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Synthesis, Characterization and Studying Biological Activity of New Schiff Base Compounds Derived from "N-[(4-hydroxy-3-methoxyphenyl) methyl]-8-methyl-(E)-6-nonenamide"

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RESEARCH ARTICLE

Synthesis, Characterization and Studying Biological Activity of New Schiff Base Compounds Derived from "N-[(4-hydroxy-3-methoxyphenyl) methyl]-8-methyl-(E)-6-nonenamide"

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ABSTRACT

This research includes synthesizing new bis-Schiff bases linked to"N-[(4-hydroxy-3-methoxyphenyl) methyl]-8-methyl-(E)-6-nonenamide" derivatives. This work involved ring opening of phathalic anhydride by hydroxyl groups in "N-[(4-hydroxy-3-methoxyphenyl) methyl]-8-methyl-(E)-6-nonenamide" then, they were reacted with thiourea to synthesize Schiff bases by the reaction of different aldehydes. The first step involved the condensation reaction between "N-[(4-hydroxy-3-methoxyphenyl)methyl]-8-methyl-(E)-6-nonenamide" (H) and phthalic anhydride to produce(H1). In the second step, compound H1 was reacted with one mol of thiourea to produce maliamic compound (H2). The third step involved the preparing of Schiff bases derivatives, by the reaction of (H2) with different aldehyde to produce compounds (H3 and H4). These compounds were characterized depending on their FT-IR, ^{1}H-NMR, and the biological study. The newly synthesized target compounds are expected to be very active biologically since their molecules are essential components of two active groups (imine and amide).

Keywords: Antimicrobial, Capsaicin, Phthalic anhydride, Ring opening, Schiff base

Introduction

For centuries, the compound "N-[(4-hydroxy-3-methoxyphenyl)methyl]-8-methyl-(E)-6-nonenamide" has been around the world as an important source of medicine, food, and seasoning.[1] It has been used to treat a variety of challenging conditions, including diabetic neuropathy and arthritis.[2] However, this compound has extensive neuroprotective, direct exacerbating effects on the mucosal layer and skin.[3] In addition, it is insoluble in water and because of these drawbacks it cannot be used as an oral drug or as a food additive. In order to enhance their bioavailability and pharmacological properties, glycosylation makes it possible to transform organic compounds that are water-insoluble but unstable into those that are water-soluble and stable in addition.[4] Functional food ingredient glycosides have recently been shown to have anti-allergic properties.[5,6] The "N-[(4-hydroxy-3-methoxyphenyl) methyl]-8-methyl-(E)-6-nonenamide" is a pepper plant-derived toxic white crystal solid. It is a white, crystalline alkaloid that is soluble in alcohol, oils, and fats. Its molecular weight is (305.40 g/mol) and its chemical formula is ($C_{18}H_{27}NO_3$). It may be useful in controlling mucositis caused by chemotherapy and radiotherapy.[7]

It is odorless, hydrophobic, and highly volatile with a melting point of (62–65) ˚C. It contains a vanillin group as a structural feature and is divided into three sections. Barbero et al.[8] Bioactivity is enhanced by teral chain lengths of 8 to 9 carbon atoms. The main group contains a vanillyl group, a hydrophobic group, and a substituted benzyl group (acetyl chain), all of

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which are essential for high potency and connected by a central amide bond with vanillyl amines of varying chain lengths. "N-[(4-hydroxy-3-methoxyphenyl) methyl]-8-methyl-(E)-6-nonenamide" is also used to treat peripheral nerve complaints caused by shingles, such as post-herpetic neuralgia.[9] The U.S. "Food and Drug" Administration (FDA) authorized the use of "N-[(4-hydroxy-3-methoxyphenyl)methyl]-8-methyl-(E)-6-nonenamide" transdermal patch (Qutenza) in 2009 as a treatment. To limit (postherpetic neuralgia).[10,11] It has been used as a sedative, but care must be taken when handling it.

It acts as an irritant and allergen when in contact with skin and eyes, or when swallowed or inhaled, and can cause stinging or inflamed skin. Moreover, if ingested in small amounts by children or in large quantities by adults, they can cause illness, vomiting, burning diarrhea, and stomach pain. Exposure to light results in blepharospasm, conjunctivitis, stridor, eye discomfort, and lacrimation.[12]

Schiff.[13] First described in 1864, Schiff bases are a condensation reaction of carbonyl compounds with primary amines. All of these compounds share an azomethine group that can be substituted in several ways and its general formula is RHC = N-R_1, where R and R_1 are alkyl groups, aryl groups, homocyclic or heterocyclic compounds. It should be noted that these compounds have other names, such as azomethines, imines, and anils. As shown by a number of studies, the presence of an electron pair in the Sp^2 orbital of the nitrogen isotope of the azomethine group is critical for both molecular and biological processes. As shown in a number of studies, the presence of an electron pair in the sp^2 orbital of the nitrogen isotope of the azomethine group is crucial for both molecular and biological processes, as this group has the property of flexibility, especially when there are functional groups close to it, for example, groups (-OH) or (-SH).[14] The flexibility of Schiff's base conjunctions has made them very useful in modern organic and scientific uses. Since Hugo Schiff's report[15] on main amine condensation with carbonyl chemicals, Schiff bases have been known. Schiff base coordination chemistry research has grown enormously in the recent years. Clinical properties were demonstrated by O-phenylenediamine Schiff bases. It was reported that Isatin Schiff bases have antiviral, anti-HIV, antiprotozoal, and antihelmintic properties. In addition to their other pharmacological properties, they also have significant anticonvulsant properties. Certain cobalt Schiff base edifices are powerful antiviral specialists. Schiff bases that are made from 4-dimethylamine benzaldehyde have the ability to kill, bacteria, antibodies and anti-inflammatory medications.

