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Aminothiazole, Schiff base: synthesis, characterization and evaluation of their antimicrobial and antioxidant activity

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Abstract

The research involves a synthesis of 2-amino-thiazole compounds (sulphanilamide, sulfacetamide, sulphamethoxazole, and 4-amino benzoic acid) with a bromine and potassium cyanide in the presence of glacial acetic acid as a solvent by using microwave irradiation method (S1-S4). Schiff bases were prepared by the reaction of compounds (S1-S4) with Benzaldehyde substitution aldehyde as (3-hydroxy-4-methoxybenzaldehyde, 4-hydroxybenzaldehyde, 4-chlorobenzaldehyde, 4-bromobenzaldehyde). The synthesized compounds were characterization using Fourier transform-infrared (FTIR), mass, proton nuclear magnetic resonance spectroscopy (1H-NMR), and Thin-layer chromatography (TLC). Some of the synthesized compounds were tested for antifungal activity against Candida species and antibacterial activity against isolates of Bacillus Puimilus using Nystatin and Neomycin sulfate as reference standard drugs. The results indicate that the synthesized compounds have the ability to inhibit the growth of the tested fungi and bacteria.

Introduction

Because of the various applications of heterocyclic compounds in medicinal chemistry research and others, they have received a lot of attention over the years [1]. Heterocyclic compounds typically have at least one heteroatom in their cyclic structures, such as nitrogen, oxygen, or sulphur, and they can be made both naturally and through laboratory synthetic methods [2]. Currently, one or more heterocyclic moieties make up at least 60% of the compounds in the most popular medications [3]. The widespread use of 2-aminothiazole as a preferred scaffold in medicinal chemistry and drug discovery research has drawn significant attention to this heterocyclic molecule [4]. One thiazole core consists one amino group make up the 2-aminothiazole moiety. Many beneficial molecules, like the vitamin thiamine B1, include the thiazole core, 2-aminothiazole [5]. The 2-aminothiazole's amino group is a functionally active group that can be attached to numerous active fragments via a variety of processes. Due to its adaptable scaffold, 2 aminothiazole has undergone extensive modification to create a variety of derivatives with superior biological activity [6]. Additionally, a large number of agents containing a 2-aminothiazole moiety have been authorized for sale. Famotidine is used to treat gastroesophageal reflux disease and peptic ulcers by preventing the secretion of gastric juices [7], abafungin is used as antifungal

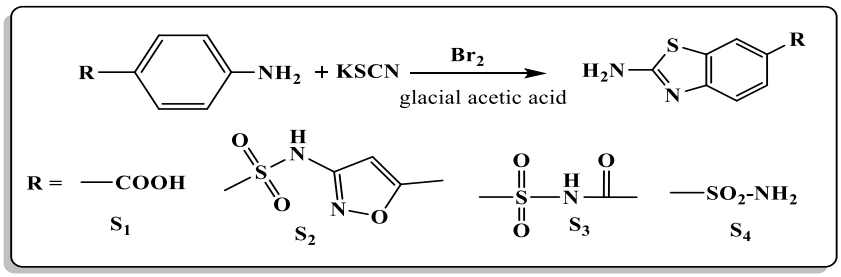
medication to treat dermatomycoses [8], recently molecule containing the heteroaromatic nucleus 2-amino-thiazole selectivity and, thus, could be useful for the development of new glutaminase inhibitors [9]. The 2-aminothiazole scaffold-bearing medication riluzole is licensed for the treatment of amyotrophic lateral sclerosis [10].

Material and Methods

Every chemical used in our study was purchased from Fluka and Sigma Aldrich. By using an electro-thermal capillary apparatus, melting points have been determined. Thin layer chromatography (TLC), and a mixture of Benzene: Methanol as the mobile phase, was used to track the reaction's progress. Infrared spectra were obtained using ATR technique Shimadzu 8400S, Fourier Transforms Infrared spectroscopy SHIMADZU in the range (400-4000) cm^{-1}. The 1H-NMR spectra was obtained on a Bruker model ultra-shield 400MHz in the laboratories of the University of Science (Basrra). Using tetra methyl silane (TMS) as internal reference and DMSO-d6 as solvent.

Synthesis of 2-amino-thiazole derivatives (S1-S4)

Dissolved (0.0027mole, 0,5g) from amine compounds (sulphanilamide, sulfacetamide, sulphamethoxazole and 4-aminobenzoic acid) in (20ml) of glacial acetic acid and add to it (0.0027mole, 0.48g) KSCN dissolved in glacial acetic acid too, stirring and add in drops (0.5 ml) from Bromine dissolved in glacial acetic acid stirring the mixture for (1 hr). TLC was performed using Methanol-Benzene (2:8). The product was treated with a 10% NaOH base solution to precipitate at PH8.3, the contents were filtered and the product was washed with water and dried, purified by recrystallization from ethanol absolute give pure product [11]. Scheme 1. Synthesis of 2-amino-thiazole derivatives (S1-S4). Table 1 Some physical properties of the synthesized compounds (S1-S4).



