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Synthesis and characterization of Novel Schiff bases and evaluation of Corrosion inhibitors and biological activity

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Abstract:

Novel Schiff bases have been prepared from (6R,7R)-3-[(acetyloxy)methyl]-7-[[(Z)-2-(2-aminothiazol-4-yl)-2-(methoxyimino)acetyl]amino]-8-oxo-5-thia-1-azabicyclo[4.2.0]-oct-2-ene-2-carboxylate sodium and 2,4-dihydroxy benzaldehyde ,3-methoxy-4-hydroxy benzaldehyde and3,4-dimethoxy benzaldehyde .The homogeneity of the compounds was chered by TLC .The structures of these Schiff bases have been characterized using IR,UV-Vis Spectroscopy, Elemental analysis(CHNS) ,melting points uncorrected ,These Schiff bases have been studies as Corrosion inhibitors for brass in acide media (0.5 N HCl) and antibacterial of the Schiff bases have also been tested against both Gram-positive and Gram –nagative bacteria .

Keyword: Synthesis, Novel, Schiff base, Corrosion inhibitor, biological activity.

Introduction:

Corrosion problems have received considerable amount of attention because of their economic and safety consequences. The use of inhibitors is one of the most practical methods for protection against corrosion [1].Corrosion inhibitors are chemicals that react with ametallic surface or the environments the metal surface is exposed to and act to protect the metal against Corrosion [2]. Most organic compounds having hetero atoms (N,O,S) in their aromatic have been successfully used as Corrosion inhibitors [3,4,5].The heterogenous Organic compounds having higher basicity and electron density on the hetero atoms have tendency to resist Corrosion . The active centers for the process of adsorption on the metals surface [6].The inhibitors are derivatives of thiazole, It is established that the lone pair of electrons on nitrogen and the protonation property of aza and thiol groups are responsible for the formation of protective on the metal surface and hence control the corrosion [7].contain this group.The compound (1) contain this group. This compound exhibit antibacterial activity in vitro particularly against *Esherichia Coli* , *Staphylococcus aureous* ,*pseudo monas aeruginosa* and *Kebsiellu Pneumonae* [8,9,10].Recent studies presented that Schiff bases were effective inhibitors for generalized

Corrosion of matel [11,12,13]. These observations promoted us to synthesize new Schiff bases to investigate inhibitive effective and evaluation of biological activity. Hence it was thought in teresting to study such type of moieties shown in Figure 1.

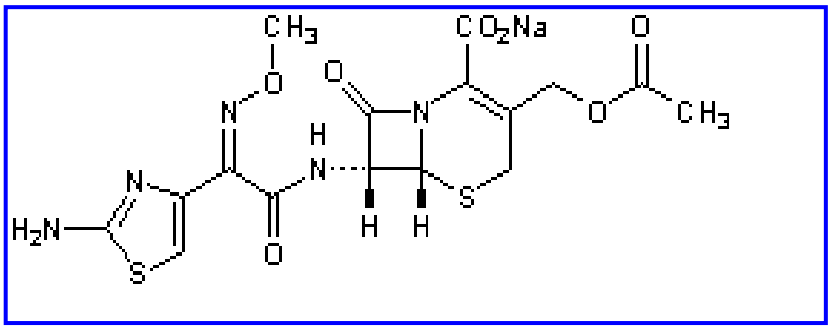


Figure (1) Chemical structure of Amine (1)

Experimental:

Material and measurement all Chemicals used were of (BDH, fluka, Merk) and used with out further purification:

All melting points were determined in open Capillary tubes using Electrothermal (Blaok 9300) apparatus were un corrected.

IR spectra were recorder as KBr disc using Shimadzo –Japan apparatus in rang (4000-400cm^{-1}).Elecronic spectra were measured in the region(200-400 nm) for solution in ethanol and chloroform at room temperature(30C°) using (Spectro Scan 80D) Uv.Vis Spectrophotometer-U.K.

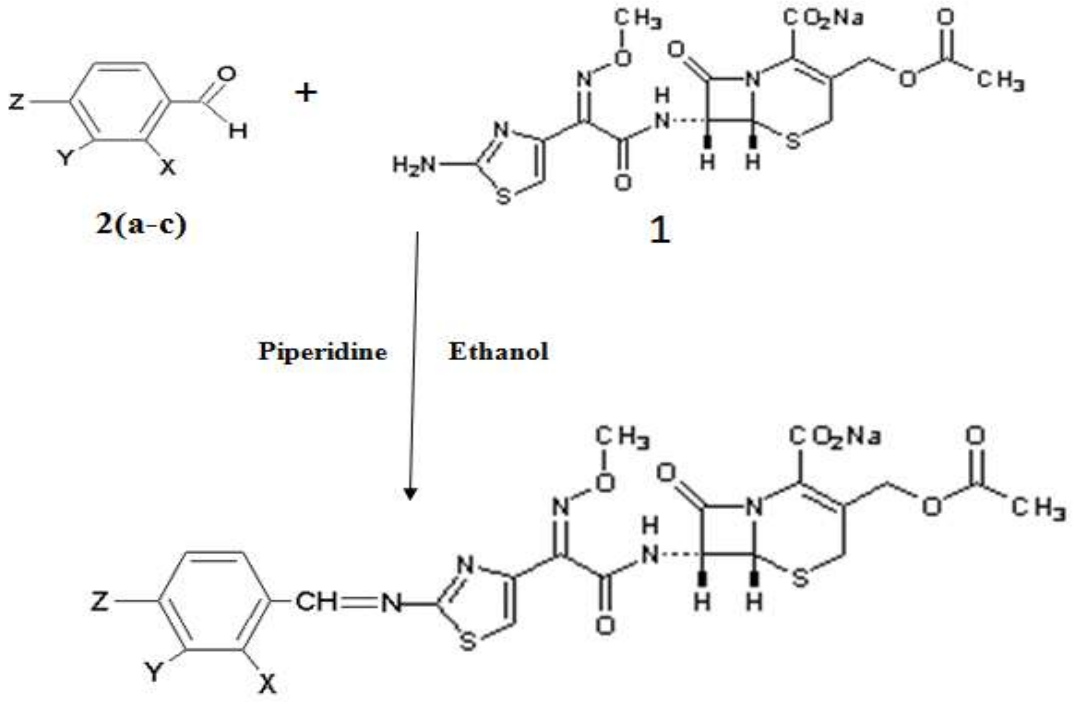
C, H and N elemental analyses were performed using a (Euro Vector EA 3000 A Italy) in Al-albait University – Jordon

