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Research Article

Synthesis and preliminary antimicrobial evaluation of new 7-amino-4-methyl-coumarin thiazolidinone conjugates

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Abstract

As a part of our ongoing project on the design and synthesis of new 4-thiazolidinone derivatives with antimicrobial activity, four new 4-thiazolidinone derivatives carrying bromo, nitro, methyl, and chloro groups on the benzene ring were synthesized by starting with the 7-amino-4-methylcoumarin moiety, linking coumarin with various phenyl isothiocynate to form the thiourea group, and then cyclizing the derivatives, characterized by IR and ^{1}HNMR, and assayed *in vitro* for their antimicrobial activity against Gram positive and Gram negative bacteria and fungi. Overall, 2-(4-methyl-2-oxo-2H-chromen-3-yl)-3-(4-nitrophenyl) thiazolidin-4-one to be the most powerful individuals in the series. Based on the observed data, it can be stated that the synthesized compounds demonstrate a variable range of antibacterial activity.

Keywords

coumarin, thiourea, phenyl isothiocynate, 4-thiazolidinone, antimicrobial activity

Introduction

The main problem in medicinal chemistry is the design and synthesis of physiologically active compounds (Kumar and Patil 2017). Many therapeutic compounds that have been found belong to a significant category of heterocycles that contain nitrogen and/or sulfur (Omar et al. 2010; Salman and Ahamad 2022; Ali and Farhan 2024). The broad range of synthetic applicability and biological activity exhibited by these heterocycles has enabled medicinal chemists to strategically design, coordinate, and execute innovative methods for the development of new pharmaceuticals (Baboo et al. 2017). At least one heterocycle motif is included in the final structure of almost 60% of the pharmaceutical industry's most successful drugs (Abbas et al. 2023). Thiazolidinones are widely acknowledged as a fundamental structure for future pharmaceuticals and therapeutic candidates (Sahu et al. 2021). 4-Thiazolidinone and its variants are a noteworthy class of bioactive chemicals. The nucleus of an atom is sometimes referred to as the "remarkable core" due to its diverse array of biological functions. The 4-thiazolidinone scaffold is very adaptable and has been utilized in several medications that are currently in clinical usage (Saleh and Farhan 2023). They have been discovered to have use as antitubercular, antibacterial, anti-inflammatory, and antiviral drugs, particularly as anti-HIV agents (Jain et al. 2012). There is a distinct category of compounds known as

coumarins, and they serve an essential purpose in the naturally occurring environment. They are a sort of secondary metabolite that are found in plants and are referred to as flavonoids. Flavonoids have been associated with a wide variety of functions within the body (Pasricha and Gahlot 2020). Coumarin compounds, regardless of their origin in nature or the laboratory, have a crucial function (Abd-Elhussain and Farhan 2024). Multiple coumarin-containing compounds have diverse biological and pharmacological effects (Farhan and Ahmed 2021), like anti-HIV (Hassan et al. 2016; Turkovic et al. 2020). anticoagulant (O'Reilly et al. 1963), antibacterial (de Souza et al. 2005), antioxidant (Kostova et al. 2011), and antifungal activities (Prusty and Kumar 2020). Upon reviewing the literature, it is evident that thiazolidinones and their derivatives exhibit diverse actions and can be synthesized using numerous methods.

Priya et al. conducted a study where they synthesized a range of thiazolidinone derivatives of 1,2-benzisoxazole-4-hydroxy-2H-chromen-2-one (Fig. 1). They achieved this by condensing the compound with aromatic aldehyde to form Schiff's base. Subsequently, they cyclized the Schiff's base with mercaptoacetic acid to obtain the thiazolidinone derivatives of benzisoxazole. The synthesized compounds underwent screening for in vitro antibacterial and antifungal activities using turbidimetric techniques (Priya and Srimathi 2016).