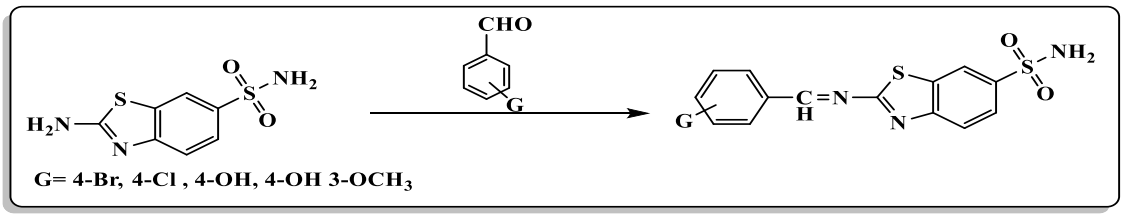
Scheme 1. Synthesis reaction of 2-amino-thiazole derivatives (S1-S4).

Table 1. Physical properties of the synthesized compounds (S1-S4).

Comp. No.	Molecular Formula	Molecular Weight	Color	M.P ^0C	Yield %	R_f
S_1	$C_8H_6N_2O_2S$	194.21	Orange	307 dec.	81	0.92
S_2	$C_{11}H_{11}N_4O_3S_2$	311.35	Orange	183-185	79	0.66
S_3	$C_9H_9N_3O_3S_2$	271.31	Brown	235-237	80	0.95
S_4	$C_7H_7N_3O_2S_2$	229.27	Yellow	270-272	81	0.79

General procedure for synthesis of Schiff bases (S_5 S_8)

Dissolved (0.001 mole, 0.35g) from S4 in (10 ml) of ethanol absolute and added to it (0.01mol) various aldehydes as (4-hydroxy-4-methoxybenzaldehyde, 4-hydroxybenzaldehyde, 4-chlorobenzaldehyde, 4-Bromobenzaldehyde) in presence of drops from glacial acetic acid, then the mixture refluxed for 10 min. in a microwave with capacity) 200 Watt, TLC was performed using Methanol-Benzene (2:8). The product was precipitated. The contents were filtered, and the product was washed with water dried and purified by recrystallization from Benzene to give product [12]. Scheme 2. synthesis of Schiff bases derivatives (S_5 S_8). Some physical properties of the synthesized compounds (S_5 S_8) as showed in table 2.



Scheme 2. Synthesis reaction of Schiff bases derivatives (S5-S8).

Table 2: Some physical properties of the synthesized compounds (S5-S8).

Comp. No.	Molecular Formula	Molecular Weight	Color	M.P ^{0}C	Yield %	R_f
S_5	$C_{14}H_{10}BrN_3O_2S_2$	396.28	Dark yellow	160-162	71	0.54
S_6	$C_{14}H_{10}ClN_3O_2S_2$	351.82	Brown	198-200	82	0.64
S_7	$C_{14}H_{11}N_3O_3S_2$	333.38	Dark yellow	233-235	70	0.61
S_8	$C_{15}H_{13}N_3O_4S_2$	363.41	Yellow	267-269	58	0.76

Antioxidant

DPPH Free radical scavenging activity: The compounds were tested for their antioxidant activity in vitro using 2,2-Diphényl-1- Picrylhydrazyle (DPPH). This enables assessing the compound's capacity for free radical scavenging action. The Gallic acid (Standard) solution, which has a concentration of 100 g/ml, was made by dissolving 1 milligram of Gallic acid in 10 mL of methanol and from it, standard solutions with various concentrations of (10, 20, 25, 50, 60 M) were created. Using the molarity law and the dilution law, the concentrations of the sample solutions (0.5, 0.2, 0.15, 0.1, and 0.05 mM) were created by dissolving 1 mg of each sample in 1 mL of (DMSO). The aforementioned quantities were created by dissolving 4 mg of 2,2-Diphényl-1-picrylhydrazyle (DPPH) in 100 mL of methanol. The mixture was then sealed in a dark canister made of aluminum foil to keep light out. [13].

Protocol for assessment of DPPH scavenging activity

For the control reading, an immediate reading of the absorbance reagent (DPPH) was taken at 517 nm. The concentration preparation was finished by adding 1.5 ml of reagent solution (DPPH) to each test tube holding the sample and concentrations of the standard solution separately. The samples are incubated for 30 minutes in the dark following the completion of the reagent addition. Using methanol as Planck's solution, the absorbance was

then measured at (517 nm) in a UV-visible spectrometer after (30 min). Using methanol as Planck's solution, the absorbance was measured at (517 nm) in a UV-visible spectrophotometer after 30 minutes [14]. The following equation is used to compute the percentage of scavenging activity that occurs in the presence of free radicals. A Control - A Test / A Control * 100 equal's percentage Antiradical activity Where:

A Control is the absorbance of the control reaction, which includes all of the ingredients except for the sample extract. The absorbance of the sample (prepared chemicals) is the "A Test."

