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Synthesis and Characterization of New Azo-Schiff Bases and Study Biological Activity of Some These Compounds

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Abstract: Aso derivative (3) was prepared by coupling reaction between diazonium salt (2) and salicylaldehyde. Aso-schiff bases 4(a-h) have been synthesized by the condensation (3) with different aromatic amines. The completion of reactions was checked by TLC. The prepared azo compounds were identified by IR and 1HNMR spectroscopy and elemental analysis. The second part of this work includes studding the effect of the some bacteria.

Key words : Synthesis , Characterization , Azo-Schiff Bases , Biological Activity .

Introduction

Schiff bases are compounds which prepared by the condensation of aldehyde or ketone with primary amines(1-3). Schiff bases are used in the synthesis of some industrial, bioactive and inorganic compounds(4-5). The compounds including good fungicdal activity(6), Anticancer(7-9), antibacterial (10-12), antifungal (13,14), antiinflammatory(15-17)and herbicidal activities(8). Azo compounds are widely used as dyes and pigments. Another application is analytical chemistry. On the other hand azo compounds shown biological activities containing antibacterial(18). Schiff bases and azo compounds are important structures in the medicinal and pharmaceutical fields(19).

Experimental

Melting points were taken on astuart Melting points apparatus. FTIR spectra were recorded on Schimadzu 8000 serise. 1HNMR were run on abruker, Ultra shield 300 MKz, Switzear land using TMS as interenal standard and CDCl3 as solvent. Uv- Vis spectra were recorded on Shimadzu Uv-1700. C.H.N analytical were recorded on a Eurovectro, EA 3000A, Italy.

A-Synthesis Azo compound

Diazonium salt (2) was prepared according to a reported method(20). Treatment of compound (2) with 2-hydroxy benzaldehyde gave compound (3), scheme (1).

Azo aldehyde (3) could also be purified in 80% yield by recrystallization from warm ethanol.

B- Synthesis new derivatives

The compound (3) treatment with different aromatic amines containing 3 drops of glacial acetic acid were heated under reflux in absolute ethanol for (3-10h), yielded the azo Schiff bases 4(a-h) in excellent yields (scheme 2).

Results and discussion

Eight new azo-Schiff bases were prepared in excellent yields via the condensation of different aromatic amines with new azo compounds(20). The diazonium compound was treatment with salicylaldehyde gave the azo compound (3) scheme (1) which was purified by recrystallization from ethanol.

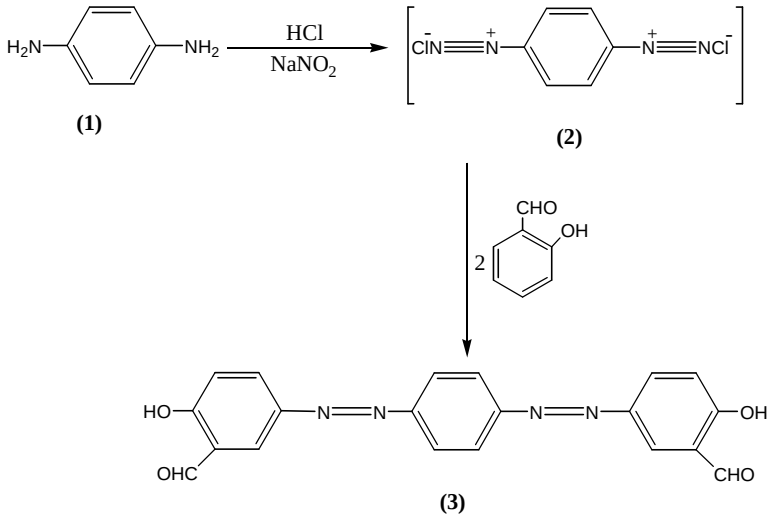
Treatment of compound (3) with different aromatic amines, in refluxing absolute ethanol yielded the new azo Schiff bases (Scheme 2, Table 1).

