Synthesis and Characterization of New Coumarin Derivatives

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ABSTRACT

New Schiff base compounds 3-acetyl-2-oxo-2H-chromen-7-yl-4(alkyloxy) benzoate thiosem icarbazide have been synthesized from the reaction between thiosemicarbazide and substituted coumarin. Target compound were characterized by FT-IR and NMR techniques heterogeneous coumarin compound. The result confirms the structure as suggested.

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1. Introduction

Coumarins or benzopyran-2-ones are group of nature-occurring lactones which were first derived from Tonka beans in 1820 [1]. Natural coumarin is also found in cereal plants licorice, strawberry, apricot, cherry, cinnamon, alfalfa, etc.[2]. Synthetic coumarin began in the early of 1820s, and started to be used in the production of perfumes, flavorings and pharmaceuticals in 1868 [3]. Coumarin derivatives are present in large quantities and more than 1300 coumarins have been identified from different natural sources [4]. Recently, all the studies have been showed that coumarins possess a variety of biological activities, such as anticoagulants [5], antibacterial [6], anti-cancer [7], anticonvulsants, diabetes, antifungal [8], and vasodilators [9]. Coumarin is known for its distinctive aroma and stable alkalinity, most manufactured coumarins have been widely used in pesticides, soaps, and perfumes [10]. Coumarin and its derivatives have wide applications against a range of viruses such as human immunodeficiency virus, influenza viruses, etc. [11-12]. The biological discipline of metal complexes with different bonds gives chemists a new horizon including organic compounds derived from coumarins [13-14]. The first application of mineral-based compounds in medicine was presented in 1911 by Paul Ehrlich [15]. Based on the information received about the biological properties of mineral complexes and coumarin derivatives, wherein several mineral complexes were prepared with the 4-methyl-7-hydroxy coumarin derivative [16-17]. Coumarins produce compounds with wide applications in organic synthesis chemistry. The most important methods of formation are: Knoevenagel condensation [18], Michael reaction [19], Birkin reaction [20],Wittig reaction [21], Bakier *et al*, [22], Clayzen rearrangement reaction [23], and Pechmann reaction [24].

2. Experimental

2.1. Material and methods

All chemical and solvents were of highest purity from commercial suppliers such as sigma-Aldrich. The chemicals used throughout the project for the synthesis of all intermediates and title compounds are listed, as follows, 1-bromoalkanes (C_6-C_{14}), 2,4-dihydroxybenzaldehyde, 4-di methylaminopyridine, ethyl acetoacetate, glacial acetic acid, 4-hydroxybenzoic acid, N,N'-dicyclohexylcarbodiimid (DCC), potassium hydroxide, piperidine, thiosemicarbazide. The prepared chemical compounds, were characterized by UV-Vis measurements of were performed using a(Shimadzu-160) UV-Vis Spectrophotometer apparatus. The samples were prepared at a concentration (1×10^{-3}) using a solvent DMSO-d_6 and a quartz cell (1cm thick) within a

wavelength (200-900) nm. Infrared spectra were recorded with FT-IR optical spectrometer Bruker Alpha, Germany, in the frequency range (4000-400) cm^{-1}, with standard KBr beam splitter. The ^{1}H-NMR and ^{13}C-NMR spectra were recorded on nuclear magnetic resonance at 298 K on a Bruker, Ultrasheild (500) MHz, DMSO-d_6 was used as solvent.

2.2. Synthesis of (4-alkyloxy)benzoic acid (1a-i):

Compound (**1a-i**) was prepared according to the previous studies [25]. (5gm, 36 mmol) of 4-hydroxybenzoic acid was dissolved in 20 ml of ethanol, and (1.21gm, 21 mmol) of KOH in 15 ml of ethanol (EtOH), the mixture of (KOH-MeOH), were collected of by ice bath. The mixture was removed and an equivalent amount of Alkylbromide (1-bromohexane) was added to the reaction mixture. The reaction mixture was then heated and condensed for 22 hours. Drops of 10 % dilute hydro chloric acid was added to neutralize the reaction solution. A white solid precipitate is formed after the solvent was evaporated. The white precipitate was filtered with a Buchner funnel, washed well with distilled water, and then recrystallized with ethanol [26].

4-(hexyloxy) benzoic acid: 1a

Yield 69 %; white solid; M.p: 146-148 °C. M.Wt: 222.28 ; IR: (KBr)(cm^{-1}): 3742 overton, 3361 (OH), 2977-2869 (C-H aliphatic), 1676 (C=O), 1606-1593 (C=C), 1240 (C-O).

4-(heptyloxy) benzoic acid: 1b

Yield 71 %; white solid; M.p: 140-142 °C. M.Wt: 236.31; IR: (KBr)(cm^{-1}): 3741 overton, 3443-3379 (OH), 2921-2855(C-H aliphatic), 1668 (C=O), 1579-1509 (C=C),

1268 (C-O).

4-(hoctyloxy) benzoic acid: 1c

Yield 80 %; white solid; M.p: 106-108 °C. M.Wt: 250.33; IR: (KBr)(cm^{-1}): 3741 overton, 3444 (OH), 2917-2817 (C-H aliphatic), 1651(C=O),1578-1510 (C=C), 1266 (C-O).

4-(nonyloxy) benzoic acid: 1d

Yield 82 %; white solid; M.p: 150-152 °C. M.Wt: 264.36; IR: (KBr)(cm^{-1}): 3743 overton, 3392 (OH), 2921-2830 (C-H aliphatic), 1679 (C=O), 1610-1520 (C=C), 1242 (C-O).

4-(decyloxy) benzoic acid: 1e

Yield 82 %; white solid; M.p: 126-128 °C. M.Wt: 278.39; IR: (KBr)(cm^{-1}): 3745 overton, 3396 (OH), 2987-2885 (C-H aliphatic), 1676 (C=O), 1608-1593 (C=C), 1244 (C-O).

4-(undecyloxy) benzoic acid: 1f

Yield 87 %; white solid; M.p: 135-137 °C. M.Wt: 292.20; IR: (KBr)(cm^{-1}): 3745 overton, 3380 (OH), 2925-2854 (C-H aliphatic), 1680 (C=O), 1606-1520 (C=C),1250 (C-O).

4-(dodecyloxy) benzoic acid: 1g

Yield 86 %; white solid; M.p: 132-134 °C. M.Wt: 306.44; IR: (KBr)(cm^{-1}): 3741 overton, 3311

3311 (OH), 2917-2853 (C-H aliphatic), 1678 (C=O), 1510-1593 (C=C), 1266 (C-O).

4-(tridecyloxy) benzoic acid: 1h

Yield 85 %; white solid; M.p: 117-119 °C. M.Wt: 320.47; IR: (KBr)(cm^{-1}): 3741 overton, 3390 (OH), 2922-2856 (C-H aliphatic), 1684 (C=O), 1609-1580 (C=C), 1244 (C-O).

4-(tetradecyloxy) benzoic acid: 1i

Yield 91 %; white solid; M.p: 98-100 °C. M.Wt: 334.49; IR: (KBr)(cm^{-1}): 3744 overton,3394 (OH), 2918-2852 (C-H aliphatic), 1684 (C=O), 1604 -1591 (C=C), 1245 (C-O).

