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SYNTHESIS, CHARACTERIZATION AND BIO-EVALUATION OF SCHIFF'S BASE PROMOTED BY CSA

S. Srinivasarao*[1], M. Someswara Rao[1], Krishnarao[1]

[1*]Department of Organic Chemistry, PRISM PG and DG College (Affiliated to Andhra University), Visakhapatnam, Andhra Pradesh, India.

ABSTRACT

In this investigation, the protocol followed by preparation of a novel derivatives of the sniffs base promoted by organic acid catalyst. The compounds.(E)-N-(5-((2-(4-chlorophenyl)-1H-benzo[d]imidazol-1-yl)methyl)-1, 3, 4-thiadiazol-2-yl)-1-phenyl meth animine (7a-7f) can be obtained from the mixture of 5-((2-(4-chlorophenyl)-1H-benzo [d] imidazol-1-yl) methyl)-1, 3, 4-thiadiazol-2-amine (5), substituted aromatic aldehyde (6) in the presence of dehydrating agent camphorsulphonicacid (CSA) as a catalyst in ethanol at 70^{o}C. The compound (5) can be prepared by the mixture of 1-(2-(4-bromophenyl)-5-methyl-1H-benzo[d]imidazol-1-yl)-2-chloroethan-1-one (4) and thiourea in Toluene and con H2SO4. The compound (4) can be synthesized from 2-(4-chlorophenyl)-1H-benzo [d] imidazole (3) with Chloro acetic acid and triethylamine is base and MDC as solvent at 350^{o}C. The 2-(4-chlorophenyl)-1H-benzo [d] imidazole (3) is obtained by the 4-Chloro benzaldehyde (2) and O-phenyl diamine (1) in the presence of ZrOCl2 in ethanol. All the newly obtained derivatives were evaluated by the advanced spectroscopic analysis such as ^{1}HNMR, ^{13}CNMR and LCMS and structural determination of titled analogous were calculated by elemental analysis. In addition to the newly synthesized compounds were examined by their anti-microbial activity.

KEYWORDS

2-(4-chlorophenyl)-1H-benzo [d] imidazole, 1-(2-(4-bromophenyl)-5-methyl-1H-benzo [d] imidazol-1-yl)-2-chloroethan-1-one, Thiosemicarbazide, 1, 3, 4-thiadiazol-2-amine, substituted aromatic carboxylic acid, CSA, Schiff's base and Anti-microbial activity.

Author for Correspondence:
Srinivasarao S,
Department of Organic Chemistry,
PRISM PG and DG College,
Visakhapatnam, Andhra Pradesh, India.
Email: naallakrishnarao@gmail.com

INTRODUCTON

The exploration of new heterocycles exhibiting an important broad range of significant biological activities is fascinating scientific endeavors. Benzimidazole is a heterocyclic aromatic organic compound. It is an important pharamacophores and a fortunate structure in medicinal chemistry. The

general synthesis of benzimidazoles and its derivatives involves condensation of phenylene diamine with substituted aromatic aldehyde. The different benzimidazoles derivatives are associated with a wide range of biological activities Pharmacological activities[1-6], Antimicrobial activity[7-12], anticonvulsant, analgesic[13], anthelmintic activity[14], Antioxidant activities[15,16], Antiviral[17], Anticancer[18-20] and analgesic activity[21]. A Schiff base (or azomethine) is an important functional group that contains a carbon nitrogen double bond with the nitrogen atom directed with aryl or alkyl group but not hydrogen. Schiff bases are usually synthesized from the condensation of primary amines and aldehydes or ketones and Schiff bases have been reported to possess antimicrobial activities. Schiff bases are analyzed by the −N=CH− (imines) group which is an important for elucidating the mechanism of transamination and racemization reactions in biological systems.