The reactions are followed by (TLC), (Benzene:MeOH). New compounds were identified by their melting points, elemental analysis, IR spectra, UV spectra and 1HNMR spectra. FTIR spectra(21) shown disappearance of (NH2) absorption bands at 3160, 3280 cm-1 and the appearance of (C=O) absorption band at (166o) cm-1. Also FTIR was used to confirm the structure of new compounds 4(a-h) no absorption band corresponding to (C=O) group was observed in the FTIR spectra of the new compounds. New bands at (1590- 1630) cm-

1due to (C=N) group, (Table 2).

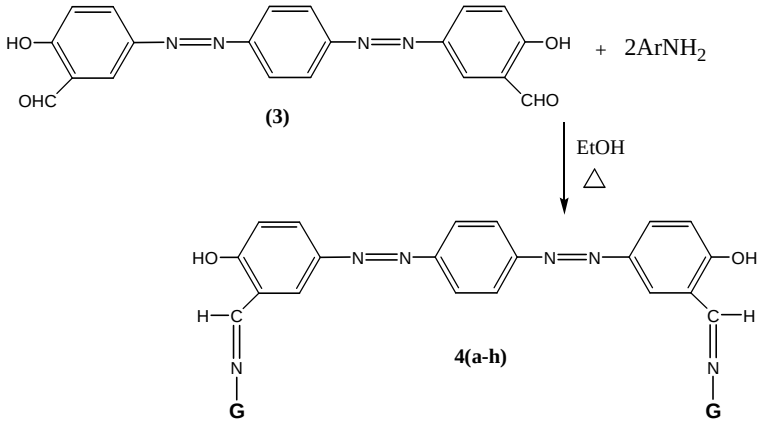
The UV spectra of new azo-Schiff bases shown absorption maxima at (260-295) nm due to the ($\pi\rightarrow\pi$□), and another absorption maxima at (305-360) nm due to the (n-→π*). Table (4).

In the 1HNMR spectra signals (6.3-8.5) ppm due to aromatic protons of new compounds (4b, 4d and 4e), (8.1-8.6) ppm belonging to the (N=CH) group of compounds (4a, 4d and 4e) and (10.1-10.6) ppm for (-OH) group of compounds were observed. These signals represent more characteristic evidence for the formation of these compounds.



(1) **(2)** **(3)**

(Scheme 1)



(3) **4(a-h)**

Comp. No.	G
4a	m-$CH_3C_6H_4$-
4b	p-ClC_6H_4-
4c	o-OHC_6H_4-
4d	m-$NO_2C_6H_4$-
4e	o-BrC_6H_4-
4f	o-ClC_6H_4-
4g	o-$CH_3C_6H_4$-
4h	2-pyridine

(Scheme 2)

Anti bacterial Activity

The new compounds were tested against one strain each of a gram positive and two gram negative. The test results presence in Tables (6) and (7), some new compounds were active against tested and another compounds are no active. All compounds are no active where used (1×10-5M), but active in the concentrations : 1×10-4 M and 1×10-3M see Tables (6) and (7).

Table (1): Physical data for new Schiff bases

NO.	M.F	M.Wt (gm/mole)	M.P C	Yield %	Rf	Time (hrs)
(4a)	$C_{34}H_{28}N_6O_2$	552	220	72	0.68	24
(4b)	$C_{32}H_{22}N_6O_2Cl_2$	593	253	78	0.81	12
(4C)	$C_{36}H_{28}N_6O_4$	608	266	68	0.71	10
(4d)	$C_{32}H_{22}N_8O_6$	614	269	62	0.59	10
(4e)	$C_{32}H_{22}N_6O_2Br_2$	682	272	75	0.76	10
(4f)	$C_{32}H_{22}N_6O_2Cl_2$	593	243	80	0.56	8
(4g)	$C_{34}H_{28}N_6O_2$	552	260	66	0.62	8
(4h)	$C_{28}H_{18}N_{10}O_2$	528	251	64	0.65	6

Table (2): Analytical data for some new Azo Schiff bases

Comp. No.	M.wt (g/mol)	Found (calc) %		
		C	H	N
(4a)	552	73.62 (73.91)	4.89 (5.07)	15.02 (15.21)
(4c)	608	70.89 (71.05)	4.25 (4.60)	13.62 (13.81)
(4d)	614	62.13 (62.54)	3.24 (3.58)	17.98 (18.24)
(4h)	528	63.12 (63.63)	2.99 (3.40)	26.30 (26.51)