2.3 Synthesis 3-acetyl-7-hydroxy-2H-Chromine-2-one: 2

Compound **2** was prepared by (Knoevenagel condensation) according our previous article previous article [27] reaction of mixture 2,4-dihydroxybenzaldehyde (4.0 gm, 29 mmol) was dissolved in ethanol in a round bottom flask and ethyl acetoacetate (3.7 gm, 29 mmol) was added gradually, then piperidine (0.5 ml) as a catalyst was added to the mixture with stirring. The reaction was condensed for 6 hours, the resulting solution was cooled in an ice bath. The yellow product separated by filtration was collected, washed with 10 mL of alcohol and dried. The resulting precipitate was recrystallized with hot ethanol [28]. Yield 89 %. Deep yellow, M.p: 230-232 °C. M. Wt. 204.18; Anal. IR, (KBr) (cm^{-1}): 3740 overton, 3475 (OH), 3059 (C-H aromatic), 2984-2881 (C-H aliphatic), 1706 (C=O) lactone ring, 1677 (C=O) ketone, 1592-1445

(C=C);^{1}H-NMR δ (ppm, DMSO-d_6): 11.14 (s, 1H, H10), 8.59 (s, 1H, H4), 7.78 (d, 1H, *J* = 8.85 Hz, H6), 6.84 (d, 1H, *J* = 8.87 Hz, H7), 6.75 (s,1H, H9), 2.55 (s, 3H, CH_3); ^{13}C-NMR δ (ppm, DMSO-d_6), 195.15 (C=O) ketone, 164.60 (C2), 159.52 (C10), 157.60 (C8), 148.18 (C4), 133.31 (C3), 119.62 (C6), 114.66 (C7), 111.15(C5), 102.36 (C9), 30.35 (CH_3).

2.4 Synthesis of 3-acetyl-2-oxo-2H-chromen-7-yl-4(alkyloxy)

benzoate (3a-i):

All these compounds were prepared following the method of [29]. A mixture of 4-hexyl oxybenzoic acid (2 gm, 8.9 mmol), 3-acetyl-2-oxo-2H-chromon-2-one (1.6 gm, 8.9 mmol), and 4-N,N-dimethylamino pyridine (DMAP). Were dissolved in a (24 ml) mixture of dimethylfromamide (DMF) and dichloromethane (DCM), stirred at 0 °C. was added N,N'-dicyclohexylcarbodiimide (DCC) (1.65 gm, 8.9 mmol) dissolved in 5ml of DCM drop wise to this solution and stirred at 0°C for 1hour, then the mixture was stirred at the room temperature for 6-8 hours. Finally, the reaction mixture was filtered, and dichloromethane (DCM) is removed by evaporation. The yellow precipitate was washed with cold distilled water and then recrystallized with ethanol [30].

3-acetyl-2-oxo-2H-chromen-7-yl-4-(hexyloxy) benzoate: 3a

Yield 76 %; yellow solid; M.p: 236-238 °C. M.Wt: 408.45; IR: (KBr)(cm^{-1}): 3321 overton, 3030 (C-H aromatic), 2975-2848 (C-H aliphatic), 1726 (C=O overlap with

ester), 1679 (C=O ketone),1614-1546 (C=C), 1234 (C-O).

3-acetyl-2-oxo-2H-chromen-7-yl-4-(heptyloxy) benzoate: 3b

Yield 80 %; yellow solid; M.p: 236-238 °C. M.Wt: 422.47; IR: (KBr)(cm^{-1}): 3320 overton,3032

(C-H aromatic), 2975-2849 (C-H aliphatic), 1725 (C=O overlap with ester), 1679 (C=O ketone), 1615-1565 (C=C), 1236 (C-O).

3-acetyl-2-oxo-2H-chromen-7-yl-4-(hoctyloxy) benzoate: 3c

Yield 78 %; yellow solid; M.p: 246-248 °C. M.Wt: 436.50; IR: (KBr)(cm^{-1}): 3320 overton, 3050 (C-H aromatic), 2955-2847 (C-H aliphatic), 1726 (C=O overlap with ester), 1679 (C=O ketone), 1616-1565 (C=C), 1233 (C-O).

3-acetyl-2-oxo-2H-chromen-7-yl-4(nonyloxy) benzoate: 3d

Yield 83 %; yellow solid; M.p: 251-253 °C. M.Wt: 450.53; IR: (KBr)(cm^{-1}): 3320 overton, 3033 (C-H aromatic), 2965-2850 (C-H aliphatic), 1727 (C=O overlap with ester), 1674 (C=O ketone), 1608-1576 (C=C), 1240 (C-O).

3-acetyl-2-oxo-2H-chromen-7-yl-4(decyloxy) benzoate: 3e

Yield 87 %; yellow solid; M.p: 256-258 °C. M.Wt: 464.55; IR: (KBr)(cm^{-1}): 3318 overton, 3032 (C-H aromatic), 2974-2848 (C-H aliphatic), 1728 (C=O overlap with ester), 1680 (C=O ketone), 1610-1537 (C=C), 1234 (C-O).

3-acetyl-2-oxo-2H-chromen-7-yl-4(undecyloxy) benzoate: 3f

Yield 90 %; yellow solid; M.p: 262-264°C. M.Wt: 478.58; IR: (KBr)(cm^{-1}): 3328 overton, 3038 (C-H aromatic), 2975-2850 (C-H aliphatic), 1730 (C=O overlap with ester), 1678 (C=O ketone), 1600-1568 (C=C), 1242 (C-O).

3-acetyl-2-oxo-2H-chromen-7-yl-4(dodecyloxy) benzoate: 3g

Yield 84 %; yellow solid; M.p: 268-270 °C. M.Wt: 492.61; IR: (KBr)(cm^{-1}): 3320 overton, 3035 (C-H aromatic), 2975-2849 (C-H aliphatic, 1725 (C=O overlap with ester), 1679 (C=O ketone), 1615-1565 (C=C), 1236 (C-O).

3-acetyl-2-oxo-2H-chromen-7-yl-4(tridecyloxy) benzoate: 3h

Yield 81 %; yellow solid; M.p: 280-282 °C. M.Wt: 506.63; IR: (KBr)(cm^{-1}): 3320 overton, 3033 (C-H aromatic), 2980-2852 (C-H aliphatic, 1729 (C=O overlap with ester), 1677 (C=O ketone), 1621-1565 (C=C), 1238 (C-O).

3-acetyl-2-oxo-2H-chromen-7-yl-4(tetradecyloxy) benzoate: 3i

Yield 87 %; yellow solid; M.p: 277-278 °C. M.Wt: 520.66; IR: (KBr)(cm^{-1}): 3320 overton, 3030 (C-H aromatic), 2975-2849 (C-H aliphatic), 1725 (C=O overlap with ester), 1679 (C=O ketone), 1615-1565 (C=C), 1236 (C-O).

