Research Article

Microwave Assisted Solvent-Free Synthesis of Some Imine Derivatives

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Some imine derivatives (**1a–7d**) were synthesized using a rapid and an environmentally friendly method with reaction of aromatic aldehydes (**a–d**) and aromatic amines (**1–7**) (in 1:1 molar ratio) in the presence of β -ethoxyethanol as a wetting reagent (2 drops) under solvent-free conditions using microwave heating.

1. Introduction

Compounds containing the $-C=N-$ (azomethine group) structure are known as Schiff bases, usually synthesized from the condensation of primary amines and active carbonyl groups [1]. The reaction is acid-catalyzed and is generally carried out by refluxing the carbonyl compound and amine, with an azeotroping agent if necessary, and separating the water as formed [2]. Schiff bases are well known for their biological applications as antibacterial, antifungal, anticancer, and antiviral agents; furthermore, they have been used as intermediates in medical substrates and as ligands in complex formation with some metal ions [1, 3]. The synthesis of imines was firstly reported by Hugo Schiff in 1864 and they have been known since then [4]. The imine compounds have been prepared using molecular sieves [5, 6], infrared irradiation [7], $Mg(ClO_4)_2$ [8], P_2O_5/SiO_2 [9], $ZnCl_2$ [10], CaO under microwave power [11], ethyl lactate as a tunable solvent [12], K10 clay [13], $TiCl_4$ [14], alumina [15], $CeCl_3 \cdot 7H_2O$ [16], ultrasound irradiation [17], polymer-supported [18], nanotube TiO_2 (in sunlight) [19], and $Ti(OEt)_4$ [20, 21].

The present work reveals the comparative aspects of condensation of some aromatic amines with aldehyde derivatives using microwave and conventional methods. The amine and aldehyde compounds as starting materials, β -ethoxyethanol (β -EE) as wetting reagent and microwave power as an

effective source of heating are used in this study. The corresponding imine compounds were prepared in high yields and short reaction times using this effective and environment friendly method.

2. Results and Discussion

In this work, we synthesized quickly and efficiently a series imine derivatives (**1a–7d**) by condensation of some aromatic amines (**1–7**) and aldehyde derivatives such as salicylaldehyde (**a**), *p*-chlorobenzaldehyde (**b**), *p*-methoxybenzaldehyde (**c**) and cinnamaldehyde (**d**) under microwave-assisted solvent-free conditions using β -EE as wetting reagent. β -EE that is a polar molecule quickly absorbs microwaves and therefore heats up and heats around effectively. As a result, β -EE, which increases the polarity of the reaction medium, has an active role in the heating of the reaction medium by microwaves.

The general reaction was summarized in Scheme 1. In addition, we tested the effect of different microwave power such as 180, 360, 600, 900 W and detected the microwave power of 180 W and 360 W are more appropriate choices for the reaction. Hence, the optimum microwave reaction conditions were determined using 180 and 360 W microwave power and neat and wetting with β -EE for compound **1a**. The highest reaction yield (94%) and shorter reaction time

SCHEME 1

TABLE 1: The optimization of microwave conditions for compound **1a**.

Microwave method			
Watt	Time (min)	Reaction conditions	Yield (%)
180	5	Wetting (with β -EE)	83
360	5	Wetting (with β -EE)	93
360	1.5	Wetting (with β-EE)	94
360	5	Neat	88
360	1.5	Neat	81

TABLE 2: The optimization of classical conditions for compound **1a**.

Classical method		
Time (min)	Yield (%)	Reaction conditions
30	64	Reflux
60	73	Reflux
120	76	Reflux
240	75	Reflux
1300*	74	Reflux

Procedure: 1 mmol aldehyde (10 mL of ethanol) and 1 mmol amine (10 mL of ethanol) are mixed a balloon and then boiled under reflux on a hot plate.

* 300 min (heated) + 1000 min (unheated).

(1.5 min.) were obtained at 360 W microwave power with β -EE. In the absence of wetting reagent, the reaction yields are 81% for 1.5 minutes and 88% for 5 minutes. It is understood that the reaction yield was increased by wetting reagent that increases the polarity of the reaction medium. The optimization of microwave-assisted conditions was given in Table 1.

In addition, we have synthesized some imine derivatives (**1a**, **4a**, **1b**, **4b**, **1c**, **4c**, **1d**, **4d**) under classical conditions using a solvent (azeotropic) and traditional heating source (hot plate) for comparison with microwave conditions. Firstly, synthesis of compound **1a** under the classical reaction conditions was tested at various periods of time (30, 60, 120, 240, 1300 min) to determine the optimum reaction time, which was determined as 120 minutes. Information on the optimization of the reaction time of compound **1a** for classic conditions was presented in Table 2.

