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Synthesis, Identification and Bio Evaluation of New Thiophene Derivatives

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Abstract

Thiophene is considered one of the most effective cyclic sulfur compounds in organic and pharmaceutical chemistry due to its high stability in various chemical reactions. It plays a key role as an antifungal agent in treating skin allergies, as well as in the preparation of polymers, rubber, and other materials. Recent research has focused on incorporating sulfur compounds into the synthesis of organic compounds because of their high stability in manufactured materials. The combination of thiophene and chalcone within a single pharmaceutical derivative or compound provides highly effective therapeutic properties against various types of bacteria, particularly those causing skin, oral, and dental infections. Sulfur derivatives, including benzothiazole and thiophene, are known for their bactericidal effects. Furthermore, the electron pair on the sulfur atom adds another biological property, giving these derivatives medicinal properties that target many infectious viruses. The current research involved the preparation of thiophene compounds linked with anile, chalcone, and di molecules of chalcone, as well as thiophene linked to cyclic derivatives such as imidazole and others. The methods for preparing these materials vary depending on the type of raw materials, the solvents used, and the reaction conditions, such as the temperature and pressure required to complete the preparation process. As is well known, to characterize and validate the synthesis of any specific chemical compound, researchers resorted to the spectroscopic methods, including proton resonance spectroscopy, carbon resonance spectroscopy, and infrared spectroscopy, in addition to biological studies. Furthermore, theoretical studies were conducted to compare with experimental results obtained to demonstrate the efficacy of the synthesized compounds.

Keywords: Anile, chalcone, infrared spectroscopy, thiazole, thiophene

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INTRODUCTION

Researchers have shown great interest in cyclic sulfur compounds due to their significant activity in the chemical field [1, 2]. Recent research has focused on incorporating sulfur compounds into the preparation of organic compounds because they are among the most stable synthetic materials [3–5]. The current research involved the preparation of thiophene compounds derived from anile [6–8], chalcone, and dichotomous chalcone, as well as thiophene [9–11] linked to azo or aniline derivatives [12–15], and cyclic derivatives, such as imidazole [16–19] and others. The methods for preparing these materials vary depending on the type of raw materials, the solvents used [20, 21], and the reaction conditions, such as the temperature and

pressure required to complete the preparation process [22]. The role of the catalyst is also crucial in obtaining a higher yield and higher purity. Many heterocyclic compounds have been found to possess numerous biological and medicinal properties [23–25]. For example, the thiophene ring is a component of some vitamins. Many drugs contain the thiophene ring, such as the antihistamine methapyrilene [26–29] and the anti-inflammatory drug tiaprofenic acid [30–33]. It has been observed that the pharmacological effects of the thienyl group are like those of the phenyl or benzyl groups [31–34]. Pyrimidine derivatives, like other heterocyclic compounds, have medicinal effects in various fields [35–38]. They have been shown to have analgesic, anti-allergic, anti-inflammatory [39–41], and antibacterial effects, and some have antidiabetic and other effects [42–44] of biological importance.

EXPERIMENTAL PART

Synthesis of Thiophene- Anile

Given the importance of thiophene compounds as a basic material in the compounds prepared in this study, several different compounds were prepared using different methods. The first compound was prepared by reacting para-toluidine with ammonium thiocyanate in the presence of bromine and glacial acetic acid. This reaction took place for (3 hours) with continuous rotation at (15°C) to form (2-aminobenzothiazole) derivative. This (2-aminobenzothiazole) derivative (0.001 mol) was then taken with (pyrimidine-2-formalthiophene) derivative (0.001 mol) by condensation with drops of (glacial acetic acid). The reaction was carried out by sublimation for (four hours), after which the solvent was evaporated and the product was purified to give a yellow precipitate of thiophene-anile.

Synthesis of Thiophene-Anile Chalcone

The second compound was prepared by reacting (0.001 mol) of (para-toluidine) with (ammonium thiocyanate) in the presence of bromine and glacial acetic acid. This reaction, conducted under continuous rotation at (15°C), lasted for (3 hours) to form (2-aminobenzothiazole) derivative. The second step involved reacting (0.001 mol) of this compound with (para-acetobenzaldehyde) by condensation with drops of (glacial acetic acid) for (three hours). After removing the solvent, purifying the compound, and drying it, (0.001 mol) of this compound was reacted at room temperature with continuous mechanical rotation for (five hours) with (0.001 mol) of (pyrimidine-2-formalthiophene) derivative using (5%NaOH) base. The solution was then filtered, purified, and dried to obtain a 76% yellowish-orange precipitate of thiophene-anile chalcone.

Synthesis of Thiophene-Imidazole Chalcone

The third compound was prepared by reacting (0.001 mol) of thiophene-anile chalcone with (0.001 mol) aminoacetic acid. This reaction, conducted under a condensation step in DMF as a solvent, lasted for (5 hours) to form a 74% orange precipitate of thiophene-imidazole chalcone.

Synthesis of Thiophene-Bis Chalcone

(0.001 mol) of (pyrimidine-2-formalthiophene) derivative reacted using (5%NaOH) base with (para-acetobenzaldehyde); then the precipitate after drying was reacted with (0.001 mol) of (p-methylacetophenone) in the presence of (5%NaOH) with stirring for (5 hrs). The solution was then filtered, purified, and dried to obtain an 80% orange precipitate of thiophene-bis chalcone.