Materials and methods

Aldrich and Merck supplied "N-[(4-hydroxy-3-methoxyphenyl) methyl]-8-methyl-(E)-6-nonenamide", phthalic anhydride, thionyl chloride, and all other chemicals, respectively. Shimadzu spectrometer (4000–400cm^{-1}) FTIR spectroscopy and ^{1}H-NMR in Dimethylsulphoxide (DMSO-*d6*) were used to characterize organic compounds and the active groups in them. Precision Digital Melting Point Instrument was used for recording melting points. A Varian-400 MHz spectrometer with TMS (Tetra methyl silane) as a reference was used to capture ^{1}H-NMR.

Ring opening of phthalic anhydride by (N-[(4-hydroxy-3-methoxyphenyl) methyl]-8-methyl-(E)-6-nonenamide) ((H1)

(1 gm., 1 mol.) of "N-[(4-hydroxy-3-methoxy phenyl) methyl]-8-methyl-(E)-6-nonenamide" was mixed in 6 mL of DMF in a 50 mL round flask fitted with a condenser and magnetic stirrer A clear red solution was obtained. (1) ml (0.01 N) of sodium hydroxide was added to the solution. (1 gm.) of phthalic anhydride was dissolved in (1) ml of DMF the mixture was refluxed at about 70 ° C for 6 hr. The product (H1) was dissolved in 15 ml of DMF, then 5 drops of $SOCl_2$ were added to the mixture, and the mixture was refluxed for 1\2 hr.[16] The reddish-orange colored compounds were obtained and dried in a vacuum oven at 60 °C for 48 hr. Afterward, it was washed with 10 ml of diethyl ether to remove any remaining component with yield (80%; m.p.348 °C).

Preparation of (E)-N-((3-methoxy-4-(oxo-l3-methoxy) phenyl)methyl)-9-methyldec-7-enamide) (H2)

An amount of (2.5 gm., 1 mol) of H1 was been dissolved in 6 ml of DMF, it was mixed with 1 mol thiourea dissolved in 12 ml of ethanol Absolut then the mixture was escalated at 70 °C for a period of 4 hr.[17] The light brown products were dried in a vacuum oven at 60 °C overnight and washed with 10 ml of diethyl ether to remove any remaining components with yield (70%; m.p.280 °C).

Synthesis of [N-(4-hydroxy-4-butylidene)-l2-azanecarbothioamideo (E)-N-((3-methoxy-4-(oxo-l3-methoxy) phenyl)methyl)-9-methyldec-7-enamide] (H3)

A mixture of propionaldehyde (2.12 gm., 0.01 mol) and (E)-N-((3-methoxy-4-(oxo-l3-methoxy)phenyl)

methyl)-9-methyldec-7-enamide) (H2) (2.7 g, 20 mmol) in 15 ml ethanol absolute and 15 ml glacial acetic acid) was refluxed for (18 hours). When the reflux reached its completion, the resulting dark brown viscous substance was washed with (10 ml) of diethyl ether. The substance was collected and dried in a vacuum oven at 60 °C for 48 hr. Yield (73%; m.p.220 °C).[18]

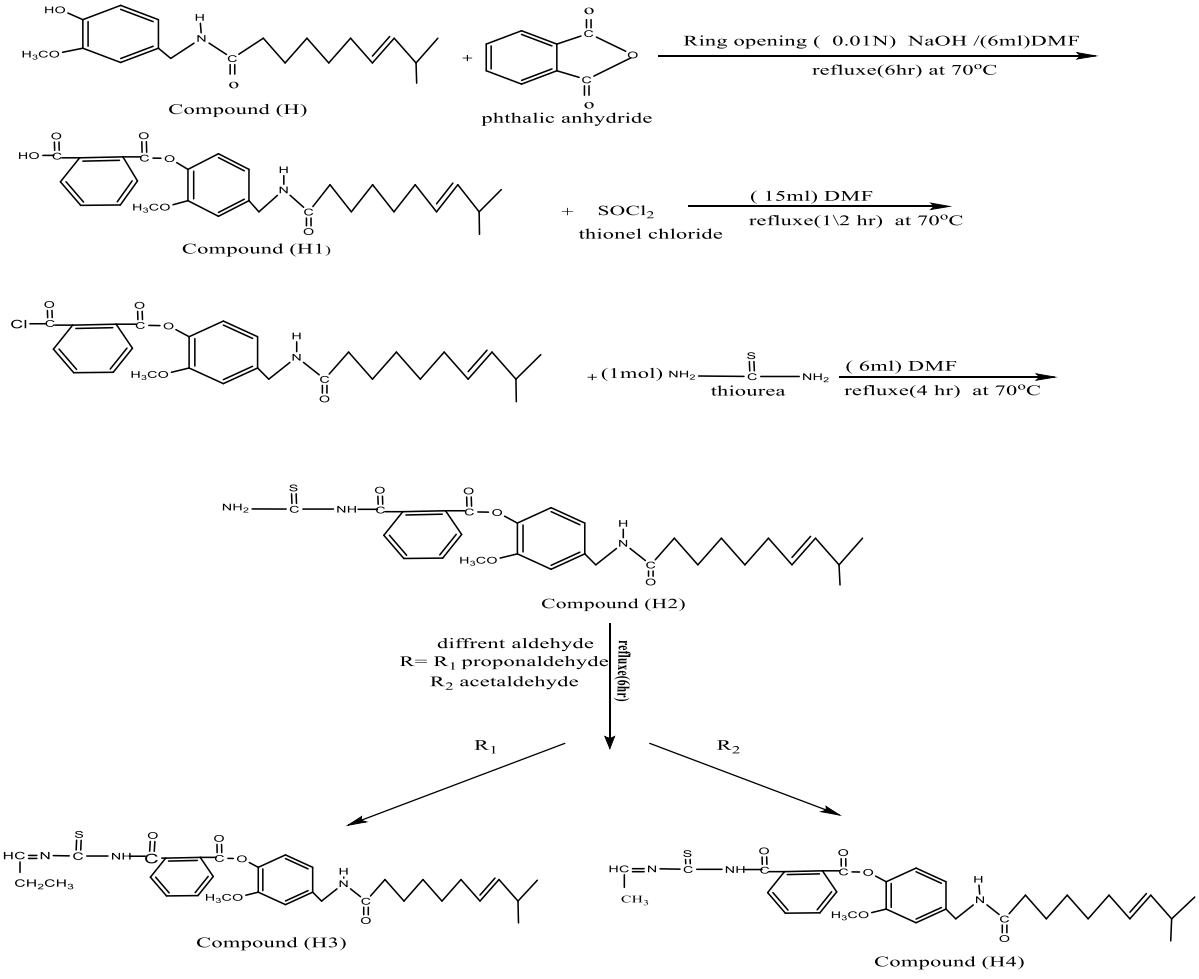
Synthesis of [N-(3-hydroxy-3-propylidene)-l2-(E)-N-((3-methoxy-4-(oxo-l3-methoxy) phenyl) methyl)-9-methyldec-7-enamide] (H4)

