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Hiba I. Abdullah
Wissam M. R. Al-Joboury
Ibrahim F.O. Aljoboury
Ibtihal K. Hameed
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Mahmod A. Abdulqader

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ORIGINAL STUDY

Synthesis and Characterization of Schiff base Compounds Produced from 6-Bromo-4-Fluoro-2 Aminobenzothazol Assessment against Antibacterial Activity

Hiba I. Abdullah [a], Wissam M. R. Al-Joboury [b], Ibrahim F.O. Aljoboury [c], Ibtihal K. Hameed [d], Khalid A. Al-Badrany [e], Mahmod A. Abdulqader [f,*]

[a] Department of Medical Laboratory Technology, University of Imam Jaafar Al-Sadiq, Balad, Iraq
[b] Department of Medical Laboratory Techniques, Al-Hawija Technical Institute, Northern Technical University
[c] Iraqi Ministry of Education, Kirkuk Education Directorate
[d] Iraqi Ministry of Education, Salahuddin Education Directorate
[e] College of Education, Chemistry Department, Tikrit University
[f] Oil Products Distribution Company (OPDC) Salahuldeen Branch, Ministry of Oil, Tikrit, Iraq

ABSTRACT

Schiff bases exhibit a wide range of biological activities, including antibacterial, antifungal, anticancer, and anti-inflammatory properties. The aim of study was to synthesized 2-amino-benzothiazole derivatives and showed their novel structure leads to potent antimicrobial activity. The Schiff base compounds derived from 6-bromo-4-fluoro-2-aminobenzothazol was prepared from the reaction of 6-bromo-4-fluoro-2-aminobenzothazol compound (W1) with potassium thiocyanate in the presence of bromine. Unsaturated Alpha-Beta (W2-6) compounds were obtained from the reaction of and aromatic benzaldehyd with para –hydroxy acetophenone in a basal medium. The interaction of alpha-beta - unsaturated compounds with 4,6-dichloro-2-aminobenzothiazole gives compounds (W7-11) 4-(1-(4,6-dichlorobenzothiazole-2-El amino) - 3-aryl) phenol. The prepared compounds have been characterized by melting points and some physical properties besides the FTIR, H-NMR spectra and quantitative analysis of elements (C.H.N.). The purity for these compounds was checked by TLC. The result showed the tested compounds (W1-8 have antibacterial activity against Gram-negative bacteria while Compounds (W9-11) showed moderate antibacterial activity against ***S. aureus and Proteus ssp***. In conclusion used the conventional method for the preparation of different substituted 2-aminobenzothiazole derivatives and showed remarkable antibacterial activity; we need further investigation to study their mechanism of actions at the molecular level.

Keywords: Schiff base, 2-Amino benzothiazoles, Chalcone, Antibacterial, 6-Bromo-4 fluoro-2-aminobenzothazol

1. Introduction

2-Aminobenzothiazole is an important class of heterocycles containing one sulfur and two nitrogen atoms, which is associated with a broad spectrum of medical and pharmacological activities, including antitumor, antibacterial, antimalarial, anti-inflammatory, and antiviral activities [1]. In recent years, an extraordinary collection of potent and low-toxicity 2-aminobenzothiazole compounds have been discovered as new anticancer agents' Heterocyclic compounds, such as benzothiazole, are of great significance in organic chemistry and drug discovery due to their diverse range of physiological functions [2].

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* Corresponding author.
E-mail address: mahmodabdulkarem1978@gmail.com (M. A. Abdulqader).

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Benzothiazole and its derivatives have been extensively studied for their potential biological activities. In the 1950s, derivatives of benzothiazole, particularly 2-aminobenzothiazoles. Benzothiazoles (BTAs) are an important class of bicyclic heterocycles that play a key role in the design of biologically active compounds [3]. At the moment, due to the threat of outbreaks of epidemics associated with the emergence and spread of various viruses (Zika, Lassa, SARS-Cov, etc.), modern research and development in medicinal chemistry and pharmacology based on benzothiazole derivatives have become especially relevant. Recently, the number of publications devoted to the synthesis and study of the pharmacological properties of 2-aminobenzothiazole derivatives has increased significantly. Several review articles are also devoted to this topic [4]. Molecules with a benzothiazole moiety have a pronounced spectrum of biological activity, exhibiting, along with antiviral, 2–5 also antimicrobial [5], anti-inflammatory [6], antidiabetic [7], analgesic [8], antioxidant [9], antidepressant [10], anticonvulsant [11], antianginal [12], antitumor [13], immunomodulatory effects. BTA derivatives also have anthelmintic [14], antimalarial [15], fungicidal [16], insecticidal [17], and herbicidal [18], effects antibacterial, anti-inflammatory, analgesic, anticonvulsant, antiviral, anthelmintic, anti-oxidant and anticancer properties [19]. Recently, researchers have sought to develop manufacturing processes through scientific research [20].

There are several methods for synthesizing benzothiazole analogues. One common approach involves the condensation process where acyl chlorides, carboxylic acids, esters, nitriles and o-aminothiophenols containing substituted aldehydes are reacted to form benzothiazole derivatives. Another widely employed method is Pd/Cu/Mn/chloranil catalyzed cyclization of o-halothioformanilide. These synthetic methods have enabled the synthesis of numerous benzothiazole derivatives with diverse structural modifications and pharmacological effects. Ongoing research continues to explore the potential applications and properties of benzothiazole compounds and their derivatives [21].

The α, β-unsaturated ketones (chalcones) are considered to be precursors of flavonoids and is flavonoids and found as naturally-occurring compounds, but it could be considered that their true importance is extended in two branches. The biological activity associated with them, including anti-inflammatory [22], antitubercular, cytotoxic, anti-HIV [23], antioxidant, analgesic, antiviral and antimicrobial [24], analgesic [25], anti-tuberculosis [26], anti-fungal [27], anti-malarial [23], Schiff bases are is important class was cligands in coordination chemistry and find extensive application in different fields [28]. Some as of these bases exhibited antimicrobials and anticancer or activities the thiazolidin-4-one ring have reported to a wide range of pharmacologic activities which include antimicrobial [29], antifungal, antitumor, antidiabetic activity [30], anti-inflammatory and stomach toxicity [31]. Previous results proved that an SH moiety at the 2 position of the heterocyclic nucleus significantly increases the antibacterial activity. The new of this work prepared and synthesize 2-amino-benzothiazole derivatives, it was structural leads to potent antimicrobial activity. The aim of study was to synthesized 2-amino-benzothiazole derivatives and showed their novel structure leads to potent antimicrobial activity. The study of the biological effectiveness of some of the compounds under study on different types of positive and negative bacteria for dye-gram.