Inhibitor Corrosion study were carried out by weight the specimens (Brass) before and after immersion in 100 ml acid solution (0.5 M HCl) for (4 days) at room temperature in the absence and presence of inhibitors compounds.

Antibacterial activity of the compounds were studies against Gram positive bacteria (*Staphylococcus aureous)* and Gram nagative bacteria(*Esherichia Coli)* by agar cup method by using (DMSO) as solvent .

Preparation of Schiff bases:

A mixture of equimolar amount (0.01) of Amine (1) and 2,4-dihydroxy benzaldehyde (2a) in ethanol and in the presence of few drops of piperidine as a catalyst was refluxed for (4hrs),the reaction is easily monitored by TLC . The reaction mixture was concentrated ,cooled and left for (24hrs) at room temperature .During this period yellow needles were formed the crystals thus formed were filtered and recrystallized form ethanol to give Schiff bases (S1) it was obtained in(60%) yield . The same method was applied for the preparation of Schiff base (S2)(65%) yield and Schiff base (S3)(60%) yield by using their respective aldehyde 3-methoxy 4-hydroxy benzaldehyde (2b) and 3,4- dimethoxy benzaldehyde (2c) The preparation of Schiff's bases was carried out according to Scheme 1.



Scheme (1)

where S1, 2a: X=Z=OH, y =H S2, 2b: X=H, Z=OH, y =OCH_3 S3, 2c: X=H, Z=y=OCH_3

Spectral Characteristics:

UV-Vis Spectra

UV-Vis Spectra of the Schiff bases are summarized in (table 1). the compound (S1) shows λmax at (326nm) in ethanol see (fig 2) , as well as compound (S2) shows λmax at (350nm) in ethanol see (fig 3), and compound (S3) shows λmax at (353nm) in ethanol see (fig 4) , This compounds(S1-S3) undergo red shift because to formed the imine bond (C=N) compared with start material (1) that showed absorption at (254nm) in the same solvent [14-16].

IR Spectra

FTIR spectra of compounds are listed (Table 2). The IR Spectra of Amine (1) contain characteristic bonds at around (3450-3350Cm^{-1}), (1760Cm^{-1}), (1665Cm^{-1}) due to υ(NH_2,NH) , υ(C=O) lactam , and υ(C=O) amide Respectively . The IR Spectra of all the compounds (S1-S3) contains characteristic bonds at around (3450Cm^{-1}), (1735Cm^{-1}), (1545Cm^{-1}) due to υ (OH), υ(C=O), and υ(C=N) Respectively. In addition to some bonds shown in the (table 2), the IR Spectra of all compounds has give in (figure 5, 6) respectively. [17-20].

Table (1) shows UV-Vis spectra and physical properties of compounds

compound	Yield(%)	M.P(°C)	Color	formula	λmax(ethanol) (nm)
1	/	/	White	$C_{16}H_{16}N_5NaO_7S_2$	254
S1	60	>250*	Yellow-red	$C_{23}H_{20}N_5NaO_9S_2$	326
S2	65	>250*	Yellow	$C_{24}H_{22}N_5NaO_9S_2$	350
S3	60	>250*	Yellow-Brunette	$C_{25}H_{24}N_5NaO_9S_2$	353

* = decomposition

Table (2) FTIR spectra data for Schiff bases (S1-S3)

Schiff bases	OH	NH	CH aliphatic	C=O lactom	C=O ester	C=C	C=N	CH aliphatic (bend)	CO
amine	/	3450-3350 br	2980 (w)	1760 (s)	1718 (s)	1608 (s)	/	1365(s)	1055 (s)
S1	3450 (br)	3339 (m)	2940 (w)	1735 (s)	1720 (s)	1620 (s)	1520	1355(s)	1050 (s)
S2	3445 (br)	3326 (m)	2970 (w)	1730 (s)	1724 (s)	1610 (s)	1535	1366 (s)	1056 (s)
S3	/	3310 (m)	2974 (w)	1728 (s)	1720 (s)	1607 (s)	1530	1340 (s)	1065(s)

br =broad , m= medium , S=strong, W=weak

Element analysis:

The obtained CHN analysis results, which are in good agreement with the proposed formulae, are summarized in (Table 3)

Table (3) Elements analysis data for Schiff bases (S1-S3)

Schiff base	FW	C%		H%		N%		S%	
		Calculated	found	Calculated	found	Calculated	found	Calculated	found
S_1	597.58	46.28	46.05	3.37	3.32	11.71	11.21	10.70	10.05
S_2	611.6	47.08	47.13	3.59	3.57	11.44	11,22	10.46	10.38
S_3	625.63	47.95	47.83	3.83	3.03	11.18	11.02	10.22	10.10

