Original Article: Green Synthesis, Characterization, and Biological Activity of Aryl Azo Schiff Bases

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ABSTRACT

This study reflected novel and eco-friendly methods approving the green synthesis of various Schiff's bases by sonication, microwave irradiation, stirring, and grinding methods using DHE and various aromatic aldehydes. We also offered the synthesis of aryl azo Schiff base in different ways and compared the way of synthesizing it. As a result, sonication and microwave irradiation proved the modest technique to synthesize aryl azo Schiff base. This technique is experimentally green, simple, of greater yielding, clean, and with compact reaction times. Also, the synthesized compounds were tested for their antibacterial activity. The most admirable results were observed and it could be a potential starting point to develop innovative lead compounds fight against a panel of some human disease-causing pathogens bacteria.

Introduction

The idea of green chemistry [1-5] and its uses [6-9] in artificial organic chemistry have developed as key solutions for the growth of sparkling and extra kindly chemical processes. Several techniques and ways have been developed for the synthesis of aryazo Schiff bases. In current years, naturally benign synthetic approaches have received significant attention and Specific solvent-free protocols have been established [10,11]. Schmeyers et al. addressed the solid-state preparation of several kinds of benzylidene aniline derivatives by grinding together benzaldehydes and solid anilines [12]. Varma et al. reported the clay catalyzed synthesis of imines and enamines under solvent-free conditions using microwave irradiation [13]. The growth of ecologically benign solvents and reaction conditions has been highly accentuated both in academic research and the chemical industry. This is mainly owing to the fact that outdated solvents, such as volatile organic solvents, have been implicated in many environmental problems.

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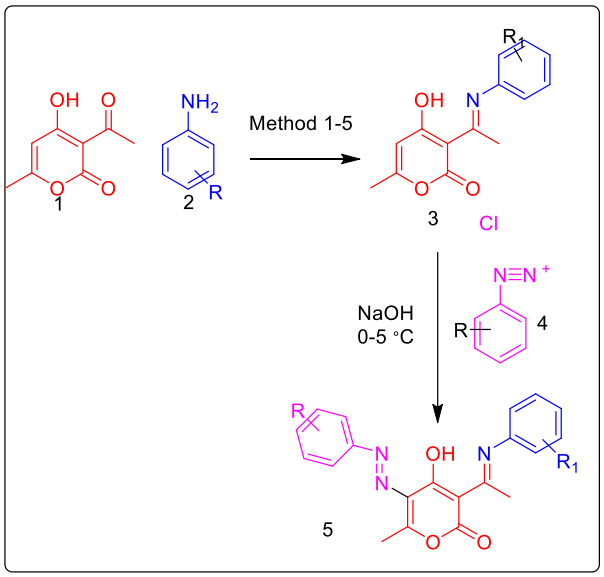
The organic synthesis uses organic solvents like benzene and chlorinated hydrocarbons, which have produced havoc on the environment because of their toxic and volatile nature [14,15]. To reduce such disasters, there is a need to use a safer reaction. Schiff bases, which are prospective chelating ligands in coordination chemistry [16], show favorable uses in drugs as anti-oxidant, antimicrobial, and anti-inflammatory [17-18]. On the industrial measure, they have an extensive variety of applications such as dyes and pigments precursors, and corrosion inhibitors [19-21].

Experimental

Materials and methods:

All the chemicals used for experimental work were of AR-grade. Throughout the experimental work, glass distilled water was used. This was obtained by double distillation of deionized water in presence of crystals of potassium permanganate. For the synthesis of Schiff bases, aryl azo Schiff bases ethanol and methanol were used as solvents. The special-purpose spectroscopic grade solvents were used invariably for all spectral analysis. The reactants such as dehydroacetic acid, aromatic amines and other chemicals used for the synthesis of Schiff bases were of AR grade. The entire experimental work was carried out using a borosilicate glass apparatus. Ultrasonicator, digital Microwave, Mortar and pestle, Rota mantle with a magnetic stirrer, Reflux condenser were employed.