Table (3): FTIR spectra for some new Azo – Schiff bases

Comp. No.	ν_{OH} cm^{-1}	$\nu_{O\text{-}H\ arom}$ cm^{-1}	$\nu_{C=N}$ cm^{-1}	$\nu_{N=N}$ cm^{-1}	$\delta C\text{-}H_{O.O.P}$ cm^{-1}	$\nu_{C\text{-}X}$ cm^{-1}
4a	3350-3310	3060-3040	1590	1610	740-810	----
4b	3380-3400	3070-3045	1580	1605	780-820	850
4C	3330-3550	3050-3020	1585	1600	800-850	----
4d	3300-3370	3060-3120	1600	1620	750-830	----
4e	3310-3350	3050-3080	1610	1625	810-880	620
4f	3250-3310	3040-3090	1585	1610	785-830	860
4g	3280-3345	3060-3100	1570	1605	730-820	----
4h	3240-3360	3040-3100	1580	1600	720-810	----

Table (4): UV-Visible spectra for new Azo- Schiff bases

Comp.	(4a)	(4b)	(4c)	(4d)	(4e)	(4f)	(4g)	(4h)
λ_{max} (nm) (Ethanol)	275- 290 330- 360	290-305 310-350	290-305 310-350	225- 240 245- 270 320- 360	270- 290 295- 325	265- 284 290- 320	212- 232 272- 293 332- 358	210- 225 270- 285 305- 345

Table (5): 1HNMR spectra for some Azo- Schiff bases

4b	4d	4e
(δ 10.5 ppm, s, -OH) (δ 8.3 ppm, s, =CH) (δ 6.5-8.5 ppm, m, ph)	(δ 10.1 ppm, s, -OH) (δ 8.1 ppm, s, =CH) (δ 6.3-7.8 ppm, m, ph)	(δ 10.6 ppm,s, -OH)) (δ 8.6 ppm, s, =CH) (δ 6.8-8.1 ppm,m,ph)

Table(6): Effect of new azo Schiff bases on the growth of tested bacteria (conc. 1×10-3M)

Bacteria / Comp.	Gram positive		Gram negative	
	S.aureus	***E.coli***	***K.pneumonia***	***P.aeruginous***
(4a)	----	6mm	----	7mm
(4b)	----	10mm	----	12mm
(4c)	----	10mm	----	7mm
(4d)	----	10mm	10mm	----
(4e)	10mm	8mm	10mm	----
(4f)	10mm	10mm	----	----
(4g)	10mm	8mm	----	10mm
(4h)	10mm	10mm	10mm	10mm

Table(7): Effect of new azo Schiff bases on the growth of tested bacteria (conc. 1×10-4M)

Bacteria / Comp.	Gram positive		Gram negative	
	S.aureus	***E.coli***	***K.pneumonia***	***P.aeruginous***
(4a)	----	----	----	5mm
(4b)	----	8mm	----	10mm
(4c)	----	8mm	----	5mm
(4d)	----	6mm	8mm	----
(4e)	----	8mm	8mm	----
(4f)	----	8mm	----	----
(4g)	10mm	----	----	----
(4h)	10mm	8mm	8mm	6mm

1-5= + (Slightly active)
6-10=++ (Moderately active)
11-15=+++ (Highly active)

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تحضير أزو– قواعد شيف الجديدة ودراسة الفعالية البايولوجية لبعضها

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الخلاصة : تم تحضير مشتق الأزو (٣) من تفاعل ازدواج ملح الديازونيوم (٢) مع السالسالديهايد، بعدها تم تحضير أزو– قواعد شيف 4(a-h) من تكاثف (٣) مع بعض الأمينات الأروماتية. تم متابعة سير التفاعلات بوساطة (TLC). شخصت مركبات الأزو المحضرة بواسطة مطيافية الأشعة تحت الحمراء والأشعة فوق البنفسجية والرنين النووي المغناطيسي ألبروتوني وكذلك التحليل الدقيق للعناصر. الجزء الثاني من البحث تضمن دراسة الفعالية البايلوجيه لبعض مركبات الأزو المحضرة على بعض أنواع من البكتريا.