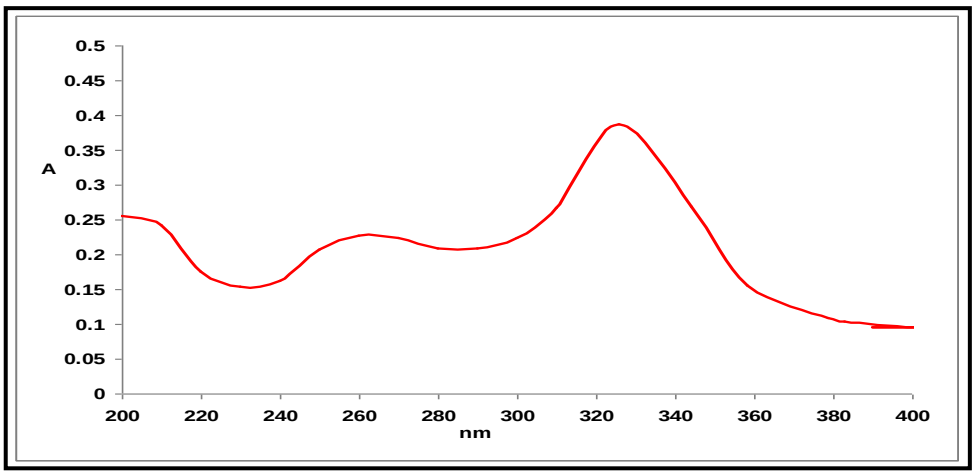


Figure (2) UV-Vis spectra for Schiff base (S1)

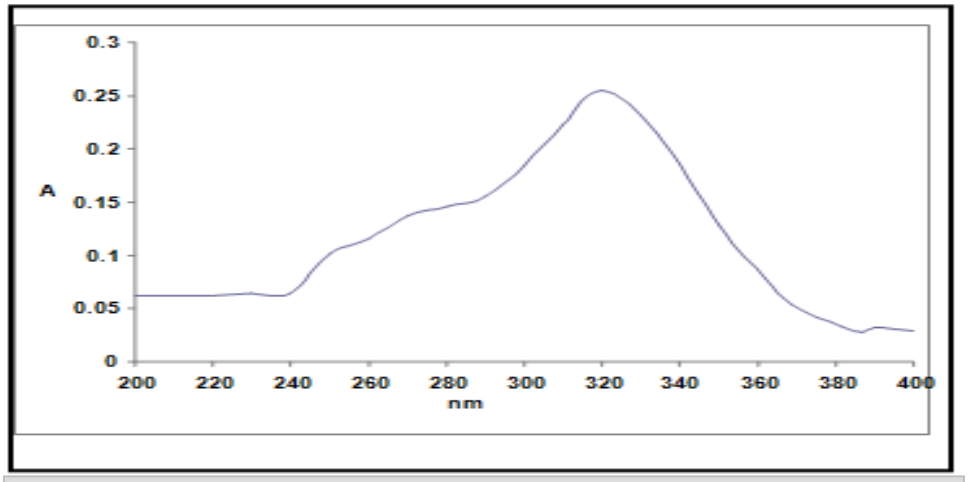


Figure (3) UV-Vis spectra for Schiff base (S2)

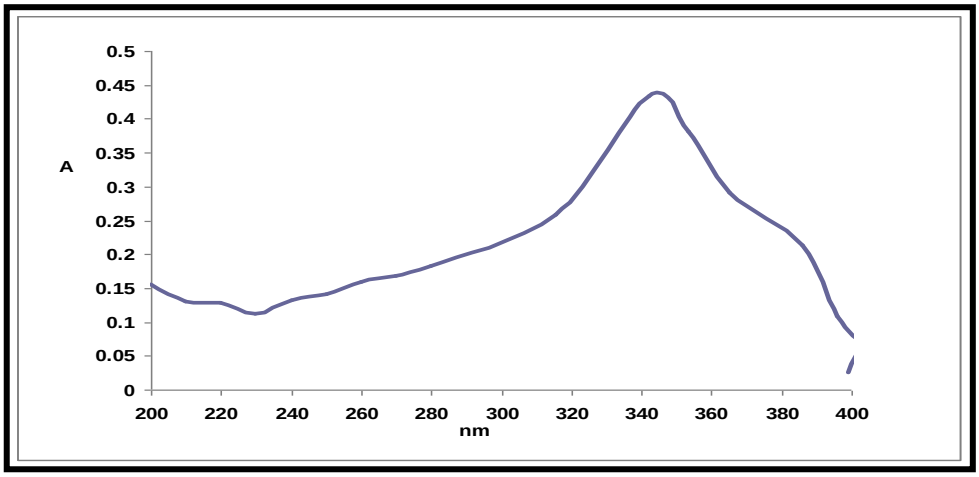


Figure (4) UV-Vis spectra for Schiff base (S3)