Scheme 1. Synthesis of Schiff base and aryazo Schiff bases

Table 1. Reaction times and yields of various methods for the synthesis Schiff's bases

Methods	Reaction condition	Time	Yield %
1	Microwave irradiation	5 min	90
2	Sonication	40 min	80
3	Stirring	20 h	60
4	Reflux	5 h	75
5	Grinding	45 min	70

General Procedure for the Synthesis of Schiff base: Step-I Synthesis of Schiff bases

Method 1: An equimolar ratio of dehydroacetic acid (2.5 mmol) (1) and the substituted primary aromatic amine (2.5 mmol) (2) were mixed thoroughly in a grinder. The reaction

mixture was then irradiated in the microwave oven by taking 3–4 mL of dry ethanol as a solvent. The reaction was completed in a short time (4–5 min) with better yields compared with the conventional procedure. The resulting product was then recrystallized from ethanol and finally dried under reduced pressure over anhydrous $CaCl_2$ in a desiccator. The progress of the reaction and purity of the product was monitored by TLC using silica gel G.

Method 2: Ultrasound-assisted synthesis of the Schiff base was prepared by the condensation of dehydroacetic acid (2.5 mmol)(1) and the substituted primary aromatic amine (2.5 mmol) (2) in a 1:1 molar ratio using an Erlenmeyer flask through a conventional Ultrasound bath by taking 2-3 drop of mL solvent (ethanol), respectively. The reaction was completed in a short period (Table 1). The solution when cooled gave colored crystals, which were washed with Ethanol and dried over anhydrous $CaCl_2$.

Method 3: The Solid starting materials were finely powdered before use. A mixture of dehydroacetic acid (2.5 mmol) (1) and the substituted primary aromatic amine (2.5 mmol) (2) was stirred in a small amount of water (5 mL) at room temperature for the mentioned time. The crystalline powder formed was collected by filtration, washed with water, and dried in a desiccator to give Schiff bases. If the reaction needed a base, K_2CO_3 (0.41 g, 3.0 mmol) would be added to the reaction mixture.

Method 4: The 0.1 mole each of dehydroacetic acid (2.5 mmol) (1) and the substituted primary aromatic amine (2.5 mmol) (2) were dissolved one after the other in 100 mL of dry ethanol taken in a round-bottomed flask. The contents of the flask were refluxed for four to five hours on 1 RMLrotamantle with a magnetic stirrer. Upon cooling, the solid mass of Schiff base that was separated was filtered, washed with portions of dry ethanol, and dried. A pure product was obtained by recrystallization from ethanol and dried in a vacuum desiccator. The purity of the products was ascertained by TLC and melting point.

Method 5: The synthesis of Schiff base ligand was prepared by taking dehydroacetic acid (2.5 mmol) (1) and the substituted primary aromatic amine (2.5 mmol) (2) in the ratio of 1:1 in a mortar and pestle. Then few drops of citric acid were added to adjust pH. Then, the reaction mixture was ground in a mortar pestle at about 45 minutes. The water produced in the reaction was removed at 70 °C under vacuum. After completion of the reaction, cold water was added and collected precipitate was filtered and dried. All solvent-free reactions were performed by grinding together 5.0 mmol of the pure amine with 5.0 mmol of the pure aldehyde in a mortar for one min. and keeping the mixture at room temperature for 1.5 h to be formed quantitatively. The water produced in the reaction was removed at 70 °C under vacuum synthesis of Schiff base ligand was prepared by taking dehydroacetic acid (2.5 mmol) (1) and the substituted primary aromatic amine (2.5 mmol) (2) in the ratio of 1:1 in a mortar and pestle. Then few drops of citric acid were added to adjust pH. Subsequently, the reaction mixture was ground in a mortar pestle for about 45 minutes. The water produced in the reaction was removed at 70 °C under a vacuum. After completion of the reaction, cold water was added and collected precipitate was filtered and dried.

Step-II: synthesis of aryl azoschiff bases:

0.1 mole of primary aromatic amine was dissolved in 23 mL of distilled water containing 23 mL of concentrated hydrochloric acid (2.5-3 equivalents) by gentle warming if necessary, and the solution was filtered, cooled in an ice bath when the amine hydrochloride crystallizes. The temperature was maintained at 0 °C and not allowed to exceed 5 °C. An ice-cold aqueous solution of sodium nitrite was obtained by dissolving 0. 11 moles of salt in 40 mL of distilled water maintained at 0°C in small increments gradually with constant stirring until the reaction mixture showed a positive test with starch iodide paper or 4,4'-diamino diphenylmethane, 2,2'-sulfone for excess of nitrous acid when the crystallized amine hydrochloride was dissolved to give a clear

