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A comparative study of microwave-assisted and conventional synthesis of novel 2-(4-diethylamino-2-hydroxyphenyl)-3-substituted-thiazolidin-4-one derivatives

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We report efficient and extremely fast procedures for the synthesis of 2-(4-diethylamino-2-hydroxy-phenyl)-3-substituted-thiazolidin-4-one using 4-diethylamino-2-hydroxybenzaldehyde, various amines and mercaptoacetic acid under microwave irradiation (μw), and similarly conventional methods are used for comparison. A considerable increase in the reaction rate has been observed with a better yield in microwave technique. The structures of the compounds were confirmed by elemental and spectral analyses (^{1}H-NMR, ^{13}C-NMR & Mass).

Key words: 4-diethylamino-2-hydroxybenzaldehyde, thiazolidin-4-one, microwave method

INTRODUCTION

Schiff bases are an important class of compounds in medicine and pharmaceutics. They show biological activities including antibacterial [1–4], antifungal [5], anticancer [6, 7], and herbicidal [8]. Furthermore, Schiff bases are utilized as starting material in the synthesis of pharmaceutically important industrial compounds such as β-lactam [9–12] viz. thiazolidin-4-one derivatives which are well known to have antifungal, antibacterial, herbicidal and host of other biological activities [13–15]. Moreover, they have been studied extensively because of their ready accessibility, diverse chemical reactivity and a broad spectrum of biological activities.

In the last few years, Microwave-induced Organic Reaction Enhancement (MORE) chemistry has gained increased popularity as a non-conventional technique for rapid organic synthesis [16], and many researchers have described accelerated organic reactions; also, a large number of papers has appeared proving the synthetic utility of MORE chemistry in routine organic synthesis [17, 18]. It can be termed as '**e-chemistry**' because it is easy, effective, economical and eco-friendly and is believed to be a step towards green chemistry. Earlier, we have reported the Microwave-induced Niementowskii reaction, i. e. synthesis of quinazolinones and 3-methyl-1H-5-pyrazolones using different solid supports [19] and fluorine containing pyrozoline derivatives over solid potassium carbonate [20], wherein the problems associated with prolonged heating were avoided. We have successfully adopted this technique for the synthesis of various 1,5-benzothiazipines [21], too. The advantages of microwave technology over conventional methods in heterocycles have been recently reviewed [22, 23]. In continuation of our earlier work [24–26] on the application of MORE [26–30] chemistry to organic synthesis, we wish to report on a simple microwave synthesis of thiazolidin-4-one derivatives 4 a–r using the Schiff base of 4-diethylamino-2-hydroxybenzaldehyde and various aromatic amines according to Scheme. All the compounds were prepared using both conventional and microwave techniques. The reaction carried out in ethanol / methanol using the conventional method requires about 6.0–8.0 h, while the microwave irradiation method requires only 1.5–3.0 min [30]. A comparative study in terms of yield and reaction period is shown in Table 1. All the compounds synthesized were characterized by elemental analysis, IR, ^{1}H-NMR, ^{13}C-NMR and mass spectrometry.

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Table 1. **Comparison of conventional and microwave synthesis for 3 a–r and 4 a–r**

