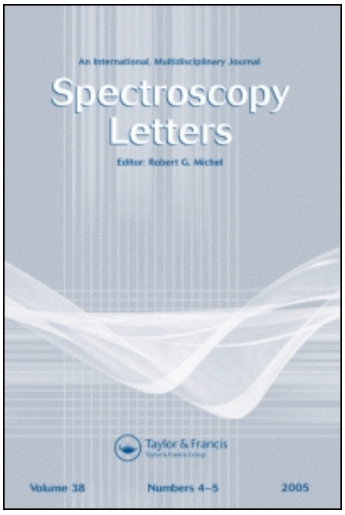

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Microwave-Irradiated and Classical Syntheses of Symmetric Double Schiff Bases of 1,1'-Bis(4-aminophenyl)cyclohexane and their Physicochemical Characterization

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Microwave-Irradiated and Classical Syntheses of Symmetric Double Schiff Bases of 1,1′-Bis(4-aminophenyl)cyclohexane and their Physicochemical Characterization

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Abstract: A series of new symmetric double Schiff bases of 1,1′-bis(4-aminophenyl)-cyclohexane and substituted aromatic benzaldehyde are synthesized by classical and microwave-irradiated techniques. The synthesis time is much shorter and yields of the Schiff bases are found to be better with the microwave-irradiation technique than classical technique. The purity of Schiff bases is checked by TLC. The structures of Schiff bases are supported by FTIR, ^{1}H NMR, and MS techniques. The biological activity of Schiff bases is checked against Gram-positive and Gram-negative microbes. Schiff bases showed moderate antibacterial activity but showed moderate to excellent antifungal activity in comparison with chosen standard drugs.

Keywords: FTIR, ^{1}H NMR, microbial activity, MS, symmetric Schiff base

INTRODUCTION

Schiff bases are most widely used as fine chemicals,[1] medical substrates, and ligands for metal complexes.[2] They are also useful as starting materials for the synthesis of important drugs like antibiotics, antiallergic, antiphologistic, and antitumor agents.[3–5] Kuzmin et al.[6] have reported anticancer activity of a set of macrocyclic Schiff bases based on 2,6-bis(2- and 4-formyl aryloxy methyl)pyridines. They have derived correlation equation between the anticancer activity and structural parameters of the molecules studied. Lozitsky et al.[7]

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have reported 4D-QSAR (Qualitative structure activity relationship) study and anticancer activity of macrocyclic Schiff bases. The Schiff bases were synthesized by the condensation of various aliphatic and aromatic diamines with 2,6-bis(2- and 4-formyl aryloxy methyl)pyridine derivatives. The results of biological activities showed that Schiff bases are active toward cell growth of nine cell cultures of human malignant tumors: leukemia, CNS cancer, prostate cancer, breast cancer, melanoma, small cell lung cancer, colon cancer, ovarian cancer, and renal cancer. They claimed that pyridine ring has a negative influence on anticancer activity, while the presence of the -C═N group with different substituents promotes anticancer activity. Schiff bases based on salicylaldehyde and other hydroxy aldehydes possess unique characteristic properties of improving both antiwear and corrosion inhibition of synthetic lubricating oils and greases. The Schiff base additive can be used for lubricating high-performance aircraft engines having sophisticated bearings and gears to perform high loading and high speeds at high temperatures.[8]

In classical synthesis of Schiff bases from amines and aldehydes or ketones using acid as a catalyst at reflux temperature,[9] solvent removal is a problem, especially dipolar high-boiling aprotic solvents or the isolation of products through liquid–liquid extraction. Recently, Schmeyers et al.[10] have reported solid-phase reaction for Schiff bases for which reaction time is longer. Tanka and Shirashi[11] have improved Schiff base formation in water suspension. The absence of solvent reduces the risk of hazardous explosion when reaction is carried out in a close vessel in a microwave oven.[12]

Recently, microwave-irradiated technique[13–15] has become popular over usual homogeneous and heterogeneous reactions[16,17] because of environmentally friendly, economical, and rapid synthesis procedure. Microwaves enhance the rate of chemical reactions[18–23] and provide pure products in quantitative yield.

The literature survey on Schiff bases of aromatic diamines revealed that no work has been reported on Schiff bases of 1,1′-bis(4-aminophenyl)cyclohexane (BAPC), which encouraged us to synthesize symmetric double Schiff bases by microwave-irradiation and classical methods.