solution of highly soluble diazonium chloride. It was filtered immediately under ice-cold conditions and used immediately for subsequent coupling. The slight excess of sodium nitrite added accidentally was decomposed by the addition of 0.01-mole urea or sulfamic acid in 5 mL of distilled water. A solution of Schiff base derived from dehydroacetic acid and corresponding amine-containing 0.1 mole of it in 100 mL of 10% sodium hydroxide was prepared and cooled to 0-5°C. To this, an ice-cold solution of diazonium chloride was added very slowly with vigorous stirring; the brownish-red colored solid aryl azo Schiff base soon separated. After adding the whole diazonium chloride solution, the mixture was allowed to stand in the ice bath for 1 hour with occasional stirring, the product was then filtered through Buchner funnel under suction, washed successively with dilute hydrochloric acid and portions of distilled water, and dried in a vacuum desiccator. It was recrystallized from alcohol/alcohol-water mixture as solvent. Its purity was ascertained by TLC and melting point.

Characterization data of compounds (3a-f).

The structural features of the ligands were elucidated with the help of elemental analysis, electronic, infrared absorption, and nuclear magnetic resonance techniques which are crucial in characterization.

a. Elemental Analysis, Colour and Melting Point of Ligands:

The C, H, N analysis of synthesized ligands L_{1-6} was carried out by micro combustion method using CHNSO, EA1108, Elemental analyzer model-CARLO-ERBA Instruments, Italy, at micro analysis division, National Chemical Laboratory, Pune. The samples weighing between 1-10 mg were used for the analysis. The C, H, N micro combustion analysis of ligand L_2 was undertaken by employing 3-5 mg sample on "CHNSO Analyser" (CE. Instru. EA 1110) at Centre for Materials for Electronics Technology (C-MET), Pune, while that of ligand L was conducted by using 3-10 mg material at Department of Chemistry, University of Pune. The color, melting point, and percentage of carbon, hydrogen, and nitrogen found in aryl azo Schiff bases, which were calculated theoretically as given in Table 2.

Table 2. Elemental Analysis, Color and Melting Point of Schiff bases

Sr. no.	Schiff base	Molecular weight	Color	Melting point ^{o}C	The elemental percentage found (Calculated)		
					C	H	N
1	L_1 = $C_{22}H_{21}O_3N_3$	375.42	Reddish brown	180	70.22 (70.35)	5.97 (5.62)	11.01 (11.18)
2	L_2 = $C_{22}H_{21}O_5N_3$	407.42	Dark brown	250	64.72 (64.82)	4.85 (5.20)	9.98 (10.30)
3	L_3 = $C_{20}H_{15}O_3N_3Br_2$	505.16	Gray brown	166	46.95 (47.52)	2.77 (2.95)	8.32 (8.35)
4	L_4 = $C_{22}H_{21}O_3N_3$	375.42	Orange brown	160	70.90 (70.35)	5.97 (5.62)	11.05 (11.18)
5	L_5 = $C_{28}H_{21}O_3N_3$	447.49	Dark violet	161	75.45 (75.20)	4.93 (4.72)	9.28 (9.36)
6	L_6 = $C_{22}H_{21}O_5N_3$	407.42	Orange red	141	65.28 (65.35)	5.55 (5.30)	10.15 (10.22)

b. Spectroscopic Study of Ligands:

The ultraviolet-visible (UV/Visible) infrared (IR), Nuclear Magnetic Resonance (NMR), and mass spectroscopy are the prominent spectroscopic techniques useful for the structure determination of organic compounds. The following are the observed max values for aryl azo Schiff bases found to be nearly the same, i.e. 326-330 nm, as shown in

Table 3. The ^{1}HNMR and IR spectral data of the aryl azo Schiff base ligands were recorded. The chemical shift values, IR frequency, and their assignments are given below.