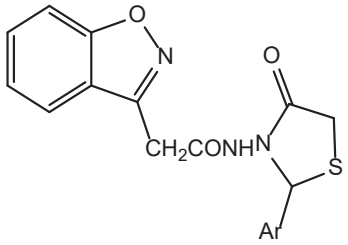


Figure 1. Thiazolidinone, derivatives of 1,2-benzisoxazole-4-,hydroxy-2H-,chromen-2-one.

Archana synthesizes a variety of different things. 2-N-(2-phenyl-1H-indol-3-yl) imino benzene/thiophenol, compound is called 3-ChloroThe (benzyl/ thiophenyl)-4-(2-1H-indol-3yl) Azetidin-2-ones and 3-substituted-benzyl/thiophenyl-4-(2-phenyl 1Hindol-3-yl) thiazolidin-4-ones were tested in vivo participants to evaluate their immediate toxicity and effectiveness in preventing seizures using MES and PTZ models (Fig. 2) (Archana 2016).

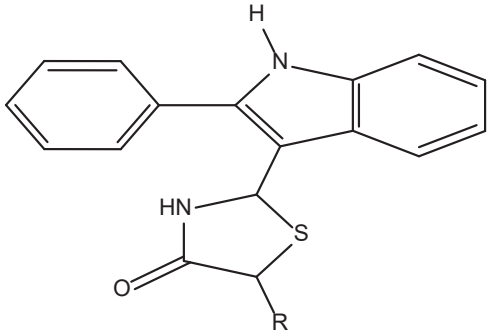


Figure 2. The compounds synthesized by Archana.

Tsyalkovsky et al. synthesized and assessed the antibacterial activity of new derivatives of 5-(R1-benzyl)-2-(R2-benzylidenehydrazono)-3-(2-furylmethyl) thiazolidin-4-one (Fig. 3). Their research uncovered that the presence of a methyl group at the 4th position of the benzyl group was crucial for the action of compounds A and B. The substitution of a methyl group with a halogen led to a reduction of the antibacterial properties (Tsyalkovsky et al. 2005).

The objective of this work is to produce thiazolidinone ring analogues by initiating with a 7-amino-4-methylcoumarin structure, connecting coumarin with different phenyl Isothiocynate compounds to create a thiourea group, and subsequently forming the derivatives through cyclization. The antimicrobial effectiveness of these derivatives will then be assessed.

Materials and methods

The chemicals, solvents, and other reagents utilized in the synthesis were purchased from commercial suppliers. We employed thin-layer chromatography (GF254, Merk, Germany) to assess the cessation of the reaction in UV-induced reactions (254 nm). Two distinct solvent solutions, A and B, were employed: Acetone:Benzene (1:9) and Chloroform:Methanol (3:1), respectively. The Stuart SMP3 melting point apparatus was utilized to ascertain the melting points in unsealed capillary tubes, and these measurements have not been altered in any manner. The infrared spectra were generated using a Shimadzu FT-IR spectrophotometer manufactured in Japan. The infrared

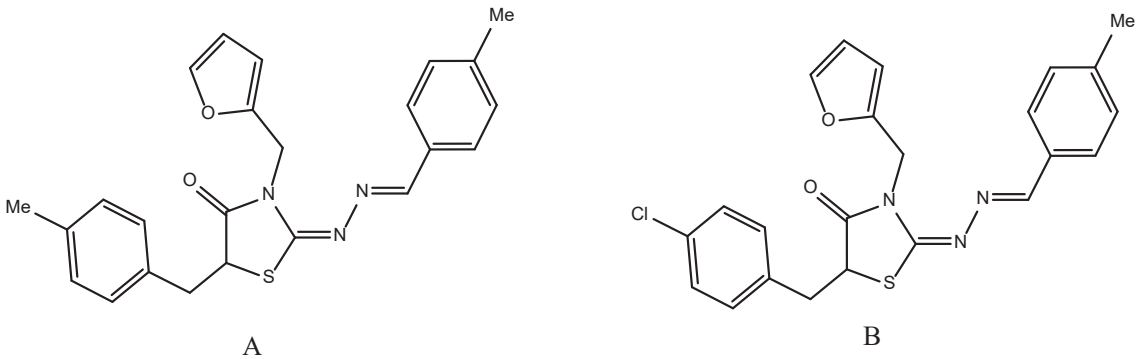


Figure 3. New derivatives of 5-(R1-benzyl)-2-(R2-benzylidenehydrazono)-3-(2-furylmethyl) thiazolidin-4-one.

