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MICROWAVE ABETTED OPERATIONAL AND ENHANCED SYNTHESIS OF -1,3-THIAZOLIDINE-4-ONE EXPRESSING ANTIMICROBIAL ACTIVITY

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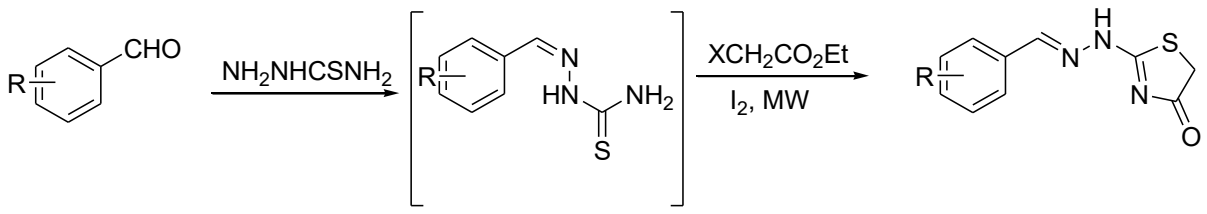
Abstract

An efficient, one pot and facile protocol was developed for the series of 2-benzylidene hydrazinyl-1,3-thiazol-4-(5H)-ones through single pot two stage sequence. Substituted aromatic aldehydes, thiosemicarbazone, and α-haloesters reacted together in presence of catalytic iodine to afford new derivatives of thiozolidinones. The strategy works under mild circumstances and desired products obtained were pure enough without the need of chromatographic separation. A library of 1,3-thiazolidine-4-one's so prepared were tested for their potential regarding antimicrobial activity opposed to *S. aureus, E. coli, B. subtilis*, as well as single strain of fungal, *Candida albicans.* Among different strains studied, compounds, **3b** and **3k** found to be potent selectively against *Staphylococcus aureus* while compounds, **3d** and **3e** have shown good activity selectively against *Bacillus subtilis*.

GRAPHICAL ABSTRACT:



INTRODUCTION:

Thiazoles and their derivatives constitute a privileged class of heterocyclic scaffolds in chemistry of medicine [1,2], attributable towards their wide spectrum of pharmacological effects, pronounced attraction towards diverse biological

targets, and their prominent role in the development of antimicrobial agents[3]. In continuation of their established significance in medicinal chemistry, thiazole and its derivatives have attracted considerable attention owing to their versatile pharmacological profile[5]. They have been extensively investigated for their therapeutic potential as antidiabetic[6], antitubercular[6], antibacterial[3], antifungal[4], anti-inflammatory[7], and potent anticancer agents exhibiting promising activity against various cancer cell lines[8]. So far numerous studies have been reported on various preparation methodology for thiazol-4(5H)-ones with the help of thioglycolic acid[9], chloroacetic acid[9], chloroacetic acid[9], ethyl chloroacetate[10], which highlights the development of selective and versatile approaches for their preparations. Microwave- assisted approach[11,12] has also been emerged to make the operational conditions straightforward in order to minimize reaction time, accelerate reaction rate and improve upon the yield. It was demonstrated so this preserve to heteroarylidene substituent at place of two thiazolin-4-one ring, coupled with a group of hydrazone at site 2, leads to a marked increase in antimicrobial efficiency[13,14]. Despite the various reports on the synthesis of 1,3-thiazolidin derivatives, there is still scope to find new analogues and methods of preparation.

In continuation with previous reports, we aimed to set an operational and efficient method to design and synthesize novel derivatives of thiazole-4-one linked to benzylidene hydrazinyl at C-2 position. Foroughifar and Ebahimi reported[9] 1,3-thiazolidinone one pot synthesis of utilize $Bi(SCH_2COOH)_3$. The library of newly synthesized 1,3-thiazolidine-4-one was evaluated for their antimicrobial potential for multiple forms of bacteria – *S. aureus, B. subtilis E. coli* as well as one fungal strain, *Candida albicans*.