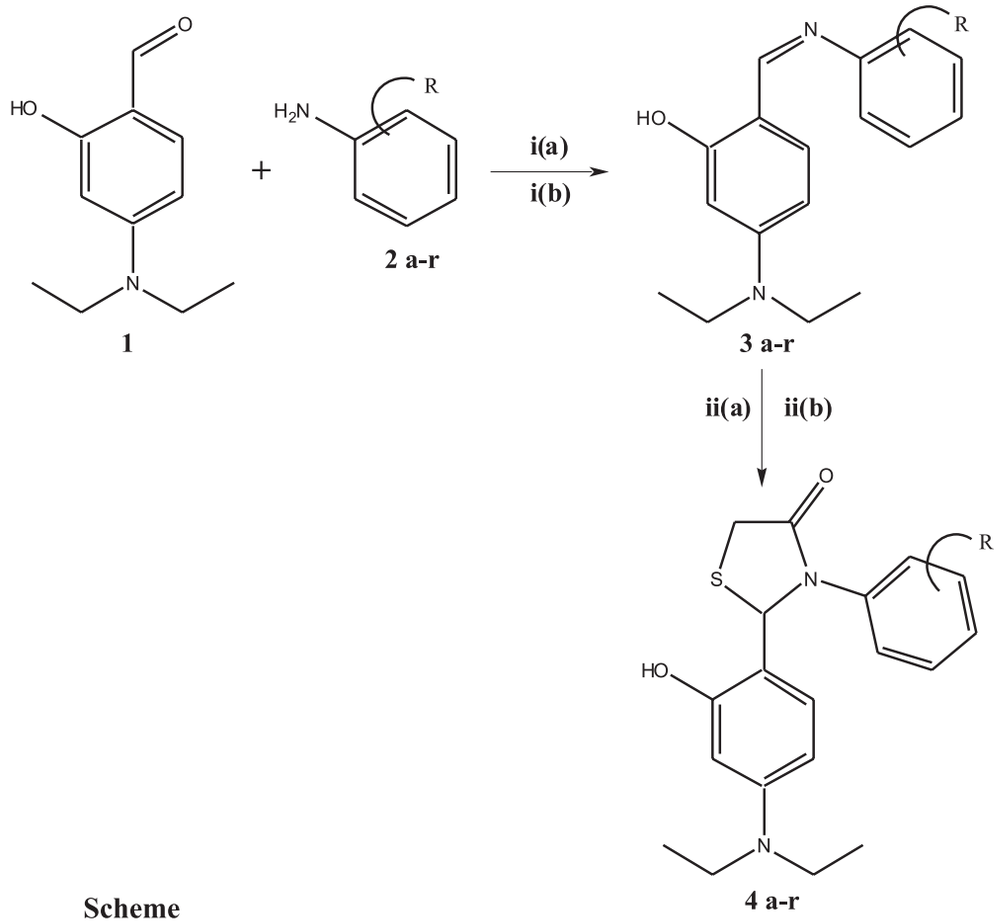
Compound	Conventional method		Microwave method				
	% yield	t/hrs	% yield	t_1/min	p_1/watt	t_2/min	p_2/watt
3 a	65–67	3	91–93	3.0	350	2.0	500
3 b	72–74	2.5	89–91	2.5	350	2.0	500
3 c	65–67	2.5	92–93	2.5	350	2.0	500
3 d	72–74	2.5	91–92	3.0	350	2.0	500
3 e	65–67	2.5	88–90	3.0	350	2.0	500
3 f	72v74	2.5	92–93	2.5	350	2.1	500
3 g	65–66	2.5	91–93	2.5	350	2.2	500
3 h	72–74	2.5	87–89	2.5	350	2.0	500
3 i	65–66	2.5	85–87	3.0	350	2.2	500
3 j	72–74	2.5	90–91	3.0	350	2.0	500
3 k	65–67	3	86–87	3.0	350	2.2	500
3 l	72–73	3	91–93	3.0	350	2.0	500
3 m	65–66	2.5	82–84	2.5	350	2.2	500
3 n	72–73	2.5	91–93	3.0	350	2.0	500
3 o	65–66	2.5	81–83	3.0	350	2.2	500
3 p	72–74	2.5	95–96	3.0	350	2.0	500
3 q	65–65	2.5	87–89	2.5	350	2.2	500
3 r	72–74	2.5	91–93	2.5	350	2.0	500
4 a	71–73	6	82–83	3.5	350	2.0	500
4 b	72–74	6	86–87	3.0	350	2.0	500
4 c	66–68	6.5	84–85	3.5	350	2.5	500
4 d	75–76	7	87–88	3.5	350	2.0	500
4 e	69–70	7	88–89	3.5	350	2.5	500
4 f	68–69	7	89–91	3.5	350	2.0	500
4 g	67–69	8	81–83	3.0	350	2.5	500
4 h	66–68	8	86–88	3.5	350	2.0	500
4 i	62–65	6.5	88–89	3.0	350	2.0	500
4 j	70–72	6	87–89	3.5	350	2.5	500
4 k	73–75	7	88–90	3.5	350	2.5	500
4 l	69–71	7	92–93	3.0	350	2.0	500
4 m	66–69	8	91–92	3.0	350	2.0	500
4 n	69–72	8	92–93	3.5	350	2.5	500
4 o	70–72	6	88–90	3.5	350	2.0	500
4 p	68–71	7	85–87	3.5	350	2.0	500
4 q	73–75	9	87–88	3.5	350	2.0	500
4 r	77–79	9	88–89	3.5	350	2.5	500

RESULTS AND DISCUSSION

Various Schiff's Base derivatives 3 a–r were prepared using 4-diethylamino-2-hydroxybenzaldehyde (1) and various amines which on cycloaddition with mercaptoacetic acid in ethanol gave 2-(4-diethylamino-2-hydroxy-phenyl)-3-substituted-thiazolidin-4-one 4 a–r (Scheme).

All the reactions under microwave irradiation were completed within 2.0–3.5 min, whereas similar reactions under conventional heating (steam bath) at refluxed temperature gave poor yields with comparatively longer reaction time periods (Table 1), demonstrating that the effect of microwave irradiation is not purely thermal. Actually, microwave irradiation facilitates the polarization of the molecules under irradiation, causing a rapid reaction to occur. This is consistent with the reaction mechanism which involves a polar transition state [23].

The impact of microwave irradiation and conventional heating on the synthesis of compounds 3 a–r and 4 a–r has been compared. Moreover, the effects of irradiation power and time on the reaction were also studied, and the results were summarized in Tables. The effects of irradiation power and time on the reaction were also studied, and the results were summarized in Tables 2 and 3). It has been found that a higher yield of compounds 3 a–r and 4 a–r can be obtained at 500 W for 2.0–2.5 min under microwave irradiation conditions. All the compounds synthesized were adequately characterized by their elemental analyses and spectral IR, ^{1}H-NMR, ^{13}C-NMR and mass data. All the structures of the above compounds were in good agreement with spectral and analytical data.



Scheme

i(a) **Conventional method:** ethanol / acetic acid, reflux, 2.5–3.0 hours

i(b) **Microwave method:** ethanol / acetic acid, μw,
t_1 = 2.5–3.0 min at p_1 = 350 w, t_2 = 2.0–2.5 min at p_2 = 500 w

ii(a) **Conventional method:** $SHCH_2COOH$ / $ZnCl_2$ / DMF, reflux, 6–8 hours

ii(b) **Microwave method:** $SHCH_2COOH$ / $ZnCl_2$ / DMF, μw,
t_1 = 3.0–3.5 min at p_1 = 350 w, t_2 = 2.0–2.5 min at p_2 = 500 w

R = H (2a, 3a, 4a), 2-CH_3(2b, 3b, 4b), 3-CH_3(2c, 3c, 4c), 4-CH_3(2d, 3d, 4d), 2-Cl(2e, 3e, 4e), 3-Cl(2f, 3f, 4f), 4-Cl(2g, 3g, 4g), 2,4-dichloro (2h, 3h, 4h), 2,5dichloro (2i, 3i, 4i), 2-NO_2(2j, 3j, 4j), 3-NO_2(2k, 3k, 4k), 4-NO_2(2l, 3l, 4l), 2-OCH_3(2m, 3m, 4m), 3-OCH_3(2n, 3n, 4n), 4-OCH_3(2o, 3o, 4o), 3,4-dimethoxy(2p, 3p, 4p), 3-Br(2q, 3q, 4q), 4-F(2r, 3r, 4r)