Our progress work to understand the role of fine electronic variations on molecular activity and the effect of substituent location in (E)-N-(5-((2-(4-chlorophenyl)-1H-benzo [d] imidazol-1-yl)methyl)-1, 3, 4-thiadiazol-2-yl)-1-phenylmethanimine. Schiff bases on the absorption spectra in organic solvents of changeable polarities and in vitro anti-microbial activity of titled analogous (7a-f) were examined by cup plate drugs biological method, various pathogenic strains Viz. S.aureus, B. substills (Gram-+ve), *S. typhi and E.coli* (Gram-ve) were employed using standard drug streptomycin for antibacterial growth respectively.

METHODS AND MATERIAL

Experimental

All the analytical chemicals and synthetic grade reagents purchased from Fine chemicals and were used without further purification. The reaction progress was monitored by thin layer chromatography. The melting point of the all the newly synthesized compounds were determined open at one end and were uncorrected using an Electrochemical Mk3 apparatus. ^{1}HNMR and ^{13}CNMR spectrum were recorded on 400MHz Bruckner spectrometer in $CDCl_3$ as a solvent and chemical shift values are recorded in units δ(ppm) relative to tetramethylsilanes (Me4Si) as an internal standard. Molecular mass of the synthesized compound were determined by LCMS spectrometer

2-(4-chlorophenyl)-1H-benzo [d] imidazole

Take dry and clean four neck 50mL RBF and mixture of chlorobenzaldehyde (1.125mmol) was added to a stirred solution of 1, 2-phenylenediamine (1.125mmol) and $ZrOCl_2$ (2.5mmol) in ethanol (25ml) for five minutes at reflux and Stirring was continued for two hours. The progress of the reaction was checked with help of TLC. After completion of the reaction (TLC, eluent Hexane: ethylacetae 5:5), the solvent was removed under reduced pressure and extracted with ethyl acetate three washings and the organic layer was washed with Braine water (25ml). Organic layers were separated and the organic layer was dried over sodium sulfate. The solvent was removed under reduced pressure and the crude product was subjected to column chromatography using petroleum ether: EtOAc (6:4), which gave desired product as a solid in 95% yield.

Pale pink compound; Yield- 95%; M.p-136^{o}C; ^{1}HNMR (400MHz, $CDCl_3$) δppm-12.107 (s, 1H, CONH), 8.217-8.078 (m, 4H, Ar-H), 7.540-7.352 (m, 4H, Ar-H), ^{13}CNMR (100MHz, $CDCl_3$) δppm: 151.99, 141.14, 139.44, 130.21, 128.24, 128.85, 127.85, 127.64, 122.68, 118. 37, 116.65; LCMS: 230.58 (M+2); Molecular Formula-$C_{13}H_9ClN_2$; Elemental Analysis: Calculated: C-68.32, H-3.98, N-12.25: Obtained: C-68.25, H-3.96, N-12.33.

2-(4-chlorophenyl)-1H-benzo [d] imidazole 2-(2-(4-chlorophenyl)-1H-benzo [d] imidazol-1-yl) acetate

2-(4-chlorophenyl)-1H-benzo [d] imidazole 2-(2-(4-chlorophenyl)-1H-benzo [d] imidazol-1-yl) acetate is dissolved in 25mL of methylene dichloride in 50mL four neck RBF and triethylamine was added. The slowly add the chloroaceticacid lot wise in appropriate time. The total mixture setup on the magnetic stirrer and continued the reaction for 5hrs at reflux. The progress of the reaction was monitored by TLC (5:5 = EtOH: n-hexane).After completion of the reaction, unconsumed

chloroacetyl chloride can be evaporated. The completion of the reaction was identified by the TLC and the crude was taken in a ethylacetae and washed with saturated solution of sodium bi carbonate and separated the organic solvent. The organic solvent distilled off under vacuum distillation final compounds obtained.

Whitesolid; Yield-92%; M.P-187^{o}C 1HNMR (400MHz, CDCl3) δppm-11.664 (s, 1H, COOH), 8.124-7.845 (m, 4H, Ar-H), 7.632-7.435 (m, 4H, Ar-H), 13CNMR (100MHz, CDCl3) δppm: 175.02, 152.54, 141.57, 134.05, 131.77, 129.58, 128.85, 128.51, 124.62, 118.45, 112.62, 50.75; LCMS: 288.71 (M+2); Molecular Formula-$C_{15}H_{11}ClN_2O_2$; Elemental Analysis: Calculated: C-62.84, H-3.87, N-9.77: Obtained: C-62.76, H-3.86, N-9.85.