EXPERIMENTAL:

MATERIALS AND METHODS

Chemicals and solvents received from commercial sources were used without further purification. ^{1}H NMR spectra and ^{13}C NMR spectra were recorded on Bruker 400 MHz and 100 MHz spectrometer respectively. Coupling constants (*J*) are reported in hertz (Hz) and chemical shifts are reported in parts per million (δ). Melting points were determined using a Thomas Hoover capillary melting point apparatus and uncorrected. Exact mass measurements were performed on Bruker impact HD Q-TOF analyzer in the ESI mode. IR spectra were recorded by a Shimadzu FT-IR 8400 Spectrometer. The routine monitoring of reactions were performed using TLC

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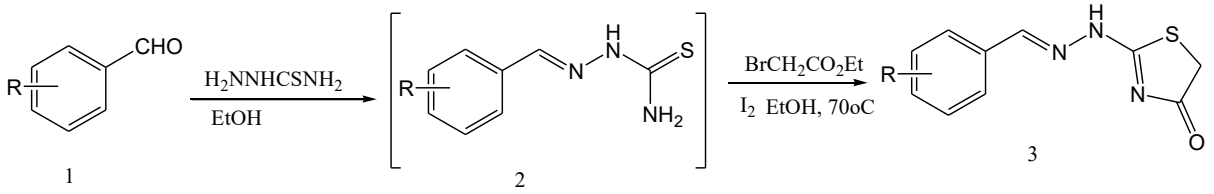
(Merck kieselgel 60 0.20 mm layer, UV254).

GENERAL PROCEDURE:

Thiosemicarbazide (455 mg, 5 mol) was introduced into a solution containing substituted benzaldehyde (530 mg, 5 mol) in anhydrous EtOH (15 ml) then obtained the refluxed reaction mixture 1.5h for assure complete consumption of benzaldehyde (TLC check). To this catalytic iodine was mixed after the adding of ethyl chloroacetate (615 mg, 5 mmol) dissolved in ethanol using pressure equalizing funnel over the period of 10 minutes. The reaction mass was continued to stir under reflux for 4-5 h (TLC check). Thereafter cooling at ambient temp. following by pouring into 50 mL of chilled water leading to solid formation. obtained product accumulated by filtration, gave washing of water and dried. After the recrystallization from ethanol desired 1,3-thiazolidine-4-one was obtained 940 mg, 86 %, in pure form for spectral analysis.

RESULT AND DISSCUSION:

The methodology worked smoothly under mild circumstances without isolation of an intermediate, 2-benzylidenehydrazine-1-carbothioamide and the use of either acid or base. After the successful preparation of 1,3-thiazolidine-4-one from benzaldehyde, the scope and feasibility of methodology was tested with substituted benzaldehydes [**Scheme 1**]. It is worth to note that electron withdrawing substituents in benzene nucleus requires less reaction time, results better yield in comparison with electron releasing substituents [15]. This may be attributed to the acceleration of first step. Also, in presence of catalytic iodine, both ethyl chloroacetate and ethyl bromoacetate react equally without affecting the yield of desired products and reaction time. Attempt to conduct this reaction sequence without catalytic iodine, resulted moderate to poor yield and required longer duration of stirring at elevated temperature [16].



Scheme 1: Preparation Of 2-Benzylidene Hydrazinyl Thiazol-4(5h)-One

Meanwhile, the methodology was reduplicated into microwave keeping using identical reaction state till affect the same transformations. compared conventional method to microwave method accomplished the same transformation and found to be superior in terms of yields (75-92%) and reaction time (10-20 min). The comparative studies for both these methods are shown in Table-1.

Table 1: Conventional and Microwave Assisted Synthesis Of 1,3-Thiazol-4(5H)-One

Sr. No	Entry	R	Conventional mode, Duration (h)	Produce	Microwave mode[a] Duration (min)	Produce
1	3a	4-OH	5	79	15	82
2	3b	4-Cl	5	82	15	90
3	3c	4-Ome	5	79	12	84
4	3d	3,4(OCH_3)$_2$	5	71	20	79
5	3e	4-N(CH_3)$_2$	5	70	20	75
6	3f	H	4	78	10	86
7	3g	4-Br	5	81	15	88
8	3h	4-NO_2	5	80	12	89
9	3i	3-OH	5	80	15	84
10	3j	4-CH_3	5	74	12	82
11	3k	3,4-(Cl)$_2$	5	84	15	92
12	3l	4-F	5	81	10	87

[a]All the synthesis were carried out in 20 mL water under MW at 100^{0}C with the power of 420W

All these synthesized samples were analyzed by FTIR, ^{1}H & ^{13}C NMR and mass spectrometry. A library of synthesized 1,3-thizol-4(5H)-one has been then tested for regarding its potential of microbial inhibition efficacy by the serial broth dilution method. various strains of bacterial, *Escherichia coli* (NCIM2065), *Staphylococcus aureus* (NCIM976), *Bacillus subtilis* (NCIM2063) were tested using streptomycin as positive control while one fungal strain, *Candida albicans* (NCIM3100) was tested using itraconazole and clotrimazole as positive controls[17]. In both the cases, DMSO acted as negative control. Activity of each compound was compared with streptomycin as standard. Among the compounds, **3b** and **3k** have

shown comparable activity against streptomycin against *E. coli*, while compounds, **3d** and **3e** have shown good activity selectively against *Bacillus subtilis.* All other compounds have shown moderate to low activity against three bacterial strains as well as fungal strain, *Candida albicans.*