MATERIALS AND METHODS

Reagents and Techniques

Solvents and chemicals used in the current investigation were of LR (laboratory reagent)grade and were used either as such or purified by fractional distillation. The Schiff bases were repeatedly recrystallized from appropriate solvent systems. Melting points were determined in open capillary tubes and were uncorrected. Purity of the compounds was checked by TLC in appropriate solvent system. IR spectra (KBr pellets) were scanned on a Shimadzu FTIR-8400 spectrometer (Japan). The NMR spectra of Schiff bases were

scanned on a Brucker FT NMR (300 MHz) spectrometer (Germany) by using $CDCl_3$ as a solvent and TMS as an internal standard. Mass spectra of the compounds were scanned on a Shimadzu GC-MS-QP 2010 spectrometer (Japan) by using EI (electron impact) (0.7 kV) detector. The ion source temperature was 220°C and interface temperature was 240°C. Microbial activity of the compounds was checked by cup-plate technique.[24]

Synthesis of 1,1′-Bis(4-aminophenyl)cyclohexane

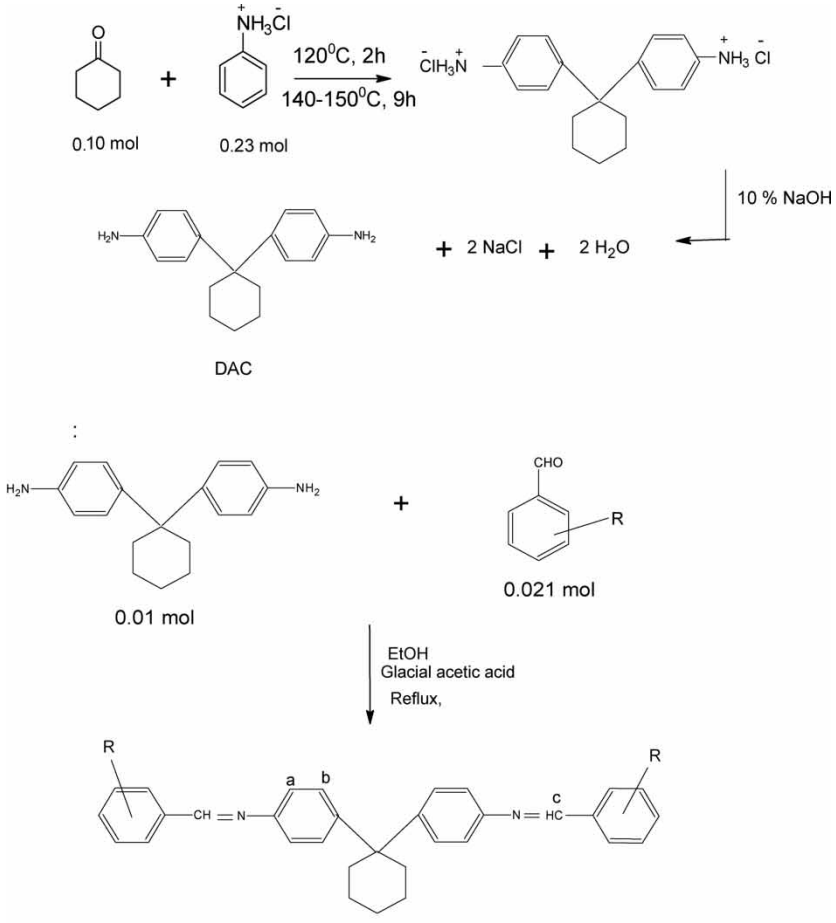
Aromatic diamines can be synthesized by acid-catalyzed condensation of aniline hydrochloride and cyclic ketones.[25–27] Thus, 0.23 mol (29.79 g) aniline hydrochloride and 0.10 mol (9.8 g) cyclohexanone were condensed with stirring at 120°C for 2 hr and then 140–150°C for 9 hr. The resultant solution of 1,1′-bis(4-aminophenyl)cyclohexane hydrochloride was cooled to 120°C, and 50 mL boiling water was added to get a deep-red solution. The solution was refluxed with activated charcoal for 20 min and filtered off charcoal. The clean solution was made alkaline by using 10% NaOH solution. BAPC was filtered, washed well with distilled water, and dried in an oven at 50°C. BAPC was recrystallized repeatedly from benzene–*n*-hexane system to harvest light-brown needle-shaped crystals. The yield was 32% and m.p. 114°C. The purity of BAPC was checked by TLC in $CHCl_3$–*n*-hexane (80:20 v/v) solvent system.

