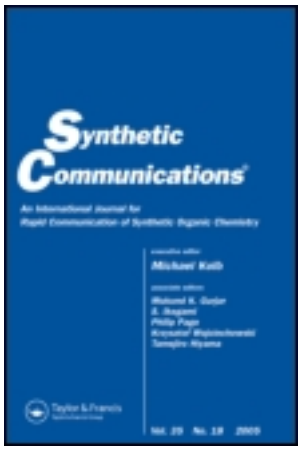

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SOLVENT-FREE SYNTHESES OF SALICYLALDIMINES ASSISTED BY MICROWAVE IRRADIATION

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SOLVENT-FREE SYNTHESES OF SALICYLALDIMINES ASSISTED BY MICROWAVE IRRADIATION

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ABSTRACT

A microwave-assisted condensation of salicylaldehyde and aryl amines without solvent were efficiently performed to form a series of salicylaldimines in high yields, which were confirmed by IR, ^{1}H NMR, ^{13}C NMR and elemental analyses. The microwave-assisted condensation provided a convenient environmental-friendship methodology for syntheses of Schiff-base in organic syntheses.

Chemists meet the controversy of synthetic procedures developing and economic to environment friendship, therefore solvent-free reactions have played strategic roles in methodologies of organic syntheses.[1] Among the most promising pathways, microwave-assisted technique has been popularly

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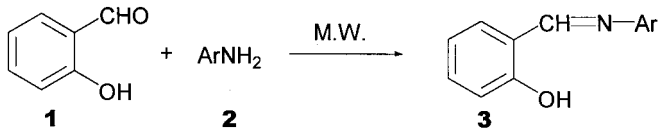
used since the earliest publication by Gedye and Majetich in 1986[2,3] Solvent-free organic synthesis mediated by microwave irradiation (M.W.) offers significant advantages, such as higher atom economy, environmental friendship, simple work-up procedure and good-to-high yield along with fairly mild conditions.[1,4] On the contrary, in classical organic syntheses, it is common to meet the problem of removing solvents especially in the case of aprotic dipolar solvent with high boiling point, or the isolation of reaction products through liquid–liquid extraction. The absence of solvent reduces the risk of hazardous explosions when the reaction takes place in a closed vessel in a microwave oven.[5]

The chemistry of the carbon–nitrogen double bond has played a vital role in the progresses of chemistry science.[6] By virtue of both the presence of a lone-pair of electrons on the nitrogen atom and the general electron-donating character of the double bond, compounds containing the azomethine group (Schiff-base compounds) have been used as fine chemicals and medical substrates, as well as ligands coordinating with metal ions in the formation of complexes. Recently multi-dentate complexes of iron and nickel showed high activities for ethylene oligomerization and polymerization.[7] In our efforts for ligands of polymerization catalysts, syntheses of Schiff-base through classical condensation of aldehydes (or ketones) and amines were pursued. However, the corresponding products were formed in different yields along with the various reaction times. Driven by industrial application of polymerization catalysts considering the Schiff-base ligands, the screening of simple and economic methods for preparation of Schiff-base is targeting in our current research project. Herein, on the base of the previous successes in microwave-assisted technique, the microwave-promoted solvent-free condensation reaction of salicylaldehyde and aryl amines displayed the convenient practicing way for forming a series of salicylaldimines in good yields (Scheme 1).

The synthesis of Schiff-base is a classical reaction. It is often carried out with acid-catalyzed and generally by refluxing the mixture of aldehyde (or ketone) and amine.[8] Recently, stoichiometric solid–solid reaction was successfully employed for Schiff-base formation.[9] However, the reaction



Scheme 1.