The compounds **1a**, **4a**, **1b**, **4b**, **1c**, **4c**, **1d**, and **4d** were obtained at 76%, 73%, 63%, 79%, 67%, 73%, 67%, and 75% yields, respectively, in 120 minutes under the classical reaction conditions, whereas in microwave conditions,

FIGURE 1: Comparison of yields under the microwave and classical conditions.

the corresponding reaction yields were 94%, 97%, 95%, 98%, 91%, 97%, 95%, and 96%, respectively, in 1.0–1.5 minutes. All the results such as reactions time, yields, and melting points of the compounds were presented in Table 3. In addition, the yields of microwave and conventional reaction conditions for some imines were expressed graphically in Figure 1.

3. Conclusion

In conclusion, we have developed a simple microwave assisted solvent-free method for the synthesis of imines using a wetting reagent (β -ethoxyethanol). The method works well for the reaction of this type amines and aldehydes. Because, we have synthesized imine compounds for good yields and fast reaction times in our method. Our method has some advances such as higher reaction yields, shorter reaction times, and green reaction conditions than classical requirements used of a solvent and conventional heat source (like hot plate) and other some microwave methods used of catalyst and solid supports. In addition, some imines were synthesized for the first time by us with this study.

4. Experimental

All of the chemicals were obtained from commercial sources or prepared according to standard methods. Melting points

TABLE 3: Synthesis of imine derivatives under microwave irradiation.

N	Ar	R ₁	R ₂	R ₃	Microwave ^a		Classical ^b		m.p °C	Lit. m.p °C
					Yield %	t/min	Yield %	t/min		
1a		OH	H	H	94	1.5	76	120	188–190	190 [22]
2a		OH	H	Cl	93	1.5	—	—	159–162	156–156.5 [23]
3a		OH	CH ₃	H	93	1.5	—	—	199–202	—
4a		H	H	H	97	1.0	73	120	50–53	50–51 [24]
5a		H	Cl	H	94	1.5	—	—	103–105	104 [12]
6a		H	CH ₃ O	H	96	1.5	—	—	83–85	82–83.5 [25]
7a		H	CH ₃	H	97	1.5	—	—	93–94	90–91.5 [25]
1b		OH	H	H	95	1.5	63	120	116–118	117 [26]
2b		OH	H	Cl	92	2.0	—	—	123–125	—
3b		OH	CH ₃	H	91	2.0	—	—	134–137	—
4b		H	H	H	98	1.0	79	120	63–66	64–65 [24]
5b		H	Cl	H	94	1.5	—	—	110–111	110–111 [24]
6b		H	CH ₃ O	H	91	3.0	—	—	124–126	124–125 [27]
7b		H	CH ₃	H	93	1.5	—	—	126–128	125 [27]
1c		OH	H	H	91	1.5	67	120	90–91	91–93 [22]
2c		OH	H	Cl	95	1.0	—	—	86–88	—
3c		OH	CH ₃	H	93	1.5	—	—	86–87	—
4c		H	H	H	97	1.0	73	120	58–60	63–63.5 [24]
5c		H	Cl	H	97	1.5	—	—	90–92	92 [16]
6c		H	CH ₃ O	H	95	1.5	—	—	147–149	145–146 [24]
7c		H	CH ₃	H	91	1.5	—	—	92–93	91–92 [24]
1d		OH	H	H	95	1.5	67	120	89–91	92 [22]
2d		OH	H	Cl	91	1.5	—	—	91–93	—
3d		OH	CH ₃	H	94	1.5	—	—	97–98	—
4d		H	H	H	96	1.0	75	120	108–111	108–109 [12]
5d		H	Cl	H	96	1.5	—	—	105–107	105 [12]
6d		H	CH ₃ O	H	97	1.5	—	—	120–121	119–120 [12]
7d		H	CH ₃	H	90	1.5	—	—	75–77	76–77 [28, 29]

^a Under solvent-free reaction using microwave.^b Procedure: 1 mmol aldehyde (10 mL of ethanol) and 1 mmol amine (10 mL of ethanol) are mixed a balloon and then boiled under reflux on a hot plate.

were determined with an Electrothermal 9100 apparatus. The infrared absorption spectrum of the compounds has been recorded in the region at 4000 and 400 cm⁻¹ range using a Bruker Vertex 80/80 v spectrophotometer using KBr pressed pellet technique at room temperature. The ¹H NMR and ¹³C NMR spectra were recorded using a Bruker AC 200 NMR 200 MHz spectrometer in CDCl₃-d₁ and DMSO-d₆ using TMS as the internal standard. Elemental analyses were conducted at the METU Central Laboratory using a LECO, CHNS-932. All syntheses were carried out in A Bosh HMT 812 C modified microwave oven.

