtOH (4:1)	3, 5, 7	60, 60, 70
10	H_2O–EtOH (1:2)/(1:4)	3, 5/3, 5	83, 83/93, 93

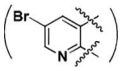
Reaction condition: succinamide (1.2 mmol), silver iodide (1.5 mmol), iodine (20 mol%), acetophenones (1 mmol) in polyethylene glycol-400–water (2:1) was stirred at r.t. and microwave-irradiated power of 350 W at 95–100 °C to α-iodo acetophenone and 1,2-diphenylamine/ortho phenyl di-amine (OPD) (1 mmol) and solvent was added to reaction mixture and irradiated power of 300 W at different temperatures.
[a]Isolated yield.

(1.2 mmol), silver iodide (1.5 mmol), iodine (20 mol%), acetophenones (1 mmol) **1** in PEG-400–water (2:1) was stirred at r.t. and microwave irradiated to α-iodo acetophenone)[46] which was used for the selected model reaction as ortho phenyl di-amine (1 mmol) **3** without solvent and different solvents were screened like water, methanol, ethanol, acetonitrile, PEG, and combination of water ethanol under the microwave irradiation power 200 to 350 W and at different temperatures 60, 70, 80, 90, and 100 °C (Table 1). We observed that better yield was obtained in polar solvents like water, methanol, and ethanol and also in PEG (Table 1, entry nos. 2, 3, 5, and 6) at 90–100 °C, power 300 W. When we mixed

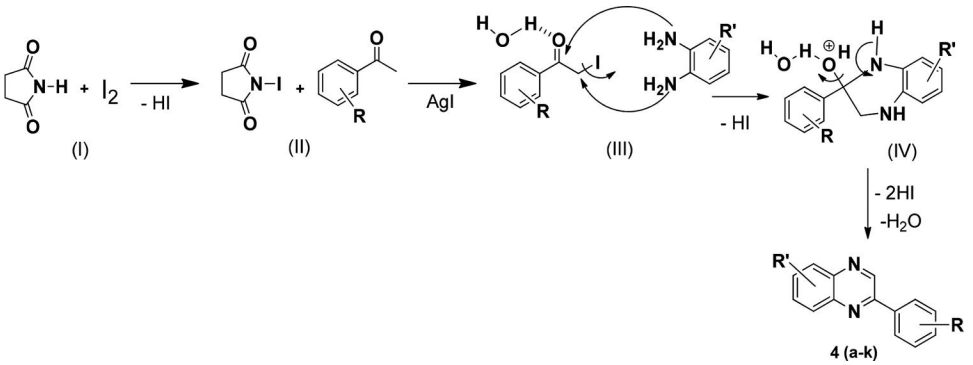
Table 2. Synthesis of functionalized quinoxalines under microwave irradiation method.



Entry	R	R′	Product	Yield[b] (%)	Time (min)	Mp (°C)	Lit. mp (°C)
1	C_6H_5	H	**4a**	97	3	73–75	73–75; Lit.[44]
2	*p*-FlC_6H_4	H	**4b**	94	5	112–113	112–118; Lit.[44]
3	*p*-ClC_6H_4	H	**4c**	96	5	129–130	128–130; Lit.[44]
4	*p*-BrC_6H_4	H	**4d**	96	3	127–129	126–129; Lit.[44]
5	*p*-MeC_6H_4	H	**4e**	96	5	85–87	84–90; Lit.[44]
6	*p*-$OMeC_6H_4$	H	**4f**	97	3	95–97	94–98; Lit.[44]
7	β-Naphthyl	H	**4g**	96	3	128–129	127–129; Lit.[44]
8	C_6H_5	CH_3	**4h**	93	5	118–119	117–120; Lit.[44]
9	*p*-BrC_6H_4	CH_3	**4i**	94	5	127–128	126–129; Lit.[44]
10	Bi-aryl	H	**4j**	96	5	117–118	116–118; Lit.[44]
11	*p*-MeC_6H_4	Br	**4k**	97	3	118–119	118–120; Lit.[44]



Reaction condition: succinamide (1.2 mmol), silver iodide (1.5 mmol), iodine (20 mol%), acetophenones (1 mmol) in polyethylene glycol-400–water (2:1) was stirred at r.t. and microwave-irradiated power of 350 W at 95–100 °C to α-iodo acetophenone and 1,2-diphenylamine/ortho phenyl di-amine (OPD) (1 mmol) and water–ethanol (1:1) was added to reaction mixture and irradiated power of 300 W at 90 °C.
[a]Isolated yield.



Scheme 2. Probable reaction mechanism for the synthesis of compound **4**.

water in ethanol, suddenly yield of product increases as the combination of solvent water–ethanol (1:1) in very short time of reaction within 3 min power 300 W at 90 °C (Table 1, entry no. 7). As we increased the quantity of water in combination water–ethanol 2:1, 4:1, there is decreased yield of product even also the time of reaction was increased (Table 1, entry nos. 8 and 9). Oppose of the quantity of solvent as water–ethanol (1:2 and or 1:4), the yield of product increases (Table 1, entry no. 10), but for the more green approach route, we select the combination of water–ethanol (1:1) for further entries of the reaction and thus, all examples were tested reasonably better to excellent yield in water–ethanol (1:1) (Table 2, Scheme 1). The electronic effect was observed that electron-donating group to aromatic nucleus gave better yield than electron-withdrawing group substituted to the same nucleus (Table 2, entry nos. 6 and 11). The unsubstituted group to aromatic carbonyl gave good to better yield (Table 2, entry no. 1). The better yield was obtained in combination of water and ethanol as green solvent with less time of reaction due to practically good paired emerged of water–ethanol using microwave energy. The hypothetical mechanistic path for the synthesis of substituted 2-phenyl quinoxaline and 7-bromo-3-(4-ethylphenyl) pyrido[2,3-*b*]pyrazine (**4**) is mentioned in Scheme 2.