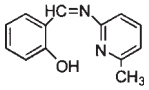
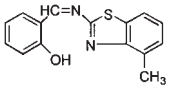
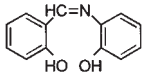
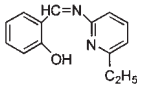
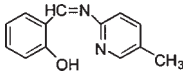
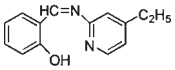
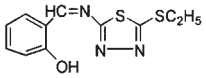
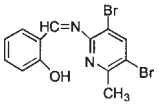
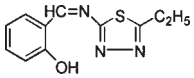
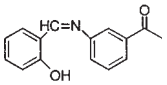
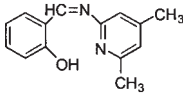
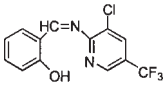
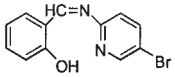
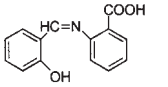
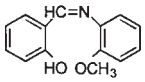
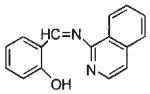
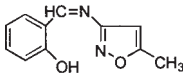
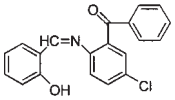
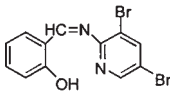
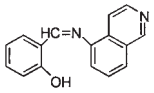
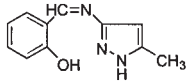
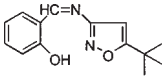
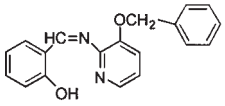
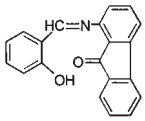
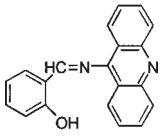
time was relative longer. Improvingly, condensation imines formation was carried out in water suspension.[10] To shorten the reaction time, microwave-mediated synthesis of heterocyclic amines with aldehydes were efficiently performed.[11] In present case, with the assistance of microwave irradiation, it was found that general Schiff-base formation of salicylaldimines proceeded fast and efficiently through the condensation reaction of salicylaldehyde and various aryl amines (Table 1). It is noteworthy that since the reaction vessel was an opened Erlenmeyer flask, the polar molecules of condensation water were immediately vaporized, which was a driven force for the reaction as well as obviated the dangerous explosion. The work-up procedure was simplified as re-crystallization of the products in an appropriate solvent, such as ethanol, for purification and separation. The products **3a–j** were literature compounds which were formed through thermal condensation of aldehyde with amines (see the corresponding numbers in the reference),[12] while the products **3k–y** were first reported with the best of our knowledge. All compounds were confirmed by their IR, ^{1}H NMR, ^{13}C NMR spectra as well as elemental analyses. Further investigation of condensation of ketones and various amines are currently in progress.

General Consideration: Melting points obtained with an electrical apparatus were uncorrected. IR spectra were recorded on a PERKIN ELMER System 200 FT-IR spectrometer; the chemical shifts of NMR spectra were measured with a Bruker BMX-300 MHz instrument and were expressed in ppm using TMS as internal standard; elemental analyses were performed by using HPMOD 1106 microanalyzer.

General Procedure for the Syntheses of Salicyaldimines 3a–y: The microwave-assisted condensations of salicylaldehyde and aryl amines were carried out in a domestic oven, Midea PJ21B-A 800 W (21 L). Salicylaldehyde **1** (3 mmol) and an equivalent aryl amines **2** were mixed together at ambient temperature in an opened Erlenmeyer flask (25 mL). The mixture was subjected to microwave for an optimized time on the "M-High" setting (616 W), except "High" setting (800 W) for compounds **3n** and **3t** (see Table 1). The crude products were re-crystallized with ethanol, while the products **3j**, **3m** and **3x** were re-crystallized with EtOH–CH_2Cl_2 (2 : 1), EtOH–CH_2Cl_2 (1 : 3) and diethyl ether, respectively.

2-[[(5-Methyl-1H-pyrazole-3-yl)imino]methyl]-phenol (3k): M.p. 145–146.5°C; IR (KBr pellet) 3430.6, 3204.7, 1614.4, 1576.6, 1499.3, 1470.2, 1433.6, 1277.9, 1026.9, 754.7 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 2.33 (s, 3H), 6.12 (s, 1H), 6.91–7.39 (m, 4H), 8.82 (s, 1H), 11.90 (s, 1H), 13.00 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$) δ 11.3, 95.2, 117.0, 118.6, 118.9, 132.2, 133.0, 141.7, 156.4, 160.8, 162.5 ppm; Anal calcd. for $C_{11}H_{11}N_3O$: C, 65.66%; H, 5.51%; N, 20.88%; Found: C, 65.65%; H, 5.49%; N, 20.86%.