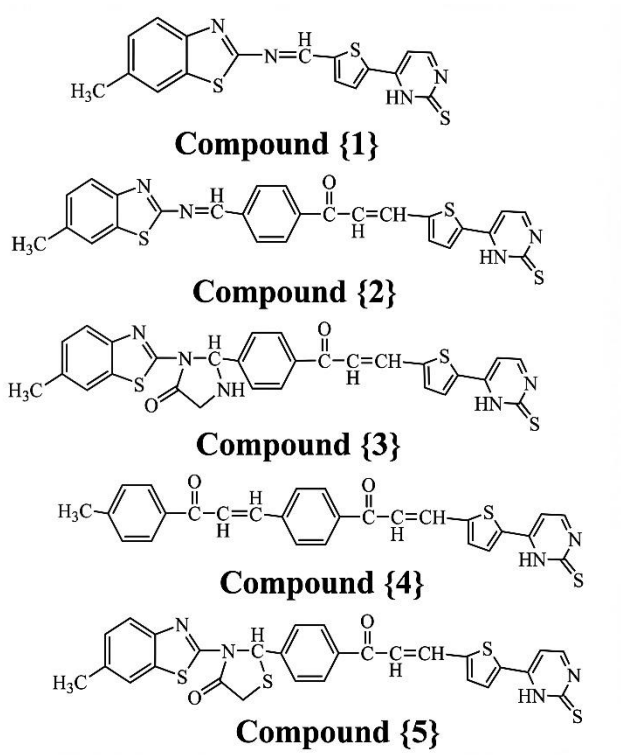
Synthesis of Thiophene-Imidazole Chalcone

The third compound was prepared by reacting (0.001 mol) of thiophene-anile chalcone with (0.001 mol) mercaptoacetic acid. This reaction, conducted under a condensation step in DMF as a solvent, lasted for (4 hours) to form a 74% deep-yellow-colored precipitate of thiophene-imidazole chalcone in Scheme 1.

RESULTS AND DISCUSSION

The infrared (IR) spectroscopy results showed absorption bands indicating the formation of new compounds, including absorption bands in the region [(3348)] cm^{-1} attributed to the ((NH) and (CH=N) anile group at (1698) in thiophene-anile. The spectrum also showed an absorption band at (1250) attributed to the vibration of the ((C=S) group resulting from the thion group in the pyrimidine ring in

the prepared derivative and absorption bands in the region [(3301)] cm^{-1} attributed to the ((NH) and (CH=N) anile group at (1630) in thiophene-anile. We also interpreted the presence of a strong, sharp bands at (1698) cm^{-1} attributed to carbonyl group in the chalcone thiophene-anile respectively. Thus, the other bands and frequencies are shown in IR spectral Figure 1.



Scheme 1. Formation of thiophene derivatives.

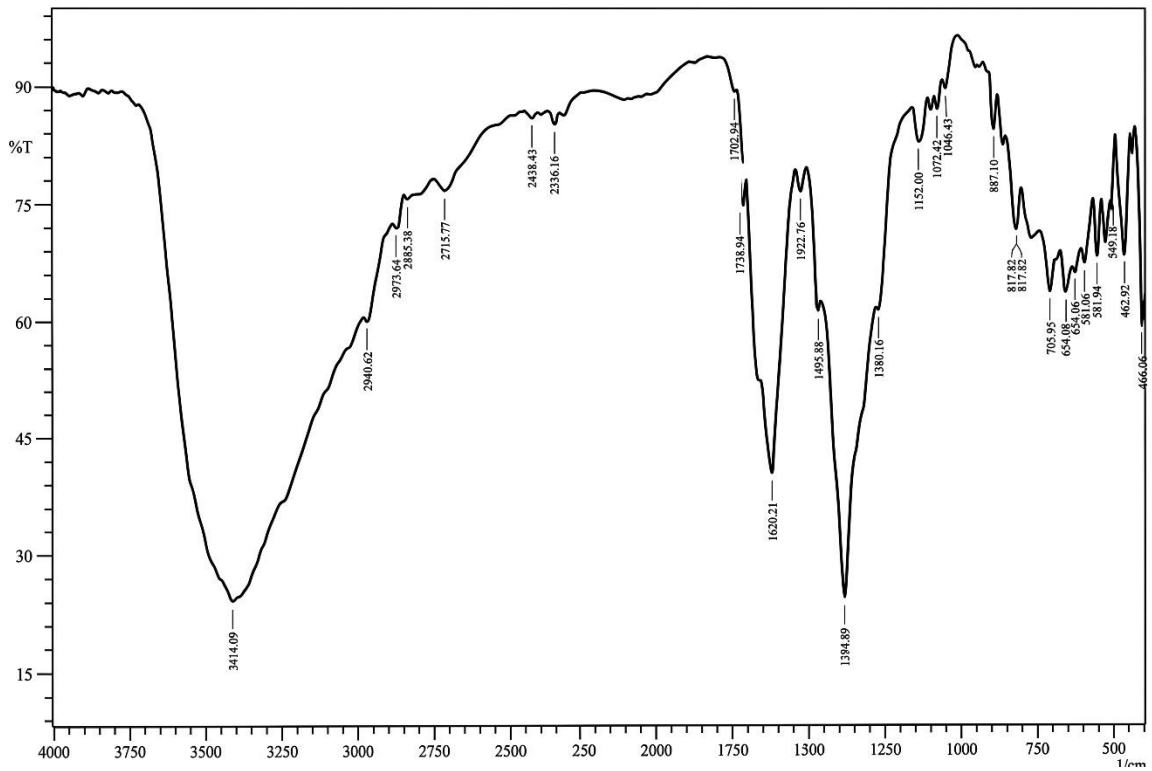


Figure 1. Infrared (IR) spectroscopy for thiophene-anile.