2.4 Synthesis 3-acetyl-2-oxo-2H-chromen-7-yl-4(alkyloxy) benzoate thiosemicarbazide (4a-i):

These series of titled compounds 3-acetyl-2-oxo-2H-chromen-7-yl-4(hexyloxy) benzoatethio semicarbazide (4a-i) was synthesis according to the modified method which was reported in the literature [31]. A mixture of 3-acetyl-2-oxo-2H-chromen-7-yl-4-(hexyloxy)benzoate (0.5 gm, 1.2 mmol) and thiosemicarbazide (0.09 gm, 1.2 mmol) was dissolved in 20 ml of absolute ethanol. The mixture was condensed with the addition of a few drops of glacial acetic acid for 6 hours at 80 °C. The product was recrystallized with hot ethanol. The analytical data for these series of compounds are summarized as follows:

3-acetyl-2-oxo-2H-chromen-7-yl-4(hexyloxy)benzoatethiosemicarbazide:4a

Yield 80 %; pale yellow; M.p: 200-202 °C. M.Wt: 481.56. Anal Calcd (%) for $C_{25}H_{27}N_3O_5S$: Found, C, 62.35; H, 5.65; N, 8.73; O, 16.16; S, 6.66; IR: (KBr)(cm^{-1}): 3360 (NH_2), 3255 (N-H), 3170 (C-H aromatic), 2927-2849 (C-H aliphatic), 1725 (C=O ester, lactone ring), 1621 (C=N), 1538-1448 (C=C), 1240 (C-O).

3-acetyl-2-oxo-2H-chromen-7-yl-4(heptyloxy)benzoatethiosemicarbazide:4b

Yield 82 %; Pale yellow; M.p: 195-197 °C. M.Wt: 495.59. Anal Calcd (%) for $C_{26}H_{29}N_3O_5S$: Found, C, 63.01; H, 5.90; N, 8.48; O, 16.14; S, 6.47; IR (KBr)(cm^{-1}): 3363 (NH_2), 3260 (N-H), 3175 (C-H aromatic), 2927-2850 (C-H aliphatic), 1725 (C=O ester, lactone ring), 1625 (C=N), 1600-1448 (C=C), 1242 (C-O).

3-acetyl-2-oxo-2H-chromen-7-yl-4(hoctyloxy)benzoatethiosemicarbazide:4c

Yield 86 %; Pale yellow; M.p: 198-200 °C. M.Wt: 509.62. Anal Calcd (%) for $C_{27}H_{31}N_3O_5S$: Found, C, 63.63; H, 6.13; N, 8.25; O, 15.70; S, 6.29; IR: (KBr)(cm^{-1}): 3365 (NH_2), 3262 (N-H), 3176 (C-H aromatic), 2927-2849 (C-H aliphatic), 1726 (C=O ester, lactone ring), 1622 (C=N), 1558 -1448 (C=C), 1240 (C-O). ^{1}H-NMR δ (ppm, DMSO-d_6): δ = 11.30 (s,1H,N-H), δ = 8.56 (s,2H ,NH2), δ = 8.38 (s, 1H, H4), δ =7.94-8.04 (d,2H,J= 9.45Hz, H12, H13), δ =7.83-7.86 (s,1H, J= 8.05 Hz, H6), δ = 7.58-7.60 (d,2H,J= 8.34 Hz, H14, H15), δ = 6.94-6.97 (d, 1H, J=8.5Hz, H7), δ = 6.74-6.76 (s,1H,H9), δ = 3.29-3.45 (t, 2H,J=6.23,OCH_2), δ = 2.59-2.61 (s, 3H,H17), δ =1.94-1.03 (m, $(CH_2)_4$), δ = 0.93-0.87 (t, 3H,CH_3).^{13}CNMR δ (ppm, chloroform): 185.20 (C20), 180.99 (C=O), 177 .24(HC=N), 172.45 (C2), 168.51 (C10), 162.91 (C4), 156.86 (C12) ,154.20(C13), 148.89 (C14), 145.97 (C15) 137.44 (C3), 131.07 (C6), 127.89 (C7), 119.42 (C5), 107.50 (C9).49.37 (OCH_2), 24 .94-33.97(CH_2)n, 20.97(C17), 14.60(CH_3).

3-acetyl-2-oxo-2H-chromen-7-yl-4(nonyloxy)benzoatethiosemicarbazide:4d

Yield 75 %; Pale yellow; M.p: 199-201 °C. M.Wt: 523.64 .Anal Calcd (%) for $C_{28}H_{33}N_3O_5S$: Found, C, 64.22; H, 6.35; N, 8.02; O, 15.28; S, 6.12; IR: (KBr)(cm^{-1}): 3366 (N-H_2), 3263 (NH), 3168 (C-H aromatic), 2930-2850 (C-H aliphatic), 1728 (C=O ester, lactone ring), 1620 (C=N), 1590-1545 (C=C), 1242 (C-O).

3-acetyl-2-oxo-2H-chromen-7yl-4(decyloxy)benzoatethiosemicarbazide:4e

Yield 87 %; Pale Yellow, M.p: 206-208 °C. M.Wt: 537.67. Anal Calcd (%) for $C_{29}H_{35}N_3O_5S$: Found, C, 64.78; H, 6.56; N, 7.82; O, 14.88; S, 5.9 3; IR: (KBr)(cm^{-1}): 3363 (NH_2), 3260 (N-H), 3176 (C-H aromatic), 2927-2849 (C-H aliphatic), 1725 (C=O ester, lactone ring), 1621 (C=N), 1590-1447 (C=C), 1241(C-O). ^{1}H-NMR δ (ppm, DMSO-d6): δ = 11.27 (s,1H,N-H), δ = 8.48 (s,2H ,NH_2), δ = 8.37 (s, 1H, H4), δ =7.93-8.03 (d,2H,*J*= 9.45Hz, H12, H13), δ =7.83-7.85 (s,1H, *J*= 8.05 Hz, H6), δ = 7.58-7.60 (d,2H, *J*= 8.34 Hz, H14, H15), δ = 6.92-6.94 (d, 1H, *J*=8.5Hz, H7), δ = 6.71 -6.77 (s,1H,H9), δ = 3.21-3.45 (t, 2H,*J*=6.23,OCH_2), δ = 2.51-2.61 (s, 3H,H17), δ =1.94-1.03 (m, $(CH_2)_6$), δ = 0.93-0.87 (t, 3H,CH). ^{13}CNMR δ (ppm, chloroform): 187.04 (C20), 181.97 (C=O), 178.10(HC=N), 172.74 (C2), 169.75 (C10), 165.30 (C4), 157.23 (C12) ,155.15(C13), 148.96 (C14), 145.00 (C15) 137.27 (C3), 132.21 (C6), 127.48 (C7), 118.20 (C5), 106.90 (C9).49.71 (OCH_2), 24.94-33.62(CH_2)n, 19.89 (C17), 14.24(CH_3).

3-acetyl-2-oxo-2H-chromen-7yl-4(undecyloxy)benzoatethiosemicarbazide:4f

Yield 75 %; Pale yellow, M.p: 202-204 °C. M.Wt: 551.70. Anal Calcd (%) for $C_{30}H_{37}N_3O_5S$: Found, C, 65.31; H, 6.76; N, 7.62; O, 14.50; S, 5.81; IR: (KBr)(cm^{-1}): 3366 (NH_2), 3259 (N-H), 3175 (C-H aromatic), 2930-2850 (C-H aliphatic), 1726 (C=O ester, lactone ring), 1620 (C=N), 1591-1445 (C=C), 1242 (C-O).

3-acetyl-2-oxo-2H-chromen-7yl-4(dodecyloxy)benzoatethiosemicarbazide:4g

Yield 87 %; dark yellow, M.p: 203-205 °C. M.Wt: 565.72. Anal Calcd (%) for $C_{31}H_{39}N_3O_5S$: Found, C, 65.82; H, 6.95; N, 7.43; O, 14.14; S, 5.67; IR: (KBr)($cm^{-1)}$: 3365 (NH_2), 3260 (N-H), 3174 (C-H aromatic), 2916-2849 (C-H aliphatic), 1725 (C=O ester, lactone ring) 1619(C=N), 1572-1460 (C=C), 1242 (C-O).