Table 1. Condensation Products of Salicylaldehyde and Aryl Amines

	Compounds	Yield (%)	Time		Compounds	Yield (%)	Time
3a		95	2 min	**3n**		82	6 min
3b		94	4 min	**3o**		87	2 min
3c		94	30 sec	**3p**		89	2 min
3d		84	4 min	**3q**		88	4 min
3e		98	3 min	**3r**		97	4 min
3f		98	4 min	**3s**		76	4 min
3g		92	4 min	**3t**		77	4 min
3h		98	3 min	**3u**		90	4 min
3i		87	30 sec	**3v**		89	3 min
3j		92	4 min	**3w**		65	2 min
3k		96	30 sec	**3x**		96	4 min
3l		88	4 min	**3y**		78	4 min
3m		68	4 min				

2-[[(3-Benzyloxypyridine-2-yl)imino]methyl]-phenol (3l): M.p. 106–107°C; IR (KBr pellet) 3436.8, 1605.3, 1580.3, 1555.6, 1453.9, 1434.8, 1382.4, 1287.8, 1212.8, 1119.5, 1021.1, 762.3 cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.21 (s, 2H), 6.79–7.48 (m, 11H), 8.10 (d, 1H), 9.44 (s, 1H), 14.17 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$) δ 70.3, 117.4, 118.3, 118.7, 119.1, 121.1, 126.7, 127.9, 128.5, 133.0, 133.6, 135.9, 139.9, 147.4, 148.5, 162.4, 162.9 ppm; Anal. calcd. for $C_{19}H_{16}N_2O_2$: C, 74.98%; H, 5.30%; N, 9.20%; Found: C, 74.97%; H, 5.33%; N, 9.17%.

2-[[(Acridine-9-yl)imino]methyl]-phenol (3m): M.p. 231–233°C; IR (KBr pellet) 3442.7, 2925.8, 1622.3, 1554.0, 1516.6, 1462.1, 1277.5, 1065.9, 753.5 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.05–8.26 (m, 12H), 8.64 (s, 1H), 12.43 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$) δ 116.4, 116.7, 117.2, 118.4, 122.2, 124.5, 128.3, 129.2, 131.9, 133.4, 148.0, 150.4, 160.0, 167.9 ppm; Anal. calcd. for $C_{20}H_{14}N_2O$: C, 80.52%; H, 4.73%; N, 9.39%; Found: C, 80.46%; H, 4.69%; N, 9.42%.

2-[[(4-Methylbenzothiazole-2-yl)imino]methyl]-phenol (3n): M.p. 99.5–102°C; IR (KBr pellet) 3440.2, 2971.9, 1617.5, 1597.9, 1566.5, 1473.1, 1280.7, 1148.9, 752.2 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.75 (s, 3H), 7.04–7.70 (m, 7H), 9.27 (s, 1H), 12.29 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$) δ 18.4, 117.6, 118.3, 119.0, 119.6, 125.1, 127.1, 133.2, 133.9, 134.4, 135.1, 150.8, 161.8, 167.1, 167.7 ppm; Anal. calcd. for $C_{15}H_{12}N_2OS$: C, 67.14%; H, 4.51%; N, 10.44%; Found: C, 67.16%; H, 4.50%; N, 10.39%.

2-[[(6-Ethylpyridine-2-yl)imino]methyl]-phenol (3o): M.p. 38.5–40°C; IR (KBr pellet) 3436.9, 2971.6, 1612.8, 1554.8, 1496.5, 1458.8, 1281.5, 1188.9, 815.5, 754.9 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.35 (t, 3H), 2.85 (q, 2H), 6.86–7.71 (m, 7H), 9.46 (s, 1H), 13.62 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$) δ 13.6, 31.1, 117.0, 117.3, 118.9, 120.8, 133.2, 133.4, 138.4, 156.6, 161.7, 163.0, 164.1, 172.2 ppm; Anal. calcd. for $C_{14}H_{14}N_2O$: C, 74.31%; H, 6.24%; N, 12.38%; Found: C, 74.36%; H, 6.22%; N, 12.34%.