Analysis (H-NMR) spectroscopy results showed peaks indicating the formation of new compounds, including peak in the region (5.3) ppm attributed to the (NH) and (CH=N) anile group at (8.63) ppm in thiophene-anile. We also interpreted the presence of clear peaks at (4.98, 4.95, 5.02, 5.00) ppm attributed to (CH=CH-CO) alkene group in the chalcone in compound 2 (thiophene-anile chalcone), compound 3 (thiophene-imidazole chalcone), compound 4 (thiophene-bis chalcone), and compound 5 (thiophene-imidazole chalcone), respectively, while all peaks were appeared in figures (2–6) for the (NH) amine and (CH=N) anile groups for all compounds with the other peaks are shown in the H-NMR spectral Figures 2–6.

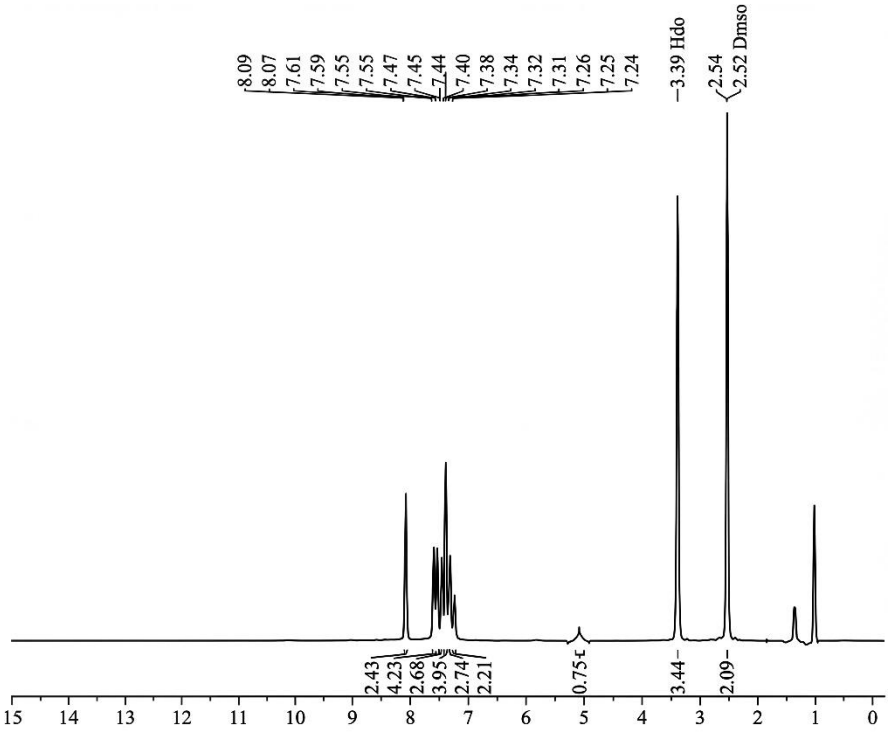


Figure 2. H-NMR for thiophene-anile.

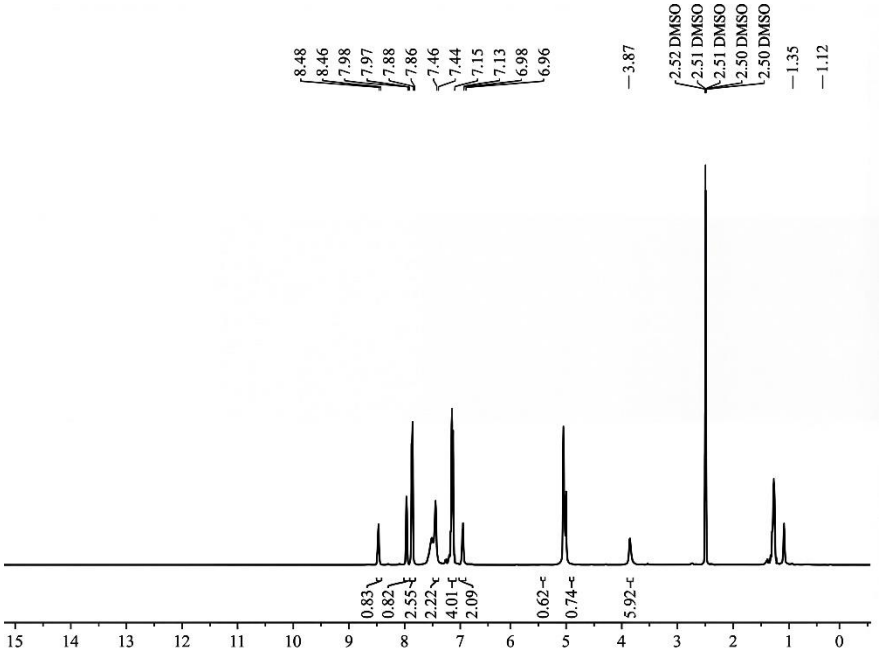


Figure 3. H-NMR for thiophene-anile chalcone.