Table 1. Chemicals were used in this work.

Chemicals	Company	Purity
Substituted aniline	Aldrich	99%
Potassium thiocyanate	Aldrich	98%
Acetic acid	Aldrich	98%
Bromine	Fluka	89%
Sodium hydroxide	Fluka	99%
Acetophenone	Fluka	98%
Benzaldehyde	Aldrich	98%
Ethanol	Aldrich	89%

2. Materials and methods

Melting points were recorded uncorrected in an open capillary tube using the Stuart melting point equipment. Infrared spectra were acquired using a Shimadzo FTIR-8100 spectrophotometer using KBr discs and 1H NMR. Spectra were measured using an MHZ spectrometer with DMSO-d6 as the solvent. TLC on silica gel-percolated alumni sheets (type 60 F254 Merck, Darmstadt, Germany) was used to monitor reaction progress and verify chemical purity.

2.1. Chemicals

The chemicals and solvents were used from Aldrich and Fluka, and they were utilized without additional purification. Table 1 shows the materials and chemicals were employed in current research.

2.2. Synthesis of 6-bromo-4-fluoro-2-aminobenzothazol (W1)

Fig. 1 depicts the structure of Nucleus 6-bromo-4-fluoro-2-aminobenzothazol. A solution of (0.1 mole) of substituted aniline and (0.4 mole) of potassium

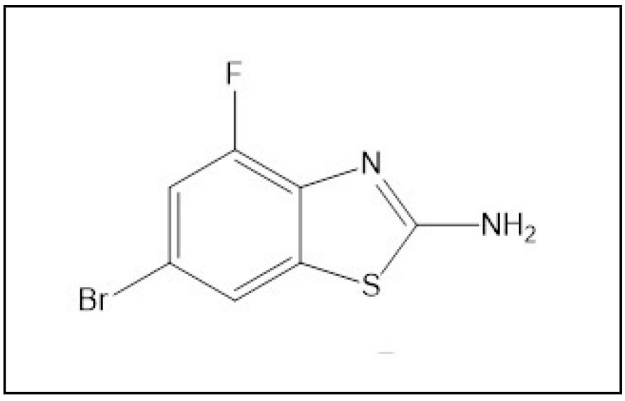

Fig. 1. Nucleus 6-bromo-4-fluoro-2-aminobenzothazol.

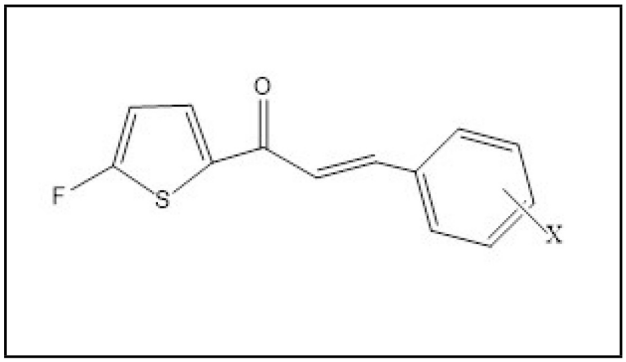

Fig. 2. Nucleus chalcones structure.

thiocyanate in (150) mL of 98% acetic acid glacial was added drupe-wise, with stirrings (16 g) (0.1 mole) of bromine dissolved in 50 mL of glacial acetic acid while the temperature was held below (10°C). After adding all of the bromine solutions, the mixture was agitated for ten hours. The combined filtered solutions are dissolved in warm water, (the temperature of water 60 to 70°C). The combined filtrate was neutralized with 10% sodium hydroxide. The precipitate was collected using a filter and dried. Recrystallization from an appropriate solvent. The physical parameters of the produced chemical (W1) include color white, M.P. (183–185°C), and yield 76% [32].

2.3. Synthesis of chalcones (W2-6)

The physical data of compounds (W2-6) are shows in Table 2. While Fig. 2 shows the Nucleus chalcones A mixture of appropriate acetophenone (0.01 mol) and aromatic benzaldehyde (0.01 mol) have been added to a solution of (10 %) sodium hydroxide 5 mL and 3 mL of ethanol. The mixture was stirred for (2–3) h at (20–40°C) and kept in a refrigerator for 12 hr. Then it was diluted with ice-cold distilled water (30 ml), filtered washed with cold water, dried in air and recrystallized from ethanol [33]. The physical data of compounds (W2-6) are shows in Table 1.

2.4. Synthesis of schiff base (W7-11)

Table 3 shows the physical parameters of compounds (W7–W11). Figs. 2 and 3 depict the structure of the Nucleus Schiff base and the path of preparation (W1-11), respectively. Dissolve (0.001 Mol, 0.219 g) of 2-amino benzothiazole in 10 ml of ethanol, then add (0.001 Mol, 0.269 g) of one of the chalcones compensators (W2-6) dissolved in 10 ml of ethanol, ascend the mixture for 6 h, filter the solution, and dry [34].

2.5. Selection of anti-bacterial activity of compounds

In this study two species of pathogenic bacteria have been used ***S. aureus and Proteus ssp***. The two species are important in the medical aspect in resistance against antibiotics and took these types of

Table 2. Physical properties of compounds (W2-6).

Compd. N0.	X	Melting point (°C)	Yield (%)	Color	Molecular Formula
W2	4-OCH_3	72–75	70	Yellow	$C_{14}H_{11}FO_2S$
W3	4-NO_2	96–98	44	Orange	$C_{13}H_8FNO_3S$
W4	4-Cl	58–60	61	White	$C_{13}H_8CLFOS$
W5	4-Br	64–67	71	Yellow	$C_{13}H_8BrFOS$
W6	4-F	78–80	78	Yellow	$C_{13}H_8F_2OS$

Table 3. Physical properties of compounds (W7-W11).