Table 2. **The effect of microwave irradiation powerK on 4 a**

Irradiation power(W)	250	300	350	400	450	500
Yield	75	77	82	83	89	92

[K] Irradiation time is 2 min

Table 3. **The effect of microwave irradiation timeG on 4 a**

Irradiation time(min)	5.0	4.5	4.0	3.0	2.5	2.0
Yield	75	77	82	83	89	92

[G] Irradiation power is 500 W

EXPERIMENTAL

All reagents, solvents and catalysts used were of analytical grade from a commercial source and used directly. All the melting points were determined in a PMP-DM scientific melting point apparatus and were uncorrected. UV spectra were determined on a Perkin–Elmer 550S spectrophotometer. IR spectra (υ_{max} in cm^{-1}) were recorded on a Shimadzu FT-IR 8300 spectrophotometer using KBr or Nujol technique; ^{1}H-NMR spectra on a Bruker's WM 400FT 400 MHz NMR instrument using $CDCl_3$ or DMSO-d_6 as solvent and TMS as internal reference (chemical shifts in δ, ppm);

^{13}C-NMR on a Varian AMX 400 (100 MHz) spectrometer as solutions in $CDCl_3$ and mass spectra on a Jeol JMS D-300 spectrometer operating at 75 eV. Column chromatography was performed with silica gel 60 (70–230 mesh) purchased from E. Merck AG. The purities of the obtained substances and the composition of the reaction mixtures were monitored by thin layer chromatography on silica gel 60 F_{254} plates (Merck) with a fluorescent indicator (Merck No. 5554). Solvents and common reagents obtained from Merck and Aldrich were reagent-grade. The elemental analysis (C, H, N) of the compounds was performed on a Carlo Erba-1108 elemental analyzer. The values established by elemental analysis were within = 0.4% in comparison to calculated values. The microwave-assisted reactions were carried out in a QPro-M Microwave Synthesis System manufactured by Questron Technologies Corporation, Ontario L4Z 2E9, Canada, where microwaves are generated by magnetron at a frequency of 2450 MHz having an output energy range of 100 to 500 W and an individual sensor for temperature control with attachment of a reflux condenser at constant stirring (thus avoiding the risk of high pressure development) and synthesis on preparative scales.

Method for preparation of 4-diethylamino-2-hydroxybenzaldehyde (1)

The starting compound was synthesized according to the literature [32].

Representative process: Conventional preparation of schiff's base derivatives (3 a–r)

An equimolar amount of 4-diethylamino-2-hydroxybenzaldehyde (1) (1.93 g, 0.01 mole) and 4-chloroaniline (2 g) (1.27 g, 0.01 mole) was refluxed in ethanol (10 mL) in the presence of a catalytic amount of acetic acid for 2.5–3.0 h. On cooling the reaction mixture, a yellow solid was crystallized from the dimethylformamide–ethanol mixture (60 : 40).

Representative process: Microwave preparation of Schiff's base derivatives (3 a–r)

An equimolar amount of 4-diethylamino-2-hydroxybenzaldehyde (1) (1.93 g, 0.01 mole), 4-chloroaniline (2 g) (1.27 g, 0.01 mole) and ethanol (10 mL) in the presence of a catalytic amount of acetic acid (1 mL) were put in an Erlenmeyer flask and irradiated under microwaves in two stages (t_1 = 2.5–3.0 min and t_2 = 2.0–2.5 min) at two different power levels (p_1 = 350 w and p_2 = 500 w) respectively (Table 1) [19, 27]. Upon completion of reaction (monitored by tlc), the solvent was removed, and the residue was recrystallized from dimethylformamide-ethanol mixture (60 : 40) to give a yellow solid, $C_{17}H_{19}N_2ClO$. M. p. 110–112 °C. IR-spectra (cm^{-1} KBr-pellets): 1626 (–N = CH–), 2990 (Alkyl CH stretching), 3370 (OH stretching), 3030 (aromatic CH stretching); ^{1}NMR-Spectra ($CDCl_3$–DMSO-d_6) (δ ppm): 1.42 (t, 3H, J = 6.8, N ($CH_2\underline{CH_3}$)$_2$), 4.29 (q, 2H, J = 7.1, N ($\underline{CH_2}CH_3$)$_2$), 5.38 (s, 1H, CH = N), 6.97–7.71 (m, 7H, Ar–H), 11.09 (s, 1H, OH).

Similarly, other compounds (3 b–r) were prepared in the above manner.

Representative process: Microwave preparation of 2-(4-diethylamino-2-hydroxy-phenyl)-3-substituted-thiazolidin-4-one (4 a–r)

An equimolar amount of 3 a (0.01 mole) and mercaptoacetic acid (0.01 mole) in ethanol with a pinch of $ZnCl_2$ (1.0 gm) as a reaction mediator [26] were put in an Erlenmeyer flask and irradiated under microwaves in two stages (t_1 = 3.0 to 3.5 min and t_2 = 2.0 to 2.5 min) at two different power levels (p_1 = 350 W and p_2 = 500 W) respectively (Table 1) [19, 27]. After completion of the reaction (monitored by tlc), it was then diluted with ice-cold water. The separated solid was crystallized from methanol to give compound 4 a as a dark-yellow solid.

Representative process: Conventional preparation of 2-(4-diethylamino-2-hydroxy-phenyl)-3-substituted-thiazolidin-4-one (4 a–r)

An equimolar amount of 3 a (0.01 mole) in ethanol and mercaptoacetic acid (0.01 mole) with a pinch of $ZnCl_2$ (1.0 gm) (as a reaction mediator) [26] were refluxed on a steam bath for about 6–8 h. After completion of the reaction (monitored by tlc), the excess solvent was removed under reduced pressure, and the remaining oily residue was treated with diethyl ether to afford a crude solid 4 a. The remaining compound was isolated by silica gel column chromatography eluted with chloroform and then crystallized from methanol.