2-[[(4-Ethylpyridine-2-yl)imino]methyl]-phenol (3p): M.p. 46–48°C; IR (KBr pellet) 3433.1, 2969.0, 1601.9, 1575.9, 1545.9, 1455.6, 1411.0, 1280.1, 1147.2, 758.1 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.29 (t, 3H), 2.68 (q, 2H), 6.93–7.51 (m, 6H), 8.38 (d, 1H), 9.43 (s, 1H), 13.52 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$) δ 14.1, 28.0, 117.4, 119.0, 119.8, 121.9, 122.3, 133.2, 133.5, 148.5, 155.7, 157.5, 161.7, 164.3 ppm; Anal. calcd. for $C_{14}H_{14}N_2O$: C, 74.31%; H, 6.24%; N, 12.38%; Found: C, 74.35%; H, 6.21%; N, 12.39%.

2-[[(3,5-Dibromo-6-methylpyridine-2-yl)imino]methyl]-phenol (3q): M.p. 149–150.5°C; IR (KBr pellet) 3442.8, 1607.1, 1574.8, 1533.7, 1448.9, 1419.7, 1280.3, 1184.7, 1054.2, 757.7 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.63 (s, 3H), 6.93–7.56 (m, 4H), 8.09 (s, 1H), 9.43 (s, 1H), 13.43 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$) δ 24.3, 114.8, 117.5, 118.7, 118.9, 119.2, 133.7, 134.4, 144.1, 152.5, 155.5, 162.1, 165.2 ppm; Anal. calcd. for

$C_{13}H_{10}Br_2N_2O$: C, 42.20%; H, 2.72%; N, 7.57%; Found: C, 42.17%; H, 2.72%; N, 7.55%.

2-[](3-Acetylbenzene-1-yl)imino]methyl]-phenol (3r): M.p. 90–92°C; IR (KBr pellet) 3436.4, 1676.4, 1618.0, 1572.4, 1498.9, 1436.0, 1271.0, 1222.0, 756.2 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.65 (s, 3H), 6.90–7.85 (m, 8H), 8.66 (s, 1H), 13.01 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$) δ 26.6, 117.1, 118.8, 119.1, 120.2, 126.0, 126.5, 129.5, 132.4, 133.4, 138.1, 148.7, 160.9, 163.6, 197.5 ppm; Anal. calcd. for $C_{15}H_{13}NO_2$: C, 75.30%; H, 5.48%; N, 5.85%; Found: C, 75.31%; H, 5.42%; N, 5.83%.

2-{[(3-Chloro-5-(trifluoromethyl)pyridine-2-yl)imino]methyl}-phenol (3s): M.p. 146.5–148°C; IR (KBr pellet) 3447.9, 1614.7, 1599.1, 1559.8, 1451.9, 1323.1, 1288.7, 1224.5, 1166.8, 1125.8, 1091.2, 915.4, 762.9 cm^{-1}; ^1H NMR ($CDCl_3$) δ 6.99–7.58 (m, 4H), 8.05 (s, 1H), 8.64 (s, 1H), 9.51 (s, 1H), 13.31 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$) δ 117.6, 118.6, 119.5, 120.8, 124.4, 127.5, 134.1, 135.3, 135.9, 143.7, 156.5, 162.5, 167.3 ppm; Anal calcd. for $C_{13}H_8ClF_3N_2O$: C, 51.93%; H, 2.68%; N, 9.32%; Found: C, 51.91%; H, 2.65%; N, 9.28%.

2-{[(2-Carboxylbenzene-1-yl)imino]methyl}-phenol (3t): M.p. 199.5–201.5°C; IR (KBr pellet) 3439.7, 3070.6, 1683.3, 1620.0, 1488.6, 1457.5, 1366.8, 1244.8, 757.0 cm^{-1}; ^1H NMR (d_6-DMSO) δ 6.41–7.68 (m, 8H), 8.84 (s, 1H), 10.25 (s, 1H), 10.70 (s, 1H) ppm; ^{13}C NMR (d_6-DMSO) δ 116.5, 119.3, 119.7, 122.5, 129.4, 129.5, 130.7, 131.4, 134.0, 136.7, 151.7, 160.8, 169.8, 191.9 ppm; Anal. calcd. for $C_{14}H_{11}NO_3$: C, 69.70%; H, 4.60%; N, 5.81%; Found: C, 69.62%; H, 4.67%; N, 5.80%.