Compd. No.	Ar	X	Melting point (°C)	Yield (%)	Rf	Color	Formulas
W7	2-ammino-4-floro-6-bromo benzothiazole	4-OCH_3	142–145	67	0.72	White	$C_{21}H_{13}BrF_2N_2OS_2$
W8	2-ammino-4-floro-6-bromo benzothiazole	4-NO_2	217–219	71	0.86	Yellow	$C_{20}H_{10}BrF_2N_3O_2S_2$
W9	2-ammino-4-floro-6-bromo benzothiazole	4-Br	236–238	78	0.76	Whit	C_{20}H10Br2F2N2S2
W10	2-ammino-4-floro-6-bromo benzothiazole	4-Cl	214–216	51	0.71	Whit	C20$H_{10}BrClF_2N_2S_2$
W11	2-ammino-4-floro-6-bromo benzothiazole	4-F	112–115	63	0.63	Yellow	$C_{20}H_{10}BrF_3N_2S_2$

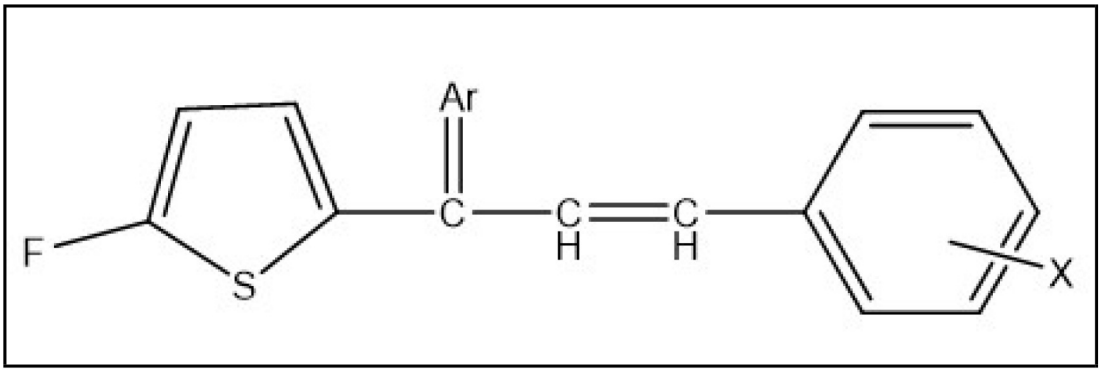

Fig. 3. Nucleus schiff base structure.

bacteria ready and isolated, this test has been done in the following ways [35].

2.6. Cultivation media

Nutrient broth has been prepared according to the methods of the manufacturing and sterilized in the auto clave in 121°C for 15 min under pressure (15 Mpa) and then poured into the dishes or tubes and leaving cooled. Molephantin, this medium is used to measure the biological activity of antibiotics and pharmaceuticals. This medium is used to measure, the diameter of inhibition zone [33].

2.7. Biological activity evaluation

Fig. 4 shown the route of prepared compounds (W1-11). The antibacterial properties of the produced compounds were assessed in vitro against numerous pathogenic representative microorganisms *(S. aureus and Proteus spp.)* using the Agar well-diffusion method (40) [36]. Ciprofloxacin was employed as a reference medicine to investigate the possible actions of these compounds. All of the compounds were evaluated at various concentration levels (0.01, 0.001, and 0.0001 mg/mL), with DMSO serving as both a solvent and a control [37]. The inhibition zone diameter in millimeters (IZD) was employed to determine antibacterial activity. To determine the minimal inhibitory concentration (MIC, μg/mL), all substances were tested and compared to the control. The analysis of antibacterial screening data revealed that 6-bromo-4-fluoro-2-aminobenzothazol (W1) [36]. Compounds (W2, W3, W5, W6, W7, and W8) had good antibacterial action against both gram-negative bacteria *(S. aureus)* [38]. Compounds (W9, W10, and W1) also demonstrated good antibacterial action against gram-positive bacteria (proteus spp.) and high activity against all microorganisms tested, in contrast to ciprofloxacin derivatives. All substances had a maximal activity of 12.5 μg/mL. The sizes of the inhibition

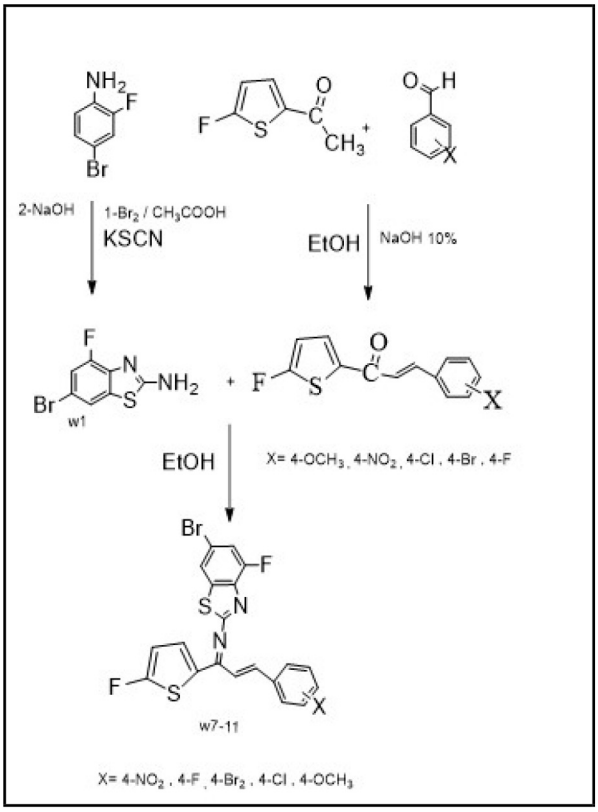

Fig. 4. Route of prepared compounds (W1-11).

zones around each hole were measured in millimeters using Prescott's approach.