Similarly, all compounds 4 b–r were prepared in the above manner using 3 b–r, respectively.

SPECTROSCOPIC & ANALYTICAL DATA OF 4 a–r

2-(4-Diethylamino-2-hydroxy-phenyl)-3-phenyl-thiazolidin-4-one (4 a)

Compound 4 a was obtained as yellow crystals (methanol); m. p. 148–150 °C. Mol. formula: $C_{19}H_{22}O_2N_2S$; UV (DMF) λ max: 301 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 1550, 781. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.28 (t, 3H, J = 7.0, N ($CH_2\underline{CH_3}$)$_2$), 4.11 (q, 2H, J = 7.1, N ($\underline{CH_2}CH_3$)$_2$), 3.24 (d, 1H, J = 15.4, 5–H_A), 4.92 (dd, 1H, J = 2.5 & 15.4, 5–H_B) [33], 6.5 (d, 1H, J = 1.4, H–2), 7.00–7.95 (m, 8H, Ar–H), 12.92 (s, 1H, OH). ^{13}C-NMR (400 MHz, DMSO-d_6): δ = 12.9 (N ($CH_2\underline{CH_3}$)$_2$), 40.2 (N ($\underline{CH_2}CH_3$)$_2$), 31.1 (S–CH_2), 51.1 (C–S), 170.6 (cyclic, >C = O), 127.7–133.0 (Ar–C). MS (EI): m/z (%) = 342 (M^+). Elemental analysis data: found, %: C, 66.75; H, 6.43; N, 8.27; S, 9.41; formula $C_{19}H_{22}N_2O_2S$ (342.4): calculated, %: C, 66.64; H, 6.48; N, 8.18; S, 9.36.

2-(4-Diethylamino-2-hydroxy-phenyl)-3-o-tolyl-thiazolidin-4-one (4 b)

Compound 4 b was obtained as light yellow crystals (methanol). M. p. 158–160 °C. Mol. Formula: $C_{20}H_{24}O_2N_2S$; UV (DMF) λ max: 285 nm. IR (KBr): 3375, 3015, 2990, 2945, 1715–1733, 781, 1550, 1320. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.30 (t, 3H, J = 6.9, N ($CH_2\underline{CH_3}$)$_2$), 4.12 (q, 2H, J = 7.1, N ($\underline{CH_2}CH_3$)$_2$), 2.05 (s, 3H, CH_3), 2.20 (s, 3H, Ar–CH_3), 3.20 (d, 1H, J = 15.9, 5–H_A), 4.92 (dd, 1H, J = 2.5 & 15.9, 5–H_B), 6.6 (d, 1H, J = 1.9, H–2), 7.10–7.95 (m, 7H, Ar–H), 12.92 (s, 1H, OH). ^{13}C-NMR (400 MHz, DMSO-d_6): δ = 13.0 (N ($CH_2\underline{CH_3}$)$_2$), 40.2 (N ($\underline{CH_2}CH_3$)$_2$), 30.1 (S–CH_2), 52.2 (C–S), 172.8 (cyclic, >C = O), 35.1 (CH_3), 120.8–140.0 (Ar–C). MS (EI): m/z (%) = 356 (M^+). Elemental analysis data: found, %: C, 67.45; H, 6.74; N, 8.77; S, 8.82; formula $C_{20}H_{24}N_2O_2S$ (356.0): calculated, %: 67.38; H, 6.79; N, 7.86; S, 8.99.

2-(4-Diethylamino-2-hydroxy-phenyl)-3-m-tolyl-thiazolidin-4-one (4 c)

Compound 4 c was obtained as light yellow crystals (methanol). M. p. 153–155 °C. Mol. formula: $C_{20}H_{24}O_2N_2S$; UV (DMF) λ max: 297 nm. IR (KBr): 3375, 3020, 2992, 2945, 1710–1733, 781, 1550, 1315. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.31 (t, 3H, J = 6.9, N ($CH_2\underline{CH_3}$)$_2$), 4.12 (q, 2H, J = 7.1, N ($\underline{CH_2}CH_3$)$_2$), 2.03 (s, 3H, CH_3), 2.18 (s, 3H, Ar–CH_3), 3.24 (d, 1H, J = 15.9, 5–H_A), 4.94 (dd, 1H, J = 2.5 & 15.9, 5–H_B), 6.6 (d, 1H, J = 1.9, H–2), 7.10–7.95 (m, 7H, Ar–H), 12.92 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 13.1 (N ($CH_2\underline{CH_3}$)$_2$), 40.2 (N ($\underline{CH_2}CH_3$)$_2$), 30.0 (S–CH_2), 52.2 (C–S), 35.5 (CH_3), 172.0 (cyclic, >C = O), 120.9–140.8 (Ar–C). MS (EI): m/z (%) = 356 (M^+). Elemental analysis data: found, %: C, 67.47; H, 6.73; N, 8.89; S, 8.81; formula $C_{20}H_{24}N_2O_2S$ (356.0): calculated, %: 67.38; H, 6.79; N, 7.86; S, 8.99.