A solution of compound (E)-N-((3-methoxy-4-(oxo-l3-methoxy)phenyl)methyl)-9-methyldec-7-enamide) (H2) (2.23 gm., 0.005 mole) and (2 gm., 0.048 mole) of acetaldehyde the reaction mixture was stirred for 12 hours to obtain light brown oil. The material was washed with 10 ml of diethyl ether and then the substance was dried in a vacuum oven at 60 °C for 48 hr. Yield (73%; m.p.240 °C).[19] Derivative compounds were synthesized by FTIR spectra as listed in Table 1. and Figs. 1 to 5, the ^{1}HNMR of the compound in Fig. 6 and Fig. 7, the biological activity studying against bacteria and antifungal in Table 2.

Results and discussion

The aim of this research is to develop novel compounds by using ring opening reactions. The hydroxyl group in "N-[(4-hydroxy-3-methoxyphenyl) methyl]-8-methyl-(E)-6-nonenamide" reacted with phthalic anhydride by attacking of hydroxyl group as nucleophilic. The result components were reacted with one mol of thiourea to give novel compounds' then the second amin groups in thiourea reacted with two aldehydes to give Schiff bases then followed by testing their biological activity.

A general reaction mechanism for "N-[(4-hydroxy-3-methoxyphenyl) methyl]-8-methyl-(E)-6-nonenamide" is shown in Scheme 1. The significant FTIR spectral data comprising the compound's pertinent vibrational bands in the region of



Scheme 1. Preperation of compound H1–H4.

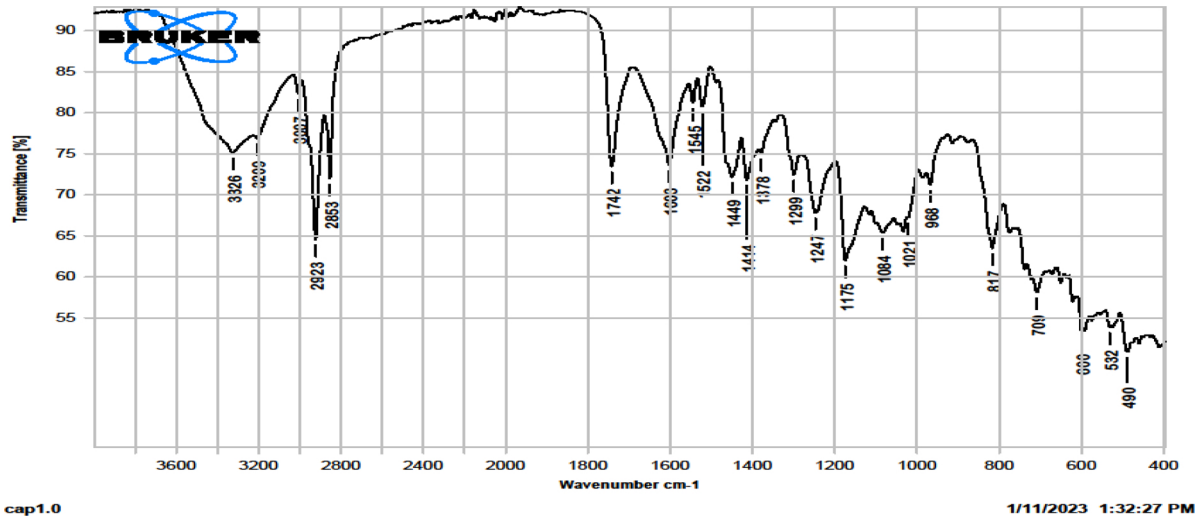


Fig. 1. FT-IR of compound H.