3. Results and discussions

3.1. Characterization of 6-bromo-4-fluoro 2-amino benzothiazoles (W1)

The structure of the is prepared compounds are confirmed by spectroscopic is methods such as; FTIR, ^{1}H-NMR. The IR spectrums of compounds (W_1) show a starting -bromo-4-fluoro 2-amino benzothiazoles required for the preparation of Schiff base are was -bromo-4-fluoro 2-amino benzothiazoles the, IR spec-

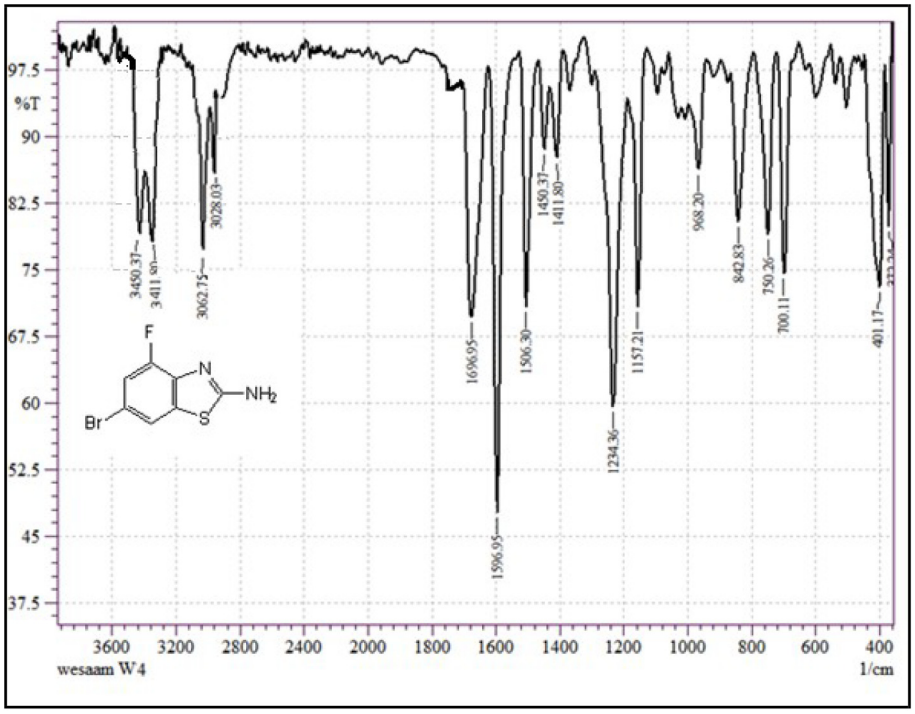

Fig. 5. FTIR spectrum of compound (W_1).

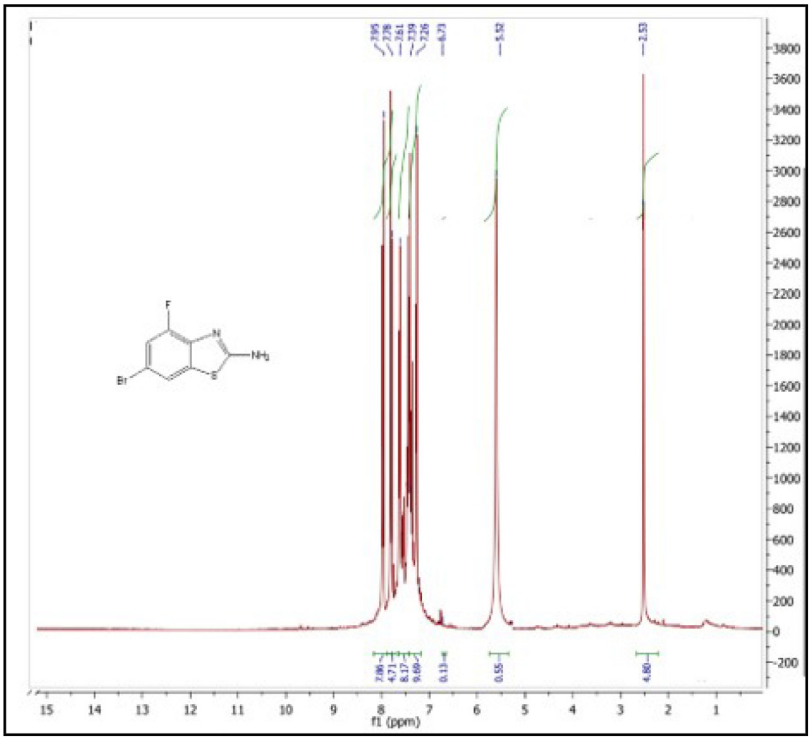

Fig. 6. ^{1}H-NMR spectrum of compound (W_1).

tra of these compounds showed a band at (1625 cm^{-1}) [39], due to stretching (C = N) group band at (1446–1581 cm^{-1}) for (C = C) group at (1267 cm^{-1}) for (C-Br) band at (1038 cm^{-1}) for (C-S-C) group bat, (3180 cm^{-1}) for (Ar-H) at (3455–3355 cm^{-1}) for (NH_2) group. IR data showed in the Fig. 5 [40]. The H^1-NMR Spectrum ($CDCl_3$) of compounds (W_1) Show signal at (2.50 ppm) for (DMSO), signal at (6.95,693 ppm) for (Ar, H), signal at (6.03 ppm) for (NH_2), the H^1-NMR showed in the Fig. 6 [41].

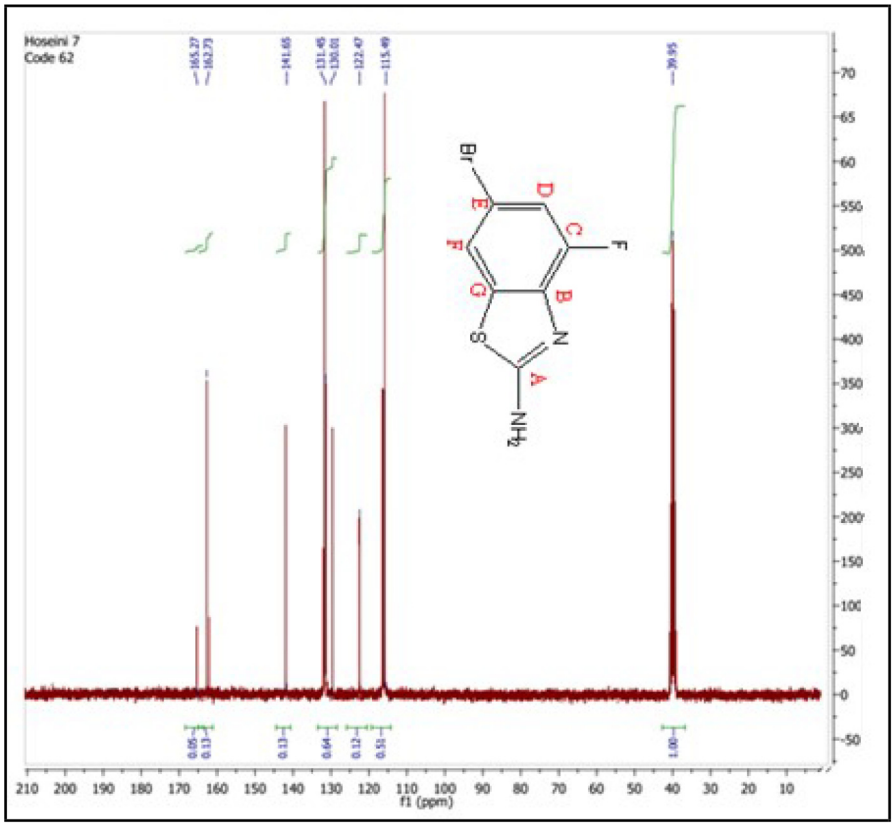

Fig. 7. CNMR spectrum of compound (W_1).