2-(4-Diethylamino-2-hydroxy-phenyl)-3-p-tolyl-thiazolidin-4-one (4 d)

Compound 4 d was obtained as light yellow crystals (methanol). M. p. 191–192 °C. Mol. formula: $C_{20}H_{24}O_2N_2S$; UV (DMF) λ max: 278 nm. IR (KBr): 3370, 3010, 2991, 2950, 1710–1733, 781, 1560, 1320. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.30 (t, 3H, J = 6.9, N ($CH_2\underline{CH_3}$)$_2$), 4.12 (q, 2H, J = 7.1, N ($\underline{CH_2}CH_3$)$_2$), 2.03 (s, 3H, CH_3), 2.20 (s, 3H, Ar–CH_3), 3.22 (d, 1H, J = 15.9, 5–H_A), 4.94 (dd, 1H, J = 2.5 & 15.9, 5–H_B), 6.8 (d, 1H, J = 1.9, H–2), 7.10–7.95 (m, 7H, Ar–H), 12.94 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 13.0 (N ($CH_2\underline{CH_3}$)$_2$), 40.2 (N ($\underline{CH_2}CH_3$)$_2$), 30.3 (S–CH_2), 52.2 (C–S), 36.0 (CH_3), 171.8 (cyclic, >C = O), 121.1–140.1 (Ar–C). MS (EI): m/z (%) = 356 (M^+). Elemental analysis data: found, %: C, 67.45; H, 6.77; N, 8.89; S, 8.92; formula $C_{20}H_{24}N_2O_2S$ (356.50): calculated, %: 67.38; H, 6.79; N, 7.86; S, 8.99.

3-(2-Chloro-phenyl)-2-(4-diethylamino-2-hydroxy-phenyl)-thiazolidin-4-one (4 e)

Compound 4 e was obtained as dark yellow crystals (methanol). M. p. 161–163 °C. Mol. formula: $C_{19}H_{21}O_2N_2SCl$; UV (DMF) λ max: 310 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 782, 1550, 698. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.32 (t, 3H, J = 6.7, N ($CH_2\underline{CH_3}$)$_2$), 4.11 (q, 2H, J = 7.0, N ($\underline{CH_2}CH_3$)$_2$), 3.20 (d, 1H, J = 15.7, 5–H_A), 4.94 (dd, 1H, J = 2.9 & 15.7, 5–H_B), 6.8 (d, 1H, J = 1.9, H–2), 7.00–7.95 (m, 7H, Ar–H), 12.94 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 12.9 (N ($CH_2\underline{CH_3}$)$_2$), 40.3 (N ($\underline{CH_2}CH_3$)$_2$), 31.1 (S–CH_2), 50.0 (C–S), 176.3 (cyclic, >C = O), 124.4–137.7 (Ar–C). MS (EI): m/z (%) = 376.5 (M^+). Elemental analysis data: found, %: C, 60.67; H, 5.64; N, 7.46; S, 8.44; Cl, 9.42; formula $C_{19}H_{21}N_2O_2SCl$ (377.0): calculated, %: 60.55; H, 5.62; N, 7.43; S, 8.51; Cl, 9.40.

3-(3-Chloro-phenyl)-2-(4-diethylamino-2-hydroxy-phenyl)-thiazolidin-4-one (4 f)

Compound 4 f was obtained as brown yellow crystals (methanol). M. p. 143–145 °C. Mol. Formula: $C_{19}H_{21}O_2N_2SCl$; UV (DMF) λ max: 324 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 782, 1550, 695. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.31 (t, 3H, J = 6.7, N ($CH_2\underline{CH_3}$)$_2$), 4.11 (q, 2H, J = 7.0, N ($\underline{CH_2}CH_3$)$_2$), 3.24 (d, 1H, J = 15.7, 5–H_A), 4.94 (dd, 1H, J = 2.9 & 15.7, 5–H_B), 6.8 (d, 1H, J = 1.9, H–2), 7.05–7.95 (m, 7H, Ar–H), 12.94 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 12.9 (N ($CH_2\underline{CH_3}$)$_2$), 40.2 (N ($\underline{CH_2}CH_3$)$_2$), 31.1 (S-CH_2), 50.1 (C–S), 176.2 (cyclic, >C = O), 124.3–137.7 (Ar–C). MS (EI): m/z (%) = 376.5 (M^+). Elemental analysis data: found, %: C, 60.63; H, 5.67; N, 7.49; S, 8.63; Cl, 9.44; formula $C_{19}H_{21}N_2O_2SCl$ (377.0): calculated, %: 60.55; H, 5.62; N, 7.43; S, 8.51; Cl, 9.40.

3-(4-Chloro-phenyl)-2-(4-diethylamino-2-hydroxy-phenyl)-thiazolidin-4-one (4 g)

Compound 4 g was obtained as dark yellow crystals (methanol). M. p. 152–155 °C. Mol. formula: $C_{19}H_{21}O_2N_2SCl$; UV (DMF) λ max: 332 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 1218, 781, 1550, 698. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.32 (t, 3H, J = 6.7, N ($CH_2\underline{CH_3}$)$_2$), 4.11 (q, 2H, J = 7.0, N ($\underline{CH_2}CH_3$)$_2$), 3.20 (d, 1H, J = 15.7, 5–H_A), 4.90 (dd, 1H, J = 2.9 & 15.7, 5–H_B), 6.5 (d, 1H, J = 1.9, H–2), 7.00–7.95 (m, 7H, Ar–H), 12.90 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 12.9 (N ($CH_2\underline{CH_3}$)$_2$), 40.3 (N ($\underline{CH_2}CH_3$)$_2$), 32.1 (S-CH_2), 50.1 (C–S), 177.2 (cyclic, >C = O), 125.3–133.9 (Ar–C). MS (EI): m/z (%) = 376.5 (M^+). Elemental analysis data: found, %: C, 60.65; H, 5.63; N, 7.47; S, 8.41; Cl, 9.46; formula $C_{19}H_{21}N_2O_2SCl$ (377.0): calculated, %: 60.55; H, 5.62; N, 7.43; S, 8.51; Cl, 9.40.

