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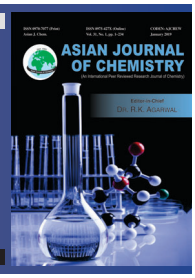

Eco-friendly Synthesis of 3-(Aryl)-2,6-diphenylpyrimidin-4(3*H*)-ones, Ethyl-1-(aryl)-1,6-dihydro-2-(aryl)-6-oxopyrimidine-4-carboxylates and 6-(4-Arylphenyl)-2-isopropylpyrimidin-4(3*H*)-one

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In present communication, we report the synthesis of pyrimidin-4(3*H*)-one derivatives by microwave irradiation in good yields and less reaction time. All titled compounds were characterized by IR, NMR and Mass spectral analyses.

Keywords: Isopropylpyrimidine, Benzimidines, Isobutyramidine hydrochloride, Microwave reaction.

INTRODUCTION

Heterocyclic compounds present in many of natural products like vitamins, antibiotics and harmones [1,2]. In biologically active compounds, pyrimidines are the most active class of compounds. Pyrimidines are the nitrogen containing heterocyclic aromatic compounds at 1,3-positions. Pyrimidine derivatives are present in our living organisms in the form of nucleic acid bases like adenine, uracil, thiamine and cytosine. Genetic information is carried by these nucleic acid bases to synthesize the various enzymes and proteins.

In the world, tuberculosis (TB) is one of the most effected mycobacterial disease [3]. In last 40 years, robust new anti-tuberculosis drugs with less side effects and new mechanisms of action have not been progressed. The annual infection rate in the developing countries is 20-50 times more than compare with the developed countries. It indicates the importance of synthesis of new antimicrobial drugs with variety of consequences like less duration of therapy with minimum expenditure and treatment with single dosage unit [4,5] than the giving multiple drugs. To overcome these drawbacks, our efforts dedicated on the development of new antibacterial compounds [6,7]. Pyrimidine derivatives represent an important place in the medicinal chemistry due to their wide range of biological activities [8,9]. Derivatives of pyrimidines are reported to have different pharmacological activities like anti-HIV [10], antitubercular [11], anti-tumor [12], antineoplastic [13], anti-inflammatory [14], diuretic [15], antimalaria [16], cardio-vascular [17], *etc*. In view of the importance of these compounds we undertook synthesis of some pyrimidine-4(3*H*)-one derivatives.

Choosing the environmentally benign conditions and solvents are as important as, synthesis of biologically active compounds. By choosing eco-friendly solvent like water or ethanol, we can minimise the waste producing in reaction or workup. By choosing the microwave reaction instead of traditional thermal heating, we can save the power by minimizing the reaction time and also the volume of solvent. Herein, we reported the synthesis of microwave-assisted novel pyrimidine derivatives by using eco-friendly solvent, ethanol.

EXPERIMENTAL

All the chemical and reagents used were purchased from Aldrich. All the solvents were of analytical grade. Thin-layer chromatography (TLC) was checked by Merck AL silica gel 60 F_{254} plates and visualized under UV light. IR spectra were recorded in KBr pellet with a Shimadzu spectrum gx FTIR instrument and all the diagnostic, intense peaks are reported. ^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ and DMSO-d_6 with a Varian Mercury plus 400 MHz instrument. All the chemical shifts were reported in δ (ppm) using TMS as an internal standard. The ^{1}H NMR chemical shifts and coupling

constants were determined assuming first-order behaviour. Mass spectra were recorded on a Shimadzu LCMS-QP 1000 mass spectrometer. Melting points were determined in open glass capillaries on a Stuart SMP30 apparatus and are uncorrected. All the reactions were performed under inert atmosphere.