2-{[(Isoquinoline-1-yl)imino]methyl}-phenol (3u): M.p. 117–118.5°C; IR (KBr pellet) 3452.1, 1614.0, 1579.2, 1551.4, 1494.7, 1392.4, 1330.3, 1280.0, 769.0 cm^{-1}; ^1H NMR ($CDCl_3$) δ 6.87–8.84 (m, 10H), 9.51 (s, 1H), 13.64 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$) δ 117.1, 119.0, 119.2, 120.3, 123.7, 124.8, 126.3, 127.6, 130.5, 133.6, 134.1, 137.6, 141.3, 156.0, 161.9, 165.8 ppm; Anal. calcd. for $C_{16}H_{12}N_2O$: C, 77.40%; H, 4.87%; N, 11.27%; Found: C, 77.35%; H, 4.84%; N, 11.25%.

2-{[(5-Chlorobenzophenone-2-yl)imino]methyl}-phenol (3v): M.p. 141–143°C; IR (KBr pellet) 3439.5, 1666.4, 1614.1, 1579.4, 1472.5, 1453.2, 1284.0, 1184.4, 756.4 cm^{-1}; ^1H NMR ($CDCl_3$) δ 6.81–7.82 (m, 12H), 8.49 (s, 1H), 11.61 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$) δ 117.1, 118.6, 119.0, 120.0, 128.2, 128.6, 128.7, 129.8, 131.3, 132.2, 132.5, 133.7, 135.6, 136.4, 145.5, 160.6, 164.0, 195.3 ppm; Anal. calcd. for $C_{20}H_{14}ClNO_2$: C, 71.54%; H, 4.20%; N, 4.17%; Found: C, 71.56%; H, 4.15%; N, 4.10%.

2-{[(Isoquinoline-5-yl)imino]methyl}-phenol (3w): M.p. 88–89°C; IR (KBr pellet) 3436.9, 1616.0, 1577.3, 1485.7, 1459.6, 1279.6, 1211.8, 1154.7, 756.3 cm^{-1}; ^1H NMR ($CDCl_3$) δ 6.98–8.01 (m, 8H), 8.59 (d, 1H), 8.68 (s, 1H), 9.27 (s, 1H), 13.10 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$) δ 116.0, 117.4,

117.9, 119.2, 119.4, 126.2, 127.4, 129.0, 131.0, 132.6, 133.9, 143.7, 145.1, 152.3, 161.2, 164.5 ppm; Anal. calcd. for $C_{16}H_{12}N_2O$: C, 77.40%; H, 4.94%; N, 11.28%; Found: C, 77.37%; H, 4.94%; N, 11.35%.

2-{[(5-*t*-Butylisoxazole-3-yl)imino]methyl}-phenol (3x): M.p. 69.5–71°C; IR (KBr pellet) 3445.4, 2972.1, 1620.6, 1598.8, 1577.1, 1457.3, 1419.5, 1278.2, 757.9 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.38 (s, 9H), 6.08 (s, 1H), 6.94–7.42 (m, 4H), 8.88 (s, 1H), 12.45 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$) δ 28.6, 33.0, 93.2, 117.4, 118.3, 119.3, 133.2, 134.5, 161.5, 166.9, 167.4, 182.9 ppm; Anal. calcd. for $C_{14}H_{16}N_2O_2$: C, 68.83%; H, 6.60%; N, 11.47%; Found: C, 68.80%; H, 6.61%; N, 11.47%.

2-{[(9-Fluorenone-1-yl)imino]methyl}-phenol (3y): M.p. 89.5–92°C, IR (KBr pellet) 3451.9, 1707.1, 1680.1, 1614.6, 1592.1, 1454.2, 1284.1, 1191.2, 756.1 cm^{-1}; ^1H NMR ($CDCl_3$) δ 6.80–7.62 (m, 11H), 8.78 (s, 1H), 13.24 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$) δ 109.5, 117.1, 117.6, 118.1, 118.8, 119.6, 120.2, 123.0, 124.1, 127.4, 129.2, 132.5, 133.3, 133.7, 134.2, 135.3, 136.0, 145.2, 161.5, 164.2 ppm; Anal. calcd. for $C_{20}H_{13}NO_2$: C, 80.25%; H, 4.38%; N, 4.68%; Found: C, 80.17%; H, 4.35%; N, 4.65%.

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