General Procedure for Preparation of 1a–7d. Aldehyde (1 mmol), amine (1 mmol) and two drops of β-ethoxyethanol as a wetting reagent were mixed in a beaker. Then the beaker was placed in the microwave oven and was exposed to microwave irradiation (360 W). The product obtained by reaction was washed with cold ethanol. Then, it was

recrystallized in ethanol. The physical and spectra data of the compounds **3a**, **2b**, **3b**, **2c**, **3c**, **2d**, and **3d** are as follows.

N-(Salicylidene)-2-hydroxy-4-methylaniline (**3a**). M.p. 199–202°C. IR: 3033, 2965, 2916, 2858, 1612 (C=N), 1592, 1527, 1486, 1422, 1278, 1221, 1141, 1113, 1012, 869, 788, 754, 720, 622, 587, 556 cm⁻¹. ¹H NMR (DMSO-d₆): δ 8.94 (s, 1H, N=CH), 13.90 (s, 1H, OH), 9.65 (s, 1H, OH). ¹³C NMR (DMSO-d₆): δ 160.54, 160.36, 150.82, 137.56, 132.36, 132.25, 131.92, 120.17, 119.46, 119.14, 118.46, 116.93, 116.48. Elemental Anal. Calcd. for C₁₄H₁₃NO₂: C, 73.99; H, 5.77; N, 6.16. Found: C, 72.81; H, 5.64; N, 6.16%.

N-(*p*-Chlorobenzylidene)-2-hydroxy-5-chloroaniline (**2b**). M.p. 123–125°C. IR: 3367, 3086, 3071, 3059, 2910, 2894, 1626 (C=N), 1588, 1569, 1478, 1424, 1370, 1274, 1234, 1194, 1154, 1087, 1011, 911, 857, 815, 799 (C–Cl), 697, 658, 608, 548, 533 cm⁻¹. ¹H NMR (CDCl₃): δ 8.66 (s, 1H, N=CH), 7.91

(d, $J = 8.4$ Hz, 2H, Ar), 7.54 (d, $J = 8.4$ Hz, 2H, Ar), 7.34 (s, 1H, Ar), 7.24 (d, $J = 8.6$ Hz, 1H, Ar), 7.02 (d, $J = 8.6$ Hz, 1H, Ar). ^{13}C NMR (CDCl_3): δ 156.75 (N=C), 150.94, 138.21, 135.86, 133.87, 130.06, 129.28, 128.77, 125.07, 116.19, 116.16. Elemental Anal. Calcd. for $\text{C}_{13}\text{H}_9\text{Cl}_2\text{NO}$: C, 58.67; H, 3.41; N, 5.26. Found: C, 57.74; H, 3.42; N, 5.26%.

N-(*p*-Chlorobenzylidene)-2-hydroxy-4-methylaniline (**3b**). M.p. 134–137°C. IR: 3367, 3063, 3049, 3028, 2907, 2857, 1629 (C=N), 1576, 1569, 1497, 1370, 1344, 1295, 1242, 1169, 1154, 1080, 1011, 947, 874, 857, 828, 808 (C–Cl), 793, 733, 687, 610, 591, 579 cm^{-1} . ^1H NMR (CDCl_3): δ 8.72 (s, 1H, N=CH), 7.92 (d, $J = 8.3$ Hz, 2H, Ar), 7.53 (d, $J = 8.4$ Hz, 2H, Ar), 7.29 (d, $J = 8.1$ Hz, 1H, Ar), 6.92 (s, 1H, Ar), 6.80 (d, $J = 8.0$ Hz, 1H, Ar), 2.42 (s, 3H, CH_3). ^{13}C NMR (CDCl_3): δ 154.09 (N=C), 152.34, 139.81, 137.40, 134.54, 132.60, 129.72, 129.16, 120.93, 115.67, 115.36, 21.48. Elemental Anal. Calcd. for $\text{C}_{14}\text{H}_{12}\text{ClNO}$: C, 68.44; H, 4.92; N, 5.70. Found: C, 67.64; H, 4.89; N, 5.69%.