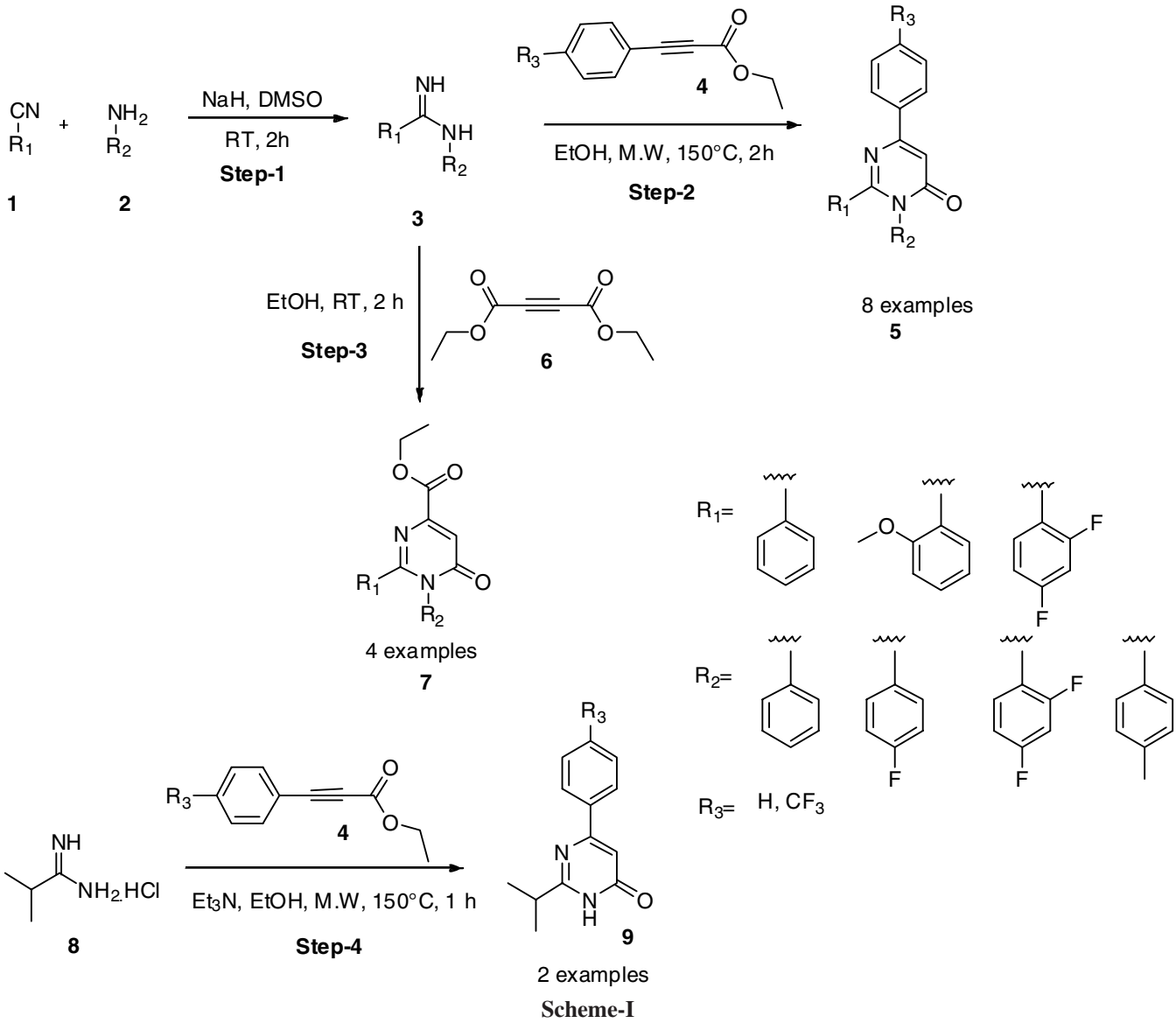
2,3,6-Aryl pyrimidin-4(3*H*)-ones (5a-h): *N*-Arynyl benzimidines (**3**) (1 eq.) and ethyl 3-phenylpropiolate (**4a**) or ethyl 3-(4-(trifluoromethyl)phenyl)propiolate (**4b**) (1.5 eq.) in EtOH (5 mL) was irradiated in microwave at 150 °C for 2 h. Reaction mixture was allowed to cooled to room temperature, resultant solid was filtered and dried. Crude material was purified by trituration using pentane (**Scheme-I**).