3-acetyl-2-oxo-2H-chromen-7yl-4(tridecyloxy)benzoatethiosemicarbazide:4h

Yield 80 %; dark yellow; M.p: 204-206 °C. M.Wt: 579.75. Anal Calcd (%) for $C_{32}H_{41}N_3O_5S$: Found, C, 66.29; H, 7.13; N, 7.25; O, 13.80; S, 5.53; IR: (KBr)($cm^{-1)}$: 3365 (NH_2), 3261 (N-H), 3160 (C-H aromatic), 2920-2850 (C-H aliphatic), 1724 (C=O ester, lactone ring), 1620 (C=N), 1590-1448 (C=C), 1238 (C-O).

3-acetyl-2-oxo-2H-chromen-7yl-4(tetradecyloxy)benzoatethiosemicarbazide:4i

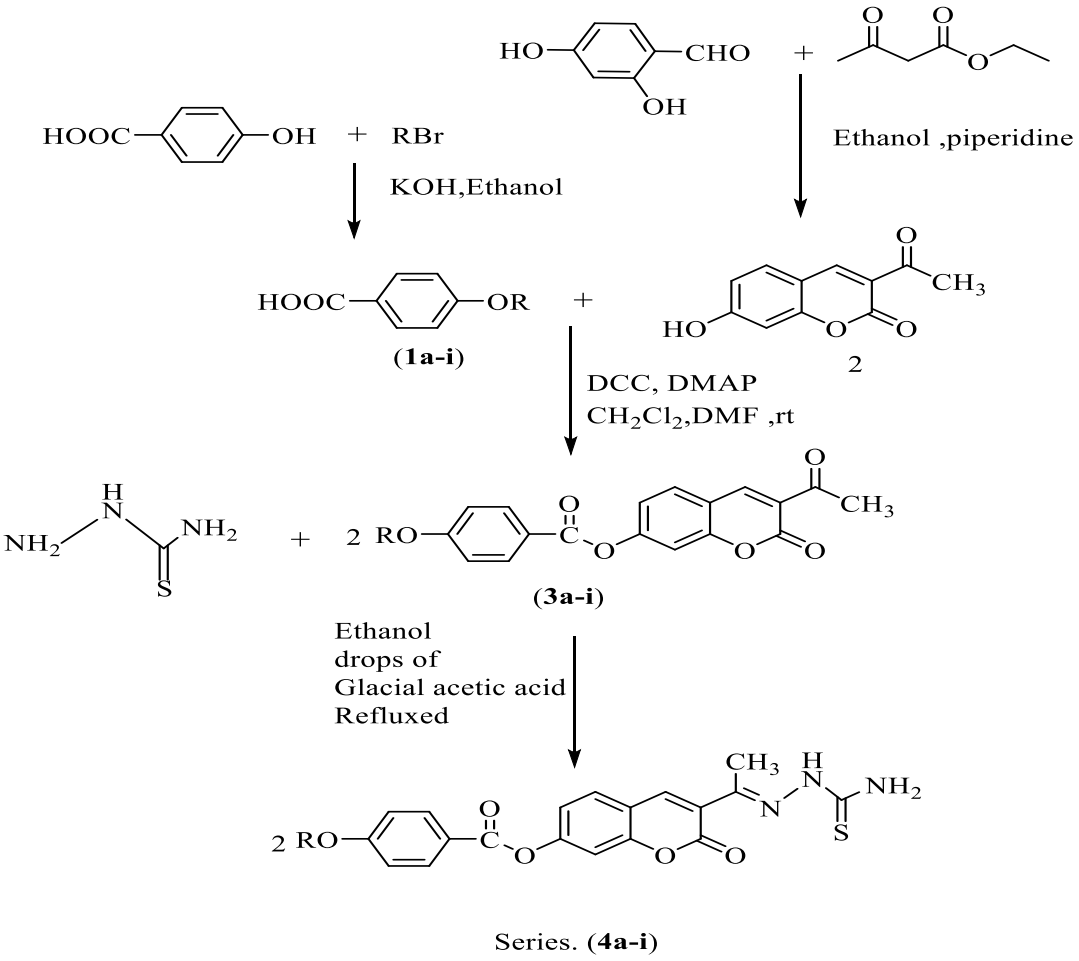
Yield 88 %; dark yellow, M.p: 197-199 °C. M.Wt: 593.75. Anal Calcd (%) for $C_{33}H_{43}N_3O_5S$: Found, C, 66.75; H, 7.30 ; N, 7.08; O, 13.47; S, 5.40; IR: (KBr)($cm^{-1)}$: 3320 (NH_2), 3260 (N-H), 31

76 (C-H aromatic) 2927-2855 (C-H aliphatic), 1725 (C=O ester, lactone ring), 1623 (C=N), 1570-14 48 (C=C), 1228 (C-O).

3. Results and discussion

The bath way towards synthesis intermediate and target compounds is shown in scheme **1**. Intermediate compounds **1a-i** were obtained from Williamson's synthesis between subs-alkylbromid and p-hyroxybenzoic acid. Coumarin compound was prepare via Knovenagel condensation reaction, fellow by esterification between compounds **1a-i** with coumarin to prepared compounds **3a-i**. The final products **4a-i** were obtained by condensation reaction between 3-acetyl-2-oxo-2H-chromen-7-yl-4 (hexyloxy) benzoate with thiosemicarbazide. The

compounds **4a-i** were characterized by analytical and various spectroscopic (UV-Visible, FT-IR, ^{1}H-NMR & ^{13}C-NMR) techniques.



Scheme 1: Synthesis of series (**4a-i**)

3.1 Spectroscopic studies

The structure of compound coumarin were confirmed by different analytical techniques such as

FT-IR, **^{1}H-NMR**, and **^{13}C-NMR**.

The FT-IR spectrum of compound 2 is shown in Fig.1. A broad band is shown 3475 cm^{-1} that can be assigned to the OH group [32]. The absorption band appears at frequency 3059 cm^{-1} weak ascribed to the aromatic (C-H) [33]. Moreover, the alkyl groups shows absorption band at of 2850-2984 cm^{-1} [34]. The strong band at the frequency of 1706 cm^{-1} it could be carbonyl group (C=O), while bond (C=C) and ether bond shows at the frequencies 1445-1592 cm^{-1} and 1205-1248 cm^{-1}, respectively [33].