(IR) tests were conducted at Baghdad University, at the College of Pharmacy. The ^{1}HNMR spectra were acquired using a Bruker Ultra-Shield 400 MHz spectrophotometer. Tetramethylsilane (TMS) was used as an internal standard, and DMSO-d6 was used as the sample solvent. The chemical shift values were reported in parts per million (ppm). The ^{1}HNMR investigations were conducted at Al-Basra University, at the College of Science.

1-General procedure for the synthesis of substituted phenyl thiourea (Sharabiyani et al. 2017)

The first compound **(Compound A**) (7-amino 4-methylcoumarin) (0.005 mol) was placed in a certain amount with substituted phenyl isothiocynate such as (bromo, nitro, methyl, or cholro phenyl Isothiocynate) (0.005 mol) separately, in absolute ethanol (25 ml), and refluxed for 12 hours at 60 °C. TLC utilizing Silica gel-G will be used to monitor the reaction. The solvent was removed, and the precipitate will be recrystallized from absolute ethanol to yield the compounds (B, C, D, E).

Compound B

1-(5-methyl-7-oxo-7,8-dihydronaphthalen-2-yl)-3-(4-nitrophenyl)thiourea

(yellow powder): 70% yield , m.p: (150 °C), R_f: 0.6 (A), and IR (KBr) n: (3433 cm^{-1}, 3348 cm^{-1}). N-H stretching (3074 cm^{-1}). C-H stretching of aromatic ring, (2985 cm^{-1}, 2958 cm^{-1}) asymmetric and symmetric CH3 stretching, (1678 cm^{-1}) C=O stretching of coumarin, (1600 cm^{-1}, 1543 cm^{-1}), C=C stretching aromatic ring, (1256 cm^{-1}) C-O-C stretching, (1211 cm^{-1}) C-N stretching and (1014 cm^{-1}) C=S stretching.

Compound C

1-(5-methyl-7-oxo-7,8-dihydronaphthalen-2-yl)-3-(p-tolyl)thiourea

(orange powder): 65% yield, m.p: (140 °C), R_f: 0.62 (A), and IR (KBr) n: (3356 cm^{-1}, 3244 cm^{-1}). N-H stretching, (3089 cm^{-1}). C-H stretching of aromatic ring, (2981 cm^{-1}) CH3 stretching, (1685 cm^{-1}) C=O stretching of coumarin, (1666 cm^{-1}, 1608 cm^{-1}) C=C stretching of aromatic ring, (1234 cm^{-1}) C-O-C stretching, (1145 cm^{-1}) C-N stretching and (1014 cm^{-1}) C=S stretching.

Compound D

1-(4-bromophenyl)-3-(5-methyl-7-oxo-7,8-dihydronaphthalen-2-yl)thiourea

(yellow powder): 50% yield, m.p: (125 °C), R_f: 0.55 (A), and IR(KBr) n: (3344 cm^{-1}, 3259 cm^{-1}) N-H stretching, (3093 cm^{-1}). C-H stretching of aromatic ring, (1685 cm^{-1}) C=O stretching of coumarin, (1689 cm^{-1}, 1504 cm-1) C=C stretching of aromatic ring, (1230 cm^{-1}) C-O-C stretching, (705 cm^{-1}) C-Br stretching, (1172 cm^{-1}) C-N stretching and (1072 cm^{-1}) C=S stretching.