Synthesis of Symmetric Double Schiff Bases by Classical Method

Schiff bases of general Scheme 1 were synthesized by condensing BAPC and substituted aldehydes in ethanol by using glacial acetic acid as a catalyst at reflux temperature for 4 hr. Thus, into a 100 mL RBF (round bottomed flask), 0.01 mol BAPC was dissolved in 15 mL ethanol containing 2 mL glacial acetic acid. To this solution, 0.021 mol aldehyde was dissolved in 10 mL ethanol and added dropwise at room temperature and then refluxed 4–7 hr. The product was isolated from chilled water, filtered, washed well with sodium bisulfite, water, and finally with ethanol and dried at 50°C in an oven. The Schiff bases are soluble in common solvents such as $CHCl_3$, CCl_4, benzene, THF, 1,4-dioxane, DMF, DMSO, and so forth. The Schiff bases were recrystallized repeatedly from appropriate solvent systems, and the purity was checked by TLC in appropriate solvent system. The reaction time and yields are reported in Table 1 along with analytical data of Schiff bases.

Solvent-Free Synthesis of Symmetric Double Schiff Bases by Microwave Irradiation

The microwave-irradiated condensation of BAPC and aldehyde was carried out in a domestic oven (L. G. 360 W, New Delhi, India). BAPC 0.01 mol



SDSB-1	: R =H	SDSB-5	: R =2 – OCH_3
SDSB-2	: R =4 - CH_3	SDSB-6	: R = 4 – OCH_3
SDSB-3	: R = 2 – OH	SDSB-7	: R = 3 – OC_6H_5
SDSB-4	: R = 4 – OH		

Scheme 1.

and 0.02 mol aromatic aldehyde were mixed together at ambient temperature in a 50 mL Erlenmeyer flask. The mixture was subjected to microwaves for an optimized time. The progress of the reaction was monitored by simultaneous TLC in appropriate solvent system, and purity was checked by TLC. The reaction time and yields are reported in Table 1.

Table 1. Analytical and experimental details of Schiff bases SDSB-1 to SDSB-7

Code	Molecular formula	Moleculer weight	Melting point (°C)	Color	Crystal. solvent system	TLC		Classical method		Microwave irradiation	
						Solvent system	R_f value	Time (hr)	% yield	Time (min)	% yield
SDSB-1	$C_{32}H_{30}N_2$	442	208	Brown	A	CF–H (90:10 v/v)	0.63	6	73	5	84
SDSB-2	$C_{34}H_{34}N_2$	470	223	Off white	CF	CF–H (85:15 v/v)	0.82	6	78	6	86
SDSB-3	$C_{32}H_{30}N_2O_2$	474	245	Light yellow	A–W	EA–H (80:20 v/v)	0.78	4.5	68	9	82
SDSB-4	$C_{32}H_{30}N_2O_2$	474	240	Yellowish brown	A	CF–H(70:30 v/v)	0.61	5	70	6	80
SDSB-5	$C_{34}H_{34}N_2O_2$	502	252	Off white	A	EA–H (75:25 v/v)	0.70	6	80	6	89
SDSB-6	$C_{34}H_{34}N_2O_2$	502	256	Light brown	CF–H	EA–H (70:30 v/v)	0.69	6	72	6	86
SDSB-7	$C_{44}H_{38}N_2O_2$	626	261	Off white	CF–H	EA–H (75:25 v/v)	0.65	7	69	9	81

A, acetone; CF, chloroform; EA, ethylacetate; H, *n*-hexane; W, water.

The reaction mechanisms for the formation of diamine and its various Schiff bases are explained as in Scheme 1.

RESULTS AND DISCUSSION

The analytical and experimental details of SDSB-1 to SDSB-7 are reported in Table 1 from which it is clear that the reaction time is much lower for the microwave-irradiation technique and also reaction yield is improved considerably. SDSB-1 to SDSB-7 are soluble in common solvents. Hydroxy Schiff bases (SDSB-3 and SDSB-4) are soluble in ethanol, whereas others are insoluble in ethanol.