Table 2: Antimicrobial Activity Of 1,3-Thiazolidinone Derivatives

Comp. no /sample	Inhibition Zone (mm); (average of triplicates)					
	Tested Microorganism (sample in DMSO, mg/mL)					
	mg analyte	DMSO (mL)	Ec*	Sa*	Bs*	Ca*
3a	3	3	12.0	9.6	10.0	9.2
3b	3	3	19.7	9.1	8.4	10.0
3c	3	3	11.3	9.3	8.4	9.2
3d	3	3	15.1	11.6	17.1	8.5
3e	3	3	13.6	10.2	14.8	8.4
3f	3	3	9.4	9.0	11.1	9.6
3g	3	3	10.9	10.0	9.8	8.6
3h	3	3	9.5	10.7	9.0	9.3
3i	3	3	9.8	8.7	9.2	8.4
3j	3	3	12.4	9.1	10.0	9.2
3k	3	3	20.3	11.5	9.8	9.3
3l	3	3	10.7	9.8	10.1	9.4
DMSO (negative control)	-	3	7	7	7	7
Streptomycin(positive control)	3	3	23.6	28.6	27.0	-
Itraconazole (positive control)	3	3	-	-	-	13.3
Clotrimazole (positive control)	3	3	-	-	-	18

Bs*: *B. subtilis;* Ca*: *C. albicans* Ec*: *E. coli;* Sa*: *S. aureus*

SPECTRAL DATA:

2-(2-(4-hydroxybenzylidene) hydrazinyl) thiazol-4(5H)-one (3a): white crystalline, mp 148-152°C (EtOH) mp 149-151°C (EtOH); IR (KBr Pellet, ν, cm^{-1}): 3633 (OH), 1712 (C=O), 1613 (C=N, schiff base), 1442 (C=N), 746 (CSC); ^{1}HNMR (400MHz, DMSO-d6): δ 3.87 (s, 2H), 6.84 (d, 1H, *J* = 7.1Hz), 7.54 (d, 1H, *J*= 7.0 Hz), 8.21 (s, 1H), 10.03 (s, 1H), 11.82 (s, 1H); ^{13}CNMR (100MHz, DMSO-d6): δ 40.3, 55.8, 114.0, 115.9, 121.5, 127.4, 142.3, 149.3, 151.2, 161.5, 197.4; HRMS: found, m/z: 235.067 $[M]^{+}$. $C_{10}H_8N_3O_2S$. calculated, m/z: 235.0697

2-(2-(4-chlorobenzylidene) hydrazinyl) thiazol-4(5H)-one (3b): white crystalline, mp 136-139 °C (EtOH) mp 138-139 °C (EtOH); IR (KBr Pellet, ν, cm^{-1}): 3217 (NH), 1702 (C=O), 1665 (C=N, schiff base), 1549 (C=N), 610 (CSC); ^{1}HNMR (400MHz, DMSO-d6): δ 3.88 (s, 2H), 7.44 (d, 1H, *J* = 7.1Hz), 7.74 (d, 1H, *J*= 7.0 Hz), 8.31 (s, 1H), 12.03 (s, 1H); ^{13}CNMR (100MHz, DMSO-d6): δ 34.3, 124.0, 125.9, 132.5, 136.4, 152.3, 169.3, 187.4; HRMS: found, m/z: 253.127 $[M]^{+}$. $C_{10}H_7N_3OSCl$. calculated, m/z: 253.1297

2-(2-(4-methoxybenzylidene) hydrazinyl) thiazol-4(5H)-one (3c): white crystalline, mp: 128-131 °C (EtOH) mp 129-131 °C (EtOH); IR (KBr Pellet, ν, cm^{-1}): 3233 (NH), 1708 (C=O), 1610 (C=N, schiff base), 1531 (C=N), 796 (CSC); ^{1}HNMR (400MHz, DMSO-d6): δ 3.75 (s, 3H), 3.82 (s, 2H), 6.94 (d, 1H, *J* = 7.1Hz), 7.64 (d, 1H, *J*= 7.0 Hz), 8.31 (s, 1H), 11.92 (s, 1H); ^{13}CNMR (100MHz, DMSO-d6): δ 33.3, 55.8, 114.8, 126.4, 132.3, 157.2, 163.5, 187.9; HRMS: found, m/z: 249.427 $[M]^{+}$. $C_{11}H_{10}N_3O_2S$. calculated, m/z: 249.4297