Results and discussion

Characterization of 2-amino-thiazole derivatives (S1-S4)

The 2-amino-thiazole derivatives were synthesized by reaction amine (sulphanilamide, sulphactamide, sulphmethaoxazole and 4-aminobenzoic acid) with KSCN and Bromine.

The IR spectrum of compounds (S1-S4) showed presence of a band at (3209-3367 cm^{-1}) assign to ν(NH_2). In addition, showed bands within (3012-3081 cm^{-1}) assign to ν(C-H) aromatic. the rest of the bands maintained their normal ranges, as shown in Table 3, which shows the results of infrared absorption of synthesis compounds (S1-S4) [15]. The spectra are shown in figures (1) for the synthesis compound (S3).

Table 3: FT-IR data for compounds (S1-S4).

Comp. No.	IR (KBr) cm^{-1}					
	ν(NH_2)	ν(C-H) Arom.	ν(C=C) Arom.	ν(C=O) ν(C=N)	ν(C-N) ν(C-S)	Others
S_1	3321 3273	3012	1579 1545	1664 1631	1350 698	C=O 1717
S_2	3321 3188	3016	1557 1526	1669 1628	1298 764	ν (C-H) 2960
S_3	3435 3367	3081	1582 1552	1672 1582	1284 764	ν (C-H) 2967
S_4	3354 3214	3025	1580 1557	1670 1626	1291 763	ν (C-H) 2967

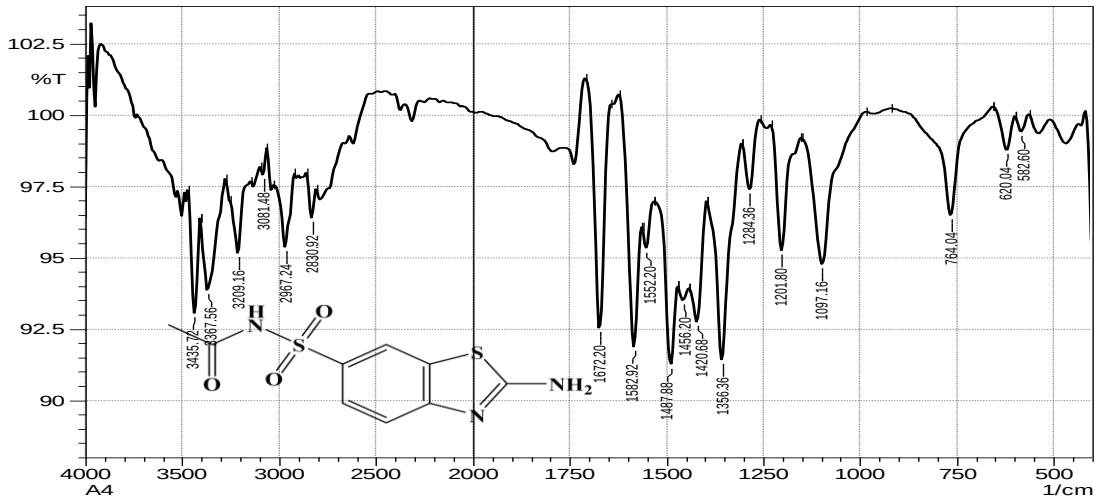


Fig. 1 FT-IR Spectrum for compound (S3).

^{1}H-NMR, spectra of compounds were comprised of a single signal at δ 7.22-7.30 (s, 2H) ppm which ascribed to the Amine thiozole protons, and a several different signals within the range [δ 7.71 – 8.98 (m, 3H)] attributed to the protons ring. 1HNMR spectra confirm the structures of synthesized compounds as shown in Table 4 and Figures 2,3: 1H NMR spectrum of compound S1, S2 [16,17].

Table 4: Chemical Shift δ ppm of (S1-S4).

Compd.	NH_2 amino-thiazole	Ar-H	Others
S1	2H s	3H m	1 H s at 12.32 OH carboxylic acid
	7.22	7.71- 8.67	
S2	2H s	3 H m	2 H s at 11.77 NH- SO_2
	7.27	7.93 – 8.58	
S4	2H s	3 H m	1 H s at 11.77 NH amide,
	7.30	7.93- 8.58	3H s at 2.09 CH_3 alip.,
			1H s at 6.23 C-H five ring