5-((2-(4-chlorophenyl)-1H-benzo [d] imidazol-1-yl) methyl)-1, 3, 4-thiadiazol-2-amine

The mixture of 1-(2-(4-bromophenyl)-5-methyl-1H-benzo[d]imidazol-1-yl)-2-chloroethan-1-one and thiourea is dissolved in acetic acid in a clean and dry 50mL RBF. The reaction was continued at 60-70^{o}C. The completion of the reaction was identified by the TLC and the crude was taken in a ethylacetae and washed with saturated solution of sodium bi carbonate and separated the organic solvent. The organic solvent distilled off under vacuum distillation final compounds obtained.

Pale-yellow solid;93%; M.P-149^{o}C; ^{1}HNMR (400MHz, $CDCl_3$) δppm: 8.144-8.102 (m, 2H, Ar-H), 7.643-7.330 (m, 6H, Ar-H); 6.587 (s, 2H, -NH); ^{13}C NMR (100MHz, $CDCl_3$) δppm: 169.15, 160.25, 150.58, 140.57, 136.47, 132.97, 130.66, 128.85, 128.04, 127.54, 125.16, 121.74, 119. 58, 31.35; LCMS (m/z); 342.51 (M+2); Molecular Formula-$C_{16}H_{12}ClN_5OS$; Elemental Analysis: Calculated: C-56.22, H-3.54, N-9.20.49: Obtained: C-56.16, H-3.52, N-20.55.

General procedure (E)-N-(5-((2-(4-chlorophenyl)-1H-benzo[d]imidazol-1-yl) methyl)-1, 3, 4-thiadiazol-2-yl)-1-phenylmethanimine

5-((5-bromo-2-(4-methoxyphenyl)-1H-benzo [d] imidazol-1-yl)methyl)-1, 3, 4-thiadiazol-2-amine(1mol) is dissolved in ethanol and camphor sulfonic acid (1.5mol) was added; the mixture was stirred for two hours in room temperature, then substituted aromatic aldehyde (1mol) was added to a mixture and was stirred and heated under reflux in conditions an water bath at 70^{o}C. The progress of reaction was monitored by thin layer chromatography (TLC). After the completion of reaction, cold water was added to the mixture. Then solid crystals were formed at the bottom of the beaker and after that, they were filtered. Finally, the solid product was washed with water, ethanol and n-hexane and dried in desiccator in R.T. The pure derivatives were obtained in good yields.

(E)-N-(5-((2-(4-chlorophenyl)-1H-benzo [d] imidazol-1-yl) methyl)-1, 3, 4-thiadiazol-2-yl)-1-phenylmethanimine (7a)

Whitesolid; Yield-87%, M.P-158-160^{o}C; ^{1}HNMR (400MHz, CDCl3) δppm-8.814 (s, 1H, =CH), 8.113-8.015 (m, 2H, Ar-H), 7.652-7.346 (m, 1H, Ar-H), 4.564 (s, 2H,-CH2); 13CNMR (100MHz, $CDCl_3$) δppm-166.27, 158.02, 151.24, 141.09, 136.04, 133.56, 131.65, 130.09, 129.74, 129.16, 128.95, 128.49, 128.06, 124.87, 122.85, 119.02, 117.94, 50.32; LCMS - 431.72; Molecular Formula-C23H16N5ClS; Elemental Analysis -Calculated: C-64.26, H-3.75, N-16.29; Obtained: C-64.20, H-3.73, N-16.35.