3-(2,4-dichloro-phenyl)-2-(4-diethylamino-2-hydroxy-phenyl)-thiazolidin-4-one (4 h)

Compound 4 h was obtained as orange yellow crystals (methanol). M. p. 161–163 °C. Mol. Formula: $C_{19}H_{20}O_2N_2SCl_2$; UV (DMF) λ max: 291 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 785, 1550, 695. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.33 (t, 3H, J = 6.9, N ($CH_2\underline{CH_3}$)$_2$), 4.14 (q, 2H, J = 7.1, N ($\underline{CH_2}CH_3$)$_2$), 3.22 (d, 1H, J = 15.9, 5–H_A), 4.94 (dd, 1H, J = 2.7 & 15.9, 5–H_B), 6.6 (d, 1H, J = 2.0, H–2), 7.20–7.85 (m,

6H, Ar–H), 12.95 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 13.9 (N ($CH_2\underline{CH_3}$)$_2$), 40.2 (N($\underline{CH_2}CH_3$)$_2$), 32.2 (CH2), 51.2 (C–S), 171.7 (cyclic, >C = O), 127.9–139.3 (Ar–C). MS (EI): m/z (%) = 411 (M^+). Elemental analysis data: found, %: C, 55.47; H, 4.86; N, 6.79; S, 7.72; Cl, 17.30; formula $C_{19}H_{20}N_2O_2SCl_2$ (411.0): calculated, %: 55.48; H, 4.90; N, 6.81; S, 7.80; Cl, 17.24.

3-(2,5-dichloro-phenyl)-2-(4-diethylamino-2-hydroxy-phenyl)-thiazolidin-4-one (4 i)

Compound 4 i was obtained as orange yellow crystals (methanol). M. p. 135–137 °C. Mol. formula: $C_{19}H_{20}O_2N_2SCl_2$; UV (DMF) λ max: 289 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 785, 1550, 690. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.33 (t, 3H, J = 6.9, N ($CH_2\underline{CH_3}$)$_2$), 4.14 (q, 2H, J = 7.1, N ($\underline{CH_2}CH_3$)$_2$), 3.24 (d, 1H, J = 15.9, 5–H_A), 4.95 (dd, 1H, J = 2.7 & 15.9, 5–H_B), 6.5 (d, 1H, J = 2.0, H–2), 7.20–7.85 (m, 6H, Ar–H), 12.95 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 13.9 (N ($CH_2\underline{CH_3}$)$_2$), 40.2 (N($\underline{CH_2}CH_3$)$_2$), 31.2 (CH_2), 51.2 (C–S), 172.1 (cyclic, >C = O),120.9–149.9 (Ar–C). MS (EI): m/z (%) = 411 (M^+). Elemental analysis data: found, %: C, 55.45; H, 4.83; N, 6.77; S, 7.71; Cl, 17.25; formula $C_{19}H_{20}N_2O_2SCl_2$ (411.0): calculated, %: 55.48; H, 4.90; N, 6.81; S, 7.80; Cl, 17.24.

2-(4-Diethylamino-2-hydroxy-phenyl)-3-(2-nitro-phenyl)-thiazolidin-4-one (4 j)

Compound 4 j was obtained as brown yellow crystals (methanol). M. p. 123–124 °C. Mol. formula: $C_{19}H_{21}O_4N_3S$; UV (DMF) λ max: 313 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 785, 1562, 1529, 1350. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.34 (t, 3H, J = 6.9, N ($CH_2\underline{CH_3}$)$_2$), 4.10 (q, 2H, J = 7.1, N ($\underline{CH_2}CH_3$)$_2$), 3.20 (d, 1H, J = 15.4, 5–H_A), 4.95 (dd, 1H, J = 2.5 & 15.4, 5–H_B), 6.8 (d, 1H, J = 1.4, H–2), 7.10–7.85 (m, 7H, Ar–H), 12.90 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 12.7 (N ($CH_2\underline{CH_3}$)$_2$), 40.2 (N ($\underline{CH_2}CH_3$)$_2$), 32.1 (S–CH_2), 50.3 (C–S), 169.7 (cyclic, >C = O), 111.1–196.8 (Ar–C). MS (EI): m/z (%) = 387 (M^+). Elemental analysis data: found, %: C, 58.95; H, 5.43; N, 10.77; S, 8.21; formula $C_{19}H_{21}N_3O_4S$ (387.0): calculated, %: 58.90; H, 5.46; N, 10.85; S, 8.28.

2-(4-Diethylamino-2-hydroxy-phenyl)-3-(3-nitro-phenyl)-thiazolidin-4-one (4 k)

Compound 4 k was obtained as brown yellow crystals (methanol). M. P. 133–135°C. Mol. formula: $C_{19}H_{21}O_4N_3S$; UV (DMF) λ max: 291 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 785, 1565, 1520, 1340. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.35 (t, 3H, J = 6.9, N ($CH_2\underline{CH_3}$)$_2$), 4.10 (q, 2H, J = 7.1, N ($\underline{CH_2}CH_3$)$_2$), 3.22 (d, 1H, J = 15.4, 5–H_A), 4.95 (dd, 1H, J = 2.5 and 15.4, 5–H_B), 6.8 (d, 1H, J = 1.4, H–2), 7.10–7.85 (m, 7H, Ar–H), 12.90 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 12.7 (N ($CH_2\underline{CH_3}$)$_2$), 40.3 (N ($\underline{CH_2}CH_3$)$_2$), 32.1 (S–CH_2), 50.2 (C–S), 169.9 (cyclic, >C = O), 111.2–196.8 (Ar–C). MS (EI): m/z (%) = 387 (M^+). Elemental analysis data: found, %: C, 58.97; H, 5.46; N, 10.79; S, 8.24; formula $C_{19}H_{21}N_3O_4S$ (387.0): calculated, %: 58.90; H, 5.46; N, 10.85; S, 8.28.