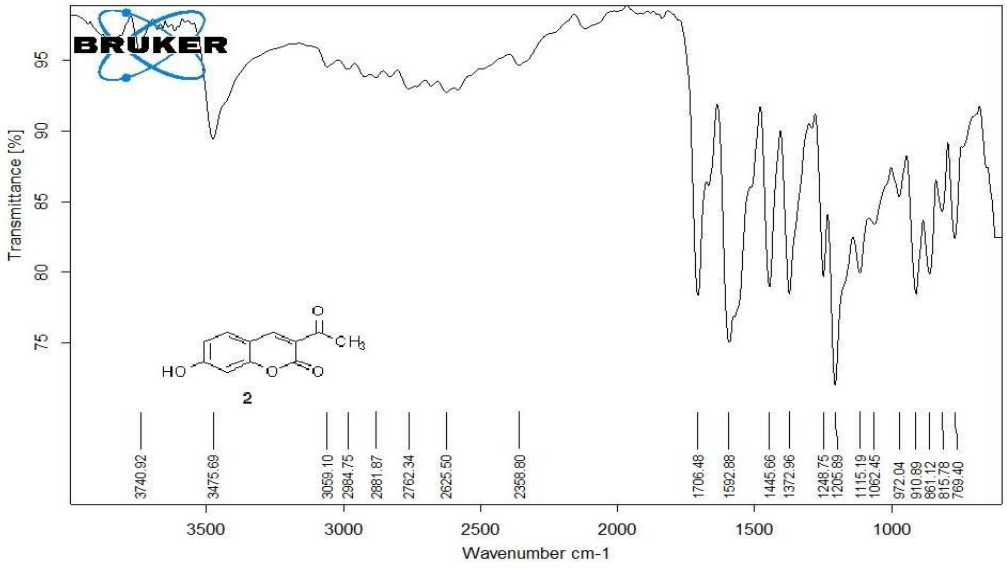


Figure 1: FT-IR spectrum of compound 2

^{1}H-NMR spectrum of compound 2. The signal at the chemical shift of δ =11.14 ppm proton can be attributed to the OH group (H10). Moreover, the signal at the chemical shift of δ = 8.59 ppm assigned to proton of (H4) of the heterocyclic in coumarin. Two double chemical shift referring to the proton (H6) and (H7) at the chemical shift of δ = 7.78-7.80 ppm and δ = 6.84-6.87 ppm, respectively. On the other hand, the spectrum shows two signals at the chemical shift δ = 6.75 ppm and δ =2.51 ppm can be attributed to the proton (H9) and (CH_3) in the coumarin, respectively (Figure 2) [35]. The ^{13}C-NMR spectrum of compound 2 shows many signals according to the carbon in the compound. The carbon signal that appears at the chemical shift of δ = 195.75 ppm is attributed to the carbon of (C=O acetyl group), while the signal at the chemical shift of δ =164.86 ppm, it is assigned to the carbonyl group of coumarin. In addition, all the aromatic carbons (C2, C10, C4, C3, C6, C7, C5, and C9) are shows at the chemical shifts of δ = 159.59 ppm, δ =157.69 ppm, δ =148.35 ppm, δ =132.71 ppm, δ = 119.31 ppm, δ = 113.95

ppm, δ = 111.27 ppm, and δ = 102.38 ppm, respectively. The methyl group shows the signal at the chemical shift δ = 30.41 ppm (Figure **3**) [35].

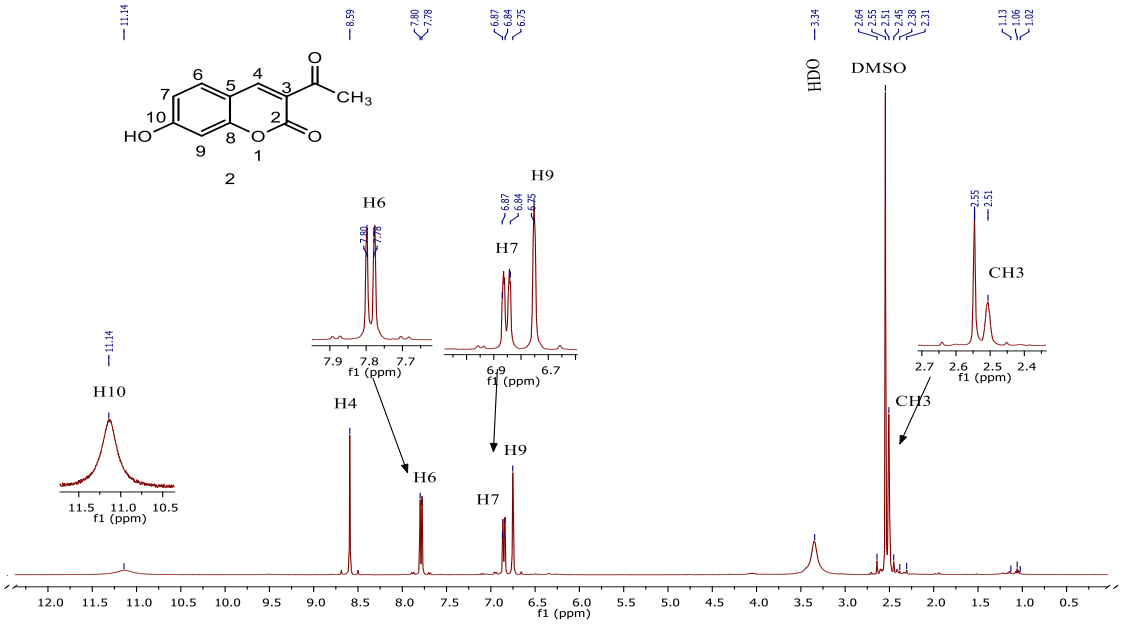


Figure 2: ^{1}H-NMR spectrum of compound 2

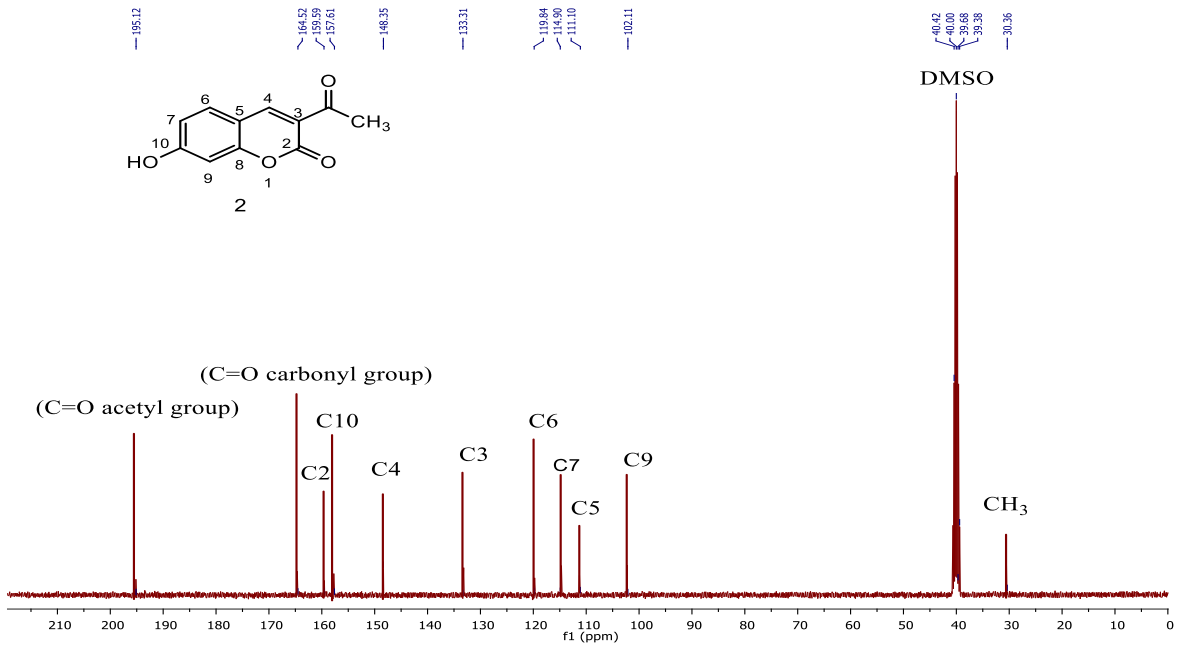


Figure 3: ^{13}C-NMR spectrum of compound 2

The UV-Vis spectrum of the prepared ligand was studied at a concentration of (1×10^{-3}M) using a solvent (DMSO-d_6), and in the range (200-900) nm at room temperature. The spectrum of the ligand 4g showed two absorption peaks. The first peak at position 269 nm (37174 cm^{-1}) is related to the ($\pi\rightarrow\pi^*$) transitions of the coumarin ring, while the second peak at position 346 nm (28901 $cm^{-1)}$ is due to the transitions of ($n\rightarrow\pi^*$), a result of the transitions of electronic charge in the aromatic ring [36] as shown in Fig. 4.