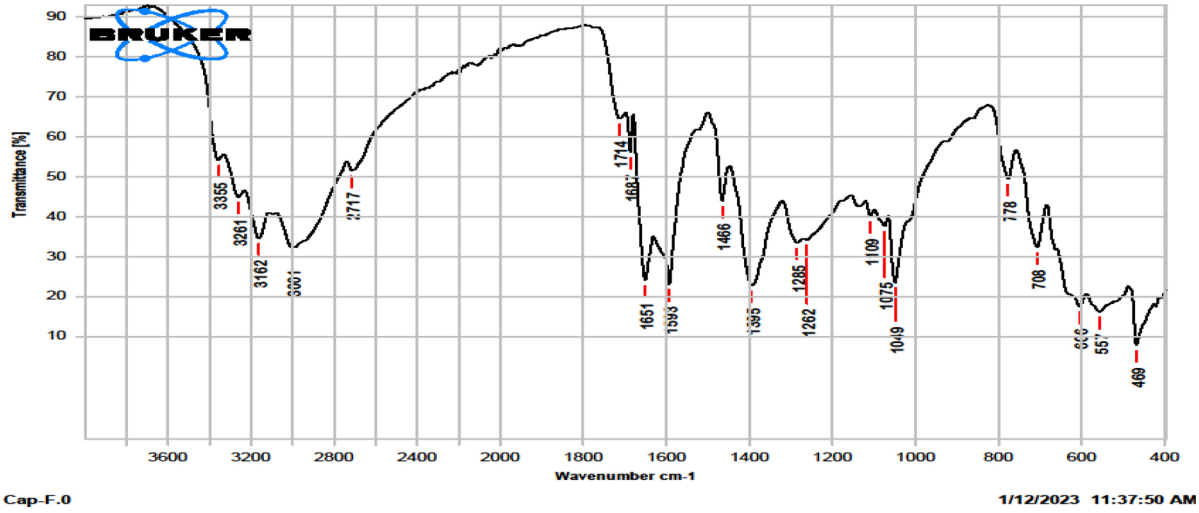


Fig. 2. FT-IR of compound H1.

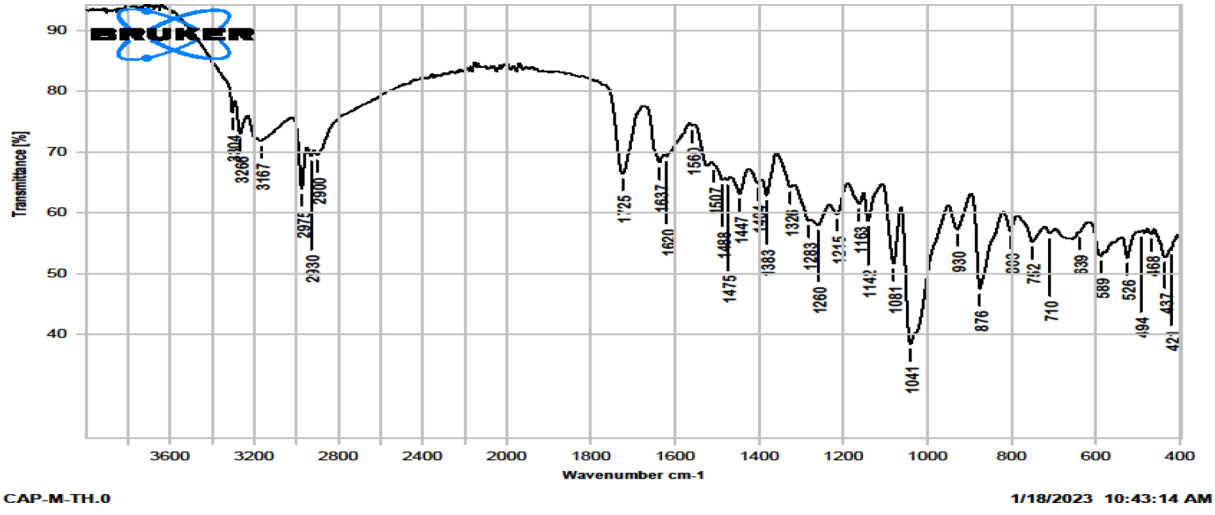


Fig. 3. FT-IR of compound H2.