IR Spectral Analysis

Infrared spectra were recorded in the range 4000–400 cm^{-1} as KBr pellets. A typical IR spectrum of SDSB-1 is presented in Fig. 1. Important IR absorption bands of the Schiff base ligands are reported in Table 2. The IR spectra of SDSB-3 and SDSB-4 exhibited a broad band at 3441.7 and 3410.7 cm^{-1} due to presence of phenolic OH groups. Phenolic C—O stretching bands for SDSB-3 and SDSB-4 appeared at 1195.8 cm^{-1} and 1165.8 cm^{-1}, respectively. SDSB-5 to 7 showed C-O-C absorption band at 1251.7, 1251.7, and 1219.9 cm^{-1}, respectively. C-H symmetric and

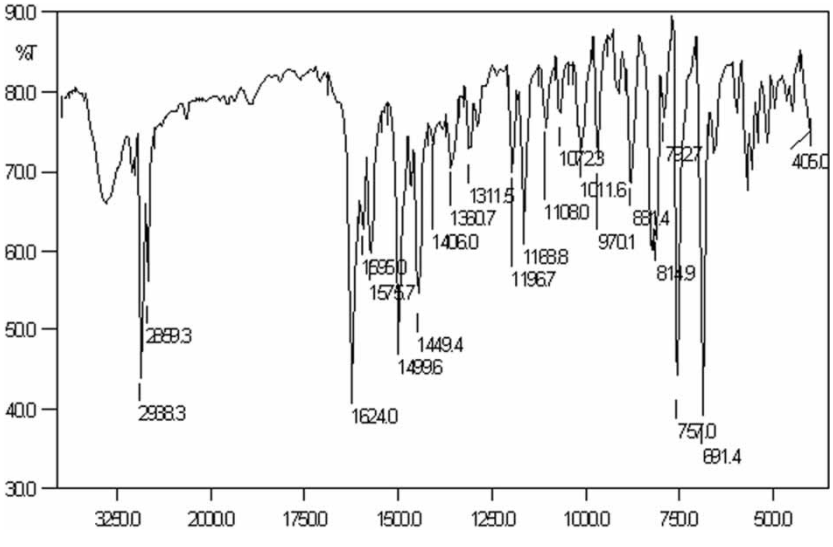


Figure 1. IR spectrum of SDSB-1.

Table 2. Important characteristic IR absorption bands of SDSB-1 to SDSB-7

Schiff base	ν(O-H)	ν(C-H)	ν(CH═N)	ν(C═C)	ν(C-N)	ν(C-O-C)	ν(C-H-oopd)	ν(C-H-ipd)
SDSB-1	—	2938.3 2859.3 m	1624.5 s	1595.0 s	1360.7 m	—	814.9 s	1168.8 1011.6
SDSB-2	—	2930.6 2842.9	1623.5 s	1594.1 s	1311.5 m	—	811.0 s	1199.6 1169.7 1107.1
SDSB-3	3441.7 br	2927.7 2842.9	1616.2 s	1596.9 s	1358.5 w	—	821.6 s	1279.7 1174.6 1115.7
SDSB-4	3410.9 br	2934.5 2856.4	1622.0 s	1600.8 s	1350.1 w	—	824.5 s	1251.7 1165.9 1108.0
SDSB-5	—	2924.8 2842.9	1619.1 s	1593.1 s	1361.7 s	1251.7	826.4 s	1286.4 1159.1 1105.1
SDSB-6	—	2933.5 2855.4	1622.0 s	1601.8 s	1306.7 m	1251.7	825.5 s	1251.7 1165.9 1107.1
SDSB-7	—	2931.6 2855.4	1629.7 s	1580.6 s 1598.9 s	1364.5 m 1318.3 s	1219.9	824.5 m	1256.5 1160.1 1069.5 1011.6

s, strong; m, medium; w, weak; br, broad.

asymmetric absorption bands due to -CH_2 and -CH_3 groups are observed in the range 2939–2925 cm^{-1} and 2860–2843 cm^{-1}, and C-H scissoring and C-H twisting bands are observed in the range 1480–1440 cm^{-1} and 1250 cm^{-1}. Vibration band due to CH═N, C═C, and C═N are observed in the ranges 1630–1617, 1602–1581, and 1364–1307 cm^{-1}, respectively. C-H out-of-plane deformation for aromatic 1,4-disubstitution is observed in the range 827–811 cm^{-1}, and C-H in-plane deformation is observed in the range 1288–1011 cm^{-1}.