Ethyl-1,6-dihydro-6-oxo-1,2-diarylpyrimidine-4-carboxylate (7a-d): *N*-arynyl benzimidines (**3**) (1 eq.) and diethyl but-2-ynedioate (**6**) (1.5 eq.) in EtOH (5 mL) was stirred at room temperature for 1 h. Reaction mixture was diluted with water, extracted with EtOAc. Organic layer was washed with water, brine dried over anhhydrous Na_2SO_4 and concentrated. Crude material was purified by column chromatography using 40-50 % ethyl acetate in pet.-ether (**Scheme-I**).

6-Aryl-2-isopropylpyrimidin-4(3*H*)-ones (9a-b): Isobutyramidine hydrochloride (**8**) (1 eq.), Et_3N (1 eq.) and ethyl 3-phenylpropiolate (**4a**) or ethyl 3-(4-(trifluoromethyl) phenyl) propiolate **(4b**) (1.5 eq.) in EtOH (5 mL) was irradiated in microwave at 150 ºC for 1 h. Reaction mixture was allowed to cool to room temperature. Resultant solid was filtered and dried (**Scheme-I**).

2,3,6-Triphenylpyrimidin-4(3*H*)-one (5a): White solid, m.p. 312-315 °C, m.w. 324.38, m.f. $C_{22}H_{16}N_2O$. ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.35 (S, 1H), 6.85-6.87 (m, 2H), 7.05-7.10 (m, 5H), 7.15-7.17 (m, 4H), 7.18-7.2 (m, 2H), 7.25-7.28 (m, 2H). ^{13}C NMR (100 MHz; $CDCl_3$): 112.08, 127.66, 128.13, 128.61, 128.77, 128.84, 129.22, 129.45, 129.49, 133.2, 134.54, 138.62, 154.6, 161.08, 169.86. Mass (M+H): 325.22

3-(4-Fluorophenyl)-2,6-diphenylpyrimidin-4(3*H*)-one (5b): Off-white solid, m.p. 314-316 °C, m.w. 342.37, m.f. $C_{22}H_{15}N_2OF$. IR (KBr, ν_{max}, cm^{-1}): 756, 852, 1159, 1405, 1511, 1652, 2852, 2922, 3058. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.34 (S, 1H), 6.76 (t, *J* = 7.6 Hz, 2H), 6.83-6.86 (m, 2H), 7.06-7.07 (d, *J* = 8.4 Hz, 2H), 7.17-7.24 (m, 5H), 7.26-7.29 (m, 3H). ^{13}C NMR (100 MHz; $CDCl_3$): 112.08, 115.77, 116.0, 127.8,



Scheme-I

128.29, 128.85, 129.14, 129.36, 129.6, 131.17, 131.26, 133.0, 134.36, 134.66, 134.7, 154.63, 160.47, 161.18, 162.97, 169.71. Mass (M+H): 343.22.

3-(2,4-Difluorophenyl)-2-(2-methoxyphenyl)-6-phenylpyrimidin-4(3*H*)-one (5c): White solid, m.p. 221-224 °C, m.w. 390.38, m.f. $C_{23}H_{16}N_2O_2F_2$. IR (KBr, ν_{max}, cm^{-1}): 851, 954, 1106, 1256, 1383, 1498, 1655, 2842, 2932, 3072. ^{1}H NMR (500 MHz, $CDCl_3$): (Rotamers): 3.49 (s) and 3.68 (s) (together 3H), 6.33 (s) and 6.37 (s) (together 1H), 6.47-6.62 (m, 3H), 6.89 (t, *J* = 7 Hz, 1H), 6.94-7.03 (m, 1H), 7.15 (d. *J* = 7 Hz, 2H), 7.21-7.29 (m, 3.5 H), 7.26-7.36 (d, *J* = 5 Hz, 1H), 7.48 (d, *J* = 7.5 Hz, 0.5 H). Mass (M+H): 391.28.