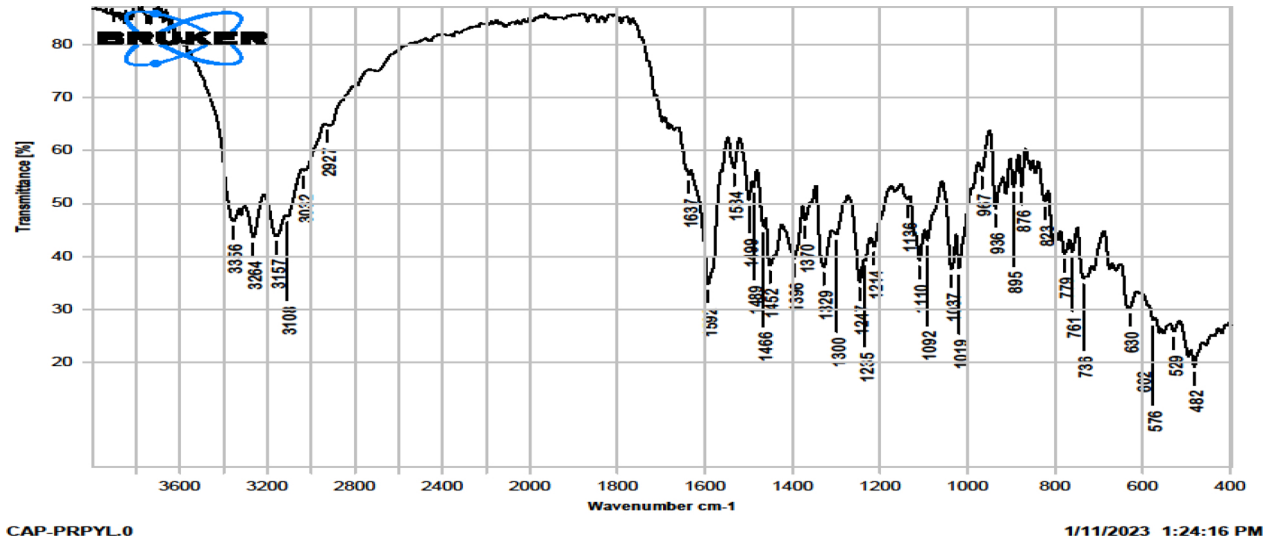


Fig. 4. FT-IR of schiff base compound H3.

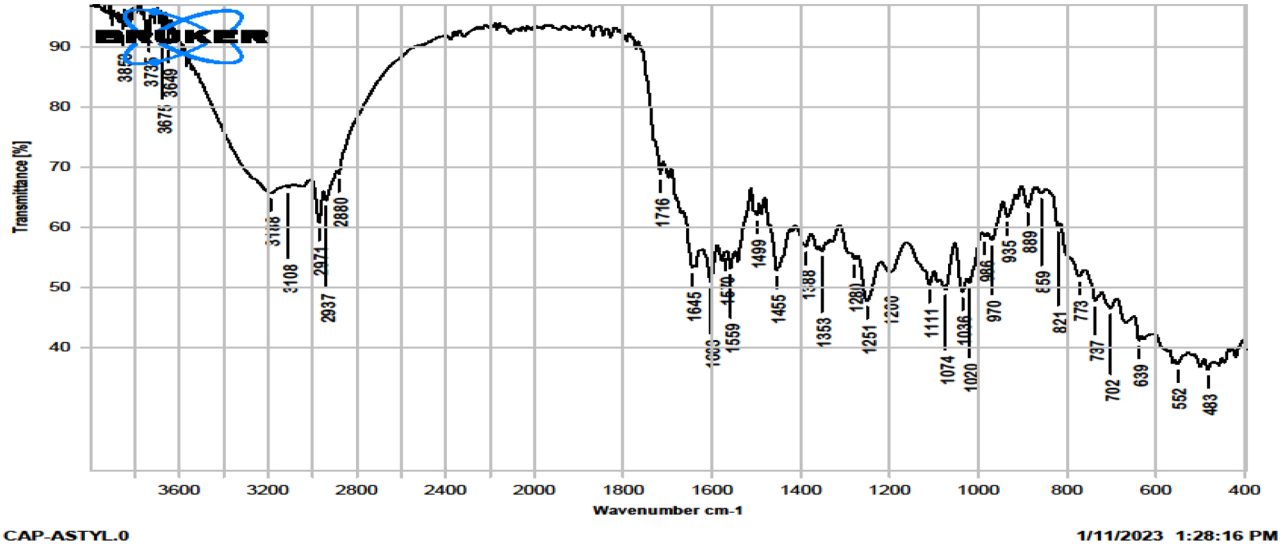


Fig. 5. FT-IR of schiff base compound H4.

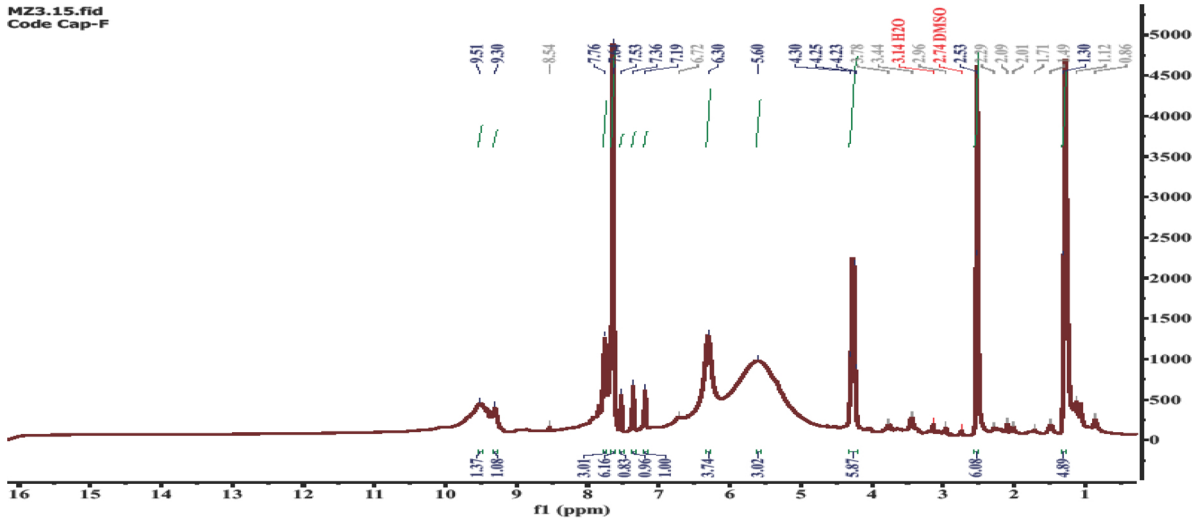


Fig. 6. ^{1}H-NMR of compound H1.