L_1: 6-methyl-5-((*E*)-*p*-tolyldiazenyl)-3-((*E*)-1-(*p*-tolylimino) ethyl)-2*H*-pyran-2,4(3*H*)-dione: FT-IR (KBr,cm^{-1}): 2600(-OH---N), 1700 (>C=O), 1657 (-C=N-), 1597 (-C=C- Ar ring stretching), 1567 (-N=N-), 1328 (-C-N, aryl azomethine stretching),1245(-C-O, enolic stretching). ^{1}H NMR ($CDCl_3$) (ppm) δ: 1.6 (s, 1H, C_3 hydrogen of DHE moiety), 2.16(s, 3H, –CH_3 of DHE moiety at C6), 2.29 (s, 3H, Ar-CH_3 of azomethine N), 2.38 (s, 3H, Ar-CH_3 of azo N), 2.56 (s, 3H,-CH_3 of azomethine C), 7.03-7.18 (dd, 4H, 2Ar-meta-H_b), 7.23-7.31(dd, 4H, 2Ar-ortho-H_a).

L_2: 5-((*E*)-(4-methoxyphenyl)diazenyl)-3-((*E*)-1-((4-methoxy phenyl)imino)ethyl)-6-methyl-2H-pyran-2,4 (3H)-dione: FT-IR (KBr, cm^{-1}): 2605(-OH---N), 1687 (>C=O), 1652 (-C=N-), 1562 (-C=C- Ar ring stretching), 1500 (-N=N-), 1250 (-C-N, aryl azomethine stretching),1175(-C-O, enolic stretching). ^{1}H NMR ($CDCl_3$) (ppm) δ: 1.8 (s, 1H, C_3 hydrogen of DHE moiety), 2.16(s, 3H, –CH_3 of DHE moiety at C_6), 2.57 (s, 3H, -CH_3 of azomethine C), 3.84 (s, 6H, 2Ar-OCH_3), 6.94-6.97 (dd, 4H, 2Ar-meta-H_b), 6.98-7.11(dd, 4H, 2Ar-ortho-H_a).

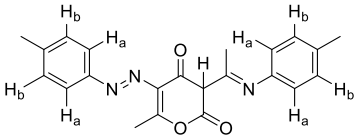
L3:5-((E)-(4-bromophenyl)diazenyl)-3-((*E*)-1-((4-bromo phenyl)imino)ethyl)-6-methyl-2H-pyran-2,4(3H)-dione: FT-IR(KBr,cm^{-1}): 2590 (-OH---N), 1690 (>C=O), 1566 (-C=N-), 1570 (-C=C- Ar ring stretching), 1480 (-N=N-), 1301(-C-N, aryl azomethine stretching),1234(-C-O, enolic stretching). ^{1}H NMR ($CDCl_3$) (ppm) δ: 1.8 (s, 1H, C_3 hydrogen of DHE moiety), 2.16(s, 3H, –CH_3 of DHE moiety at C_6), 2.59 (s, 3H, -CH_3 of azomethineC), 7.04-7.58 (dd, 4H, 2Ar-meta-H_b), 7.58-7.61(dd, 4H, 2Ar-ortho-H_a).

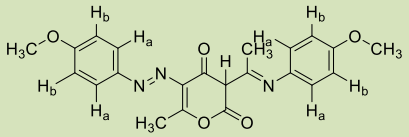
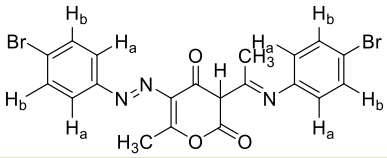
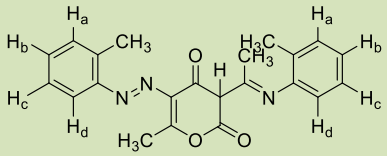
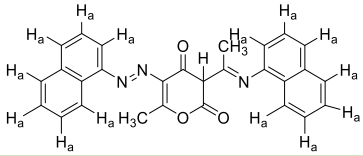
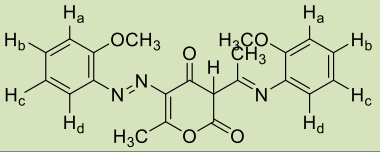
L4:6-methyl-5-((*E*)-o-tolyldiazenyl)-3-((*E*)-1-(o-tolylimino) ethyl)-2*H*-pyran-2,4(3*H*)-dione: FT-IR (KBr, cm^{-1}): 2581(-OH---N), 1701 (>C=O), 1667 (-C=N-), 1560 (-C=C- Ar ring stretching), 1460 (-N=N-), 1352(-C-N, aryl azomethine stretching),1242(-C-O, enolic stretching). ^{1}H NMR ($CDCl_3$) (ppm) δ: 1.65 (s, 1H, C_3 hydrogen of DHE moiety), 2.18(s, 3H, –CH_3 of DHE moiety at C_6), 2.25 (s, 3H, 2Ar-Ortho-CH_3 of azomethine N), 2.80 (s, 3H, 2Ar-Ortho-CH_3 of azo N), 2.52 (s, 3H, -CH_3 of azomethine C), 7.06-7.39 (m, 8H, 2Ar-H_a,H_b, H_c, H_d).