2,6-Diphenyl-3-*p*-tolylpyrimidin-4(3*H*)-one (5d): Pale brown solid, m.p. 324-326 °C, m.w. 338.40, m.f. $C_{23}H_{18}N_2O$. ^{1}H NMR in (400 MHz, $CDCl_3$): δ 2.19 (s, 3H), 6.33 (s, 1H), 6.71-6.73 (d, *J* = 8.4 Hz, 2H), 6.84-6.86 (d, *J* = 8.4 Hz, 2H), 7.05-7.07 (d, *J* = 8.8 Hz, 2H), 7.18-7.24 (m, 6H), 7.28-7.30 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (100 MHz; $CDCl_3$): 20.94, 112.03, 127.6, 128.07, 128.81, 129.06, 129.12, 129.19, 129.33, 129.38, 133.29, 134.65, 135.96, 138.69, 154.78, 161.23, 169.92. Mass (M+H): 339.26.

6-(4-(Trifluoromethyl)phenyl-2,3-diphenylpyrimidin-4(3*H*)-one (5e): White solid, m.p. 334-336 °C, m.w. 392.37, m.f. $C_{23}H_{15}N_2OF_3$. IR (KBr, ν_{max}, cm^{-1}): 850, 1127, 1328, 1410, 1652, 2917, 3058, 3441. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.33 (s, 1H), 6.88 (d, *J* = 8 Hz, 2H), 7.10-7.18 (m, 5H), 7.22-7.25 (m, 3H), 7.28-7.30 (m, 2H), 7.47 (d, *J* = 8 Hz, 2H). ^{13}C NMR (100 MHz; $CDCl_3$): 112.28, 122.05, 124.76, 125.12, 125.15, 127.7, 129, 129.03, 129.18, 129.39, 129.63, 131.08, 131.41, 131.74, 134.23, 136.69, 138.28, 153.22, 161.32, 169.45. Mass (M+H): 393.32.

6-(4-(Trifluoromethyl)phenyl)-3-(4-fluorophenyl)-2-phenylpyrimidin-4(3*H*)-one (5f): Off-white solid, m.p. 344-346 °C, m.w. 410.36, m.f. $C_{23}H_{14}N_2OF_4$. IR (KBr, ν_{max}, cm^{-1}): 851, 1126, 1328, 1409, 1504, 1653, 2922, 3061, 3450. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.31 (s, 1H), 6.77-6.82 (m, 2H), 6.89-6.93 (m, 2H), 7.16-7.20 (m, 3H), 7.29-7.74 (m, 4H), 7.5-7.52 (d, *J* = 8 Hz, 2H). Mass (M+H): 411.25.

6-(4-(Trifluoromethyl)phenyl)-3-(2,4-difluorophenyl)-2-(2-methoxyphenyl)pyrimidin-4(3*H*)-one (5g): white solid, m.p. 239-241 °C, m.w. 458.38, m.f. $C_{24}H_{15}N_2O_2F_5$. ^{1}H NMR (500 MHz at 100 °C, DMSO-d_6): δ 3.6 (s, 3H), 6.2 (s, 1H), 6.76-6.81 (m, 2H), 6.90-6.95 (m, 2H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.39-7.4 (m, 2H), 7.5 (d, *J* = 8 Hz, 2H), 7.62 (d, *J* = 8 Hz, 2H). ^{13}C NMR (100 MHz; $CDCl_3$) (Rotamers): 54.49, 55.11, 103.71, 103.95, 104.2, 104.44, 109.94, 110.23, 110.9, 111.16, 111.89, 111.96, 120.53, 121.96, 122.36, 122.48, 123.32, 123.42, 124.67, 125.07, 125.48, 125.51, 128.77, 128.88, 129.27, 130.36, 130.8, 130.9, 131.45, 131.66, 131.71, 131.84, 131.93, 135.98, 152.69, 152.91, 154.81, 155.22, 155.9, 158.28, 158.41, 160.42, 160.56, 161.67, 169.47. Mass (M+H): 459.28