(E)-4-(((5-((2-(4-chlorophenyl)-1H-benzo [d] imidazol-1-yl) methyl)-1, 3, 4-thiadiazol-2-yl) imino) methyl) phenol (7b)

Whitesolid; Yield-95%, M.P-167-169^{o}C; 1H NMR (400MHz, $CDCl_3$) δppm, 9.346 (s-OH, 1H), 8.756 (s, 1H, =CH), 8.152-8.046 (m, 2H, Ar-H), 7.694-7.482 (m, 10H, Ar-H), 4.594 (s, 2H, -CH_2); 13C NMR (100 MHz, CDCl3) δppm-165.74, 159.11, 156.62, 151.36, 141.19, 138.08, 134.28, 132.06, 129.67, 129.04, 128.77, 128.18, 122.66, 120.2, 118.94, 117.2, 51.75; LC-MS– 447.38; Molecular Formula-$C_{23}H_{16}N_5ClOS$; Elemental Analysis-Calculated: C-61.95, H-3.62, N-15.71 Obtained: C-61.89, H-3.60, N-15.79.

(E)-N-(5-((2-(4-chlorophenyl)-1H-benzo [d] imidazol-1-yl) methyl)-1, 3, 4-thiadiazol-2-yl)-1-(4-methoxyphenyl) methanimine (7c)

Whitesolid; Yield-92%, M.P-187-189^{o}C; 1HNMR (400MHz, CDCl3) δppm-8.846 (s, 1H, =CH), 8.142-8.024 (m, 2H, Ar-H), 7.662-7.421 (m, 10H,

Ar-H), 4.646 (s, 2H, -CH2-), 3.746 (s, 3H, CH3); 13C NMR (100 MHz, CDCl3) δppm- 167.07, 160.84, 157.33, 150.04, 141.45, 136.45, 133.35, 130.15, 129.67, 128.58, 128.17, 127.72, 127.14, 123.09, 122.14, 120.02, 119.41, 56.82, 51.62; LC-MS – 461.29; Molecular Formula-C24H18N5ClOS; Elemental Analysis-Calculated: C-62.67, H-3.94, N-15.23; Obtained: C-62.60, H-3.92, N-15.29

(E)-4-(((5-((2-(4-chlorophenyl)-1H-benzo [d] imidazol-1-yl) methyl)-1, 3, 4-thiadiazol-2-yl) imino) methyl)-N, N-dimethylaniline (7d)

Whitesolid; Yield-88%, M.P-195-197^{o}C; ^{1}HNMR (400MHz, $CDCl_3$) δppm-8.884 (s, 1H, =CH), 8.114-8.014 (m, 2H, Ar-H), 7.674-7.214 (m, 10H, Ar-H), 4.746 (s, 2H,-CH2), 2.462 (s, 6H, 2CH3); ^{13}CNMR (100MHz, $CDCl_3$) δppm-167.24, 158.16, 152.62, 148.74, 141.02, 136.82, 133.07, 130. 42, 129.22, 128.46, 128.18, 126.62, 123.04, 120.62, 119.21, 50.46, 40.74; LCMS (m/z)–474.25; Molecular Formula-$C_{25}H_{21}N_6$ Close Elemental Analysis -Calculated: C-63.48, H-4.48, N-17.77;Obtained: C-63.46, H-4.46, N-17.85.

(E)-1-(4-bromophenyl)-N-(5-((2-(4-chlorophenyl)-1H-benzo [d] imidazol-1-yl) methyl)-1, 3, 4-thiadiazol-2-yl) methanimine (7e)

Whitesolid; Yield-89%, M.P-174-176^{o}C; ^{1}HNMR (400MHz, $CDCl_3$) δppm-8.942 (s, 1H, =CH), 8.142-8.052 (m, 2H, Ar-H), 7.642-7.324 (m, 10H, Ar-H), 4.721 (s, 2H, -CH2); ^{13}CNMR (100MHz, $CDCl_3$) δppm-169.21, 160.04, 152.46, 141.66, 138.24, 135.24, 133.36, 131.02, 129.52, 128.72, 128. 46, 128.42, 126.44, 124.21, 122.02, 120.14, 118.73, 53.02; LCMS (m/z): 497.35 Molecular Formula-$C_{23}H_{15}BrN_5Cl$ S; Elemental Analysis - Calculated: C-54.29, H-2.97, N-13.76 Obtained: C-54.20, H-2.95, N-13.82.