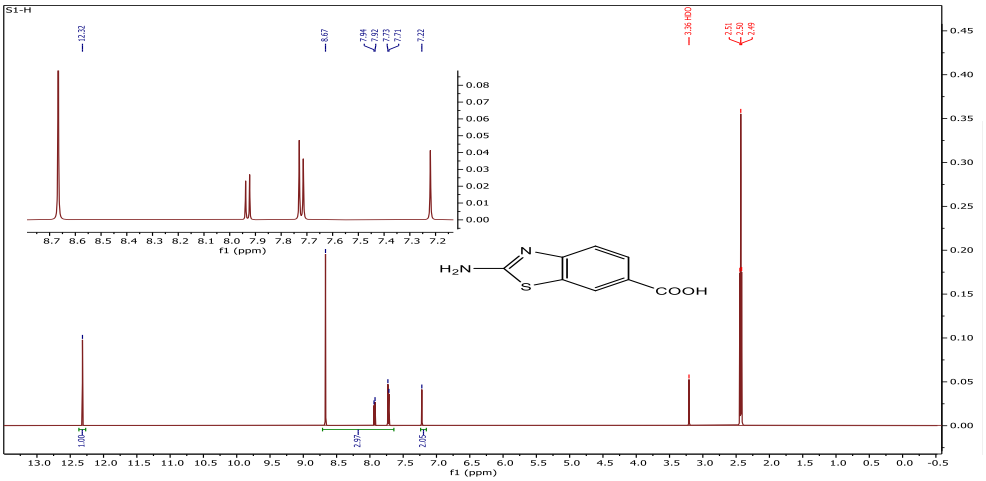


Fig. 2 ^{1}H-NMR Spectrum for compound (S1).

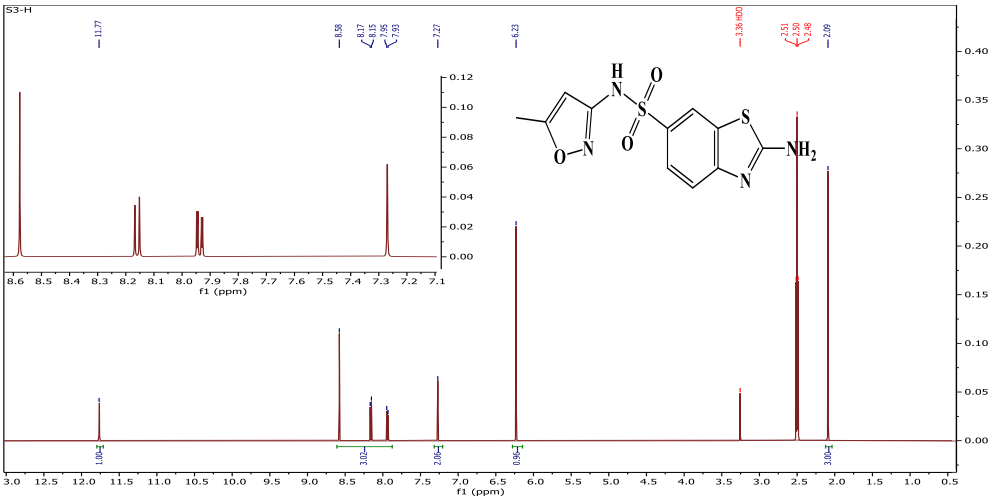


Fig. 3 NMR-H^{1} Spectrum for compound (S_2).

Characterization of Schiff bases (S_5 S_8)

Imine (Schiff's bases) were synthesized by reaction of prepared compound S4 and derivatives aldehyde [18].

The IR spectrum of compounds (S5-S8) showed the absence of a $\nu(NH_2)$-SO_2 band at (3331-3443 cm^{-1}), and assign of ν(C=N) at (1631-1639 cm^{-1}), also showed bands within assign to ν(C-H) aromatic at (3076-3088 cm^{-1}), and showed assign to ν(C=C) aromatic at (1448-1593 cm^{-1}). The show of other bands within (1231-1255 cm^{-1}) assign to ν(C-N). The rest of the bands maintained their normal ranges, as shown in Table 5, which shows the results of infrared absorption of synthesis compounds (S5-S8) [19]. The spectra are shown in figure (4) for the synthesis compound S8.

Table 5: FT-IR spectral data for compounds (S5-S8).

Comp. No.	IR (KBr) cm^{-1}				
	$\nu(NH_2)$-SO_2	ν(C-H) Arom.	ν (C-N) ν (C=N)	ν(C=C) Arom.	Others
S_5	3425, 3331	3088	1255, 1639	1587, 1448	C-Br at 470
S_6	3428, 3333	3086	1231, 1637	1593, 1448	C-Cl at 518
S_7	3443, 3329	3076	1245, 1631	1591, 1448	OH $_{phenolic}$ at 3423
S_8	3423, 3342	3080	1247, 1633	1573, 1450	OH at 3624, CH_3 at 2941