Compound E

1-(4-chlorophenyl)-3-(5-methyl-7-oxo-7,8-dihydronaphthalen-2-yl)thiourea

(pale yellow powder): 50% yield, m.p: (132 °C), R_f 0.5 (A), and IR(KBr) n: (3348 cm^{-1}, 3244 cm^{-1}) N-H stretching, (3089 cm^{-1}). C-H stretching of aromatic ring, (1685 cm^{-1}) C=O stretching of coumarin, (1666 cm^{-1}, 1608 cm^{-1}) C=C stretching of aromatic ring, (1234 cm^{-1}) C-O-C stretching, (675 cm^{-1}) C-Cl stretching, (1184 cm^{-1}) C-N stretching, and (1064 cm^{-1}) C=S stretching.

2-cyclization of thiourea derivatives to form thiazolidin-4-one derivatives (Güzel-Akdemir et al. 2018; Alheety et al. 2020)

In a round bottom flask (100 ml), ethyl-bromoacetate (0.0012) and anhydrous sodium acetate (0.003 mol) were added to a solution of the appropriate thiourea derivative compounds (B, C, D, and E) separately (0.005 mol) in absolute ethanol (20 ml), and the reaction mixture was heated under reflux for 12 hrs. The precipitated solid was filtered, washed with water, dried, and recrystallized from absolute ethanol.

compound Ia (2-((4-methyl-2-oxo-2H-chromen-7-yl)imino)-3-(4-nitrophenyl) thiazolidin-4-one)

(off white powder): 40% yield, m.p: (179 °C), Rf: 0.4 (A), and IR(KBr) n: (3074 cm^{-1}) C-H stretching of aromatic ring, (2981 cm^{-1}, 2924 cm^{-1}) asymmetric and symmetric CH3 stretching (1724 cm^{-1}), C=O stretching of thiozolidinone, (1635 cm^{-1}) C=O stretching of coumarin, (1581 cm^{-1}) C=N stretching, (1508 cm^{-1}, 1442 cm^{-1}) C=C stretching of aromatic ring, (1161 cm^{-1}) C-O-C stretching, (1103 cm^{-1}) C-N stretching, (748 cm^{-1}) C-S stretching. ^{1}H NMR (500 MHz, DMSO-d_6) d: δ 2.41(s, 3H) of coumarin's C (=O) -C=C-CH3), δ 4.27 (s,2H) protons of the thiazolidinone ring, δ 6.47 (s, 1H) proton of coumarin's –C(=O)- C=C and δ 7.14–8.24(m,7H) protons of aromatic rings.

compound Ib (2-((4-methyl-2-oxo-2H-chromen-7-yl)imino)-3-(p-tolyl)thiazolidin-4-one)

(white powder): 37% yield, m.p: (160 °C), Rf: 0.44 (A), and IRKBr) n: (3066 cm^{-1}) C-H stretching of aromatic ring, (2947 cm^{-1}, 2981 cm^{-1}) symmetric and asymmetric CH3 stretching, (1720 cm^{-1}) C=O stretching of thiozolidinone, (1643 cm^{-1}) C=O stretching of coumarin, (1597 cm^{-1}) C=N stretching, (1442 cm^{-1}, 1543 cm^{-1},) C=C stretching of and aromatic ring, (1126 cm^{-1}) C-O-C stretching, (1064 cm^{-1}) C-N stretching, (644 cm^{-1}) C-S stretching. ^{1}H NMR (500 MHz, DMSO-d_6) d: δ 2.42–2.48 (m, 6H) protons of

coumarin's C (=O) -C=C-CH3), δ 4.25 (s,2H) protons of the thiazolidinone ring, δ 6.31 (s, 1H) proton of coumarin's –C(=O)- C=C and δ 6.49–7.94(m,7H) protons of aromatic rings.

copound Ic (3-(4-bromophenyl)-2-((4-methyl-2-oxo-2H-chromen-7-yl)imino)thiazolidin-4-one)