2-(4-Diethylamino-2-hydroxy-phenyl)-3-(4-nitro-phenyl)-thiazolidin-4-one (4 l)

Compound 4 l was obtained as brown yellow crystals (methanol). M. p. 144–145 °C. Mol. formula: $C_{19}H_{21}O_4N_3S$; UV (DMF) λ max: 298 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 785, 1577, 1500, 1345. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.34 (t, 3H, J = 6.9, N ($CH_2\underline{CH_3}$)$_2$), 4.10 (q, 2H, J = 7.1, N ($\underline{CH_2}CH_3$)$_2$), 3.24 (d, 1H, J = 15.4, 5–H_A), 4.92 (dd, 1H, J = 2.5 and 15.4, 5–H_B), 6.8 (d, 1H, J = 1.4, H–2), 7.10–7.85 (m, 7H, Ar–H), 12.90 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 12.8 (N ($CH_2\underline{CH_3}$)$_2$), 40.3 (N ($\underline{CH_2}CH_3$)$_2$), 32.1 (S–CH_2), 50.3 (C–S), 169.8 (cyclic, >C = O), 111.8–196.8 (Ar–C). MS (EI): m/z (%) = 387 (M^+). Elemental analysis data: found, %: C, 58.97; H, 5.45; N, 10.77; S, 8.22; formula $C_{19}H_{21}N_3O_4S$ (387.0): calculated, %: 58.90; H, 5.46; N, 10.85; S, 8.28.

2-(4-Diethylamino-2-hydroxy-phenyl)-3-(2-methoxy-phenyl)-thiazolidin-4-one (4 m)

Compound 4 m was obtained as light brown crystals (methanol). M. p. 158–160 °C. Mol. formula: $C_{20}H_{24}O_3N_2S$; UV (DMF) λ max: 320 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 789, 1577, 1255. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.30 (t, 3H, J = 7.0, N ($CH_2\underline{CH_3}$)$_2$), 4.12 (q, 2H, J = 7.1, N ($\underline{CH_2}CH_3$)$_2$), 3.24 (d, 1H, J = 15.4, 5–H_A), 4.95 (dd, 1H, J = 2.5 and 15.4, 5–H_B), 3.82 (s, 6H, OCH_3), 6.8 (d, 1H, J = 1.5, H–2), 7.00–7.75 (m, 7H, Ar–H), 12.94 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 13.0 (N ($CH_2\underline{CH_3}$)$_2$), 40.2 (N ($\underline{CH_2}CH_3$)$_2$), 30.1 (S–CH_2), 54.6 (C–S), 172.0 (cyclic, >C = O), 58.7 (OCH_3) 129.0–135.0 (Ar–C). MS (EI): m/z (%) = 372 (M^+). Elemental analysis data: found, %: C, 64.46; H, 6.44; N, 7.49; S, 8.72; formula $C_{20}H_{24}N_2O_3S$ (372.0): calculated, %: 64.49; H, 6.49; N, 7.52; S, 8.61.

2-(4-Diethylamino-2-hydroxy-phenyl)-3-(3-methoxy-phenyl)-thiazolidin-4-one (4 n)

Compound 4 n was obtained as light brown crystals (methanol). M. p. 163–164 °C. Mol. formula: $C_{20}H_{24}O_3N_2S$; UV (DMF) λ max: 300 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 789, 1567, 1250. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.30 (t, 3H, J = 7.0, N ($CH_2\underline{CH_3}$)$_2$), 4.12 (q, 2H, J = 7.1, N ($\underline{CH_2}CH_3$)$_2$), 3.22 (d, 1H, J = 15.4, 5–H_A), 4.95 (dd, 1H, J = 2.5 & 15.4, 5–H_B), 3.80 (s, 6H, OCH_3), 6.8 (d, 1H, J = 1.5, H–2), 7.00–7.75 (m, 7H, Ar–H), 12.94 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ= 13.1 (N ($CH_2\underline{CH_3}$)$_2$), 40.2 (N ($\underline{CH_2}CH_3$)$_2$), 30.2 (S–CH_2), 55.4 (C–S), 172.1 (cyclic, >C = O), 60.0 (OCH_3) 129.9–140.1 (Ar–C). MS (EI): m/z (%) = 372 (M^+). Elemental analysis data: found, %: C, 64.51; H, 6.53; N, 7.57; S, 8.72; formula $C_{20}H_{24}N_2O_3S$ (372.0): calculated, %: 64.49; H, 6.49; N, 7.52; S, 8.61.

2-(4-Diethylamino-2-hydroxy-phenyl)-3-(4-methoxy-phenyl)-thiazolidin-4-one (4 o)

Compound 4 o was obtained as light brown crystals (methanol). M. p. 183–185 °C. Mol. formula: $C_{20}H_{24}O_3N_2S$; UV (DMF) λ max: 275 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 789,

1559, 1260. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.31 (t, 3H, J = 7.0, N ($CH_2\underline{CH_3}$)$_2$), 4.12 (q, 2H, J = 7.1, N ($\underline{CH_2}CH_3$)$_2$), 3.22 (d, 1H, J = 15.4, 5–H_A), 4.92 (dd, 1H, J = 2.5 & 15.4, 5–H_B), 3.82 (s, 6H, OCH_3), 6.6 (d, 1H, J = 1.5, H–2), 7.00–7.75 (m, 7H, Ar–H), 12.94 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 13.0 (N ($CH_2\underline{CH_3}$)$_2$), 40.2 (N ($\underline{CH_2}CH_3$)$_2$), 30.2 (S–CH_2), 54.6 (C–S), 172.2 (cyclic, >C = O), 59.2 (OCH_3) 128.2–138.0 (Ar–C). MS (EI): m/z (%) = 372 (M^+). Elemental analysis data: found, %: C, 64.52; H, 6.47; N, 7.49; S, 8.69; formula $C_{20}H_{24}N_2O_3S$ (372.0): calculated, %: 64.49; H, 6.49; N, 7.52; S, 8.61.