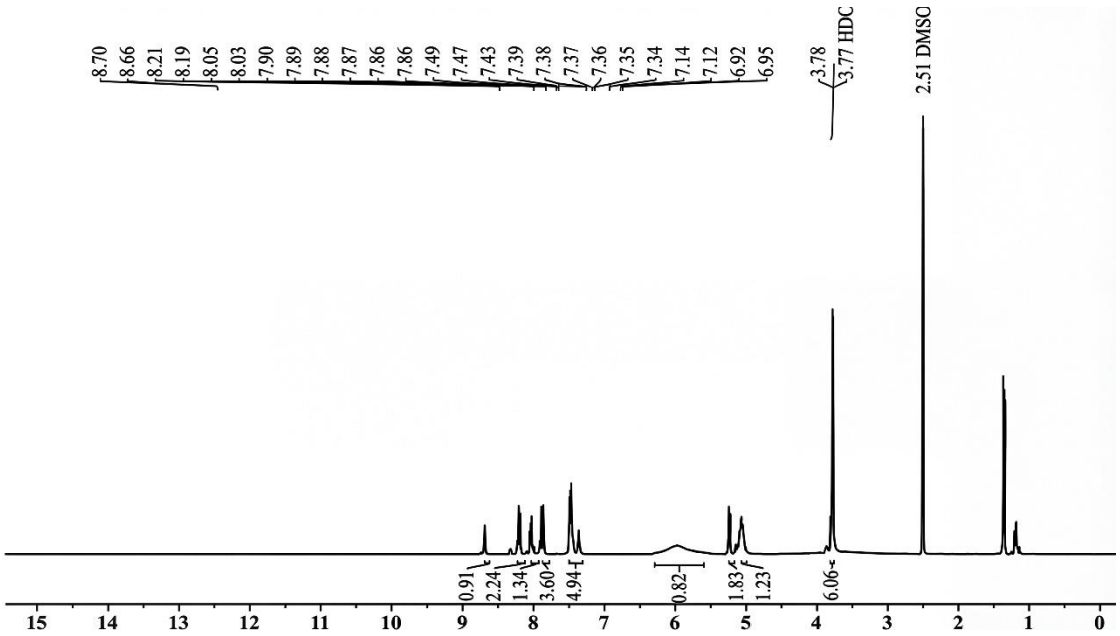


Figure 4. H-NMR for thiophene-imidazole chalcone.

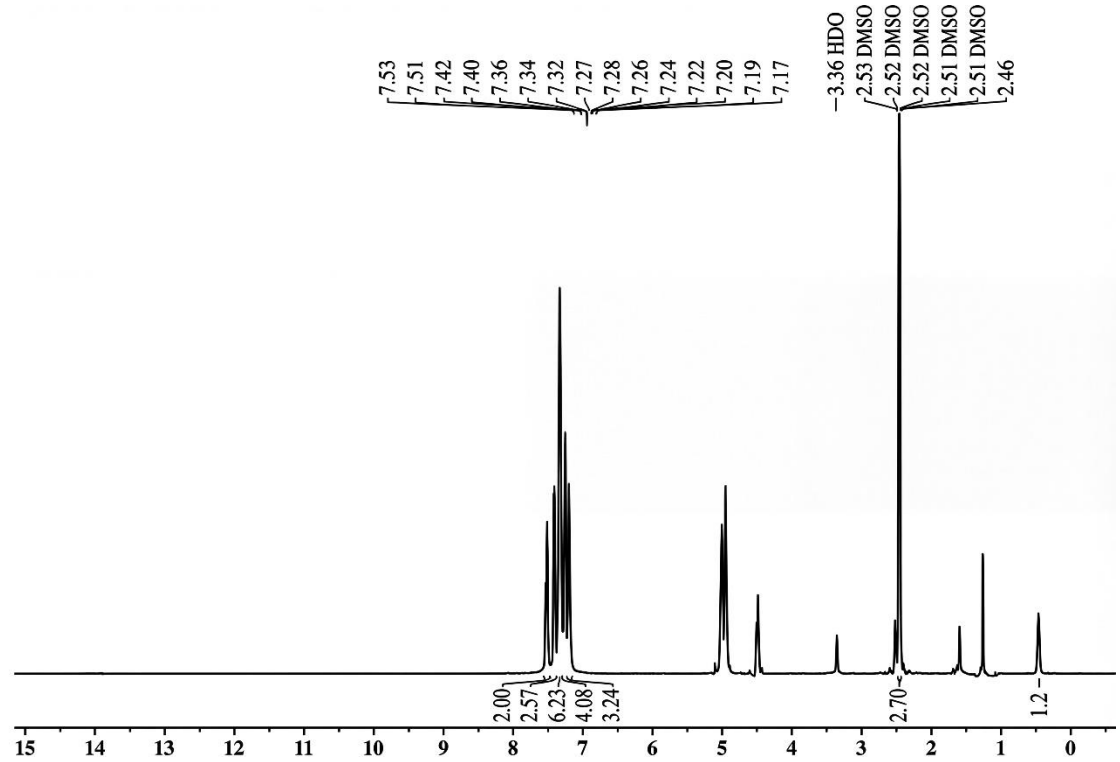


Figure 5. H-NMR for thiophene-bis chalcone.