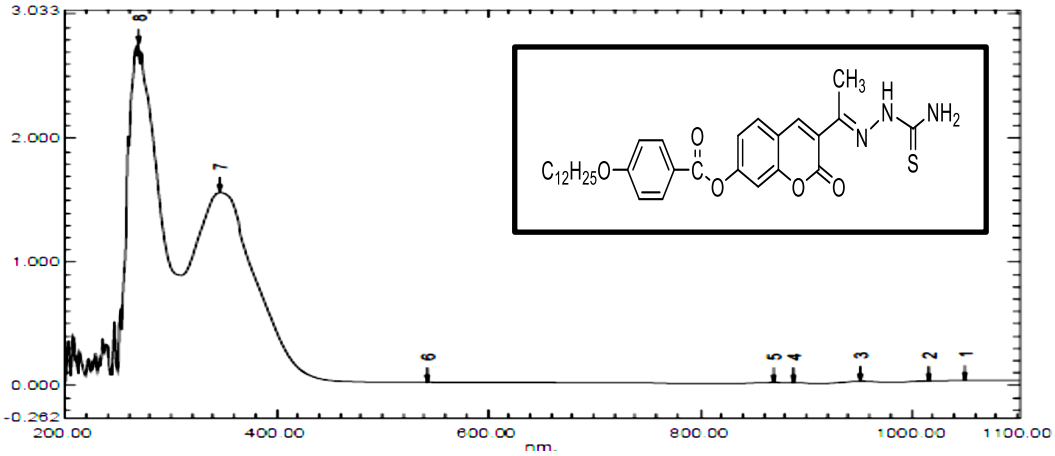


Figure 4: Spectrum UV-Vis of ligand 4g

The FT-IR data of the series compounds 4a-i were recorded in the experimental section, while the selected FT-IR spectra of compound 4c and 4g were presented in Figs. 5 and 6, respectively. The spectra show many similarities were observed in terms of absorption frequency as well as band shapes. The absorption of the (N-H) and (NH_2) group is shown broad peak at the range of 3260-3262 and 33 22-3365 cm^{-1}, respectively [37]. Moreover, the spectra appeared weak absorption in the range of 3173-3176 cm^{-1} that is related to the stretching vibration (C_{ph}-H) [38]. The (υ CH_3as, υ CH_3s) and (υCH_2as, υCH_2s) groups show absorptions at the frequency 2916-2927 cm^{-1} and 2846-2849 cm^{-1}, respectively [39]. The (C=O) of lactone is found at 1700-1726 cm^{-1} [40]. The absorptions in the frequency of range 1619-1623 cm^{-1} could be attributed to the (C=N) group [41]. On the other hand, strong absorption appears at the range 1435-1572 cm^{-1} was attributed to the stretching (C=C) of the phenyl ring [42]. While, in the fingerprint region the frequencies appear from 1238-1241 cm^{-1} which is attributed to the ether extended group (C-O) [38].