Table 4. FTIR spectral data of Compounds (W2-W6).

		FTIR cm^{-1} (KBr)				
Compd. No.	X	-C=O	(Ar-H)	(C=C)	(C=C)	Others cm^{-1}
W2	4-OCH_3	1670	3011	1591	1527	2840 for C-H aliphatic
W3	4-NO_2	1652	3025	1598	1512	1345 for -NO_2
W4	4-Cl	1665	3008	1592	1539	830 for Cl
W5	4-Br	1661	2997	1602	1525	779 for C-Br
W6	4-F	1674	3006	1583	1479	823 for C-F

The 13C NMR spectra of all compound (W1) were characterized characterized (cf. Exper. Part) [42]. The resonances in the range 166.5–ppm due to carbon thiazoles rings (C-A), showed signal $\delta =$ 134 ppm due to (carbon-B), as well as signal $\delta =$ (148) due to (carbon-C), showed signal $\delta =$ 116 ppm due to (carbon-D) [43], as well as signal $\delta =$ (148) due to (carbon-E), and signal at $\delta =$ 129 ppm due to (carbon-F) as well as singlet signal at $\delta =$ 39.49–40.49 ppm due to $DMSOd^6$ [33]. The 13C NMR showed in the Fig. 7.

3.2. Characterization of chalcones (W2-6)

Table 4 shows FTIR spectral data of Compounds (W2-W6). The synthesis of chalcones derivatives were performed [44]. The reaction of acetophenon with aromatic aldehydes yielded the compounds (W2-6), the IR spectra of compounds (W_5) showed characteristic (C=O) stretching at (1674 cm^{-1}) and (C=C) stretching frequencies at (1580 cm^{-1}), band at (1479 cm^{-1}) for (C=C) group band at (3006–3085 cm^{-1}) for (Ar-H) group Fig. 8 [45]. ^1H NMR Spectrum of compound (W_5), Fig. 9 showed the following signals: a singlet signal at (δ 2.50 ppm) due to a proton of (DMSO), signal at (6.95,693 ppm) for (Ar, H), sharp signal at δ 2.66 ppm due to a proton of phenyl, signal at (7 to 8.5 ppm) for (HC=CH)) Fig. 10 [46]. The 13C NMR spectra of all compound (W5) were characterized characterized (cf. experiment. part). The resonances in the range 166.5–ppm due to carbon thiazoles rings (C-A), showed signal $\delta =$ 134 ppm due to (carbon-B), as well as signal $\delta =$ (148)

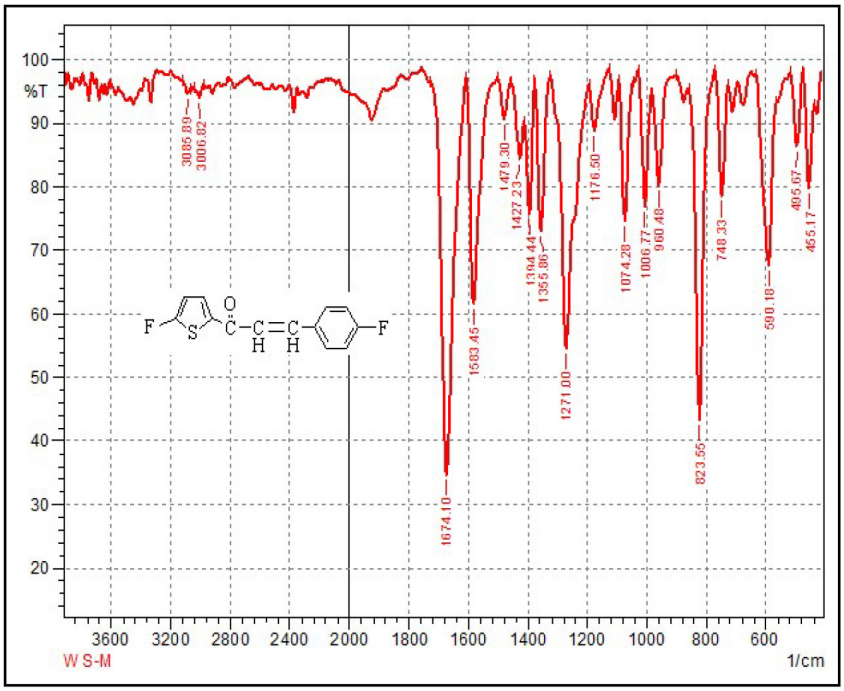

Fig. 8. FTIR spectrum of compound (W5).

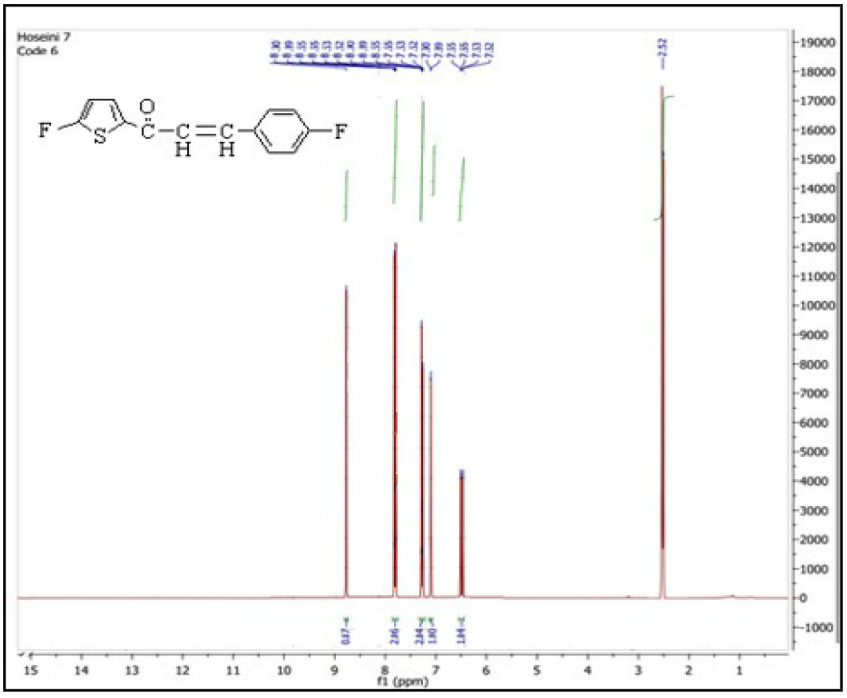

Fig. 9. 1H-NMR spectrum of compound (W5).