6-(4-(Trifluoromethyl)phenyl)-2-phenyl-3-*p*-tolylpyrimidin-4(3*H*)-one (5h): Off-white solid, m.p. 302-304 °C, m.w. 406.40, m.f. $C_{24}H_{17}N_2OF_3$. IR (KBr, ν_{max}, cm^{-1}): 850, 1129, 1321, 1411, 1539, 1655, 2920, 3055. ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.19 (s, 3H), 6.31 (s, 1H), 6.74 (d, *J* = 8.4 Hz, 2H), 6.88 (d, *J* = 8.1 Hz 2H), 7.17-7.25 (m, 5H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 8.1 Hz, 2H). ^{13}C NMR (100 MHz; $CDCl_3$): 20.99, 112.36, 122.1, 124.81, 125.14, 125.18, 127.7, 129.0, 129.2, 129.35, 129.60, 129.66, 131.06, 131.39, 134.38, 135.66, 136.83, 139.21, 153.31, 161.44, 169.48. Mass (M+H): 407.29.

Ethyl 1,6-dihydro-6-oxo-1,2-diphenylpyrimidine-4-carboxylate (7a): Yellow solid, m.p. 284-286 °C, m.w. 320.34, m.f. $C_{19}H_{16}N_2O_3$. ^{1}H NMR (500 M Hz, $CDCl_3$): δ 1.02 (t, *J* = 7.5 Hz, 3H), 4.03-4.07 (q, *J* = 7 Hz, 2H), 6.62 (s, 1H), 7.11-7.12 (m, 2H), 7.16-7.19 (m, 2H), 7.24-7.25 (m, 3H), 7.27-7.32 (m, 3H). Mass (M+H): 321.23.

Ethyl 1-(4-fluorophenyl)-1,6-dihydro-6-oxo-2-phenylpyrimidine-4-carboxylate (7b): Pale brown solid, m.p. 295-297 °C, m.w. 338.33, m.f. $C_{19}H_{15}N_2O_3F$. IR (KBr, ν_{max}, cm^{-1}): 852, 1014, 1216, 1509, 1647, 1740, 2923, 2996, 3050, 3327. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.11 (t, *J* = 7 Hz, 3H), 4.08-4.13 (q, *J* = 7 Hz, 7.5 Hz, 2H), 6.64 (s, 1H), 6.96-7.0 (m, 2H), 7.1-7.12 (m, 2H), 7.2-7.25 (m, 3H), 7.27-7.3 (m, 2H). Mass (M+H): 339.23.

Ethyl 1-(2,4-difluorophenyl)-1,6-dihydro-2-(2-methoxyphenyl)-6-oxopyrimidine-4-carboxylate (7c): Pale yellow solid, m.p. 211-213 °C, m.w. 386.35, m.f. $C_{20}H_{16}N_2O_4F_2$. ^{1}H NMR (500 MHz, $CDCl_3$): 1.18 (t, *J* = 7 Hz) and 1.24 (t, *J* = 7 Hz) (together 3 H), 3.52 (s) and 3.69 (s) (together 3 H), 4.15 (q, *J* = 7 Hz) and 4.22 (q, *J* = 7 Hz) (together 2H), 6.55-6.65 (m, 2H), 6.75-6.94 (m, 4H), 7.29-7.3 (m, 1.5 H), 7.39 (d, *J* = 8 Hz, 0.5 Hz). Mass (M+H): 387.24.

Ethyl 2-(2,4-difluorophenyl)-1-(4-fluorophenyl)-1,6-dihydro-6-oxopyrimidine-4-carboxylate (7d): Pale brown solid, 319-321 °C, m.w. 374.31, m.f. $C_{19}H_{13}N_2O_3F_3$. ^{1}H NMR (500 MHz, $CDCl_3$): 1.21 (t, *J* = 7 Hz, 3H), 4.16-4.20 (q, *J* = 7 Hz, 2H), 6.80-6.83 (m, 3H), 6.92-6.95 (t, *J* = 8.5 Hz, 2H), 7.12-7.13 (m, 1H), 7.29-7.32 (m, 2H). Mass (M+H): 375.26.