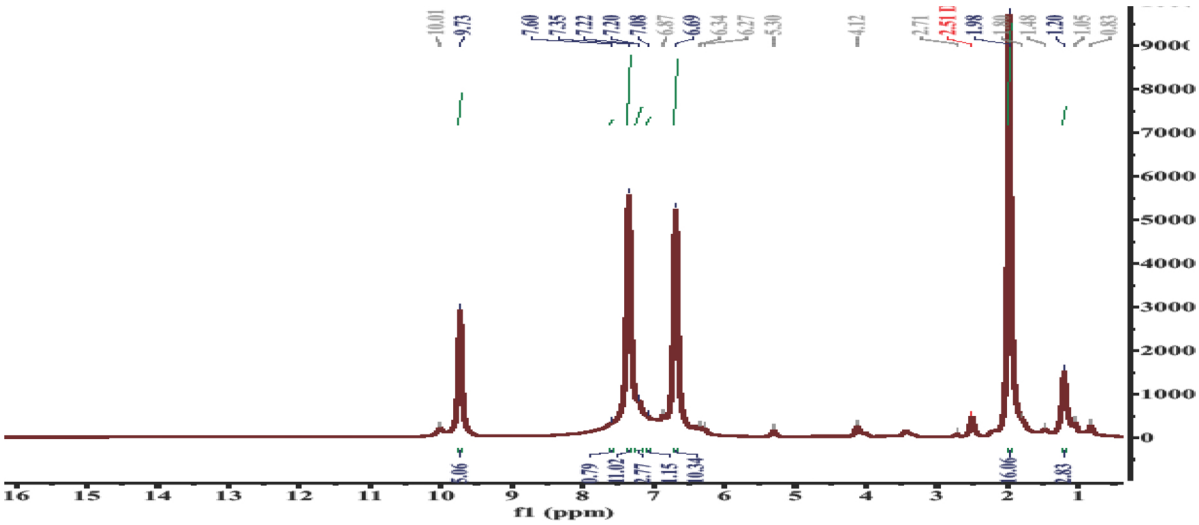


Fig. 7. ^{1}H-NMR of compound H2.

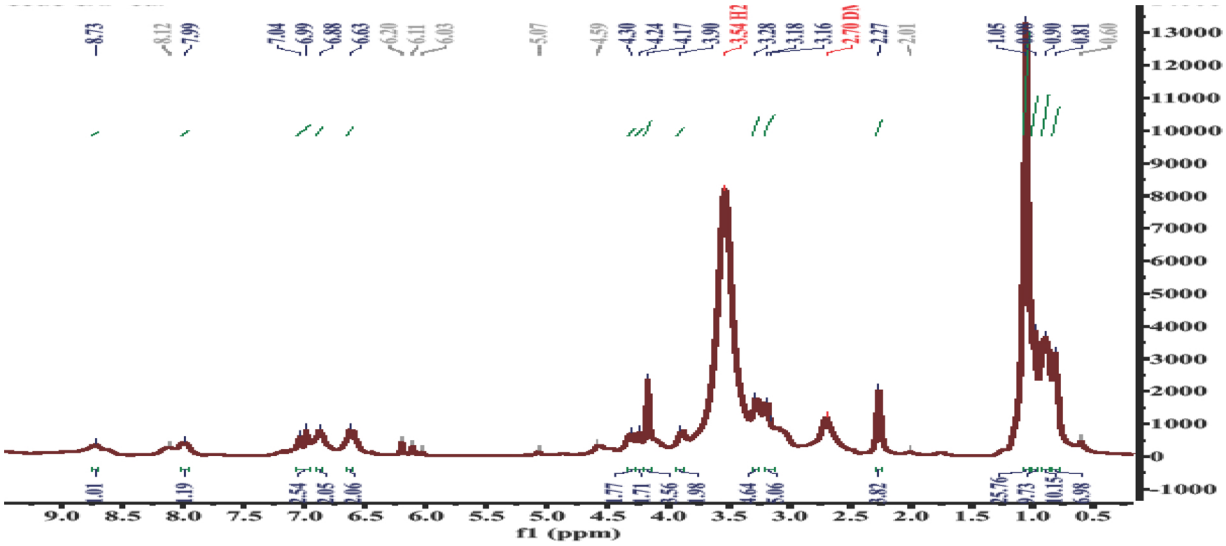


Fig. 8. ^{1}H-NMR of schiff base compound H3.

4000–400 cm^{-1} compound (H1) in Fig. 2. Showed peaks at 3355 cm^{-1} broad band attributed to the absorption of the –OH group, and 3230 cm^{-1} denoting to N-H. The appearance of two bands at 2880–2971 cm^{-1} denoting C-H Alph., as well as the appearance of one band at 3001 cm^{-1} Which, corresponds to C-H Aro., bands at 1651 cm^{-1} denote C=O amide, 1678 cm^{-1} denoting to C=O ester. 1714 cm^{-1} denoting to C=O carboxylic. The second compound was prepared from the reaction of carboxylic with NH_2 in thiourea.The infrared spectra of the compound H2 in Fig. 3. Explain the existence of a band in (3328) cm^{-1} denoting (O-H). The appearance of two bands of 2937–2971 cm^{-1} denoting C-H Ali., and 3001 cm^{-1} indicates C-H Aro., (1600) cm^{-1} denoting to (C=O) amide, while (1645) cm^{-1} denoting to (C=O) ester, while C=C an extended vibration of the benzene rings in this compound appeared as strong bands at 1455 cm^{-1}.the third compound was prepared from reaction of propionaldehyde with thiourea derivatives in absolute ethanol to give compound [H3]. This Schiff base [H3] was recognized by FTIR and ^{1}HNMR spectroscopy. FTIR spectra was shown of H3 in Fig. 4. The disappearance of bands refers to the amine and carbonyl group of the starting materials together with the appearance of new absorption at 1592 cm^{-1} due to the azomethine group and stretching band at 1637 cm^{-1} which refers to C=O of amide and 3356, 3264 cm^{-1} for NH_2. The fourth compound was prepared from the reaction of acetaldehyde with thiourea derivatives in absolute ethanol to give compound [H4]. The