due to (carbon-C), showed signal $\delta = 116$ ppm due to (carbon-D), as well as signal $\delta = (148)$ due to (carbon-E), and signal at $\delta = 129$ [47]. As shown in Figs. 8 to 10.

3.3. Characterization of schiff bases (W7-W11)

Fig. 3 shown the FTIR spectral data of compounds (W7-11). Schiff Base derivatives have been produced

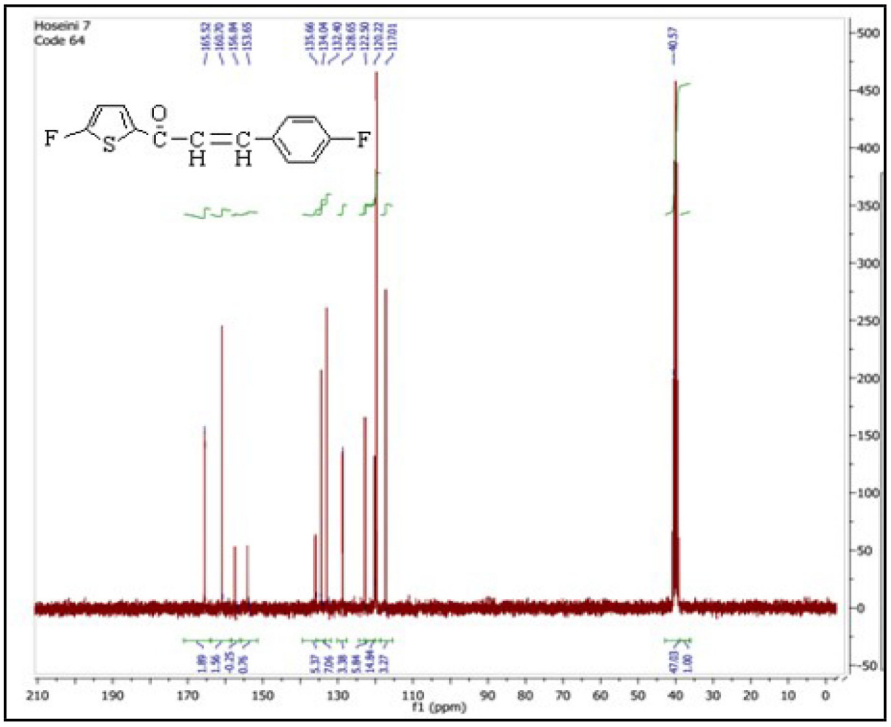

Fig. 10. CNMR spectrum of compound (W5).

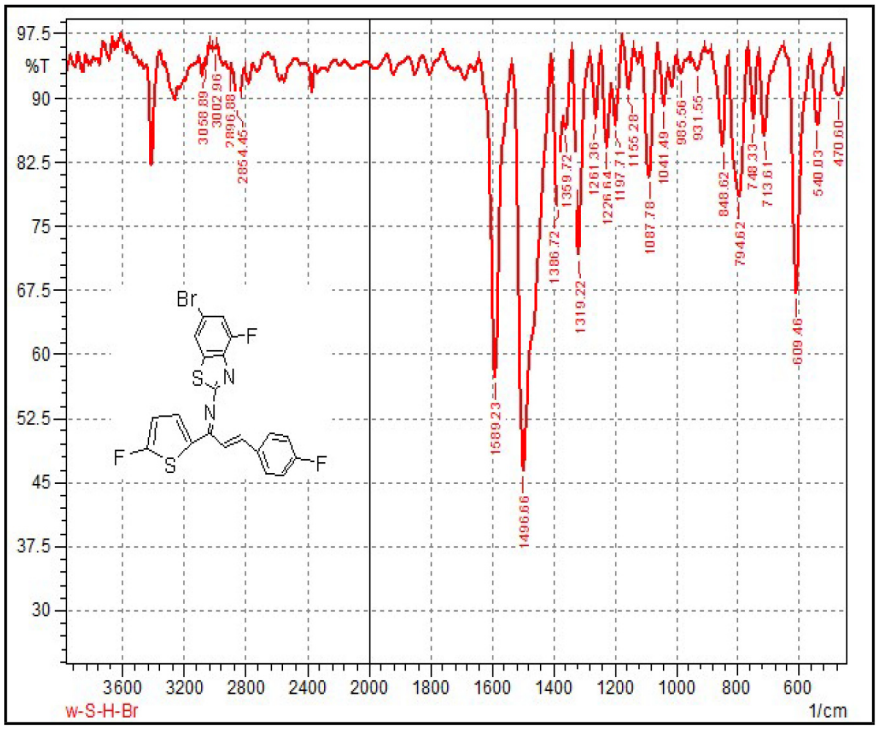

Fig. 11. FTIR spectrum of compound (W10).

by reacting compound (W10) with various aromatic amines. The infrared spectrum revealed beams at the frequency (1599–1643 cm^{-1}) pertaining to the stretching of the pinche (C=N), beams stretching the pinches (C C) at the frequency (1487–1543 cm^{-1}), and beams at the frequency (3097 cm^{-1}) returning [48]. The aromatic pinch stretch (Ar - H), a beam at frequency (1033 cm^{-1}) belonging to the stretch stretch of