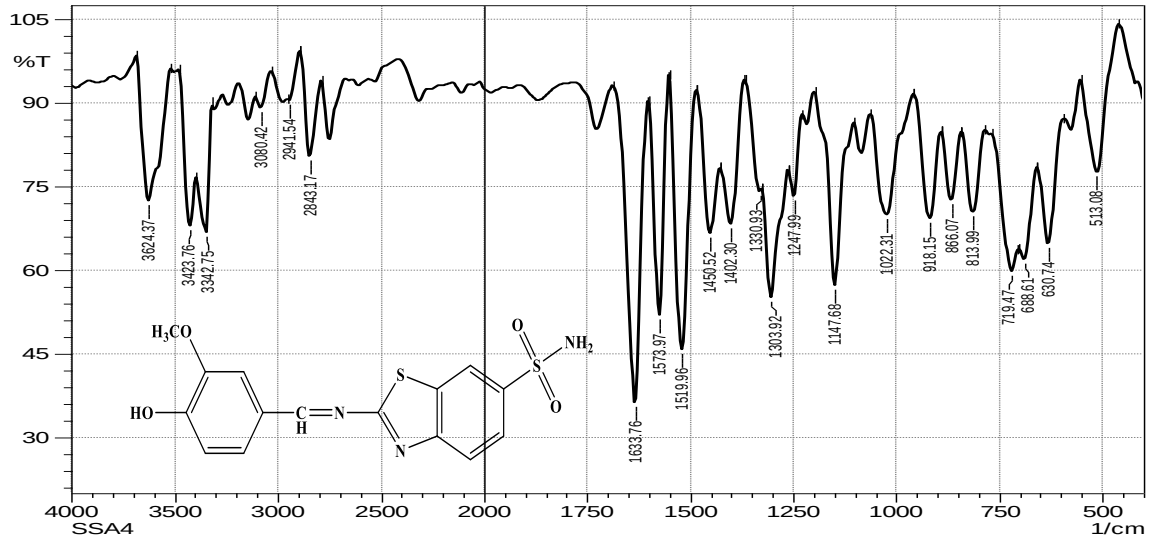


Fig. 4. FT-IR Spectrum for compound (S_8).

^{1}H-NMR, spectra of compounds was comprised of a single signal at δ 7.05- 7.12 (s, 2H) ppm which ascribed to the NH_2-SO_2 protons, and a several different signals within the range [δ 7.28- 8.64] attributed to the protons ring, and a single signal at δ 8.74-8.87 (s, 1H) ppm which ascribed to the Imine protons, 1HNMR spectra confirm the structures of synthesized

compounds as shown in Table 6 and Figures 5, 6: 1H NMR spectrum of compound S6, S8 [20, 21].

Table 6: Chemical Shift δ ppm of (S6-S8).

Comp.	NH_2- SO2	H-C=N	Ar-H	Others
S6	2H s / 7.05	1 H s / 8.87	7 H m / 7.55-8.64	……
S8	2H s 7.12	1 H s 8.74	6 H m 7.28- 8.54	1 H s at 9.93 OH 3H s at 3.83 CH_3 alip.,

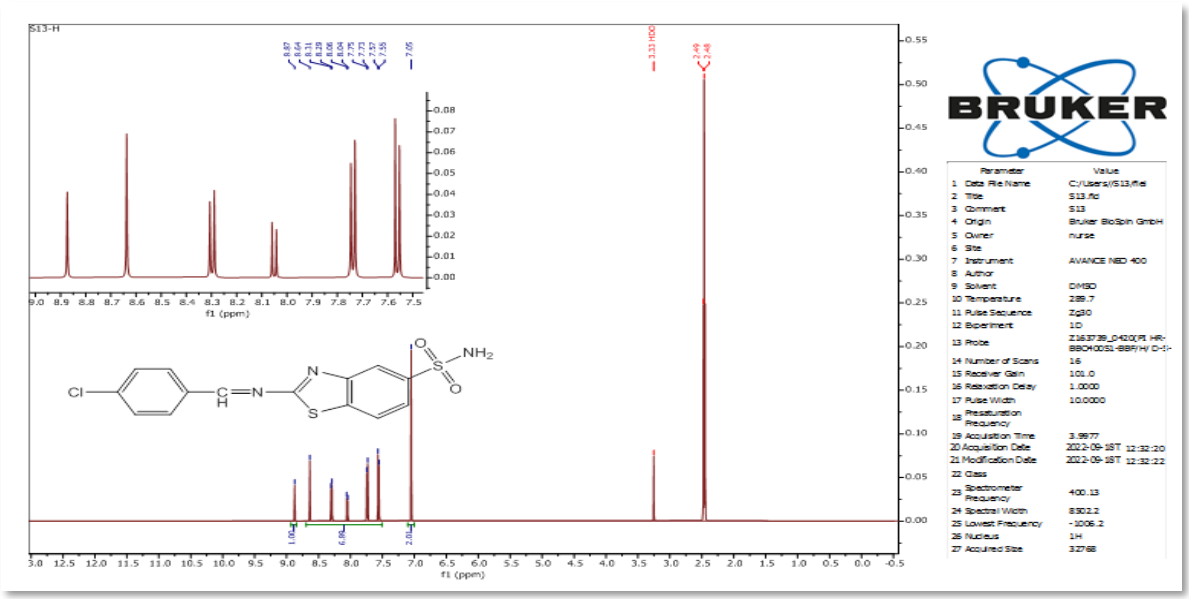


Fig. 5 NMR Spectrum for compound (S_6).