(pale yellow powder): 30% yield, m.p: (129 °C), Rf: 0.45 (A), and IR(KBr) n: (3059 cm^{-1}) C-H stretching of aromatic ring, (2866 cm^{-1}, 2978 cm^{-1}) symmetric and asymmetric CH3 stretching, (1716 cm^{-1}) C=O stretching of thiozolidinone, (1651 cm^{-1}) C=O stretching of coumarin, (1604 cm^{-1}) C=N stretching, (1570 cm^{-1}, 1539 cm^{-1}) C=C stretching of aromatic ring, (1188 cm^{-1}) C-O-C stretching, (1029 cm^{-1}) C-N stretching, (717 cm^{-1}) C-S stretching. ^{1}H NMR (500 MHz, DMSO-d_6) d: δ 2.41 (s, 3H) protons of (O=C-CH3), δ 4.25 (s,2H) protons of the thiazolidinone ring, δ 6.31 (s, 1H) proton of coumarin's C (=O) -C=C-CH3) and δ 6.47–7.93(m,7H) protons of aromatic rings.

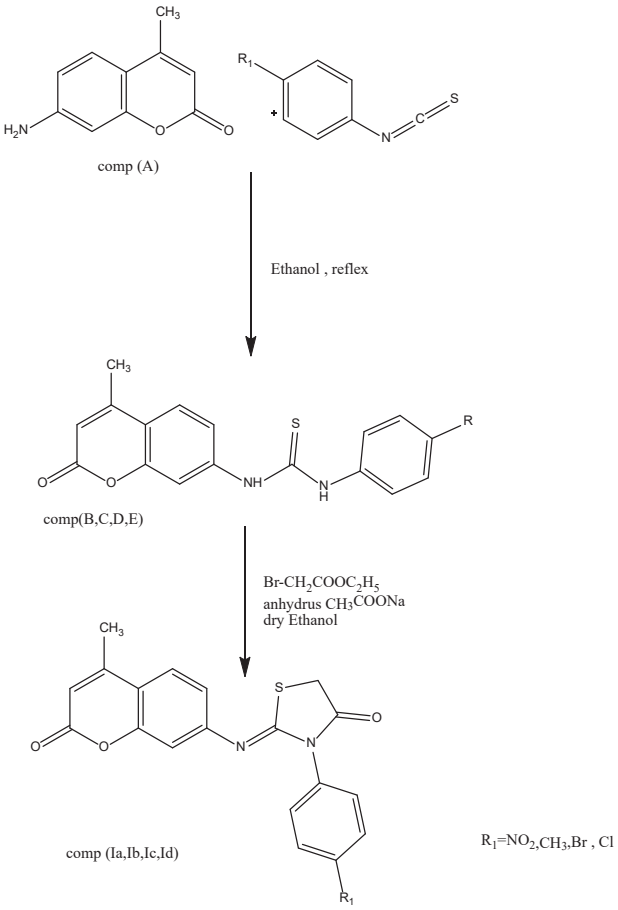
compound Id (3-(4-chlorophenyl)-2-((4-methyl-2-oxo-2*H*-chromen-7-yl)imino) thiazolidin-4-one)

(yellow powder): 32% yield, m.p: (137 °C), Rf: 0.4 (A), and IR(KBr) n: (3059 cm^{-1}) C-H stretching of aromatic ring, (2947 cm^{-1}, 2993 cm^{-1}) asymmetric and symmetric CH3 stretching (1716 cm^{-1}) C=O stretching of thiozolidinone, (1650 cm^{-1}) C=O stretching of coumarin, (1597 cm^{-1}) C=N stretching, (1415 cm^{-1}, 1543 cm^{-1}) C=C stretching of aromatic ring, (1126 cm^{-1}) C-O-C stretching, (1068 cm^{-1}) C-N stretching, (644 cm^{-1}) C-S stretching. ^{1}H NMR (500 MHz, DMSO-d_6) d: δ 2.40 (s, 3H) protons of coumarin's C (=O) -C=C-CH3), δ 4.51 (s,2H) protons of the thiazolidinone ring, δ 6.12 (s, 1H) proton of coumarin's –C(=O)- C=C and δ 6.28–7.76(m,7H) protons of aromatic rings.