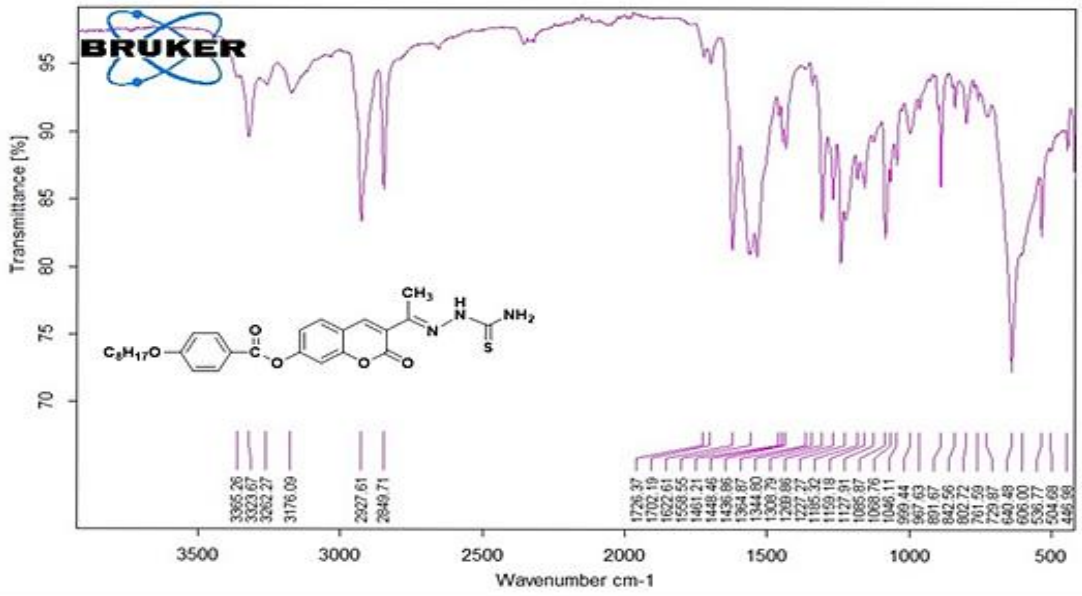


Figure 5: FT-IR spectrum of compound 4c

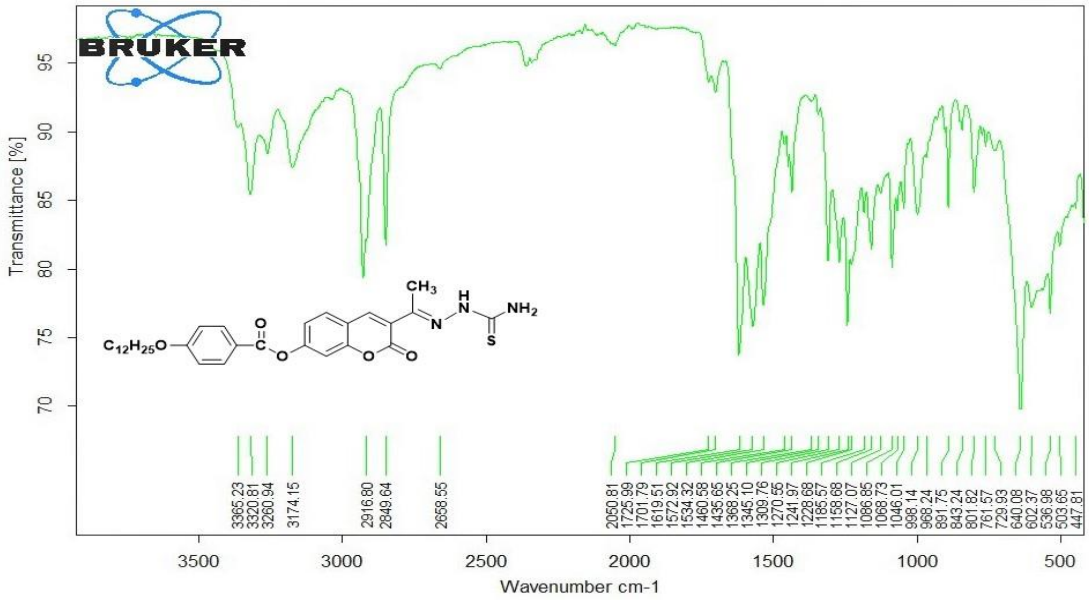


Figure 6: FT-IR spectrum of compound 4g

The data of the ^{1}H-NMR spectra of compound 4c and 4e was chosen to interpret the signal locations, as shown in (Figures 7 and 8) in the following sections. The ^{1}H-NMR spectra showed a signal at δ = 8.48-8.56 ppm and δ = 11.27-11.30 ppm attributed to the protons of (H19, H18) respectively [43]. The signal appears at the chemical shift δ = 8.37-8.38 ppm corresponds to a proton (H4) in coumarin rings [39]. Moreover, the aromatic protons appears signals (H12, H13), (H6), (H14, H15), and (H7) at the chemical shift of δ =7.94-8.04 ppm, δ =7.80-7.86 ppm, δ = 7.58-7.60 ppm, and δ = 6.92-6.97 ppm, respectively. While the signal appears protons (H9) where located at the chemical shift δ = 6.71-6.77 ppm [39]. The triplets attributed for the methylene protons (OCH_2) in the long chain at δ = 3.21-3.45 ppm [39]. The protons of (CH_3) appear in the coumarin ring at the chemical shift δ = 2.51-2.61ppm [41].The resonance of the protons (CH_2) appeared at the range of δ =1.94-1.03 ppm, and δ =1.88-1.06 ppm, respectively for compounds **4c** and **4e**. Furthermore, it observed in the upfield δ = 0.93-0.87 ppm which can be assigned to the methyl protons of the alkyl terminal chains [41].

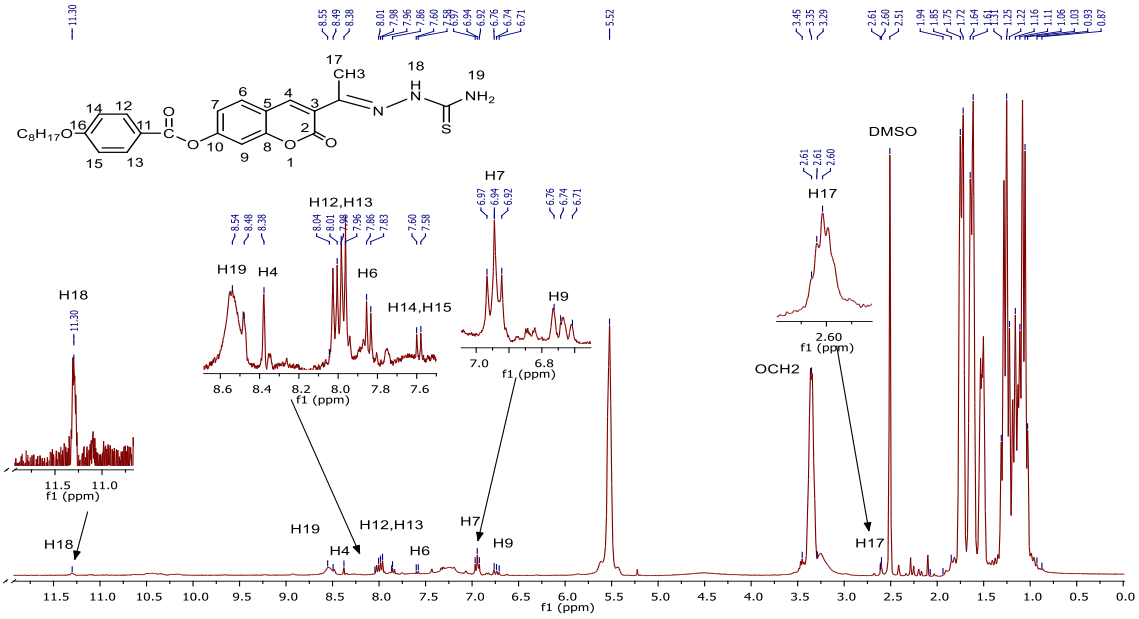


Figure7: ^{1}H-NMR spectrum of compound 4c

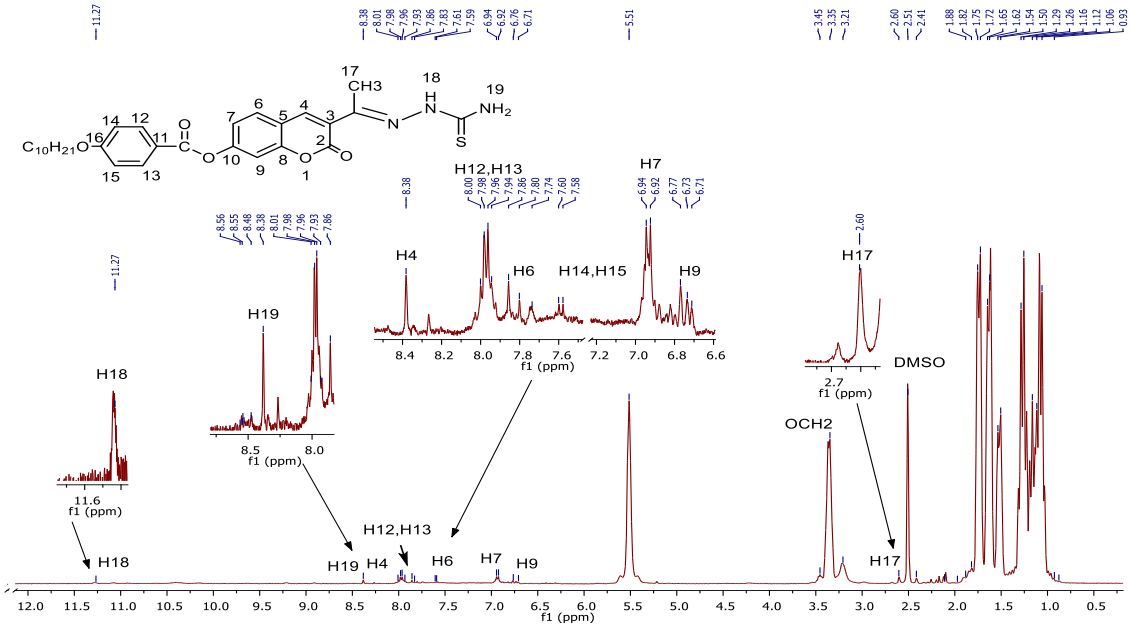


Figure 8: ^{1}H-NMR spectrum of compound 4e

The ^{13}C-NMR spectra of 4c and 4e compounds was assigned in the experimental section as an illustrative example of chemical signals, as shown in (Figures 9 and 10). The ^{13}C-NMR