Analysis (C-NMR) spectroscopy results showed peaks indicating the formation of new compounds, including peak in the region (112–138) ppm attributed to protons of (Aromatic ring) and (CH=N) anile group at (154.7) ppm in thiophene-anile. We also interpreted the presence of clear peaks at (172, 170, 175, 177) ppm attributed to (CH=CH-CO) carbonyl group in the chalcone chalcone in compound 2 (thiophene-anile chalcone), compound 3 (thiophene-imidazole chalcone), compound 4 (thiophene-bis chalcone), and compound 5 (thiophene-imidazole chalcone) respectively, while all peaks were appeared in figures 7–10 for the (CO-N) amide and (CH=N) anile groups for all compounds with the other peaks are shown in the C-NMR spectral Figures 7–10.

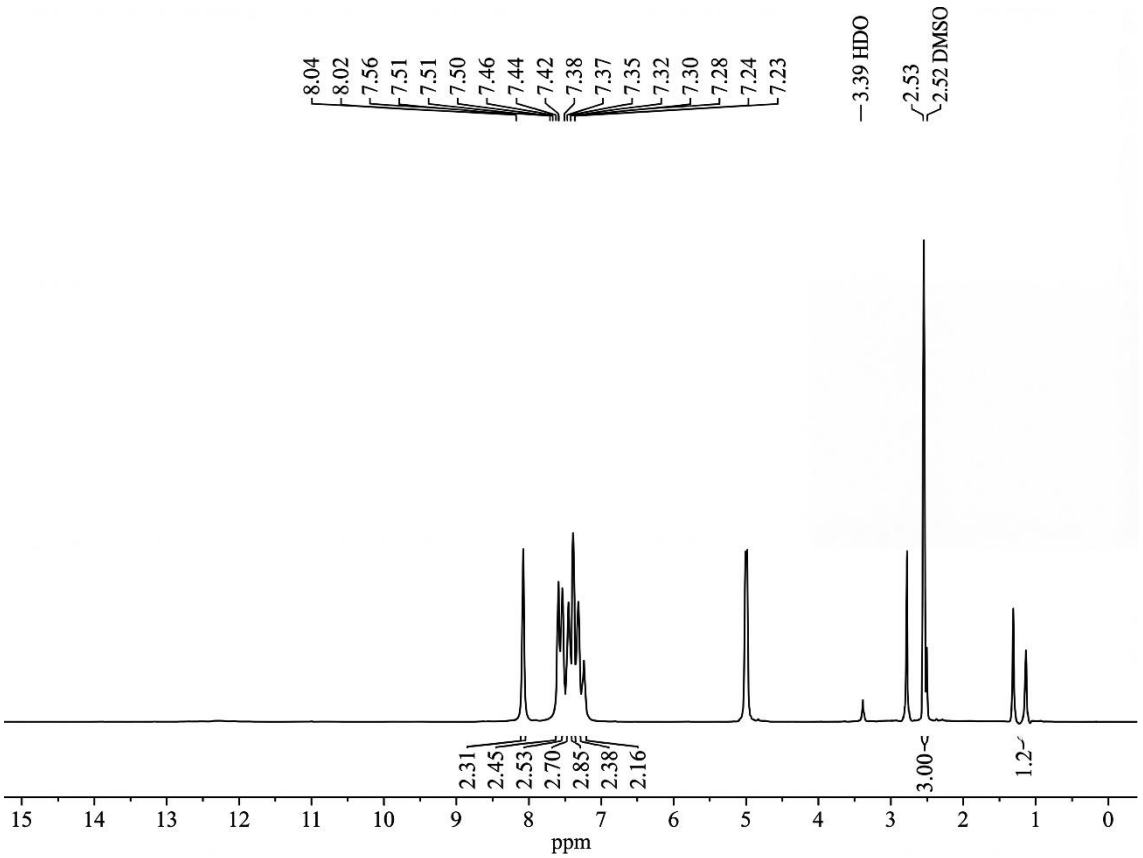


Figure 6. H-NMR for thiophene-imidazole chalcone.