(E)-N-(5-((2-(4-chlorophenyl)-1H-benzo [d] imidazol-1-yl) methyl)-1, 3, 4-thiadiazol-2-yl)-1-(4-nitrophenyl) methanimine (7f)

Yellowsolid; Yield-85%, M.P-178-180^{o}C; ^{1}HNMR (400MHz, $CDCl_3$) δppm-8.964 (s, 1H, =CH), 8.352-8.024 (m, 6H, Ar-H), 7.692-7.356 (m, 6H, Ar-H), 4.841 (s, 2H, -CH_2); ^{13}CNMR (100MHz, $CDCl_3$) δppm-168.79, 161.42, 151.64, 148.42, 142.76, 141.66, 136.25, 133.37, 129.62, 128.75, 128.42, 127.62, 125.74, 123.02, 121.72, 119.62, 118.34, 50.24 LC-MS–476.18 (M+2); Molecular Formula-$C_{23}H_{15}N_6ClO_2S$; Elemental Analysis - Calculated: C-58.17, H-3.18, N-17.70 Obtained: C-58.10, H-3.16, N-17.78.

RESULTS AND DISCUSSION

Chemistry

In this investigation, the protocol followed by preparation of a novel derivatives of the sniffs base promoted by organic acid catalyst. The compounds. (E)-N-(5-((2-(4-chlorophenyl)-1H-benzo [d] imidazol-1-yl) methyl)-1, 3, 4-thiadiazol-2-yl)-1-phenyl meth animine (7a-7f) can be obtained from the mixture of 5-((2-(4-chlorophenyl)-1H-benzo [d] imidazol-1-yl) methyl)-1,3,4-thiadiazol-2-amine (5), substituted aromatic aldehyde (6) in the presence of dehydrating agent CSA as a catalyst in ethanol at 70^{o}C. The compound (5) can be prepared by the mixture of 1-(2-(4-bromophenyl)-5-methyl-1H-benzo[d] imidazol-1-yl)-2-chloroethan-1-one (4) and thiourea in Toluene and con H_2SO_4. The compound (4) can be synthesized from 2-(4-chlorophenyl)-1H-benzo [d] imidazole (3) with Chloro acetic acid and triethylamine is base and MDC as solvent at 35^{0}C. The 2-(4-chlorophenyl)-1H-benzo[d]imidazole (3) is obtained by the 4-Chloro benzaldehyde (2) and O-phenyl diamine (1) in the presence of $ZrOCl_2$ in ethanol.