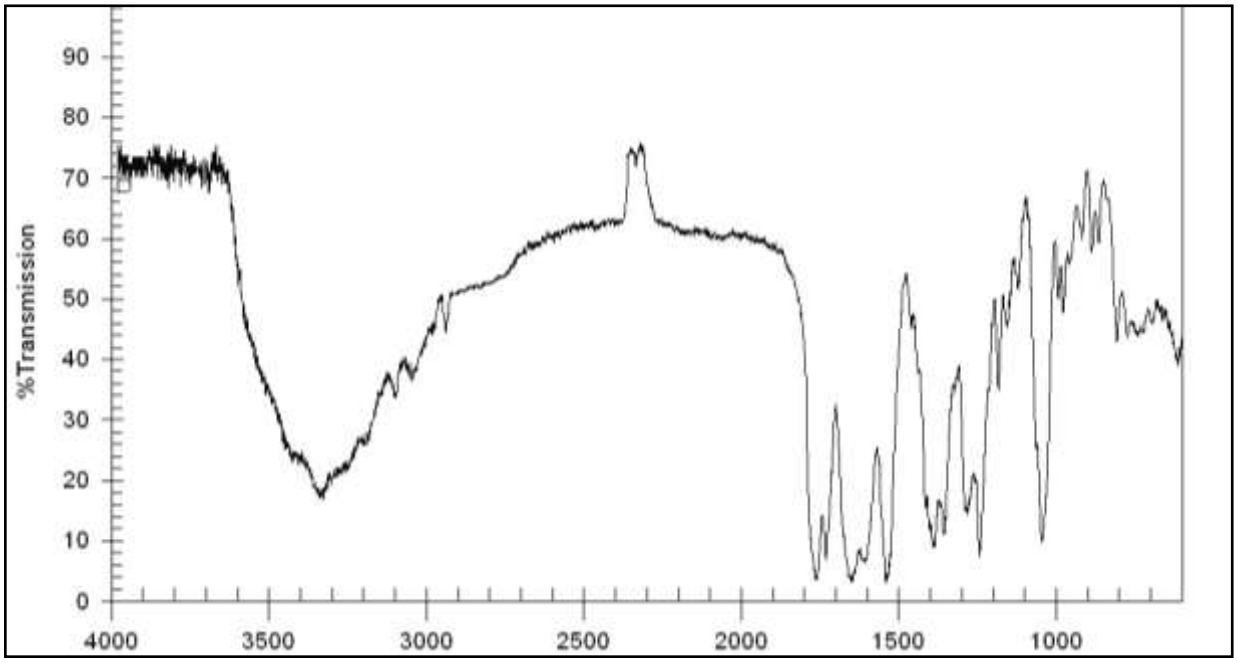


Figure (5) FTIR spectra of Amine (1)

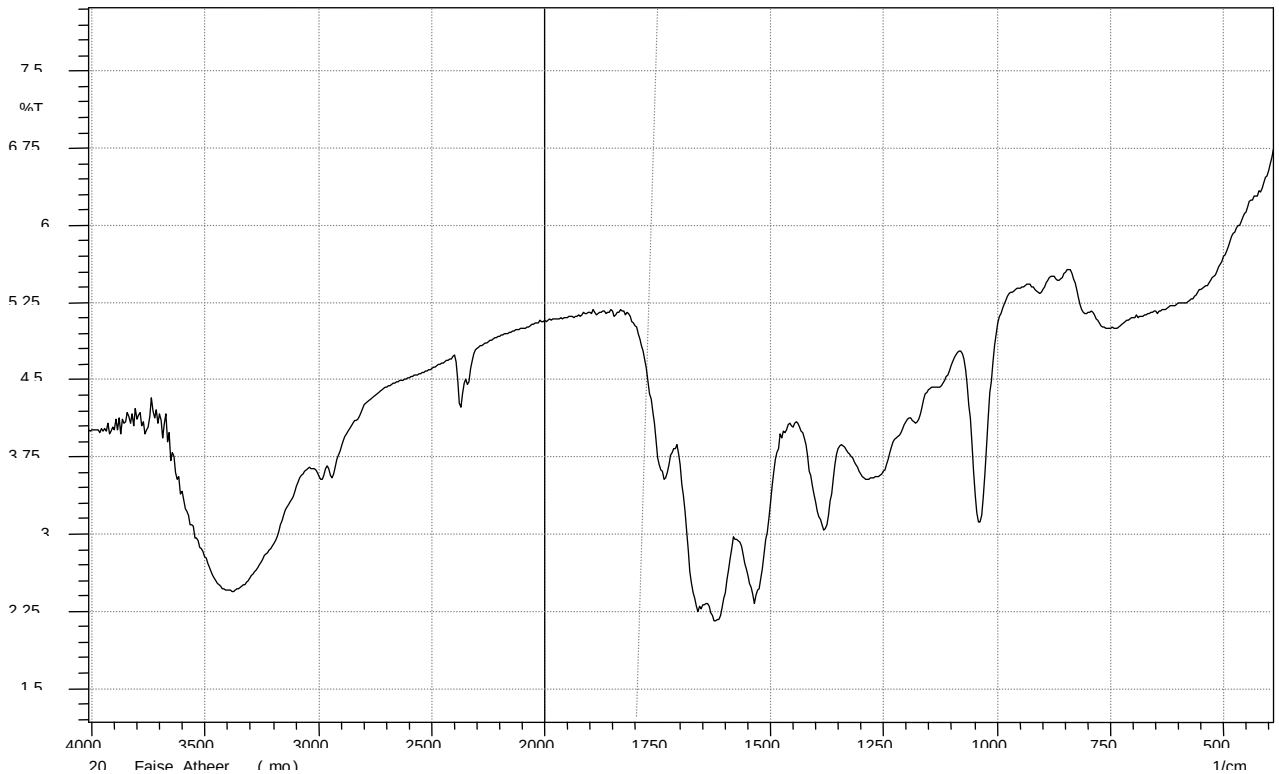


Figure (6) FTIR spectra of Schiff base (S1)

Weight loss study [21-24]

Samples of brass were used for weight loss measurement studies at (25◦C) for (4 days) .The figures (7-10) shows the variation of weight loss of inhibitors (Schiff bases).

The Corrosion rate of all inhibitors was calculated as:-

$$CR = \frac{W \cdot K}{A \cdot d \cdot t}$$

Where CR: Corrosion rate, W: amount of weight loss , K: constant (534) , A: area of Brass , d: density of sample (8.52), t: time (hour).

The figures (11-14) show the variation of Corrosion rate of Schiff bases compounds (inhibitors).