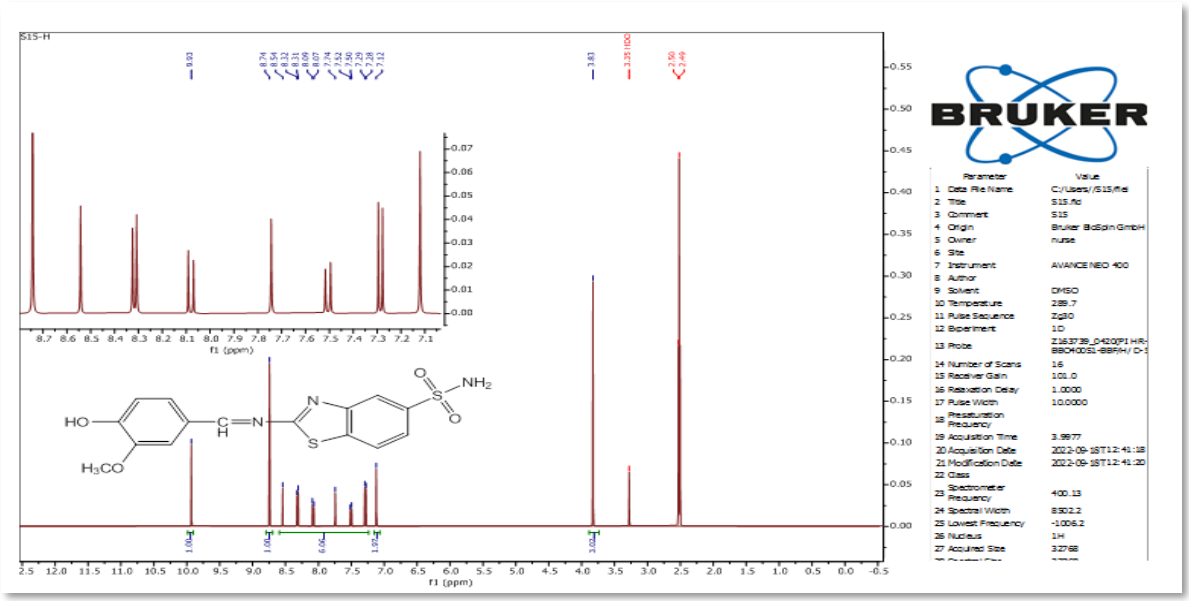


Fig. 6 NMR Spectrum for compound (S8).

Antimicrobial and Anti-fungal

The results of the laboratory test for bacterial activity are as follows, tables 7 and 8:

1. The four aminothiazole derivatives (S1, S2, S3, and S4) all have inhibitory activity for the types of bacteria used, but in different proportions, where the highest among them is S1 and the lowest of this group is S4. In the final result, it was found that among all samples, compound S1 has the highest inhibition of all types of compounds used bacteria [22].

2. Although the concentration of the prepared compounds was doubled, the effectiveness does not reach the level of the standard substance.

Table 7: Bacterial activity of the prepared compounds against Gram-positive bacteria

Type of compounds prepared	Sample codes	Gram positive bacteria					
		Staphylococcus aureus			Bacillus cereus		
Standard	St	18.6			19.6		
concentrations used		200%	100%	50%	200%	100%	50%
aminothiazole	S1	18.1	14	11.2	19.1	14.8	12.2
derivatives	S2	17.1	12.2	10.3	17.4	13	11.3
	S3	14	10	8.2	15.3	11	9
	S4	13.7	9.8	8	14.7	10.7	8.8

Table 8: Bacterial activity of prepared the compounds against Gram- negative bacteria

Type of compounds prepared	Sample codes	Gram-negative bacteria					
		Shigellosis			Pseudomonas		
Standard		17.3			16.5		
concentrations used		200%	100%	50%	200%	100%	50%
aminothiazole	S1	17	14.1	12.2	16.6	13.8	11.9
derivatives	S2	15	12.2	10.3	14.3	11.4	9.6
	S3	13.6	10.7	8.9	12.7	9.6	8.1
	S4	11.9	8.9	7	10.8	7.2	6.2

Evaluation of fungal biological activity of some prepared compounds

The prepared compounds have different biological efficacy against fungi, as the biological efficacy of all the prepared compounds in this study is evaluated on one type of fungus, Candida albicans. For antibiotics, the fungal biological effectiveness of the prepared compounds was evaluated using the method of drilling and measuring the level of inhibition (inhibition zone), and all banned compounds were used in the study of the fungal biological activity. The standard material (Nystatin) is an effective antifungal and is used as a standard approved material in the quality control laboratories of the General Company for Pharmaceutical Industry in Samarra. The results of the in vitro test for fungal activity are as follows, table 9.

• The aminothiazole derivatives S1, S4 have an inhibitory effect on the fungus. [23]

• It is found that among all samples, compound S1 is the highest inhibition of the fungus.

• Although the concentration of the prepared compounds is doubled, the effectiveness does not reach the level of the standard substance [24, 25].