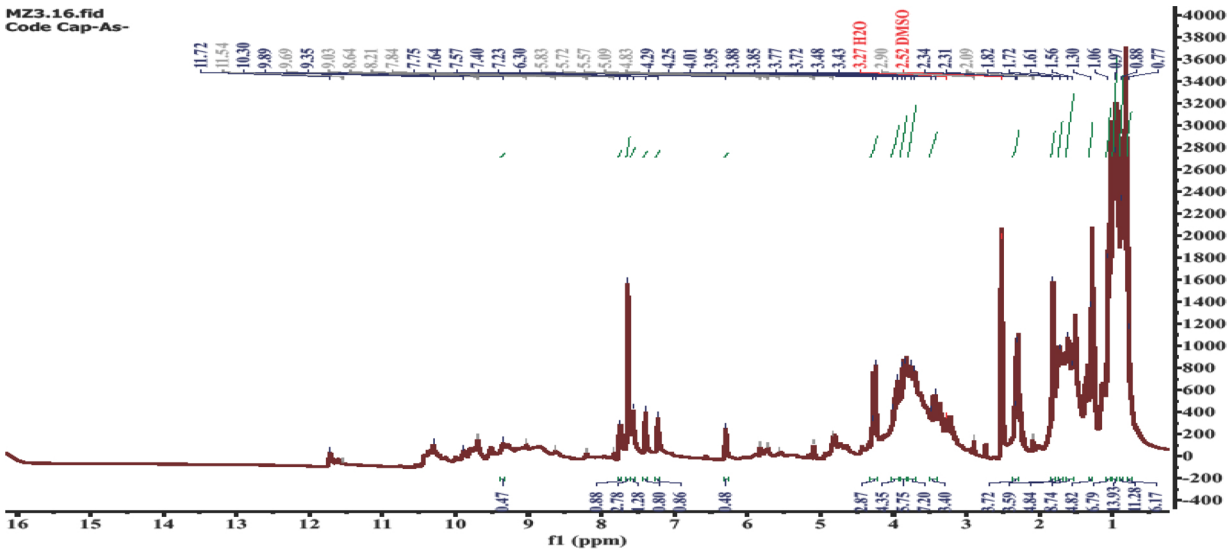


Fig. 9. ^{1}H-NMR of schiff base compound H4.

Table 1. FT-IR data for compounds H1–H4.

	(O-H) cm^{-1}	(N-H) cm^{-1}	(C-H) cm^{-1} Aro.	(C-H) cm^{-1} Ali.	(C=O) cm^{-1} amide	(C=O) cm^{-1} ester	(C=O) cm^{-1} carboxylic	(C-N) cm^{-1}	(C=C) cm^{-1}
H	3326	3200	3007	2853–2923	1600	–	1742	1378	1522
H1	3355	3230	3001	2880–2971	1651	1687	1714	1395	1466
H2	3304	3268	3167	2975–2930	1637	1620	1725	1383	1447
H3	3356	3264	3157	2927–2998	1637	–	–	1396	1466
H4	3328	3108	3001	2937–2971	1600	1645	1716	1388	1455

infrared spectra of the compound [H4] in Fig. 5 show medium intensity bands in the range of 3328 cm^{-1} which indicate the expansion vibrations of the hydroxyl groups. The appearance of two bands of 2937–2971 cm^{-1} which corresponds to C-H Alph, and 3001 cm^{-1} indicates (C-H) Aro., 1600 cm^{-1} which belongs to C=O amide, while 1645 cm^{-1} which belongs to C=O ester, 1716 cm^{-1} which belongs to (C=O) carboxylic, while (C=C) an extended vibration of the benzene rings in this compound appeared as strong bands at (1455) cm^{-1} .The data of FTIR of compounds are tabulated in Table 1.

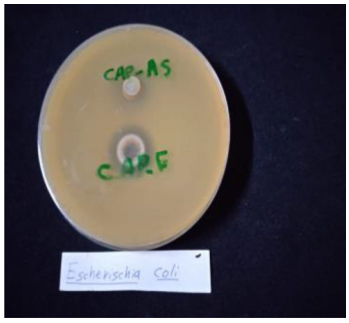
Proton nuclear magnetic resonance (^{1}H-NMR)