2-(2-(3,4-Dimethoxybenzylidene) hydrazinyl) thiazol-5(4H)-one (3d): pale brown solid, mp 241-243 °C (EtOH) ; IR (KBr Pellet, ν, cm^{-1}): 3622 (N-H), 1701 (C=N, schiff base), 1610 (C=O), 1448 (C=N), 742 (CSC); ^{1}HNMR (400MHz, DMSO-d6): δ 3.79 (s, 3H), 3.80 (s, 3H), 3.87 (s, 2H), 7.03 (d, 1H, *J* = 7.1Hz), 7.29 (d, 1H, *J*= 7.0 Hz),7.35 (s, 1H), 8.31 (s, 1H), 11.9 (bs, 1H); ^{13}CNMR (100MHz, DMSO-d6): δ 40.3, 55.8, 114.0, 115.9, 121.5, 127.4, 142.3, 149.3, 151.2, 161.5, 197.4; HRMS: found, m/z: 279.066 $[M]^{+}$. $C_{12}H_{12}N_3O_3S$. calculated, m/z: 279.0677

2-(2-(N,N-dimethylaminobenzylidene) hydrazinyl) thiazol-5(4H)-one (3e): yellow Solid; m.p. 196-198 °C (EtOH); IR (KBr Pellet, ν, cm^{-1}): 3728(N-H), 1703 (C=N Schiff base), 1589 (C=O), 1506 (C=N), 729 (CSC); ^{1}H NMR (400 MHz, DMSO-d6): δ 2.99(s, 6H), 3.44 (s, 1H), 6.71 (d, 2H, *J* = 6.8 Hz), 7.50 (d, 2H, *J* = 6.7 Hz), 8.08 (s, 1H); ^{13}CNMR (100MHz, DMSO-d6): δ 39.4, 40.1, 112.3, 123.8, 128.0, 142.2, 150.6, 160.4, 199.0; HRMS: found, m/z: 262.330 $[M]^{+}$. $C_{12}H_{13}N_4OS$. calculated, m/z: 262.298.

2-(2-benzylidenehydrazinyl) thiazol-4(5H)-one (3f): white crystalline, mp: 252-254 ℃ (EtOH) mp 254-256 ºC (MeOH); IR (KBr Pellet, ν, cm^{-1}): 3728(N-H), 1709 (C=N Schiff base), 1649 (C=O), 1584 (C=N), 754 (CSC); ^{1}H NMR (400 MHz, DMSO-d6): δ 3.91(s, 2H), 7.55 (d, 2H, *J* = 6.8 Hz), 7.70 (d, 2H, *J* = 6.7 Hz), 8.48 (s, 1H), 11.97 (s, 1H); ^{13}CNMR (100MHz, DMSO-d6): δ 33.4, 128.3, 129.8, 131.0, 134.2, 156.6, 175.4, 199.0; HRMS: found, m/z: 220.340 $[M]^{+}$. $C_{10}H_8N_3OS$. calculated, m/z: 220.398.

2-(2-(4-bromobenzylidene) hydrazinyl) thiazol-4(5H)-one (3g): white crystalline, mp 133-136 ℃ (EtOH) mp 132-134 ºC (EtOH); IR (KBr Pellet, ν, cm^{-1}): 3215 (N-H), 1712 (C=N, schiff base), 1601 (C=O), 1465 (C=N), 743 (CSC); ^{1}HNMR (400MHz, DMSO-d6): δ 3.86 (s, 2H), 7.65 (d, 1H, *J* = 7.1Hz), 7.72 (d, 1H, *J*= 7.0 Hz),8.35 (s, 1H), 12.01 (s, 1H); ^{13}CNMR (100MHz, DMSO-d6): δ 33.1, 122.3, 127.4, 128.4, 131.1, 142.4, 149.6, 161.3, 174.1; HRMS: found, m/z: 298.036 $[M]^{+}$. $C_{10}H_7N_3OSBr$. calculated, m/z: 298.0357