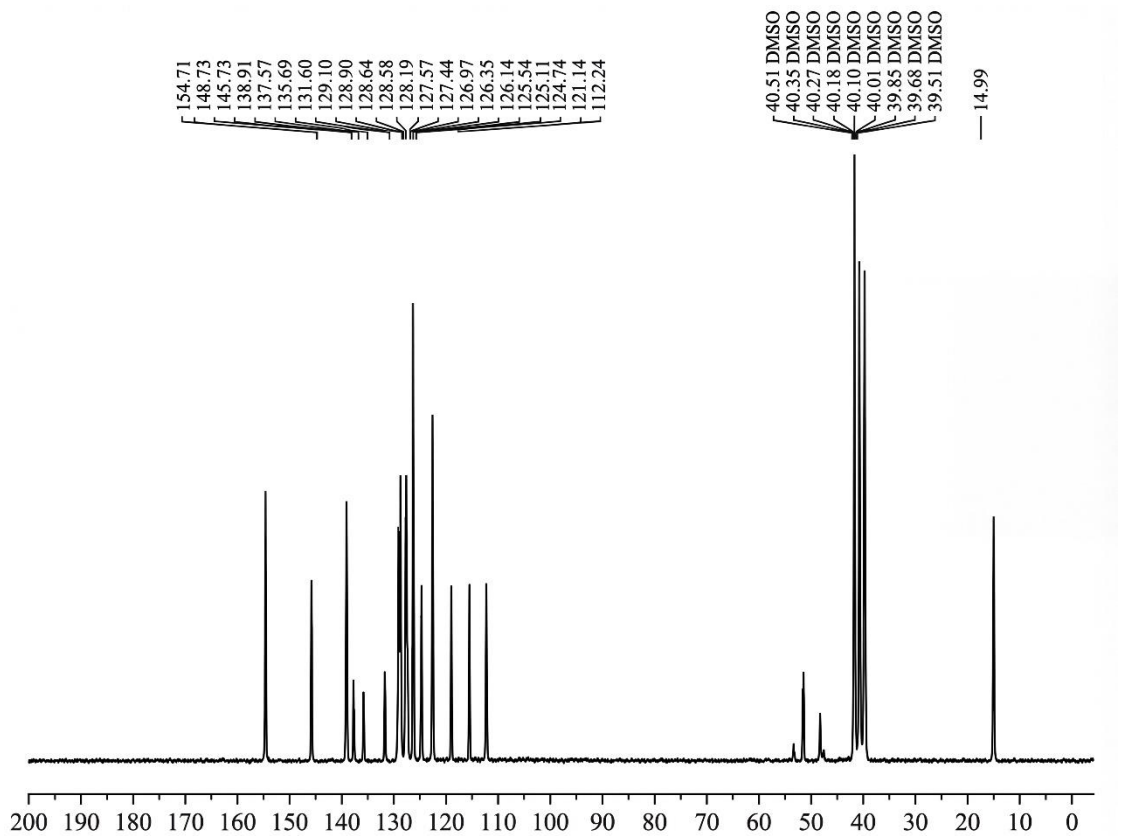


Figure 7. C-NMR for thiophene-anile.

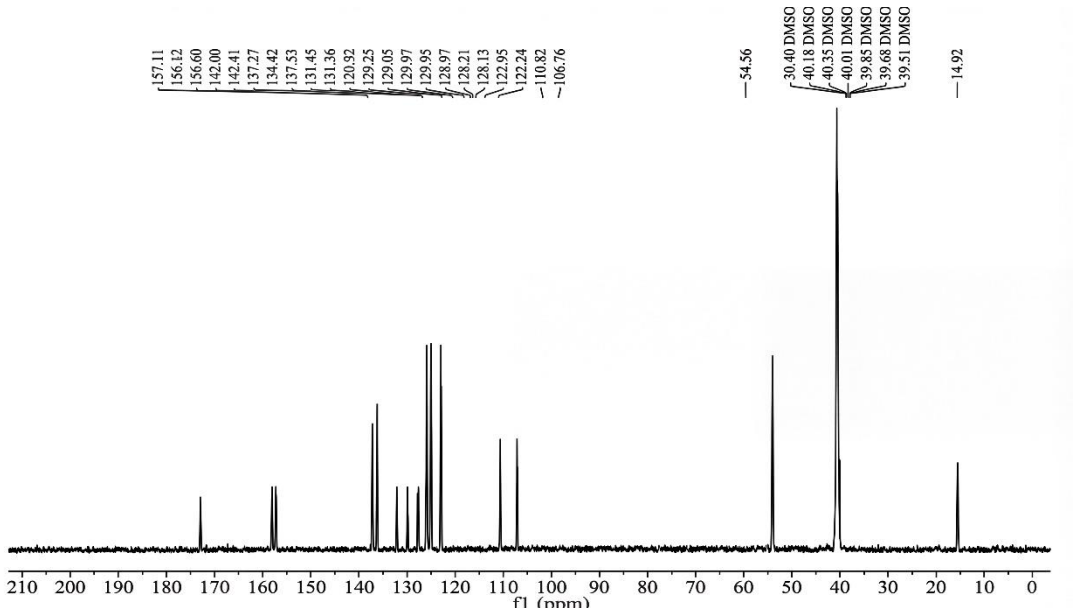


Figure 8. C-NMR for thiophene-anile chalcone.