The Inhibitors Efficiency of all inhibitors (S1-S3) was calculated as:-

$$IE(\%) = \frac{(CR_\circ - CR_1) \cdot 100}{CR_\circ}$$

Where CR◦= Corrosion rate with out inhibitors (blank)

CR1= Corrosion rate with inhibitors

IE = Inhibitors Efficiency.

The figure (15-18) shows the variation of Inhibitors Efficiency of Schiff bases compounds (S1-S3) (inhibitors).

The values of weight loss, corrosion rate, inhibitor efficiency and surface area (θ) for Schiff bases (S1-S3) are give in (table 4).

Table (4) shows values of weight loss, corrosion rate, inhibitor efficiency and surface area (θ) for Schiff bases (S1-S3) and amine

	Day1				Day2				Day3				Day4			
	wt	CR	IE%	θ	wt	CR	IE%	θ	wt	CR	IE%	θ	wt	CR	IE%	θ
HCl	0.0693	90.14	/	/	0.1165	75.77	/	/	0.1566	67.90	/	/	0.1812	58.92	/	/
amine	0.0159	20.68	77.05	0.77	0.0275	17.88	76.39	0.76	0.0516	22.37	67.07	0.67	0.0637	20.71	64.84	0.64
S1	0.0017	2.21	97.54	0.975	0.0053	3.44	95.45	0.95	0.0093	4.03	94.06	0.94	0.0128	4.16	92.93	0.93
S2	0.0016	2.08	97.69	0.97	0.0070	4.55	93.99	0.93	0.0096	4.16	93.86	0.93	0.0116	3.77	93.59	0.93
S3	0.0022	2.86	96.82	0.96	0.0085	5.52	92.70	0.92	0.0119	5.15	92.40	0.92	0.0136	4.42	92.49	0.92