L5:6-methyl-5-((E)-naphthalen-1-yldiazenyl)-3-((E)-1-(naphthalen-1-ylimino)ethyl)-2H-pyran-2,4(3H)-dione : FT-IR (KBr, cm−1): 2582 (-OH---N), 1704 (>C=O), 1660 (-C=N-), 1565 (-C=C- Ar ring stretching), 1480 (-N=N-), 1355 (-C-N, aryl azomethine stretching),1215(-C-O,enolic stretching), 850 (Naphthalene ring deformation), 767(Naphthalene ring). ^{1}H NMR ($CDCl_3$) (ppm) δ: 1.55 (s, 1H, C_3 hydrogen of DHE moiety), 2.17(s, 3H, –CH_3 of DHE moiety at C6), 2.63 (s, 3H, -CH_3 of azomethine C), 7.22-7.90 (m, 14H, Ar-H of napthyl)

L6: 5-((E)-(2-methoxyphenyl)diazenyl)-3-((E)-1-((2 methoxy phenyl)imino)ethyl)-6-methyl-2H-pyran-2,4(3H)-dione: FT-IR (KBr, cm−1): 2705(-OH---N), 1694(>C=O), 1661(-C=N-), 1564(-C=C- Ar ring stretching), 1502(-N=N-), 1325(-C-N, aryl azomethine stretching),1250(-C-O, enolic stretching).^{1}H NMR ($CDCl_3$) (ppm) δ: 1.55 (s, 1H, C_3 hydrogen of DHE moiety), 2.16(s, 3H, –CH_3 of DHE moiety at C_6), 2.54 (s, 3H, -CH_3 of azomethine C), 3.82 (s, 6H, 2Ar-OCH_3), 6.97-7.40 (m, 8H, 2Ar-H_a,H_b, H_c, H_d).

Table 3. Observed λ_{max} UV-Vis spectral data

Sr. no.	Schiff base	Structure of	£$_{max}$ for >C=O (nm)	λ_{max} for >C=N (nm)	£$_{max}$ for naphthyl ring (nm)
1	L_1		326	225	--

2	L_2		328	225	--
3	L_3		358	225	--
4	L_4		326	260	--
5	L_5		330	225	514
6	L_6		326	225	--

Results and Discussion

The structures of the synthesized Schiff bases and aryl azo Schiff bases were elucidated based on IR, ^{1}H-NMR, UV-Vis, and mass spectral data which are given in Table1-4. The observed λ_{max} values for aryl azo Schiff bases were found to be nearly the same i.e. 326-330 nm (Table 3). The >C-N (azomethine)) $\pi \rightarrow \pi^*$ transitions were expected in the range 220 to 230 nm. The ligand L shows λ max at 358 nm. The ligand L_5 in addition to a band at 330 nm exhibited a band at 514 nm probably due to the presence of a condensed naphthyl ring in the ligand which is dark purple-violet. The weak band at around 300 nm shown by carbonyl compounds was considered to have its origin in the lone pair of electrons of the oxygen atom.