Results and discussion

Chemistry

The synthesis of 4-thiazolidinones involved a two-step process. The initial stage involved the synthesis of thiourea derivatives by the reaction between 7-amino-4-methyl-coumarin and substituted phenyl isothiocynate, namely bromo, nitro, methyl, or chloro phenyl isothiocynate. This reaction took place separately in 99% ethanol (25 ml) and was refluxed for a duration of 12 hours at a temperature of 60 °C. The compounds (Ia–Id) were synthesized by reacting the compounds from step 1 with ethyl-bromoacetate (0.0012) and anhydrous sodium acetate (0.003 mol) individually in absolute ethanol (20 ml). The reaction mixture was then heated under reflux. The solvent was evaporated, and the solid will be purified through recrystallization using absolute ethanol to obtain the final chemicals, as described in Scheme 1.



Scheme 1. Chemical synthesis route for the chemicals (Ia–Id).

The thiourea derivatives (B, C, D, and E) were characterized by the appearance of two bands in the range of **3244 cm^{-1} to 3348 cm^{-1}** due to N-H stretching and the appearance of a band at **1014 cm^{-1} to 1072 cm^{-1}** due to C=S stretching. The IR spectra of the 4-thiazolidinone derivatives showed a stretching vibration of the C=O lactam amide in an area of **1716 cm^{-1} to 1724 cm^{-1}**, C=N stretching at range **1597 cm^{-1} to 1604 cm^{-1}**, C-N stretching at range **1103 cm^{-1} to 1068 cm^{-1}** and the disappearance of N-H stretching bands of thiourea.

According to the interpretation of the ^{1}H-NMR spectra, the compounds (Ia–Id) exhibited a doublet peak in the region of **4.21–4.51 ppm** due to protons of CH2 of the thiozolidinone ring. All aromatic protons appeared in their expected region.

Antimicrobial evaluation

The antibacterial activities of the synthesized derivatives (Ia-Id) were measured using a well diffusion technique with G(+ve) and G(-ve) bacteria and compared to ciprofloxacin and amoxicillin. Fluconazole was used as an antifungal standard to test for the antifungal activity of the final compounds. As stated in Table 1, DMSO was employed as a solvent and a control. Compound Ia showed the best antibacterial activity against gram-positive bacteria. Compound Ic showed the highest antifungal activity than other compounds against *C. albicans*.

Table 1. Antimicrobial activity of the final compounds.

Compound No.	Inhibition zone (IZ) in mm				
	E. coli (G-ve)	*P. aeroginosa* (G-ve)	*S. aureus* (G+ve)	*S. pyogenes* (G+ve)	*C. albicans*
Ciprofloxacin*	20	21	25	23	–
Fluconazole **	–	–	–	–	26
DMSO	–	–	–	–	–
Ia	11	10	15	16	10
Ib	16	12	13	13	15
Ic	12	13	10	9	17
Id	14	15	8	10	11

* standard for bacterial strains, ** standard for fungi. No activity, mildly active (inhibition zone 5–10 mm), moderately active (inhibition zone 10-20 mm), and very active (inhibition zone more than 20 mm).

Conclusion

Four novel 4-thiazolidinone derivatives were synthesized in this study, employing FT-IR spectroscopy and ^{1}HNMR techniques. These compounds were thoroughly characterized and assessed for their antimicrobial efficacy. The compounds demonstrated antibacterial activity against the specified Gram-positive and Gram-negative bacterial strains as well as the fungal strain. In summary, it was shown that 2-(4-methyl-2-oxo-2H-chromen -3-yl)-3-(4-nitrophenyl) thiazolidin -4-one had the highest level of potency among the compounds in the series. Based on the investigations on antimicrobial activity, it can be inferred that all the substances possess multiple levels of antimicrobial activity. Therefore, this study offers a starting point for the creation and assessment of other 4-thiazolidinone derivatives with antibacterial properties, which could potentially lead to the identification of effective medicines.

Ethics statements

The researchers abstained from employing animals in the study.

Authors contribution

The writers affirm their contribution to the paper. All authors critically evaluated the findings and gave their endorsement to the final draft of the publication.

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