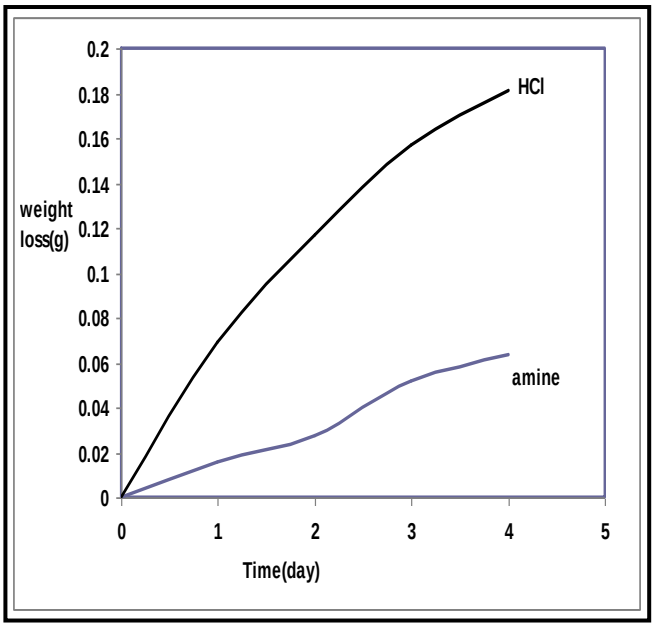


Fig. (7) Show of weight loss of Amine compound

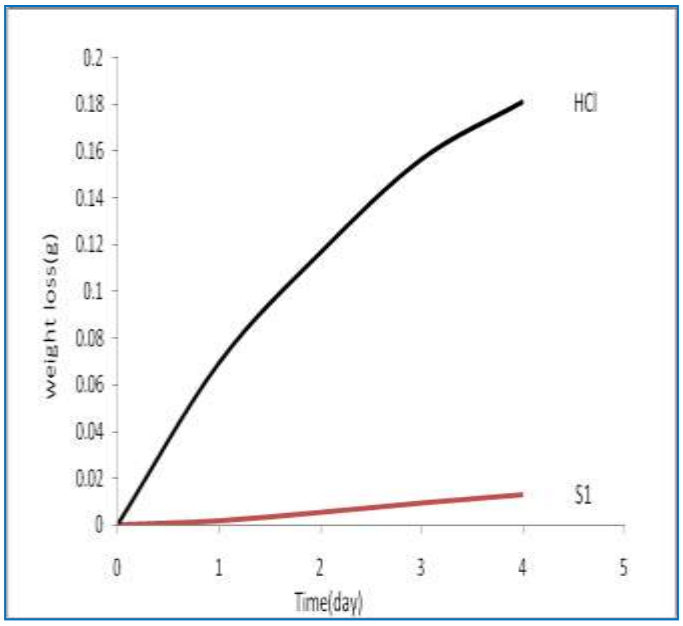


Fig. (8) Show of weight loss of S1 compound

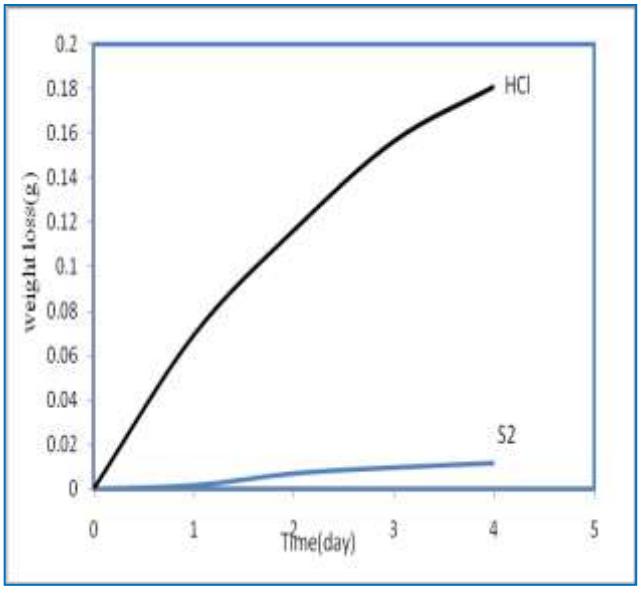


Fig. (9) Show of weight loss of S2 compound

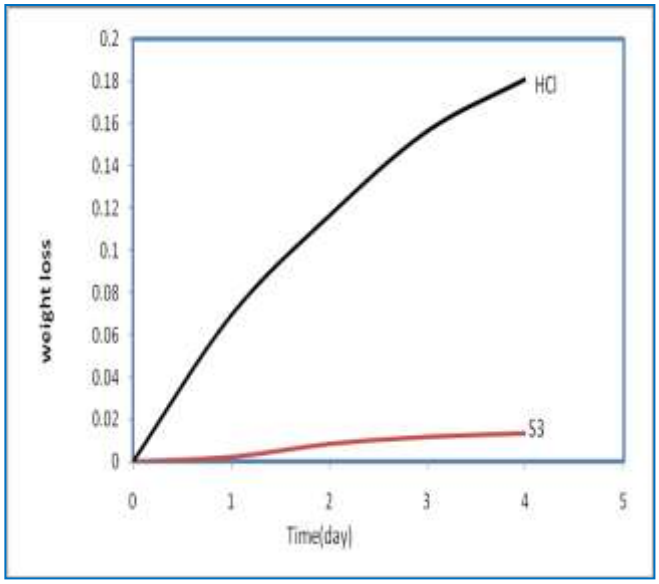


Fig. (10) Show of weight loss of S3 compound

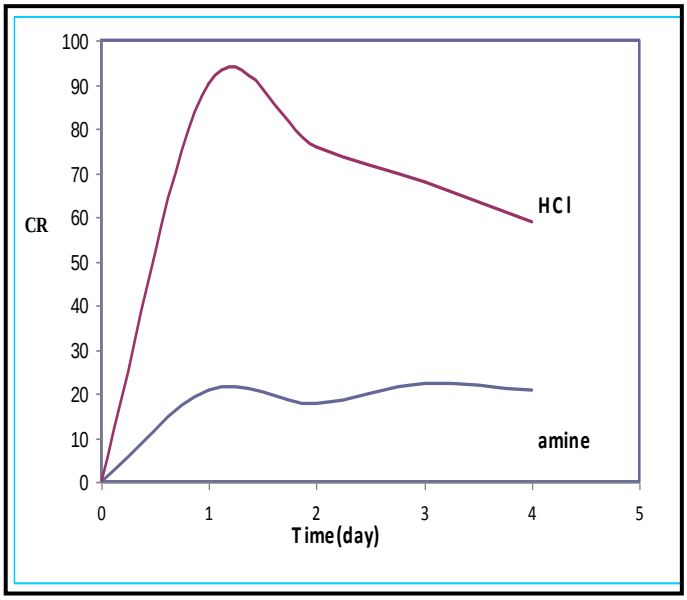


Fig. (11) Show of Corrosion rate of Amine compound

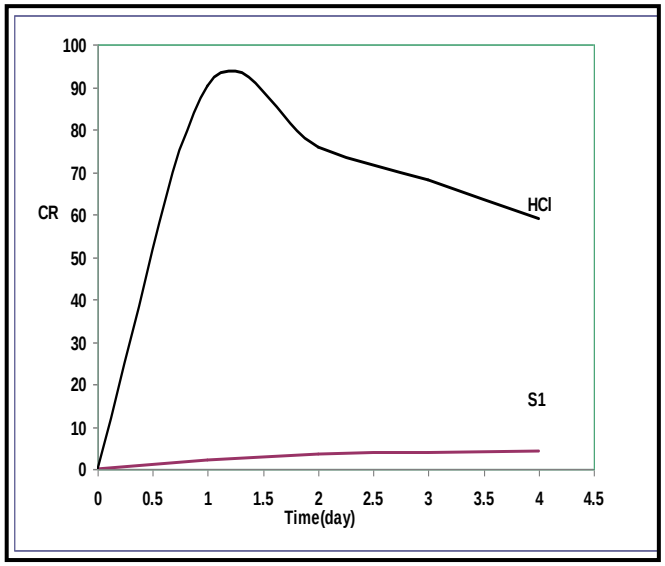


Fig. (12) Show of Corrosion rate of S1 compound

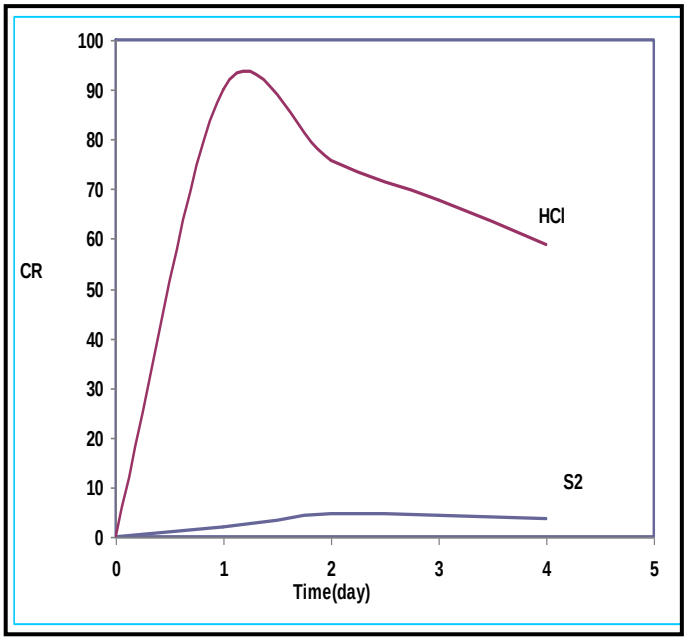


Fig. (13) Show of Corrosion rate of S2 compound

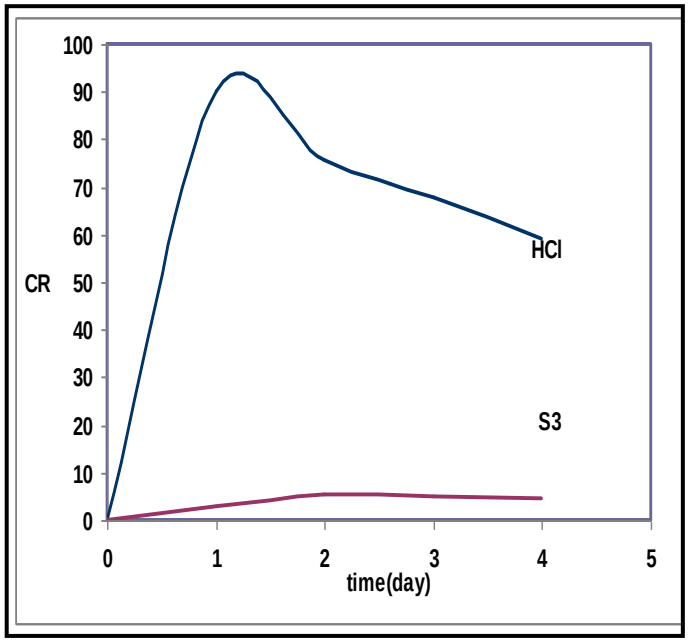


Fig.(14) Show of Corrosion rate of S1 compound

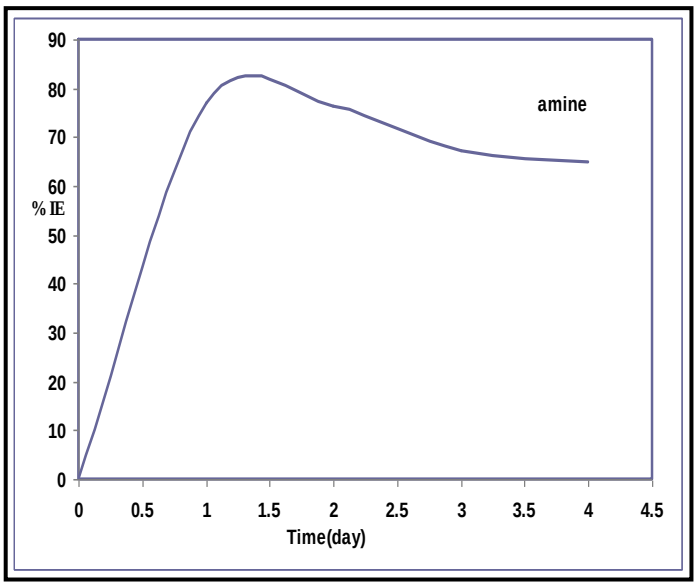


Fig. (15) Show of inhibitor efficiency of Amine compound

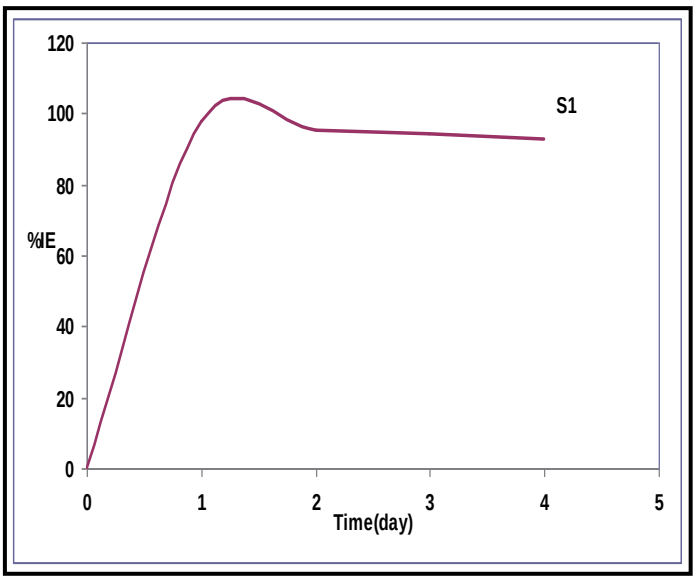


Fig. (16) Show of inhibitor efficiency of S1 compound

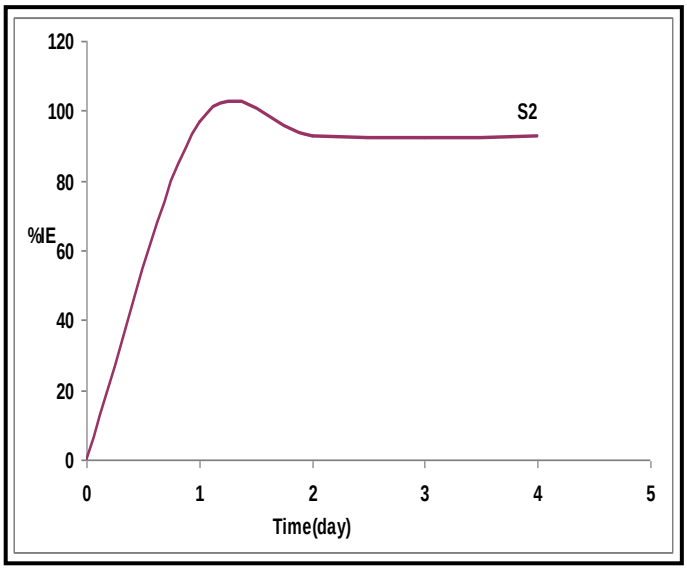


Fig. (17) Show of inhibitor efficiency of S2 compound