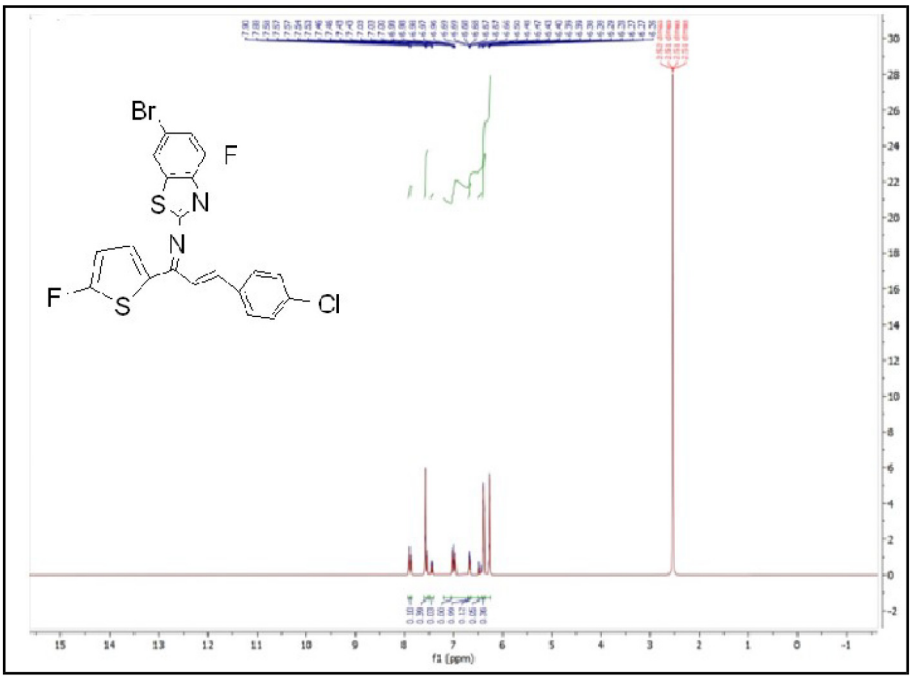

Fig. 12. 1H-NMR spectrum of compound (W10).

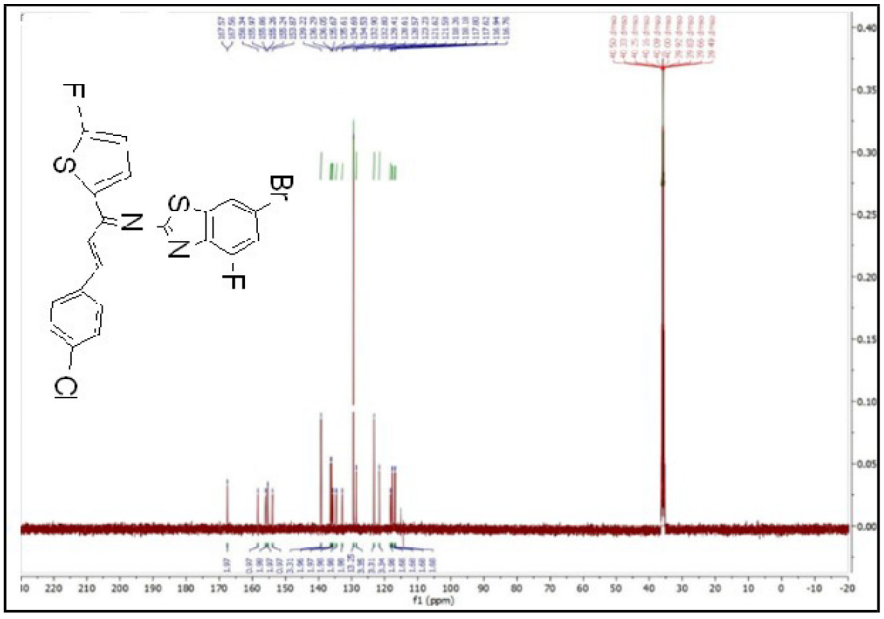

Fig. 13. CNMR spectrum of compound (W10).

the pinch (C - S - C), and the bundle stretching of the pinups (C-Cl) at frequency (813 cm^{-1}), and showed the resonance spectrum Nuclear magnetic H1-NMR of compound E47, a signal at frequency ($\delta = 6.2$ ppm) of a proton (F, CH=C), a signal at frequency (=7.4 ppm) of a proton *(H, CH-phenyl)* [49]. The C13-NMR spectrum of compound E47 showed a signal at frequency (=115 ppm) returning to C (CH=CH), a signal within the range (δ = 123–130 ppm) referring to carbon atoms in benzene rings, a sign at frequency (=149 ppm) returning to C in (C=N), a sign at frequency (=157 ppm) referring to C (C-F), and a signal at frequency (δ = 167 ppm) referring to [50]. As shown in Figs. 11 to 13 and Table 5 [51].

Table 5. FTIR spectral data of compounds (W7-11).

Compd. No.	X	FTIR cm^{-1} (KBr)				
		C-S-C	(Ar-H)	(C=N)	(C=C)	Others cm^{-1}
W7	4-OCH_3	1022	3077	1645	1514	2922 for C-H aliphatic
W8	4-NO_2	1008	3077	1617	1511	1339 for -NO_2
W9	4-Br	11103	3075	1637	1532	784 for Cl
W10	4-Cl	1087	3087	1689	1496	794 for C-F
W11	4-F	1094	3009	1630	1507	834 for C-Br

Table 6. Antibacterial activity of the prepared compounds (W1-W11) and control antibiotic.

Comp. No.	*Proteus ssp* Conc. mg/mL			*Proteus ssp* Conc. mg/mL		
	0.01	0.001	0.0001	0.01	0.001	0.0001
W_1	23	20	18	24	20	18
W_2	32	24	21	13	11	10
W_3	28	29	20	16	14	13
W_4	21	20	13	18	15	14
W_5	22	20	21	21	20	20
W_6	25	21	23	19	13	10
W_7	22	23	21	19	20	20
W_8	23	15	14	24	23	20
W_9	21	23	20	16	15	10
W_{10}	21	22	18	12	11	10
W_{11}	17	21	20	20	15	13
Ciprofloxacin	2	3	3	2	2	4
Blank disk	0	0	0	0	0	0