NMR Spectral Analysis

A typical NMR spectrum of SDSB-1 is presented in Fig. 2. The chemical shifts, types of protons, and coupling constant (J) of SDSB-1 to SDSB-7 are reported in Table 3. The integrated area under the NMR peaks have revealed the labeled protons in each Schiff base. The solvent $CDCl_3$ appeared at about 7.25–7.24 ppm either as a separate peak or overlapped with peak because of aromatic protons. Thus, NMR spectral data indicated expected number and types of the protons in a given Schiff base molecule.

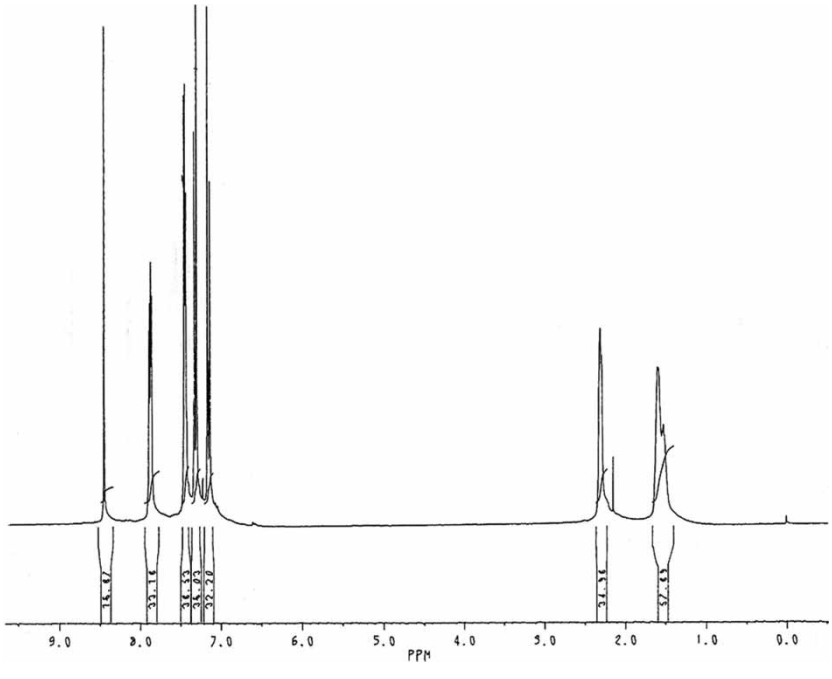
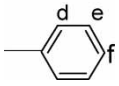
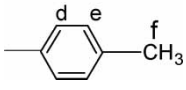
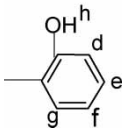
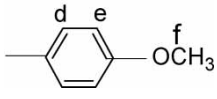
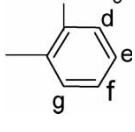


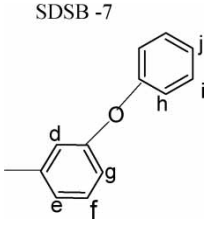
Figure 2. NMR spectrum of SDSB-1.