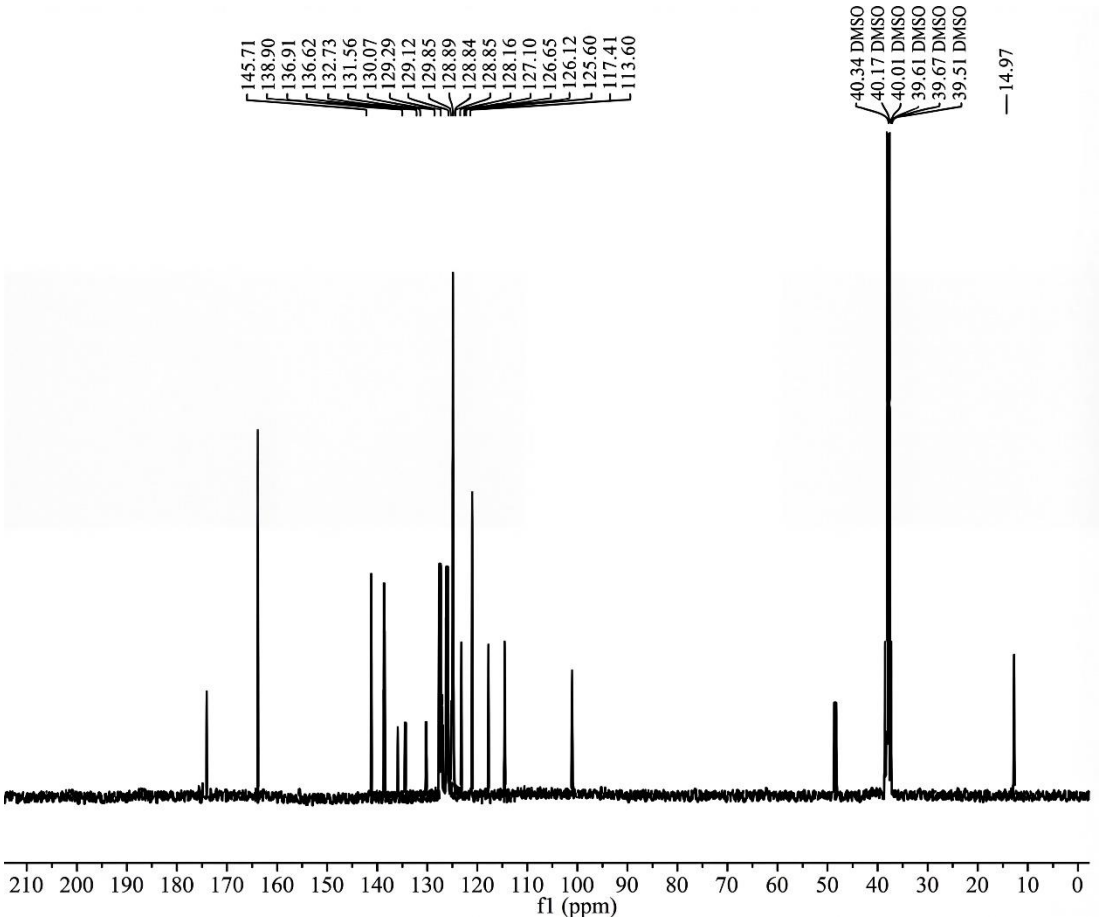


Figure 9. C-NMR for thiophene-imidazole chalcone.

Study of Bio Properties via Theoretical Program

Chem-informaticians developed different molecular descriptors mined from chemical structures via theoretical computational program to supply us with bio-information about probability bio activity of prepared compounds in Tables 1, 2.

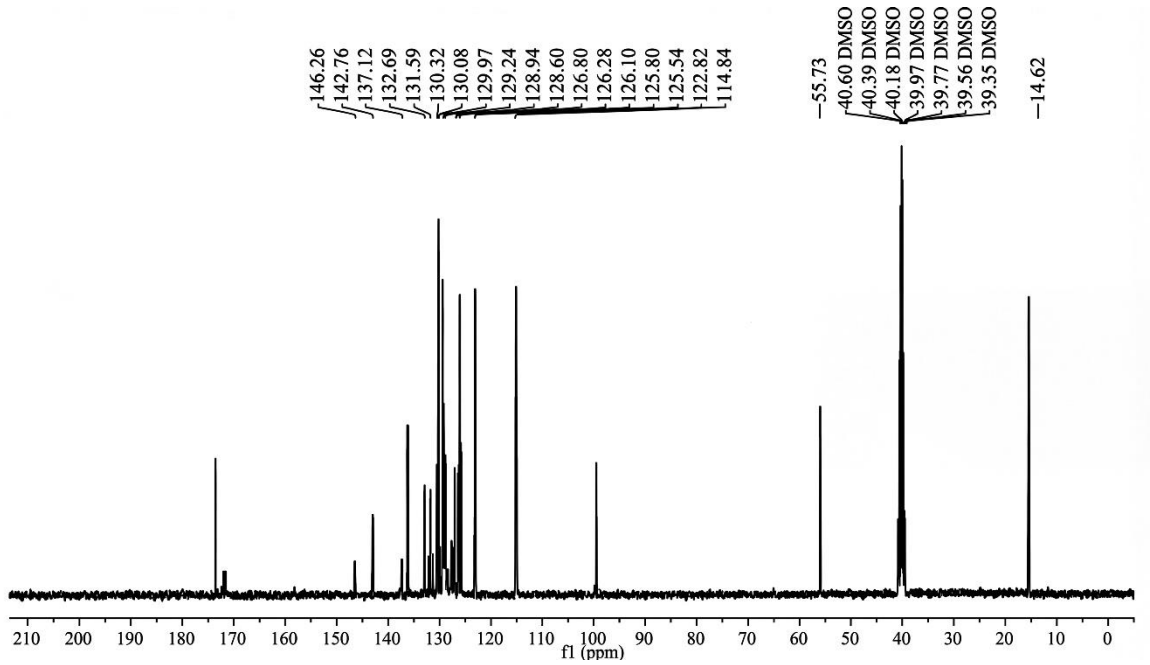


Figure 10. C-NMR for thiophene-bis chalcone.

Table 1. Bio properties of thiophene-imidazole chalcone.