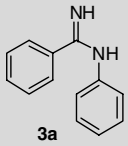
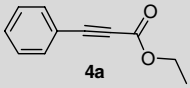
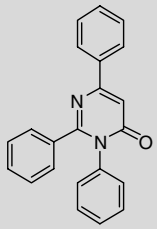
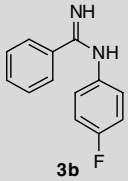
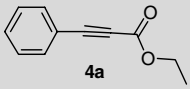
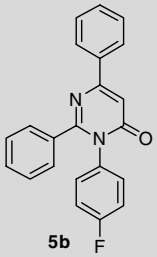
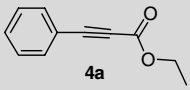
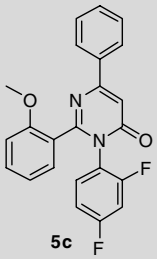
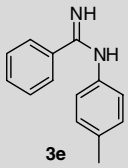
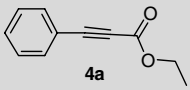
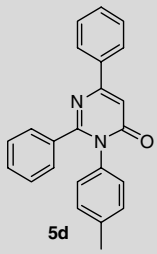
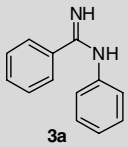
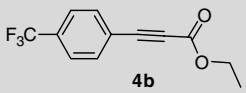
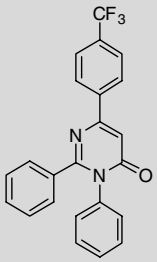
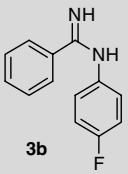
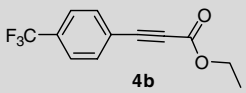
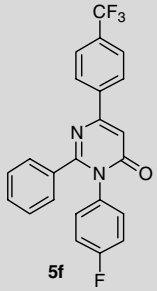
2-Isopropyl-6-phenylpyrimidin-4(3*H*)-one (9a): Off-white solid, m.p. 302-304 °C, m.w. 214.26, m.f. $C_{13}H_{14}N_2O$. IR (KBr, ν_{max}, cm^{-1}): 843, 949, 1174, 1231, 1388, 1670, 2974, 3440. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.4 (d, *J* = 7 Hz, 6H), 3.07 (m, 1H), 6.79 (s, 1H), 7.46-7.48 (m, 3H), 8.02-8.05 (m, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): 20.65, 34.60, 106.76, 127.22, 128.73, 130.68, 136.68, 163.28, 166.25, 166.31. Mass (M+H): 215.23.

6-(4-(Trifluoromethyl)phenyl)-2-isopropylpyrimidin-4(3*H*)-one (9b): Pale brown solid, m.p. 204-206 °C, m.w. 282.26, m.f. $C_{14}H_{13}N_2OF_3$. IR (KBr, ν_{max}, cm^{-1}): 835, 1115, 1326, 1667, 2974, 3144, 3438. ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.43 (d, *J* = 6.8 Hz, 6H), 3.0-3.07 (m, 1H), 6.81 (s, 1H), 7.73 (d, *J* = 8.4 Hz, 2H), 8.13 (d, *J* = 8.4 Hz, 2H), 12.36 (br, 1H). ^{13}C NMR (100 MHz; $CDCl_3$): 20.58, 34.6, 107.88, 125.63, 127.52, 132.11, 132.43, 139.99, 161.73, 166.02, 166.77. Mass (M+H): 283.11.