Table 3. ^{1}H NMR spectral characterization of Schiff bases SDSB-1 to SDSB-7

Sample	NMR chemical shifts (ppm)
SDSB-1 	1.581 (6H s, $\beta + \gamma$, -CH_2), 2.304 (4H s, α, -CH_2) 7.162–7.135 (4H, d, Ar-H_b, J = 10.9) 7.326–7.298 (4H, d, Ar-H_a, J = 8.4) 7.452–7.403 (6H, m, Ar-H_{e+f}, J = 6.5 & 2.7) 7.896–7.856 (4H, m, Ar-H_d, J = 7.4 & 5.4), 8.446 (2H_c s, -CH═N-)
SDSB-2 	1.578 (6H s, $\beta + \gamma$, -CH_2), 2.297 (4H s, α, -CH_2) 2.387 (4H s, f, -CH_3), 7.148–7.120 (4H, d, Ar-H_b, J = 8.3) 7.312–7.231, 7.776–7.749 (8H, m, Ar-H_{d+e}, J = 8.1 & 8.3) 8.408 (2H_c s, -CH═N-)
SDSB-3 	1.59 (6H s, $\beta + \gamma$, -CH_2), 2.301 (4H s, α, -CH_2) 6.936–6.883 (2H, dd, Ar-H_f, J = 8.3) 7.016–6.966 (2H, dd, Ar-H_f, J = 8.7) 7.222–7.187 (4H, d, Ar-H_b, J = 9.1) 7.371–7.319 (8H, m, Ar-$H_{(a+e+g)}$, J = 7.07 & 8.7) 8.578 (2H_c s, -CH═N-), 13.365 (2H, s, Ar-OH (h))
SDSB-5 	1.580 (6H s, $\beta + \gamma$, -CH_2), 2.300 (4H s, α, -CH_2) 3.856 (6H_f, s, -OCH_3), 6.98–6.95 (4H, d, Ar-H_b, J = 8.7) 7.146–7.177 (4H, d, Ar-H_d, J = 8.7) 7.313–7.284 (4H, d, Ar-H_a, J = 8.7) 7.843–7.815 (4H, d, Ar-H_e, J = 8.7), 8.385 (2H_c s, -CH═N-)
SDSB-6 	1.594 (6H s, $\beta + \gamma$, -CH_2), 2.303 (4H s, α, -CH_2) 3.898 (6Hh, s, -OCH_3), 6.962–6.913 (2Hh, d, Ar-H_d, J = 8.4) 7.046–6.998 (2H, t, Ar-H_f, J = 7.4 & 7.7) 7.171–7.143 (4H, d, Ar-H_b, J = 8.5), 7.245 ($CDCl_3$), 7.317–7.267 (4H, d, Ar-H_a, J = 9) 7.441–7.367 (2H, m, Ar-H_e, J = 8.4 & 8.7) 8.147–8.114 (2H, d, Ar-H_g, J = 9.6) 8.916 (2H_c s, -CH═N-)

(*continued*)

Table 3. Continued

Sample	NMR chemical shifts (ppm)
SDSB -7 	1.568 (6H s, $\beta + \gamma$, -CH_2), 2.289 (4H s, α, -CH_2) 7.05–7.024 (4H, d, Ar-H_h, J = 7.7) 7.147–7.094 (8H, m, Ar-H_{g+i+j}) 7.244 ($CDCl_3$), 7.312–7.275 (4H, d, Ar-H_b, J = 5.9) 7.347–7.321 (4H, d, Ar-H_a, J = 9.9) 7.4343–7.374 (2H, d, Ar-H_f, J = 8) 7.541–7.534 (2H, d, Ar-H_d, J = 1.8) 7.608–7.582 (2H, d, Ar-H_e, J = 7.5), 8.397 (2H_c s, -CH═N-)

Mass Spectral Analysis

A typical mass spectrum of SDSB-1 is presented in Fig. 3. Mass spectral fragments of BAPC and SDSB-1 to SDSB-7 are reported in Table 4. The molecular ion peaks M^+, $(M-1)^+$, $(M+1)^+$, $(M+2)^+$, and some of the main fragments are reported in Table 5. An attempt is made to assign each

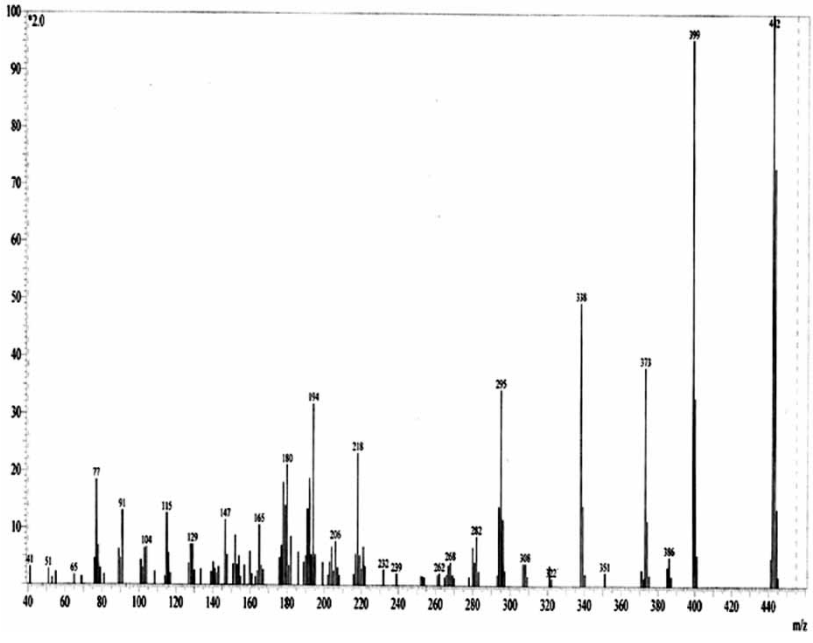


Figure 3. Mass spectrum of SDSB-1.