spectra of the compound 4c and 4e indicates the appearance of four recorded signals in the region of δ =185.20-187.04 ppm, δ =180.99-181.97 ppm, δ = 177.24-178.10 ppm, and 172.45-177.74 ppm are belonging to the of the atom carbon (C20), the carbonyl ester, the (HC=N) group and the carbonyl carbon (C2) in the heterocyclic coumarin, respectively [34,45-47]. Moreover, the resonances due to the aromatic carbons at the chemical shifts of δ = 168.51-169.75 ppm, δ =162.91-165.30 ppm, δ = 156.86-157.23 ppm, δ = 154.20 -155 .15 ppm , δ = 148.89-148.96 ppm, δ = 145.00 -145.97 ppm, δ = 137.27-137.44 ppm, δ =131.07-132.21 ppm, δ = 122.48-122.89 ppm, δ = 118.20-119.42 ppm, and δ = 106.90-107.50 it can be attributed to carbon (C10, C4, C12, C13, C14, C15, C3, C6, C7, C5, and C9) respectively [39]. While the signal appeared at the chemical shift range δ = 49.37-49.71 ppm that was attributed to the presence of an ether linked (OCH_2) group [38], and the chemical shift which located at range δ = 24.94-33.62 ppm is assigned to (CH_2)n group [48]. the signal which located at range δ =19.89-20.97 ppm is assigned to (CH_3) group in the coumarin ring [49]. The alkyl terminal chains appear at the chemical shift δ = 14.24-14.60 ppm [50].

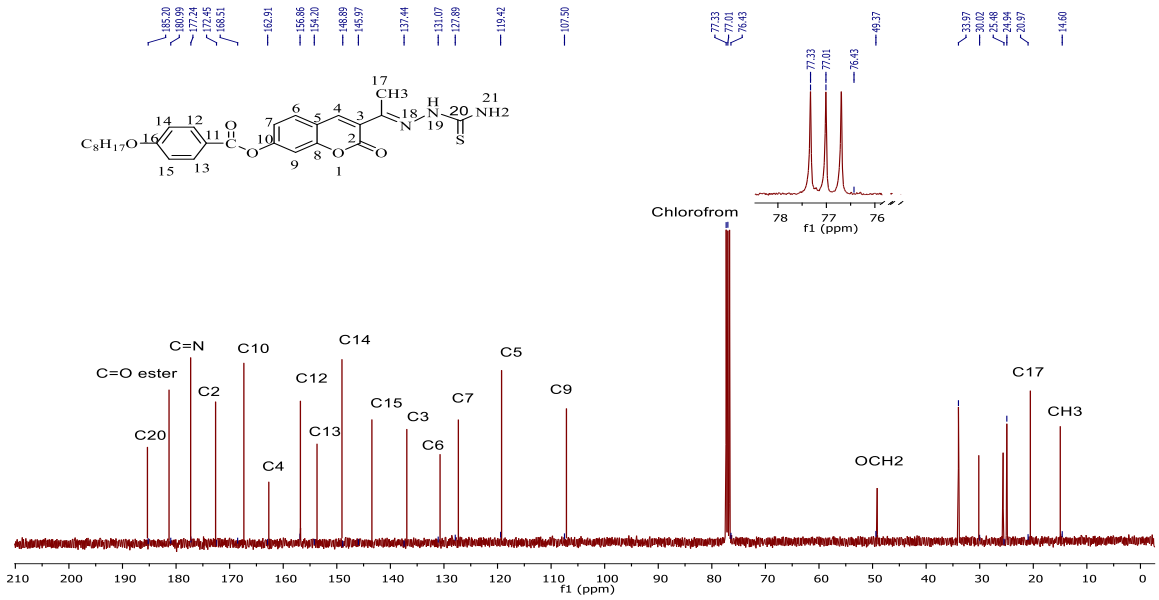


Figure 9: ^{13}C-NMR spectrum of compound 4c

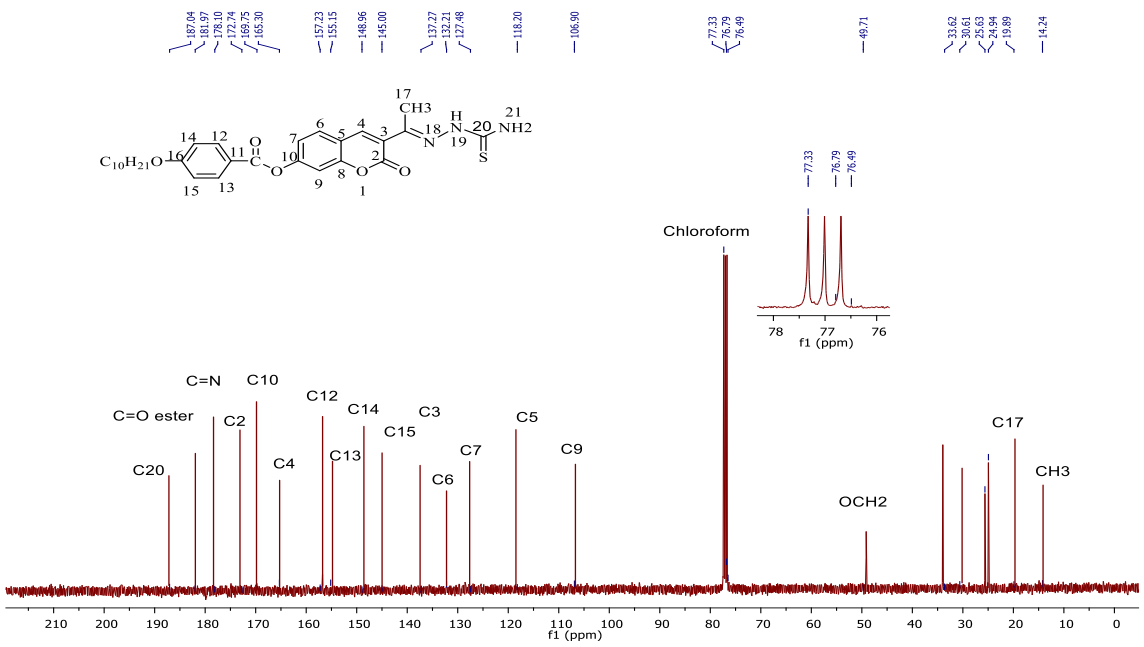


Figure 10: ^{13}C-NMR spectrum of compound 4e

4. Conclusions

A new homogeneous compounds based on coumarin are **s**ynthesied and characterized.The new ligands were synthesized from 3-acetyl-2-oxo-2H-chromen-7-yl-4-(hexyloxy) benzoate with thiosemicarbazide in the presence of a catalytic amount of glacial acetic acid in refluxing ethanol. These compounds contain alkyl terminal chains R = C_6-C_{14}, it was analyzed and characterized by Spectroscopy techniques. In the present study, the final compounds gave gradient colors from Pale yellow to dark yellow with afforded a good yield as indicated in the product percentage.

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توليف وتشخيص مركبات الكومارين الجديدة

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المستخلص

تم تصنيع مركبات قواعد شيف الجديدة (3-أسيتيل-2-أوكسو-2H-كرومومين-7-4-yl (ألكيلوكسي) بنزوات إيثيوسيميكاربزيد) من التفاعل بين (ثايوسيميكربازايد) والكومارين البديل. تم تمييز المركب المستهدف بواسطة تقنيات FT-IR و NMR مركب كومارين غير متجانس. النتيجة تؤكد الهيكل كما هو مقترح.