Table 9: Fungicidal activity of the prepared compounds against Candida fungus

Type of compounds prepared	Sample codes	Candida Albicans		
Standard		16		
concentrations used		50%	100%	200%
aminothiazole derivatives	S1	13.1	14.2	15.4
	S4	11	12	13.3

Antioxidant ability

Any compound becomes active when its color changes from purple to yellow, and the concentrated hue is a good indicator of efficiency. Additionally, the substances (S1, S4, S5, S7, and S8) are better sources of antioxidants than gallic acid (the industry standard). Table 10 exhibits it. The present results show the free radical scavenging activity of the efficiency compounds compared to Gallic acid as a standard. It is clear from the collected data that the free radical scavenging activity of the compounds under investigation increased with the increase of concentration showing [26, 27]. This result supports the findings in Figure 7, which show that the S7 has the best capacity for scavenging free radicals.

Table 10: Antioxidant activity percentage of (S1-S8) compounds

St	S1	S4	S5	S7	S8	
%45.72	%29.13	%19.61	%22.56	%32.1	%26.28	Percentage at 25 µg /ml
%52.15	%34.97	%24.51	%27.12	%40.1	%30.31	Percentage at 50 µg /ml
%59.86	%40.12	%31.32	%34.72	%48.13	%36.24	Percentage at 100 µg /ml
%76.41	%44.71	%34.61	%42.86	%57.45	%47.22	Percentage at 200 µg /ml
%94.62	%61.35	%45.47	%56.23	%71.78	%70.52	Percentage at 400 µg /ml
%97.38	%71.52	%51.52	%63.75	%80.14	%80.74	Percentage at 500 µg /ml
31.97 µg/ml	249.17 µg /ml	466.45 µg /ml	319.4 µg /ml	159.11 µg /ml	225.26 µg /ml	IC50 µg /ml

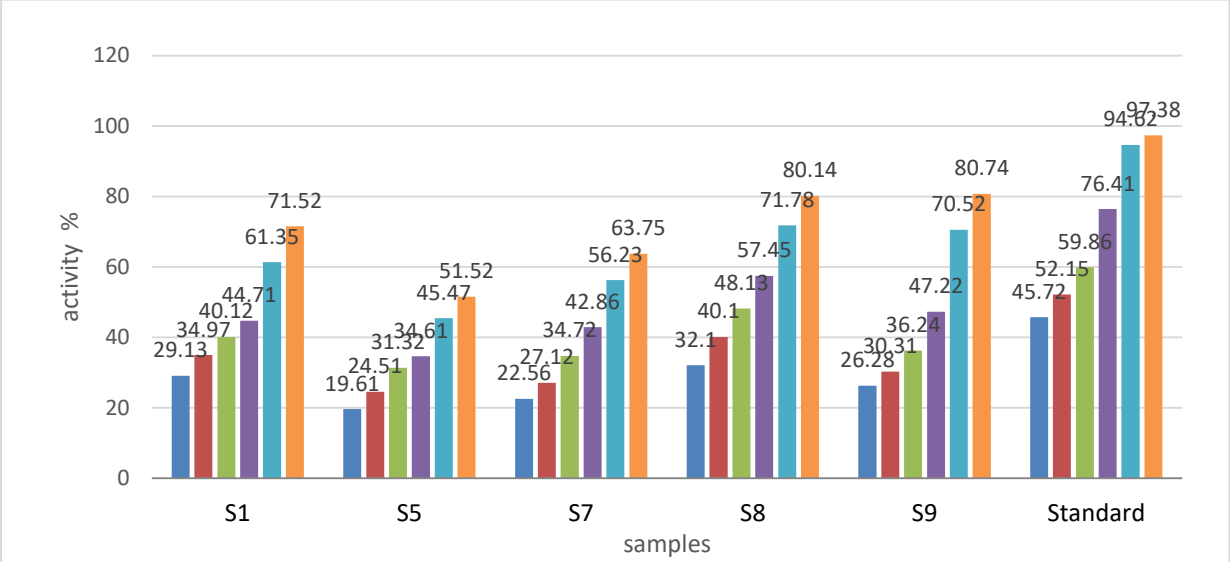


Fig. 7 antioxidant activity of (S1-S8) compounds.

Table 11 and Figure 8 show the specific IC50 values for each compounds.

Table 11: IC50 values of antioxidant activity of (S1-S8) compounds.