Table 4. Important mass spectral fragments of BAPC and SDSB-1 to SDSB-7

Schiff base	m/e
BAPC	268, 267, 266, 265, 224, 223, 197, 195, 144, 130, 106, 92
SDSB-1	444, 443, 442, 441, 400, 399, 386, 373, 338, 295, 282, 218, 194, 181, 180, 178, 165, 147, 129, 115, 104, 91, 77
SDSB-2	472, 471, 470, 469, 428, 427, 401, 352, 335, 310, 309, 296, 232, 220, 208, 194, 178, 165, 154, 115, 105, 91, 77
SDSB-3	477, 476, 475, 474, 432, 431, 405, 355, 354, 337, 312, 299, 238, 237, 210, 196, 178, 165, 152, 128, 115, 102, 91, 77, 65, 51, 41
SDSB -4	477, 474, 475, 474, 431, 412, 371, 370, 355, 327, 311, 301, 250, 234, 210, 207, 194, 180, 163, 144, 130, 115, 106, 77, 65, 45
SDSB-5	504, 503, 502, 459, 433, 384, 368, 341, 315, 301, 266, 254, 234, 224, 223, 207, 195, 178, 165, 130, 119, 106, 91,77
SDSB-6	504, 503, 502, 459, 433, 384, 351, 341, 266, 265, 250, 224, 223, 207, 195, 165, 119, 106, 91, 77, 44
SDSB-7	628, 627, 626, 607, 583, 570, 557, 446, 430, 403, 374, 354, 314, 313, 298, 286, 272, 250, 228, 191, 181, 165, 141, 129, 115, 104, 91, 77, 65, 51

fragment mentioned in Table 5 through Scheme 2. Pyrolysis of a compound is a complex process and involves a variety of reactions such as ionization, decomposition, branching, cross-linking, and rearrangement. Upon loss of electron results molecular ion, while loss of azomethine proton gives $(M-1)^+$ ion peak in the mass spectrum. $(M-1)^+$ ion undergoes further fragmentation to give fragments 1 and 2. Fragment 1 further undergoes fragmentation to give fragments 3 and 4. Similarly, fragment 2 produces fragments 4 and 6, while fragment 3 produces fragments 5, 6, and 7. In case of SDSB-4, fragment 4 further decomposes into fragment $C_7H_6O^+$ ($m/e = 106$) and nitrogen radical. Besides main fragments, many more fragments are possible due to the above-mentioned possible reactions. At the end, all fragments are converted into low-molecular-mass gaseous products.