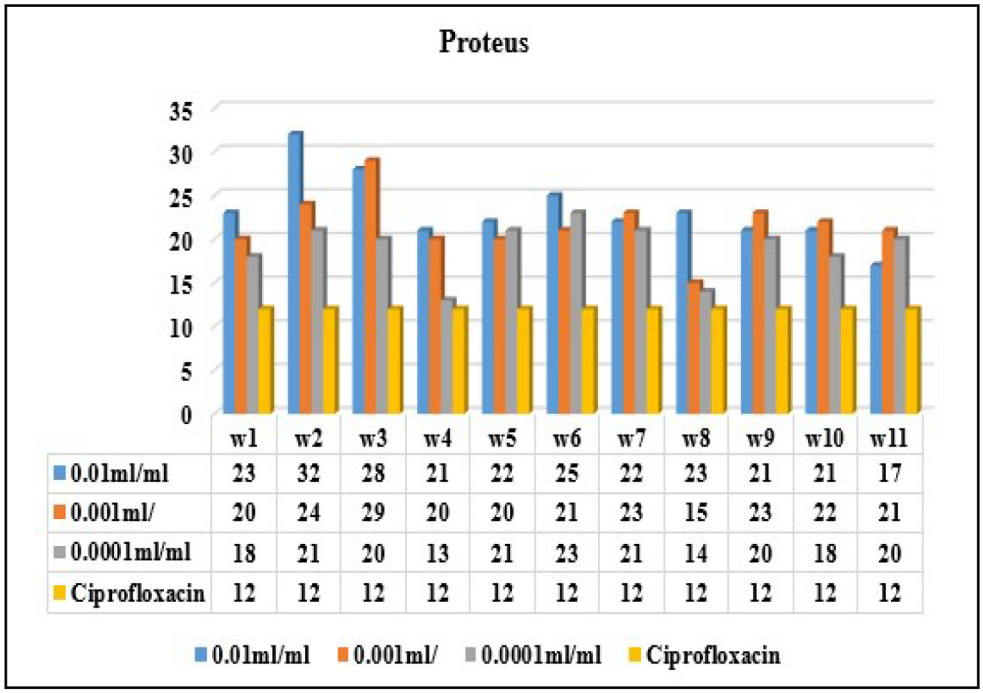

Fig. 14. Evaluation of inhibitory activity of compounds prepared for (*Proteus ssp*).

3.4. Antibacterial activity

Table 6 shows the Antibacterial activity of the prepared compounds (W1-W11) and control antibiotic. Fig. 14 shows the evaluation of inhibitory activity of compounds prepared for (*Proteus ssp*) [52]. While Fig. 15 shows the evaluation of inhibitory activity of compounds prepared for *(S. aureus*) [53]. The produced compounds (W1-W11) were evaluated against several bacterial strains, including *Gram-positive*

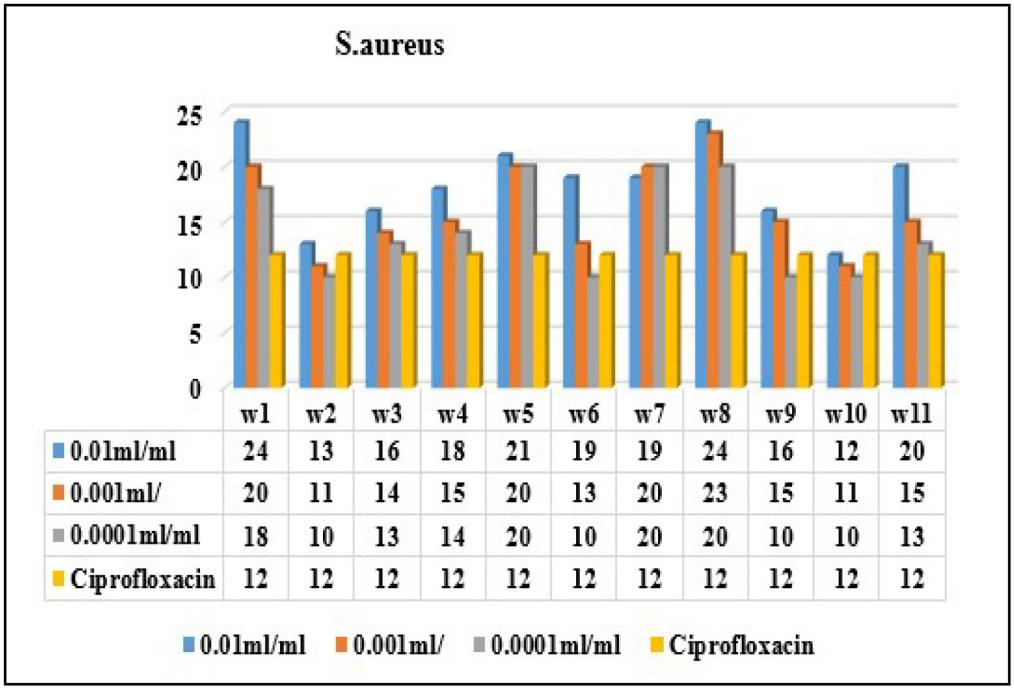

Fig. 15. Evaluation of inhibitory activity of compounds prepared for *(S. aureus)*.

bacteria S. aureus and Gram-negative bacteria Proteus ssp, using the cup plate agar diffusion method [54]. Microbial cultures were cultured at 37°C for 8 h, then diluted with 0.8% sterile saline. The concentration of utilized medicines in DMSO was maintained at 100 μg/mL [55]. Ciprofloxacin was utilized as the negative control [56]. Biological activity was determined by measuring the inhibition diameter of the growth of bacteria around the disk in use [57]. Small activity 15 to 18 mm, moderate activity 18 to 20 mm and high activity 21 to 25 mm, MIC: minimum inhibition concentration (μg/mL) [58].

4. Conclusion

The correctness and validity of the prepared compounds were determined by spectroscopic and physical measurements, with the infrared spectrum accurately proving the presence of active aggregates, and this confirmation increased the NMR spectrum of the proton and the carbon spectrum, which accurately agreed on the correctness of the prepared compounds' structures. These chemicals remain stable at laboratory temperature and do not breakdown or alter color. The synthesized compounds exhibited high and good inhibitory action against Gram-positive and Gram-negative bacteria, and the results were compared to those of amoxicillin, which served as a control sample.

Conflict of interest

The author declares no conflict interest.

Ethical approval

Not applicable.

Data availability

The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

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Author contributions

Hiba I. Abdullah: formal analysis, validation, data curation, writing—original, Wissam M. R. Al-Joboury: data curation, writing—original, Ibrahim F.O. Aljoboury: conceptualization, methodology, writing review and editing, Ibtihal K. Hameed: research administration, conceptualization, Khalid A. Al-Badrany: methodology, writing—review and

editing, Mahmod A. Abdulqader: supervision, research administration.

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