Water solubility	
Log *S* (ESOL)	-2.89
Solubility	3.50e-01 mg/ml ; 1.30e-03 mol/l
Class	Soluble
Log *S*	-3.66
Solubility	5.92e-02 mg/ml ; 2.21e-04 mol/l
Class	Soluble
Log *S* (SILICOS-IT)	-1.48
Solubility	8.85e+00 mg/ml ; 3.30e-02 mol/l
Class	Soluble
Pharmaco -kinetics	
GI absorption	High
BBB permeant	Yes
P-gp substrate	No
CYP1A2 inhibitor	Yes
CYP2C19 inhibitor	Yes
CYP2C9 inhibitor	No
CYP2D6 inhibitor	Yes
CYP3A4 inhibitor	No
Log K_p (skin permeation)	-6.11 cm/s
Drug likeness	
Lipinski	Yes; 0 violation
Ghose	Yes
Veber	Yes
Egan	Yes

Muegge	Yes
Bioavailability Score	0.55
Medicinal chemistry	
PAINS	0 alert
Brenk	4 alerts: a cyclic_C=C-N, Cycles, sulfur Nitrogen _double bond, oxygen ,sulphur_nitrogen_single_bond
Lead likeness	Yes
Synthetic accessibility	3.04

Table 2. Physicochemical properties and lipophilicity properties of thiophene-imidazole chalcone.

Physicochemical Properties	
Num. heavy atoms	19
Num. arom. heavy atoms	0
Fraction Csp3	0.64
Num. rotatable bonds	3
Num. H-bond acceptors	10
Num. H-bond donors	9
Molar Refractivity	100.08
TPSA	151.23 Å²
Lipophilicity	
Log $P_{o/w}$ (iLOGP)	2.19
Log $P_{o/w}$ (XLOGP3)	3.30
Log $P_{o/w}$ (WLOGP)	1.95
Log $P_{o/w}$ (MLOGP)	0.84
Log $P_{o/w}$ (SILICOS-IT)	1.57
Consensus Log $P_{o/w}$	1.97

Table 3. Bio Evaluation of derivatives and inhibition diameter (mm).

Derivatives	**Bacillus subtlis (bacteria)**	**Candida albicans (fungi)**
Thiophene- Anile	8	6
Thiophene-Anile Chalcone	12	8
Thiophene-Imidazole Chalcone	14	14
Thiophene-Bis Chalcone	14	10
Thiophene-Imidazole Chalcone	18	14

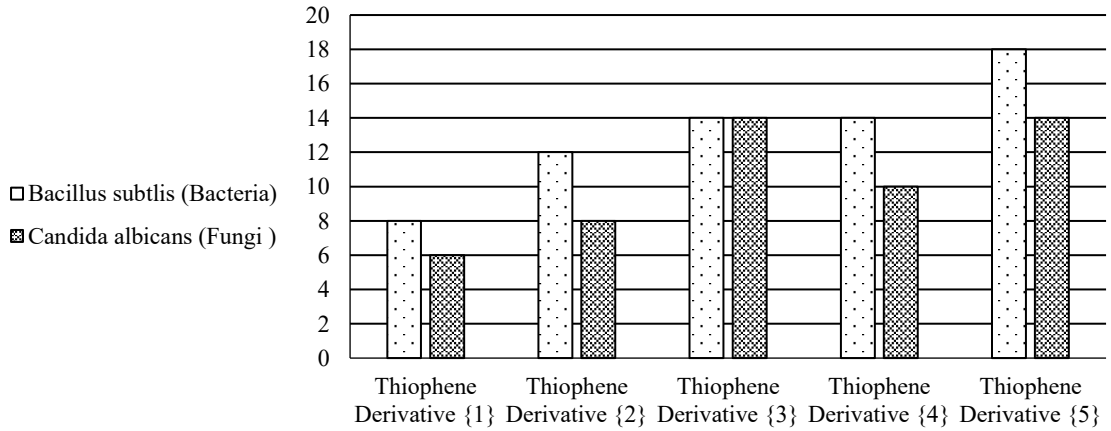


Figure 11. Effect of derivatives on candida albicans and bacillus subtilis.

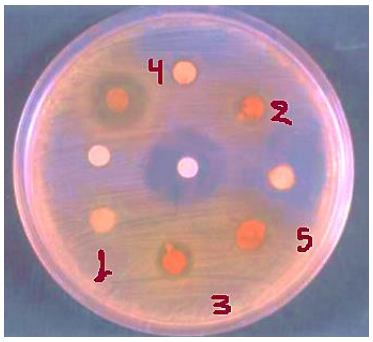


Figure 12. Inhibition diameter of bacillus subtilis.

Bio Evaluation

This type of assay was used for fungi and bacteria, and the biological tests for bacterial growth showed the following results, listed in Table 3 and Figures 11, 12, within the different concentrations prepared for the derivatives. An average of three concentrations (at concentrations of 10^{-4}, 10^{-3}, 10^{-2} molar) and three readings for each prepared derivative were taken to ensure the accuracy of the tests for calculating the diameter of inhibition of bacterial growth.

CONCLUSIONS

All diagnostic analyses were conducted to confirm the prepared derivatives, their chemical composition, and their biological properties. Physical properties, such as color, precipitate type, and product yield were also studied, in addition to demonstrating their effectiveness in laboratory settings based on theoretical studies, due to their content of several nitrogen and sulfur atoms, which are known for their biological activity.

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