2-(4-Diethylamino-2-hydroxy-phenyl)-3-(3,4-dimethoxy-phenyl)-thiazolidin-4-one (4 p)

Compound 4 p was obtained as brown crystals (methanol). M. p. 197–199 °C. Mol. formula: $C_{21}H_{26}O_4N_2S$; UV (DMF) λ max: 277 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 791, 1557, 1255. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.32 (t, 3H, J = 7.0, N ($CH_2\underline{CH_3}$)$_2$), 4.11 (q, 2H, J = 7.0, N ($\underline{CH_2}CH_3$)$_2$), 3.24 (d, 1H, J = 15.5, 5–H_A), 4.92 (dd, 1H, J = 2.5 & 15.5, 5–H_B), 3.90 (s, 6H, OCH_3), 6.6 (d, 1H, J = 1.4, H–2), 7.10–7.75 (m, 6H, Ar–H), 12.92 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 13.1 (N ($CH_2\underline{CH_3}$)$_2$), 40.2 (N ($\underline{CH_2}CH_3$)$_2$), 32.1 (S–CH_2), 55.9 (C–S), 177.8 (cyclic, >C = O), 57.7 (OCH_3) 121.2–139.8 (Ar–C). MS (EI): m/z (%) = 402 (M^+). Elemental analysis data: found, %: C, 62.75; H, 6.43; N, 6.87; S, 7.91; formula $C_{21}H_{26}N_2O_4S$ (403.0): calculated, %: 62.66; H, 6.51; N, 6.98; S, 7.96.

3-(2-Bromo-phenyl)-2-(4-diethylamino-2-hydroxy-phenyl)-thiazolidin-4-one (4 q)

Compound 4 q was obtained as brown crystals (methanol). M. p. 210–212 °C. Mol. formula: $C_{19}H_{21}O_2N_2SBr$; UV (DMF) λ max: 335 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 791, 1556, 698, 600. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.32 (t, 3H, J = 6.7, N ($CH_2\underline{CH_3}$)$_2$), 4.13 (q, 2H, J = 6.9, N ($\underline{CH_2}CH_3$)$_2$), 3.24 (d, 1H, J = 15.7, 5–H_A), 4.92 (dd, 1H, J = 2.5 & 15.7, 5–H_B), 6.6 (d, 1H, J = 1.9, H–2), 7.10–7.95 (m, 7H, Ar–H), 12.92 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 14.0 (N ($CH_2\underline{CH_3}$)$_2$), 40.3 (N ($\underline{CH_2}CH_3$)$_2$), 30.1 (S–CH_2), 60.0 (C–S), 178.0 (cyclic, >C = O), 129.3–154.2 (Ar–C). MS (EI): m/z (%) = 421 (M^+). Elemental analysis data: found, %: C, 66.75; H, 6.43; N, 8.27; S, 9.41; Br, 18.86; formula $C_{19}H_{21}N_2O_2SBr$ (421.0): calculated, %: 54.16; H, 5.02; N, 6.65; S, 7.61; Br, 18.96.

3-(4-Fluoro-phenyl)-2-(4-diethylamino-2-hydroxy-phenyl)-thiazolidin-4-one (4 r)

Compound 4 r was obtained as brown crystals (methanol). M. p. 217–218 °C. Mol. formula: $C_{19}H_{22}O_2N_2SF$; UV (DMF) λ max: 298 nm. IR (KBr): 3370, 3010, 2990, 1715–1733, 791, 1557, 1059. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm: 1.95 (t, 3H, J = 6.9, N ($CH_2\underline{CH_3}$)$_2$), 4.15 (q, 2H, J = 6.9, N ($\underline{CH_2}CH_3$)$_2$), 3.22 (d, 1H, J = 15.9, 5–H_A), 4.92 (dd, 1H, J = 2.5 & 15.9, 5–H_B), 6.6 (d, 1H, J = 1.9, H–2), 7.20–7.95 (m, 7H, Ar–H), 12.94 (s, 1H, OH). ^{13}C NMR (400 MHz, DMSO-d_6): δ = 12.9 (N ($CH_2\underline{CH_3}$)$_2$), 40.2 (N ($\underline{CH_2}CH_3$)$_2$), 30.8 (S–CH_2), 51.8 (C–S), 180.0 (cyclic, >C = O), 117.9–166.0 (Ar–C), J_{F-C} = 2.8 Hz; MS (EI): m/z (%) = 361 (M^+). (Here, ^{13}C NMR spectra were done on the basis of the long-range coupling constants ^4J (C–F)). Elemental analysis data: found, %: C, 63.35; H, 5.83; N, 7.87; S, 8.81; formula $C_{19}H_{21}N_2O_2SF$ (360.0): calculated, %: 63.31; H, 5.87; N, 7.77; S, 8.91.

CONCLUSIONS

A new method for the synthesis of 2-(4-diethylamino-2-hydroxy-phenyl)-3-substituted-thiazolidin-4-one 4 a–r, using microwave irradiation, offers significant improvements over the existing procedures and thus the synthesis of a variety of thiazolidin-4-one derivatives. Also, this simple and reproducible technique affords various thiazolidin-4-one derivatives with short reaction times, excellent yields, and without formation of undesirable side products.

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NAUJŲ 2-(4-DIETILAMINO-2-HIDROKSIFENIL)-3-PAKEISTŲ TIAZOLIDIN-4-ONO DARINIŲ TRADICINĖS SINTEZĖS IR SINTEZĖS ŠVITINANT MIKROBANGOMIS PALYGINAMASIS TYRIMAS

S a n t r a u k a

Aprašytos efektyvios ir labai greitos 2-(4-dietilamino-2-hidroksifenil)-3-pakeistų tiazolidin-4-onų sintezės iš 4-dietilamino-2-hidroksibenzaldehido, įvairių aminų ir merkaptoacto rūgšties, švitinant mikrobangomis. Atliktosios sintezės palygintos su atitinkamais tradiciniais sintezės būdais. Švitinant mikrobangomis, reakcijos vyksta žymiai sparčiau, o reakcijos produktų išeiga yra didesnė. Gautų junginių struktūros patvirtintos elementine ir spektrine (^{1}H-BMR, ^{13}C-BMR ir masių spektrometrija) analizėmis.