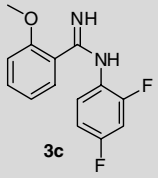
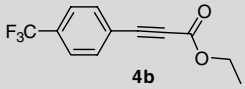
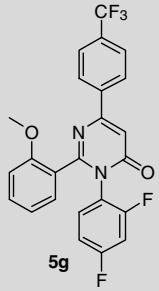
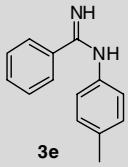
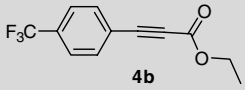
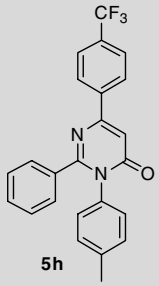
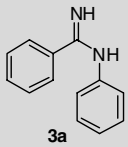
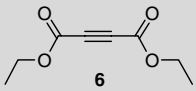
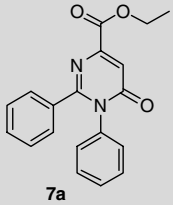
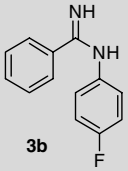
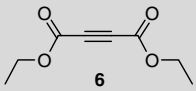
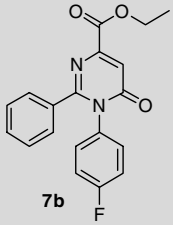
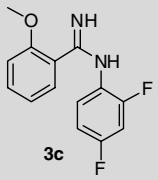
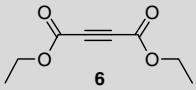
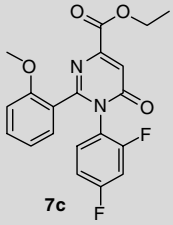
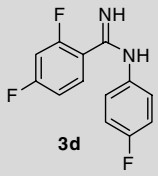
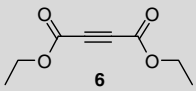
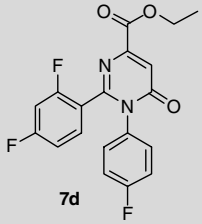
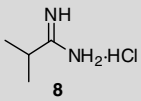
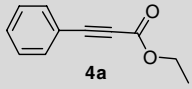
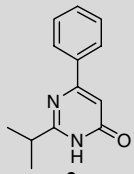
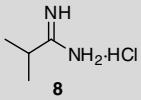
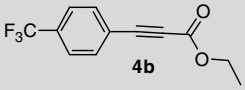
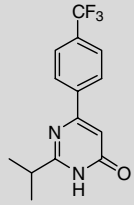
RESULTS AND DISCUSSION

Herein, we report a new synthesis method of pyrmidinone derivatives devoid of catalyst/reagent with less reaction time and quantitative yield. We achieved good yield for pyrimidinoe derivatives by condensation followed by cyclization of ethyl-3-phenylpropiolate or diethylacetylene dicarboxylate with N-phenylbenzimidine or isobutyramidine (Table-1). All the reactions are carried out in environmentally benign solvent EtOH. Although several reports available for the title molecules (**5a**-**h**) in reagent free condition, the reaction takes place in 4-12 days [18]. Here we drastically reduced the reaction time

TABLE-1

Entry-1	Entry-2	Product	Isolated yields (%)
 **3a**	 **4a**	 **5a**	81
 **3b**	 **4a**	 **5b**	79
 **3c**	 **4a**	 **5c**	84
 **3e**	 **4a**	 **5d**	85
 **3a**	 **4b**	 **5e**	75
 **3b**	 **4b**	 **5f**	72

from 4-12 days to 1 to 2 h by micro wave irradiation. The reaction of ethyl-3-phenylpropiolate with N-phenylbenzamidine or isobutyramidine, required product is precipitated after the reaction is completed, we can get the pure compound by simple filtration. So that we can avoid the environmentally harmful solvents for column chromatography. Reaction of

 **3c**	 **4b**	 **5g**	70
 **3e**	 **4b**	 **5h**	88
 **3a**	 **6**	 **7a**	55
 **3b**	 **6**	 **7b**	51
 **3c**	 **6**	 **7c**	45
 **3d**	 **6**	 **7d**	42
 **8**	 **4a**	 **9a**	72
 **8**	 **4b**	 **9b**	74

diethyl acetylene di carboxylate with phenylbenzamidine is happening at ambient temperature without using any reagent and clear when compared with ethyl 3-phenylpropiolate reaction. Compounds **4a** and **4b** were prepared from corresponding phenyl acetylenes following as reported in the literature [19].

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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