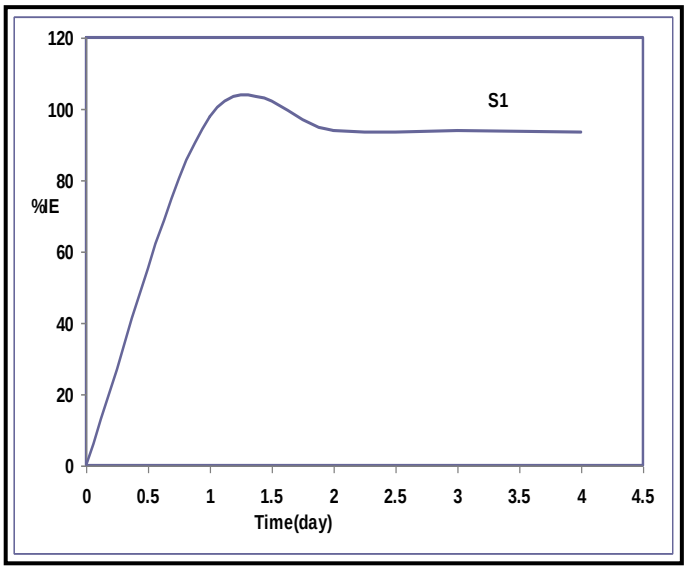


Fig. (18) Show of inhibitor efficiency of S1 compound

Biological activity:

The antibacterial activity of the test compounds (S1-S3) determined by cup plate method using tow type of bacteria: Gram positive bacteria (*Staphylococcus aureous*) and Gram nagative bacteria (*Esherichia Coli*). It was found that all these compounds were considered inhibitors for bacterial with different inhibited Diameter of inhibition zone among them because of contain these compounds on group's increases activity it against the bacteria's. Results are shown

in (table 5). The figures (19, 20) shown effective the Schiff bases on bacteria's [25, 26].

Table (5) Diameter of inhibition zone (in mm) against bacteria (E.coli, ST)

Schiff bases	E.coli	ST
S1	20 mm	15 mm
S2	24 mm	18 mm
S3	15 mm	13 mm

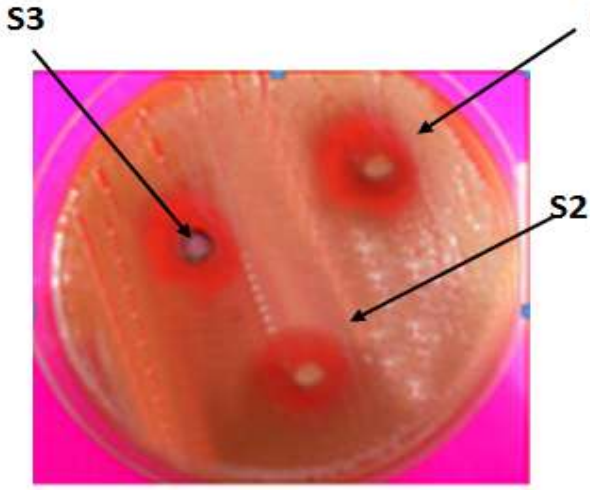


Fig. (19) inhibited effective of Schiff bases (S1-S3) against bacteria E.coil

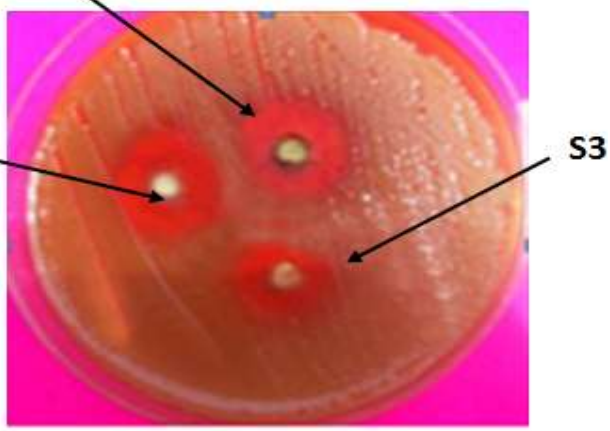