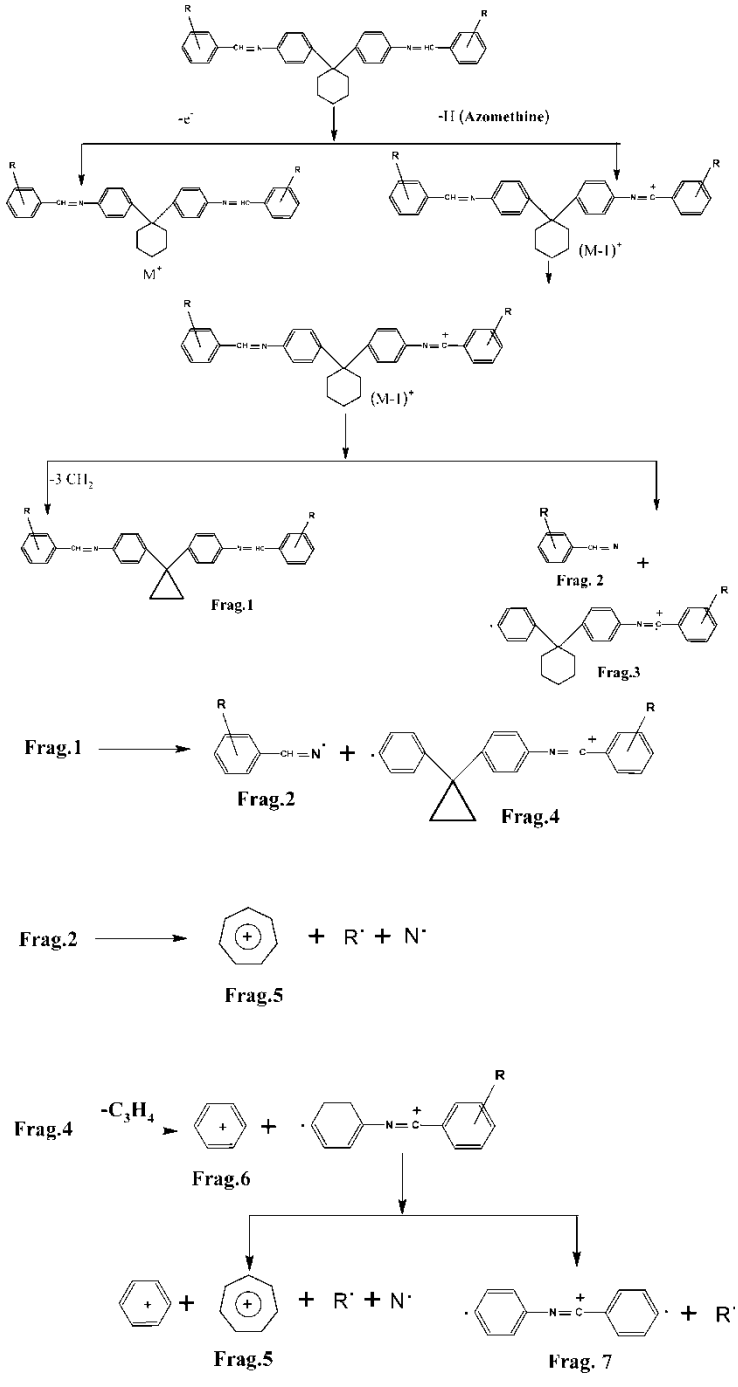
Microbial Activity

All of the seven Schiff bases, the standard drugs, and solvent DMF have been screened for their microbial activity against *Escherichia coli, Bacillus megaterium, Proteus vulgaris, Staphylococcus aureus and Aspergillus niger* by cup-plate method at 37°C. The sample volume was 0.1 mL 50 μg. The plates were incubated for 24 hr at 37°C, and zones of inhibition for each microbial growth were measured relative to control DMF. A comparative zone of inhibition (mm) for Schiff bases and standard drugs (amoxicillin, ampicillin, ciprofloxacin, and erythromycin) are reported in Table 6, from which it is clear that SDSB-1 to SDSB-7 have shown moderate antibacterial

Table 5. Molecular M^+ ion, $(M\text{-}1)^+$ peaks, and some important fragments of SDSB-1 to SDSB-7

Schiff base	M^+	$(M-1)^+$	Fragment 1	Fragment 2	Fragment 3	Fragment 4	Fragment 5	Fragment 6	Fragment 7
SDSB-1	442	441	399	104	338	296	91	77	178
SDSB-2	470	469	427	118	352	309	91	77	178
SDSB-3	474	473	431	120	355	311	91	77	178
SDSB-4	474	473	431	106	355	311	91	77	178
SDSB-5	502	501	459	119 ($-CH_3$)	368	325	91	77	178
SDSB-6	502	501	459	119 ($-CH_3$)	368	325	91	77	178
SDSB-7	626	625	583	196	430	387	91	77	178

M^+, molecular ion peak; $(M-1)^+$, due to loss of azomethine proton.



Scheme 2.

Table 6. Microbial activities of Schiff bases SDSB-1 to SDSB-7

	Zones of inhibition				
Sample	*Escherichia coli*	*Becillus megaterium*	*Bacillus subtilis*	*Proteus vulgaris*	*Aspergillus niger*
SDSB-1	08	09	09	08	12
SDSB-2	10	08	10	09	14
SDSB-3	09	11	09	10	15
SDSB-4	09	10	09	09	15
SDSB-5	10	09	12	11	18
SDSB-6	11	10	12	10	18
SDSB-7	08	09	08	08	17
Amoxicillin	18	19	22	22	18
Ampicillin	17	21	19	23	20
Ciprofloxacin	22	20	21	24	19
Erythromycin	—	—	—	—	18

activity in comparison with standard drugs, while they possess good to comparable antifungal activity except SDSB-1, which showed moderate antifungal activity in comparison with standard drug.

CONCLUSIONS

On the basis of experimental findings, it is concluded that the reaction time is shortened from 4–7 hr to 5–9 min and reaction yield is improved from 69–80% to 80–89% with the use of microwave irradiation. SDSB-1 to SDSB-7 showed moderate antibacterial activity but showed good to comparable antifungal activity in comparison with chosen standard drugs.

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