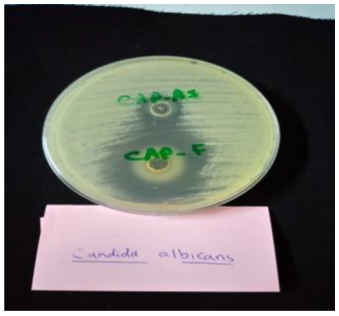
^{1}H-NMR spectroscopy with (DMSO-*d6*) as a solvent and TMS as an internal standard was used to screen the prepared compounds.The ^{1}H-NMR spectrum of) H1 (Fig. 6, which indicates the presence of signal assignments in the corresponding formula, which shows the following peaks: (1.49, S, H, CH_3), (2.01, S, H, CH_2), (2.53, S, H, OCH_3), (3.14, S, NH), (7.61–8.05 H aromatic), (6.3, S, H, OH). The signal (9.51 ppm) might be the result of two protons of carboxylate (–COO). The 1H-NMR spectrum of compound (H2) Fig. 7, which indicates the presence of signal assignments in the corresponding formula, which shows the following peaks: (1.2–1.86, S, H, CH_2, CH_3), (1.98, S, H, OCH_3), (2.71, S, NH), (7.20–7.60 Haromatic), (6.69, S, H, OH), at (9.37 ppm) result of one of (CH=N) group proton and a signal of singlet at (10, 01 ppm) due to NH group proton. The ^{1}H-NMR spectrum of compound (H3) Fig. 8, which indicates the presence of signal assignments in the corresponding formula, which shows the following peaks: (1.05–2.01, S, H, CH_2, CH_3), (2.27, S, H, OCH_3), (3.28, S, NH), 7.04–7.99 Haromatic, (6.99, S, H, OH), at 8.12 ppm result of one of (CH=N) group proton and a signal of singlet at (8.73 ppm) due to NH group proton. The H-NMR for compound H4 eared in the range 6.30–7.52 ppm for the aromatic protons sharp signal at 5.4 ppm for one proton could be attributed to the group of OH ring a singlet signal at 8.7 ppm result of one of CH=N group proton and a signal of singlet at 9.35 ppm due to NH group proton.

Biological activity

Candida albicans are caused by the human fungal pathogens that cause candidiasis. *Candida albicans* is a pathogenic yeast that is a component of the human intestinal microbiota. It is also capable of living

Table 2. The effectiveness of bacteria against the prepared compounds H1, H4.





Comp.	*Staphylococcus*	*E. Coli*	*Candida*
H1	25	24	32
H4	14	22	18

outside of the human organism. This study involves the evaluation of the antifungal activities against *Candida albicans* in three different concentrations of the compounds and showed moderate to good antifungal activity. Table 2 the antibacterial activity of these compounds was studied against *Staphylococcus* and *Escherichia coli* were tested (in vitro). Because there was no obvious change in bacterial growth, DMSO was used as a reference by agar spread technique at a concentration of 1 ppm and the plates were incubated for 24 hours at 37 °C. The inhibition zone was measured, and the results are shown Table 2 Showed that all compounds had a higher growth inhibition zone diameter and showed slight activity against *Candida albicans* than bacteria.[19]

Conclusion

The current research seeks to synthesize and characterize some new maleamic linked to Schiff bases and their reactions with "N-[(4-hydroxy-3-methoxyphenyl) methyl]-8-methyl-(E)-6-nonenamide". Some of these synthesized substances produced appropriate FT-IR and ^{1}H-NMR results that corresponded to data given in the construct to sources. The biological activity of synthesized compounds was determined to know their importance and the extent of their use in medical applications, where the antibacterial activity of these compounds against Staphylococcus and Escherichia coli (in vitro) was studied. The results showed that all compounds had a higher growth inhibition zone diameter and showed slight activity against Candida albicans bacteria.

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Author's declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for re-publication, which is attached to the manuscript.
- Authors sign on ethical consideration's approval.
- No animal studies are present in the manuscript.
- No potentially identified images or data are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Baghdad.

Author's contribution

This work was carried out in collaboration with all the authors. H. S. H. prepared the compounds, conducted the tests, and wrote and edited the manuscript with the idea of revision. S. H. A. analyzed the data with idea reviews. All authors read and approved the final manuscript.

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تحضير ,تشخيص ودراسة النشاط البايلوجي لمركبات قاعدة شيف جديدة مشتقة من (ن-(4- ميثوكسي-3-ميثوكسي فنيل) مثيل)- 8- مثيل - 6- نانونئميد).

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الخلاصة

تضمن هذا البحث تحضير قواعدشيف جديدة مرتبطة بمشتقات (ن-(4- ميثوكسي-3-ميثوكسي فنيل) مثيل)- 8- مثيل -6-نانونئميد). هناك بعض الخطوات التي تتضمن فتح حلقة من أنهيدريد الفثاليك بواسطة مجموعات الهيدروكسيل الموجودة في المركب (ن-(4- ميثوكسي-3-ميثوكسي فنيل) مثيل)- 8- مثيل -6-نانونئميد),وقد ثم تفاعلها مع الثيوريا لتكوين قواعد شيف عن طريق تفاعل الألديهايدات المختلفة. تضمنت الخطوة الأولى تفاعل التكثيف بين المركب (ن-(4- ميثوكسي-3-ميثوكسي فنيل) مثيل)- 8- مثيل -6-نانونئميد) (H) مع أنهيدريد الفثاليك لينتج المركب (H1). في الخطوة الثانية ، تم تفاعل المركب H1 مع الثيوريا لإنتاج مركب مالي اميك (H2) .في الخطوة الثالثة ، تم تفاعل الناتج (H2) مع الديهايدات مختلفة لتكوين قواعد شيف جديدة (H3 and,H4) . تم تمييز المركبات المحضرة اعتمادًا على تقنيات FT-IR و H-NMR1 ومن المتوقع أن تكون المركبات المستهدفة المُصنَّعة حديثًا نشطة للغاية من الناحية البيولوجية لأن جزيئاتها مكونات أساسية لمجموعتين نشطتين (إيمين وأميد).

الكلمات المفتاحية: مضادات الميكروبات، الكابسيسين، أنهيدريد الفثاليك، فتحة الحلقة، قاعدة شيف.