Fig. (20) inhibited effective of Schiff bases (S1-S3) against bacteria ST

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تحضير ودراسة قواعد شف جديدة وتقييمها كمثبطات للتاكل ومضادات للبكتريا

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الملخص:

تم في هذا البحث تحضير قواعد شف جديدة من المركب (6R,7R) اسيتايلوكسي مثيل -7-(Z) (2)--2امينوثايازول 4- بل)-2-(ميثوكسي امينيو)اسيتايل)امينو 8-اوكسو-5-ثايا-1-ازاباي سايكليو(4.2.0) اوكتو2-ين-2-كاربوكسيلات الصوديوم , مع كل من الالديهايدات 4,2- ثنائي هيدروكسي بنزالديهايد , 3 - ميثوكسي -4-هيدروكسي بنزالديهايد والمركب 4,3- ثنائي ميثوكسي بنزالديهايد كما تم التاكد من المركبات المحضرة من خلال متابعتها بطريقة TLC وشخصت بالطرق الطيفية من خلال اطياف الاشعة الفوق البنفسجية-المرئية والاشعة تحت الحمراء وتحليل العناصر الدقيق (CHNS). كما قيست لها درجة الانصهار . كما درست المركبات المحضرة (قواعد شف) كمثبطات للتاكل لمادة البراص في الاوساط الحامضية (0.5N HCl), كما قيمت قدرة هذه المركبات على التثبيط البكتيري لنوعين من البكتريا الموجبة والسالبة.