Finally, the structures of compounds **4a** were confirmed by spectral and analytical data, for example, IR, ^{1}H NMR, ^{13}C NMR, and mass. The spectral data were consistent with the previous literature report.[8–10,44]

Conclusion

In the present work, we developed expeditious; an effective, environmentally benign, multi-component microwave-assisted synthesis of quinoxaline derivatives (substituted 2-phenyl quinoxaline and 7-bromo-3-(4-ethylphenyl) pyrido[2,3-*b*]pyrazine) in water–PEG and water–ethanol as green solvent from easily available starting materials and *in situ*-generated α-iodo acetophenone in combination with green solvent PEG-400 and water (2:1) under microwave irradiation method. Here, we avoided hazardous, volatile, cost-effective organic solvents, reagents, and lacrimatic α-chloro and α-bromocarbonyl compounds is the advantage of present research work.

Experimental section

Materials

The chemicals with high purity were purchased from Alfa Aesar. Melting points of synthesized products were recorded on OptiMELT digital melting point apparatus and were uncorrected. IR spectra were recorded on an FTIR (Bruker). ^{1}H NMR and ^{13}C NMR spectra were recorded on a 300 and 75 MHz Bruker spectrometer in solvent $CDCl_3$ as part per million (ppm) downfield from a tetramethylsilane as an internal standard. HRMS and mass spectra were recorded using QUART-MASS JEOL-Accu TOF JMS-T 100LC mass spectrometer. The microwave reactions were carried out in Micro SYNTH Lab station of Ethusi Milestone. Spectral data for the entire synthesized products (**4a–k**) as depicted in tabulated form and in a supporting data.

General procedure for the preparation of 2-phenyl quinoxaline (4a)

A mixture of succinamide (1.2 mmol) in water–PEG-400 (1:2), silver iodide (1.5 mmol), catalytic amount of iodine, and substituted acetophenone (1 mmol) stirred at room temperature till homogeneous to added iodine (20 mol%) and were microwave irradiated power at 350 W at 95–100 °C. Progress of the reaction was monitored on thin-layer chromatography (TLC) to obtain α-iodo acetophenone, then 1,2-diphenylamine (1 mmol) and water-ethanol (1:1) were added to the reaction mixture and irradiated for appropriate time (Table 1) and power 300 W at 90 °C. The progress of reaction was monitored by TLC. The reaction mixture was extracted with ethyl acetate/Et_2O, the etheral layer was concentrated by rotary evaporator and the crude product was purified by the preparative TLC on silica gel using a mixture of petroleum ether and ethyl acetate as an eluent to provide the corresponding pure product **4a**; yield = 97% and 10 other examples (**4b–k**) were prepared using the same protocols, yield = 93–97% (Table 2).

Spectral characterization data of compounds 4a and 4k

2-Phenyl quinoxaline (4a)

Solid: Yield 97%; mp 73–75 °C; 73–75 Lit.[44] mp (°C); ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.50–7.60 (s, 3H, Ar-H); 7.72–7.82 (m, 2H, Ar-H); 8.12–8.21 (m, 4H, Ar-H); 9.32 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 125.02, 127.03, 129.19, 129.62, 129.85, 130.53, 132.39, 135.65, 141.71, 142.24, 142.84, 150.68; IR (KBr) ν_{max}/cm^{-1}: 2910, 2840, 1915, 1710, 1620, 1521, 1401, 950, 822, 708; MS (ESI): *m/z* (%) 207 $[M+H]^+$; HRMS-EI: calcd for 206.0843; found, 206.0837.

7-Bromo-3-(4-methylphenyl)pyrido[2,3-b]pyrazine (4k)

Solid: Yield 97%; mp 118–119 °C; 118–120 Lit.[44] mp (°C); ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 2.46 (s, 3H, CH_3); 7.31–7.39 (d, J = 8.307 Hz, 4H, Ar-H); 7.4–7.6 (m, 1H, Ar-H); 8.18–8.25 (d, J = 8.30 Hz, 2H, Ar-H); 8.59–8.7 (d, J = 2.26 Hz, 1H, Ar-H); 9.14–9.19 (d, J = 2.26 Hz, 1H, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 21.326, 124.59, 125.33,126.39, 127.89, 28.17, 129.88, 129.92, 131.27, 132.83, 133.15, 124.59, 127.76, 128.88, 129.23, 129.29, 129.68, 133.37, 134.15, 141.69, 142.39, 148, 3.70; IR (KBr) ν_{max}/cm^{-1}: 2929, 2856, 1785, 1587, 1430, 1362, 1120, 987, 873, 823, 752; MS (ESI): *m/z* (%) 300 $[M+H]^+$; HRMS-EI: calcd for 299.007; found, 299.000.

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