Code	St	S1	S4	S5	S7	S8
IC50 (Mm)	31.97	249.17	466.45	319.4	159.11	225.26

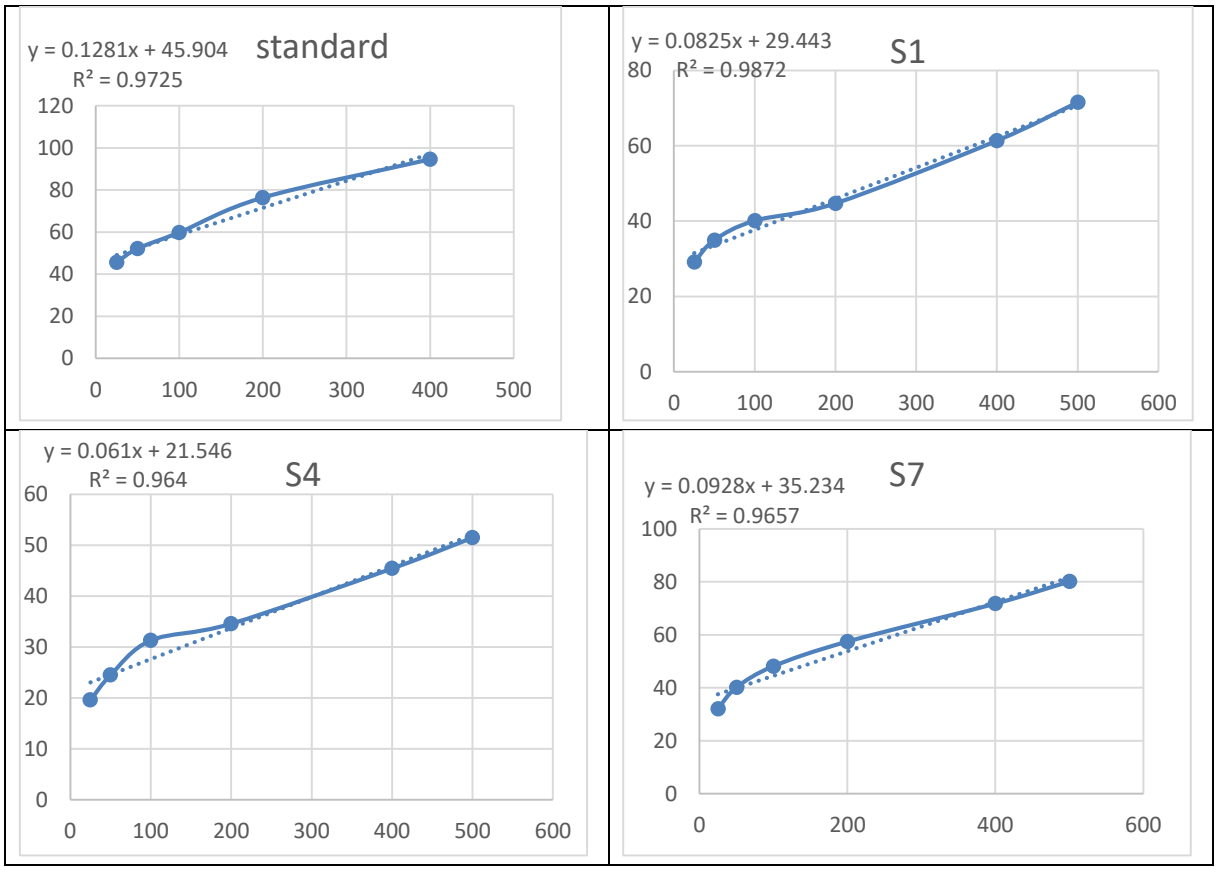


Fig. 8 IC50 antioxidant activity of (S1-S7) compounds.

Conclusion

This study aims to synthesis new 2-aminothiazole compounds and their use in the Synthesis of new Schiff bases and investigate this compound. Schiff bases derived from 2-aminothiazoles were prepared and study the biological, bacterial and antioxidant activity of the prepared compounds were measured it was noted that the prepared compounds had high bacterial and biological effects in addition to their effectiveness as antioxidants.

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تحضير وتشخيص مركبات الامينوثيازول وقواعد شف وتقييم نشاطها البيولوجي كمضادات للميكروبات ومضادات للأكسدة

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الخلاصة:
يتضمن البحث تحضير 2-أمينو-ثيازول من مركبات (سلفانيلاميد، سلفاسيتاميد، سلفاميثوكسازول، 4-أمينو بنزويك أسيد) مع البروم وسيانيد البوتاسيوم في وجود حامض الخليك الثلجي كمذيب باستخدام طريقة التشعيع بالميكروويف (4 -S1 س). تم تحضير قواعد شف عن طريق تفاعل المركبات (S1-S4) مع مشتقات البنزالديهيد مثل (3-هيدروكسي-4-ميثوكسي بنزالديهيد، 4-هيدروكسي بنزالديهايد، 4-كلورو بنزالديهايد، 4-بروموبنزالديهايد). تم تشخيص المركبات المحضرة باستخدام الأشعة تحت الحمراء (FTIR)، وطيف الكتلة، والتحليل الطيفي بالرنين المغناطيسي النووي (H-NMR1)، وكروماتوجرافيا الطبقة الرقيقة (TLC). تم اختبار بعض المركبات المحضرة من أجل الفعالية المضادة للفطريات ضد أنواع المبيضات والنشاط المضاد للبكتيريا ضد عزلات Bacillus Puimilus باستخدام Nystatin و Neomycin sulfate كأدوية مرجعية. تشير النتائج إلى أن المركبات المحضرة لديها القدرة على تثبيط نمو الفطريات والبكتيريا المختبرة بالإضافة الى نشاطها البيولوجي كمضادات للاكسدة.