The IR spectra clearly showed a strong band (C=N) at 1657-1567 cm^{-1}, (-O-H) at 2700-2500 cm^{-1}, (C=O) at 1704-1687 cm^{-1}, (C=N stretching of Azomethine group) at 1657-1567 cm^{-1}, (N=N, stretching of aryl azo) at 1567-1502 cm^{-1}. The stretching vibration of aromatic–C=C- was observed within the 3166-3062 cm^{-1} range. Azomethinehydrogens of Schiff bases showed three absorption bands at 2986-2874, cm^{-1} (N=C-H),1286-1228 cm^{-1} (N=C-H), and 1056-1019 cm^{-1} (N=C-H). These IR data confirmed the presence of specific functional groups in the final products. The ^{1}H NMR spectra of ligand L_{1-6} are discussed as follows: In ligand L_1, the methyl group was substituted at the para position of both phenyl rings attached to azomethine nitrogen and azo nitrogen. Each of these phenyl rings had four bonded aromatic hydrogens, two ortho H and two meta H, atoms and gave rise to doublets at δ 7.23-7.31 4(H) and at δ 7.03-7.18 (4H), respectively. Due to methyl substituent at para position, the upfield shift of proton at meta position (H) was greater than that of proton at ortho position (H). In CH_3-Ar type of systems, the methyl protons were expected to have δ a value of 2.3. The singlets at δ 2.29 (3H) and 2.38 (3H) appeared in the NMR spectra. The six protons of two –CH_3 groups at the para position of phenyl rings attached to azomethine nitrogen and azo nitrogen respectively. In L_2, the NMR spectrum showed a singlet at δ 3.84(6H). This was assigned to the six protons of two methoxy substituents at the para position of two phenyl rings. Due to the Bromo substituent at the para position of phenyl moieties of ligand L_3, the δvalues of aromatic

protons were shifted downfield because of the deshielding effect. The doublets at δ 7.58- 7.61 (4H), and δ 7.04-7.58 (4H) in the spectrum were attributed due to aromatic protons H -*ortho* and H-*meta* protons, respectively. In ligand L_4, the methyl group was substituted at the ortho position of both phenyl rings attached to azomethine nitrogen and azo nitrogen. Each of these two phenyl rings had four bonded aromatic hydrogens, H_a, H_b, H_c, and H_d, which were shielded due to methyl substitution causing upfield shift and exhibited a multiplet at δ 7.07-738 (8H). The ligand L_5, with two naphthyl moieties attached to azomethine nitrogen and azo nitrogen, showed multiplet at δ 7.25-7 95 (14H) in its spectrum because of fourteen aromatic hydrogens of two naphthyl moieties. This is because of the different electronic environments of protons or naphthyl moiety.

Antibacterial activity: All synthesized compounds (1-6) were tested their antibacterial activity in Dayanand Science College, Latur in the Department of Microbiology, using two gram-positive bacteria (Staphylococcus aureus, Streptococcus spp.) and two gram-negative bacteria (*Escherichia coli and Klebsiella pneumonia*) by the diffusion technique. Filter paper disc of (6 mm) was impregnated with specified concentrations (500 µg/mL, 750 µg/mL and 1000µg/mL) in DMSO solvent. The results of measuring the diameter of the inhibition area after 24 hours at 37 °C are represented in Table 6. It was noticed from the results that the zone of inhibition increased with an increase in concentration, hence for high concentration (1000 µg/mL). At (1000 µg/mL), compound $L_{3,5}$ had inhibition zone 14-18 mm against all bacterial strains, while compound $L_{1,4}$ had the lower activity.

Table 4. Inhibition zone of Azo-Schiff base derivatives (1-6) against tested bacteria

Compd.	Conc.(µg/mL)	S. aureus	Str. spp	K. pneumonia	E. coli
L_1	1000	11	12	14	13
	750	8	10	10	10
	500	-	8	-	-
L_2	1000	10	11	15	14
	750	-	-	12	10
	500	-	-	10	8
L_3	1000	10	11	16	15
	750	-	-	14	12
	500	-	-	12	8
L_4	1000	10	12	13	13
	750	8	10	10	10
	500	-	8	-	-
L_5	1000	16	14	18	18
	750	9	11	16	16
	500	-	9	12	12
L_6	1000	12	12	15	13
	750	-	-	13	11
	500	-	-	10	-

Conclusion

Different methods and ways of reflux have been used to synthesize Schiff base derivative of the well-known aryl azo Schiff base dye; only irradiation in a microwave reactor in a moderate to high temperature gave the desired product. When compared with all methods, method 1 had great virtue. It is the same outfit for industrial production which consumes the minimum time to varnish the synthesis of Schiff base. The Microwave irradiation and sonication method to synthesize Schiff bases do not only use the minimum time but also have the highest yield. From the above experiment, we can know clearly that sonication and

microwave irradiation is the modest method to prepare the Schiff base. Sonication is attractive and microwave irradiation is an increasingly current method of heating that substitutes the traditional one because it is a cheap, clean, and appropriate method. Often, it affords greater yields and results in a shorter reaction time.

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Conflict of Interest

The authors declared that they do not have any conflict of interest regarding this research article.

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