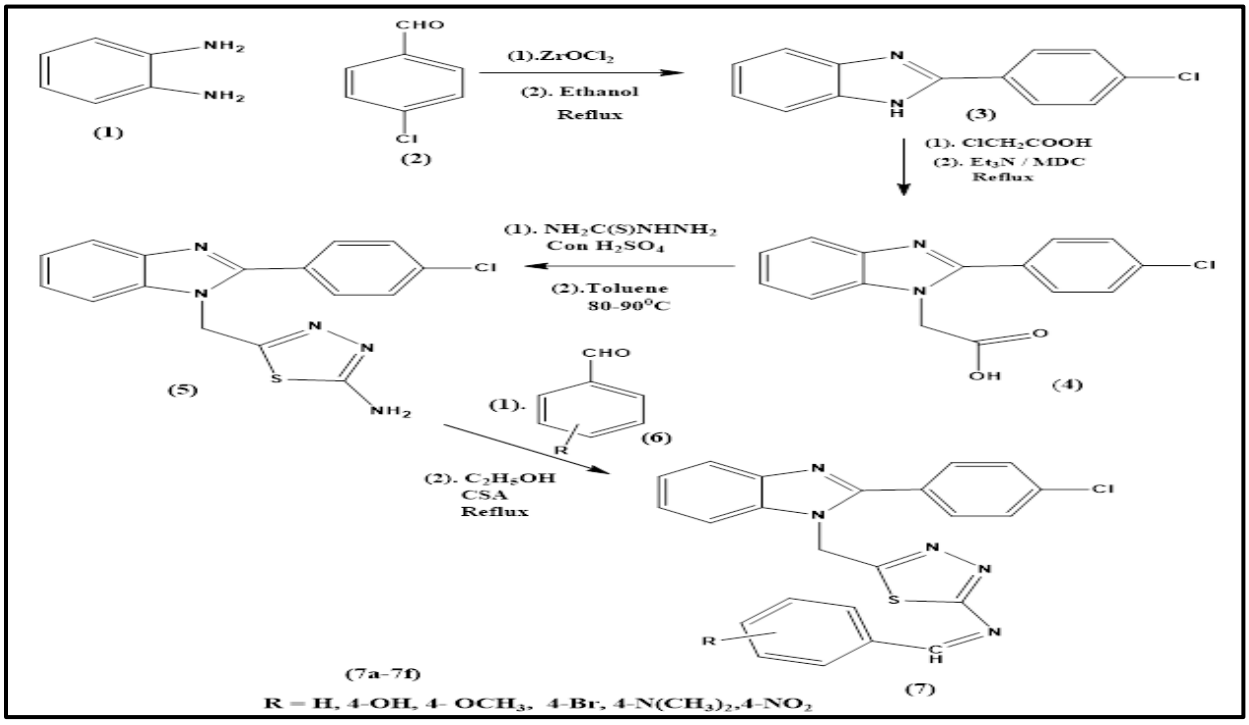
Characterization of the compounds

The structures of the desired analogous (7a-7f) compounds were characterized by ^{1}HNMR, ^{13}C NMR, mass spectral and elemental analyses. Similarly, 1H NMR spectrum of the titled derivatives showed in various aromatic protons appears at δ 7.054ppm and aromatic protons benzimidazoles appear at δ 8.214. The methoxy protons showed at 3.746. The olefin protons appear at δ 8.9424 and N-H protons appeared at 12.107 of imidazole and also appeared at protons of COOH group of OH at 11.664 and 9.346. The mass spectrum of "7e" showed molecular ion peak at m/z = 497.356 (M+2), which is in agreement with the molecular formula$C_{23}H_{15}BrN_5ClS$.

Antimicrobial activity

All the desired compounds were evaluated by anti-bacterial activity as well as antifungal activity. The

electron withdrawing group of compounds and electron releasing group compounds exhibited various potent activities against bacterial as well as fungal strains. Therefore, electron withdrawing group of compounds showed low biological potent activity compared with electron releasing groups. All halogen compounds "d, e, and g" exhibited well to excellent activity. The compounds that containing electron donating group showed to moderate activity as shown in Table No.1.



Scheme No.1

Table No.1: Antimicrobial activity screening activity titled compounds scaffold

S.No	Compound Code	*Zone of inhibition in (mm)					
		Bacteria				Fungi	
		S.aureus	***E.coli***	***S. typhi***	***B.substills***	***A. Niger***	***C. albicans***
1	7a	05	07	07	08	07	06
2	7b	16	15	16	16	14	15
3	7c	20	20	19	18	14	14
4	7d	18	20	20	19	15	16
5	7e	20	21	20	20	15	17
6	7f	10	14	13	17	09	08
7	streptomycin	25	25	22	22	NA	NA
8	Ketoconazole	NA	NA	NA	NA	20	20
9	DMSO	---	----	---	---	---	---

CONCLUSION

The reaction condition carried at reflux condition for all the newly desired synthesized derivatives. The percentage of the titled analogous was obtained from 85-95%. The electron releasing group derivatives group with halogen group of the compound "7b" got maximum yield than that of the derivatives possesses electron attracting group. The rates of the reaction of the desired compounds were developed by catalyst such as CSA. All the compounds are evaluated by anti- microbial activity against gram (+ve), gram (+ve) and fungal. The compound possess halogens exhibited excellent potent active. Otherwise the compounds having electron releasing group which showed better potent active than that of the electron attracting group.

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CONFLICT OF INTEREST

The author declares no conflict of interest.

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