2-(2-(4-nitrobenzylidene) hydrazinyl) thiazol-4(5H)-one (3h): white crystalline, mp 143-146 ℃ (EtOH) (mp 145-146 ºC (EtOH); IR (KBr Pellet, ν, cm^{-1}): 3213 (N-H), 1709 (C=N, schiff base), 1602 (C=O), 1478 (C=N), 642 (CSC); ^{1}HNMR (400MHz, DMSO-d6): δ 3.92 (s, 2H), 8.03 (d, 1H, *J* = 7.1Hz), 8.18 (d, 1H, *J*= 7.0 Hz),8.25 (s, 1H), 11.91 (s, 1H); ^{13}CNMR (100MHz, DMSO-d6): δ 42.1, 52.2, 114.3, 114.4, 122.4, 127.1, 142.4, 149.6, 151.8, 161.3, 192.1; HRMS: found, m/z: 246.026 $[M]^{+}$. $C_{10}H_7N_4O_3S$. calculated, m/z: 246.0257

2-(2-(3-hydroxybenzylidene) hydrazinyl) thiazol-4(5H)-one (3i): white crystalline, mp 197-200 ℃ (EtOH) mp 196-198 ºC (EtOH); IR (KBr Pellet, ν, cm^{-1}): 3446 (OH), 1702 (C=N, schiff base), 1699 (C=O), 1589 (C=N), 759 (CSC); ^{1}HNMR (400MHz, DMSO-d6): δ 3.84 (s, 2H), 6.83 (t, 2H),7.56 (m, 2H), 8.35 (s, 1H), 11.86 (s, 1H); ^{13}CNMR (100MHz, DMSO-d6): δ 32.4, 37.4, 129.5, 138.4, 140.3, 150.3, 159.2, 161.4, 179.5; HRMS: found, m/z: 235.066 $[M]^{+}$. $C_{10}H_8N_3O_2S$. calculated, m/z: 235.0677

2-(2-(4-methylbenzylidene) hydrazinyl) thiazol-4(5H)-one (3j): pale yellow Solid, mp 265-258 ℃ (EtOH) mp 268-270 ℃ (DMF:MeOH); IR (KBr Pellet, ν, cm^{-1}): 1709 (C=N, schiff base), 1629 (C=O), 1569 (C=N), 739 (CSC); ^{1}HNMR (400MHz, DMSO-d6): δ 2.90 (s, 3H), 3.88 (s, 2H), 7.27 (t, 2H),7.65 (m, 2H), 8.35 (s, 1H), 11.94 (s, 1H); ^{13}CNMR (100MHz, DMSO-d6): δ 22.4, 32.4, 127.5, 128.4, 130.3, 140.3, 156.2, 165.4, 173.5; HRMS: found, m/z: 234.066 $[M]^{+}$. $C_{11}H_{10}N_3OS$. calculated, m/z: 234.0677

2-(2-(3,4-dichlorobenzylidene) hydrazinyl) thiazol-4(5H)-one (3k): white crystalline, mp 302-306 °C (EtOH) mp 306-308 °C (DMF:MeOH); IR (KBr Pellet, ν, cm^{-1}): 1711 (C=N, schiff base), 1634 (C=O), 1579 (C=N), 749 (CSC); ^{1}HNMR (400MHz, DMSO-d6): δ 3.90 (s, 2H), 7.74 (t, 2H),7.95 (m, 2H), 8.13 (s, 1H), 12.04 (s, 1H); ^{13}CNMR (100MHz, DMSO-d6): δ 33.4, 126.5, 128.4, 130.3, 131.3, 131.2, 132.4. 153.2, 173.5; HRMS: found, m/z: 287.066 $[M]^{+}$. $C_{10}H_6N_3OSCl_2$. calculated, m/z: 287.0677

2-(2-(4-fluorobenzylidene) hydrazinyl) thiazol-4(5H)-one (3l): yellow Solid, mp 282-284 °C (EtOH) mp 280-282 °C (DMF:MeOH); IR (KBr Pellet, ν, cm^{-1}): 1714 (C=N, schiff base), 1638 (C=O), 1583 (C=N), 754 (CSC); ^{1}HNMR (400MHz, DMSO-d6): δ 3.92 (s, 2H), 7.32 (t, 2H),7.82 (m, 2H), 8.42 (s, 1H), 11.96 (bs, 1H); ^{13}CNMR (100MHz, DMSO-d6): δ 33.5, 126.5, 128.4, 130.3, 131.3, 152.2, 164.5, 173.4; HRMS: found, m/z: 238.066 $[M]^{+}$. $C_{10}H_7N_3OSF$. calculated, m/z: 238.0677

CONCLUSION:

In summary, newly developed method provides a reliable, time- saving, and eco-friendly access for 1,3-thiazolidine-4-one derivatives. Microwave assisted synthesis offers better yield and requires short reaction time than conventional heating methods. Considering its operational simplicity and scalability, this approach holds promise for the preparation of biologically significant heterocyclic frameworks.

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CONFLICT OF INTERESTS:

The authors declare that there is no competing interest.

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