N-(*p*-Methoxybenzylidene)-2-hydroxy-5-chloroaniline (**2c**). M.p. 86–88°C. IR: 3323, 3017, 2970, 2954, 2927, 2840, 1626 (C=N), 1599, 1569, 1511, 1453, 1312, 1250, 1165, 1027, 804, 650, 594 cm^{-1} . ^1H NMR (CDCl_3): δ 9.88 (s, 1H, OH), 8.55 (s, 1H, N=CH), 7.85 (d, $J = 8.5$ Hz, 2H, Ar), 7.23 (s, 1H, Ar), 7.11 (d, $J = 8.6$ Hz, 1H, Ar), 6.99 (d, $J = 8.5$ Hz, 2H, Ar), 6.90 (d, $J = 8.6$ Hz, 1H, Ar), 3.88 (s, 3H, OCH_3). ^{13}C NMR (CDCl_3): δ 162.92, 157.76 (N=C), 150.70, 136.69, 130.85, 128.49, 127.81, 124.91, 116.08, 115.77, 114.33, 55.49. Elemental Anal. Calcd. for $\text{C}_{14}\text{H}_{12}\text{ClNO}_2$: C, 64.25; H, 4.62; N, 5.35. Found: C, 62.92; H, 4.57; N, 5.45%.

N-(*p*-Methoxybenzylidene)-2-hydroxy-4-methylaniline (**3c**). M.p. 86–87°C. IR: 3344, 3066, 3018, 2974, 2934, 2907, 2840, 1622 (C=N), 1593, 1567, 1509, 1481, 1423, 1378, 1245, 1162, 1026, 969, 909, 861, 835, 809, 761, 666, 617, 559 cm^{-1} . ^1H NMR (CDCl_3): δ 8.59 (s, 1H, N=CH), 7.83 (d, $J = 8.7$ Hz, 2H, Ar), 7.16 (d, $J = 8.1$ Hz, 1H, Ar), 6.97 (d, $J = 8.7$ Hz, 2H, Ar), 6.81 (s, 1H, Ar), 6.68 (d, $J = 7.8$ Hz, 1H, Ar), 3.86 (s, 3H, OCH_3), 2.31 (s, 3H, CH_3). ^{13}C NMR (CDCl_3): δ 162.52, 155.25 (N=C), 152.02, 138.71, 133.68, 130.36, 129.13, 120.74, 115.34, 115.26, 114.30, 55.45, 21.41. Elemental Anal. Calcd. for $\text{C}_{15}\text{H}_{15}\text{NO}_2$: C, 74.67; H, 6.27; N, 5.81. Found: C, 74.63; H, 6.41; N, 6.21%.

N-(Cinnamylidene)-2-hydroxy-5-chloroaniline (**2d**). M.p. 91–93°C. IR: 3344, 3064, 3013, 2964, 2911, 2881, 2839, 1621 (C=N), 1577, 1501, 1483, 1445, 1366, 1240, 1154, 1082, 979, 837, 804, 742, 683, 661, 593 cm^{-1} . ^1H NMR (CDCl_3): δ 8.41 (d, $J = 8.2$ Hz, 1H, N=CH), 7.54 (d, $J = 5.2$, 2H, Ar), 7.43–7.00 (m, 5H, Ar & CH=CH), 7.29 (s, 1H, Ar), 7.22 (d, $J = 8.1$ Hz, 1H, Ar), 6.90 (d, $J = 8.6$ Hz, 1H, Ar). ^{13}C NMR (CDCl_3): δ 159.36, 150.95 (N=C), 145.23, 136.47, 135.35, 129.99, 128.99, 128.36, 128.11, 127.69, 124.94, 115.99, 115.86. Elemental Anal. Calcd. for $\text{C}_{15}\text{H}_{12}\text{ClNO}$: C, 69.91; H, 4.69; N, 5.43. Found: C, 68.80; H, 4.72; N, 5.36%.

N-(Cinnamylidene)-2-hydroxy-4-methylaniline (**3d**). M.p. 97–98°C. IR: 3350, 3061, 3012, 2963, 2912, 2880, 2840, 1624 (C=N), 1577, 1541, 1498, 1447, 1368, 1257, 1157, 1092, 975, 836, 804, 749, 686, 630, 552 cm^{-1} . ^1H NMR (CDCl_3): δ 8.57

(d, $J = 6.6$ Hz, 1H, N=CH), 7.66 (dd, $J = 6.0, 1.9$ Hz, 2H, Ar), 7.54–7.12 (m, 5H, Ar & CH=CH), 7.30 (d, $J = 7.9$ Hz, 1H, Ar), 6.93 (s, 1H, Ar), 6.80 (d, $J = 8.1$ Hz, 1H, Ar), 2.43 (s, 3H, Ar). ^{13}C NMR (CDCl_3): δ 156.85, 152.28 (N=C), 143.26, 139.38, 135.71, 133.20, 129.53, 128.92, 128.73, 127.46, 120.75, 115.48, 115.04, 21.46. Elemental Anal. Calcd. for $\text{C}_{16}\text{H}_{15}\text{NO}$: C, 80.98; H, 6.37; N, 5.26. Found: C, 79.22; H, 6.18; N, 5.90%.

Conflict of Interests

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

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