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Scientific paper

Synthesis of New Coumarin Scaffold Bearing 2-Iminochromene Moiety

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Abstract

Coumarin is classified as one of interesting therapeutic starting research points to obtain remarkable compounds with high efficacy. So, during this research, new 2-iminochromene derivatives bearing coumarin moiety were synthesized. At first, cyanoacetohydrazone of 3-acetylcoumarin was prepared and used as the starting material. The 2-iminochromene derivatives were synthesized through the ring closure of cyanoacetohydrazone derivative with 4-hydroxysalicylaldehyde, 5-aryldiazosalicylaldehydes, 2-hydroxynaphthaldehyde and 7-hydroxychromone-6-carboxaldehyde derivative. All of the newly synthesized coumarin derivatives were obtained in excellent yields; so, the new synthesized coumarin derivatives will participate in the enrichment of the chemical libraries.

Keywords: Coumarins; 2-Iminochromenes; Benzopyrones; Chromenes; Chromen-2-ones.

1. Introduction

Coumarins (chromen-2-one derivatives) and 2-iminochromene derivatives are classes of natural benzopyrone. They are found in different varieties of the plant species. Coumarin is classified as one of interesting therapeutic starting research points and is being studied to obtain remarkable compounds with high efficacy and low toxicity. In the field of drug development and discovery, many various coumarin derivatives of synthetic origin are being developed as important compounds because they possess a wide range of pharmacological properties.[1–5]

Chromene derivatives have antibacterial and antifungal activities. Their promising antifungal activities make them potentially useful in agri-food and as pharmaceutical agents.[4–8] Chromene derivatives have great roles in treating of various cancers such as leukemia, lymphoma and renal cell carcinoma. Moreover, they reduce the effects of radiation therapy.[9,10] Chromene derivatives have been effective against several viruses, such as HIV, influenza, hepatitis, Dengue and Chikungunya.[11,12] Chromene derivatives were reported as analgesic, anti-pyretic and anti-inflammatory agents; where they recover fluid and edema in harmful tissues.[13,14] Some chromene derivatives were marketed as drugs to inhibit blood clotting such as phenprocoumon, choleraicin A, warfarin, acenocoumarin, the antibiotic novobiocin and hymecromone (umbelliferone).[15,16]

As shown in Figure 1, beside the application of chromene derivatives in the field of medicinal chemistry, some chromene derivatives have applications in various fields such as optical brighteners, markers, photosensitizers, lasers, fluorescent dyes, cosmetics, perfumes, pigments, dyes, solar cells and optical data storage devices.[17–19]

Depend on the above facts, it was aimed to synthesizing new 2-iminochromene derivatives bearing coumarin moiety wishing to discover new chromene derivatives that may have significant activities.

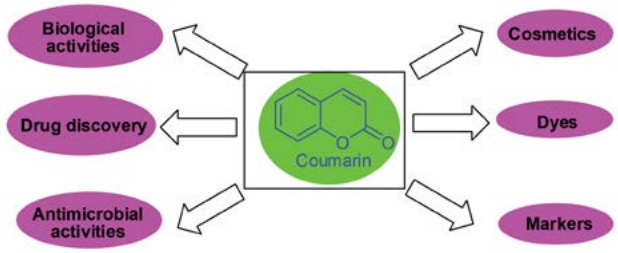
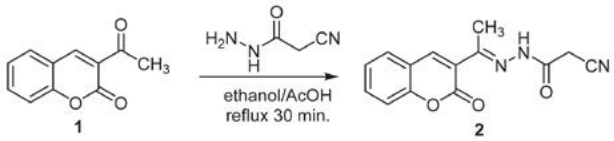


Figure 1. Applications of coumarin nucleus

2. Results and Discussion

The cyanoacetohydrazone **2** as starting material was prepared as illustrated in Scheme 1. First, 3-acetylcoumarin (**1**) was prepared *via* ring closure of salicylaldehyde

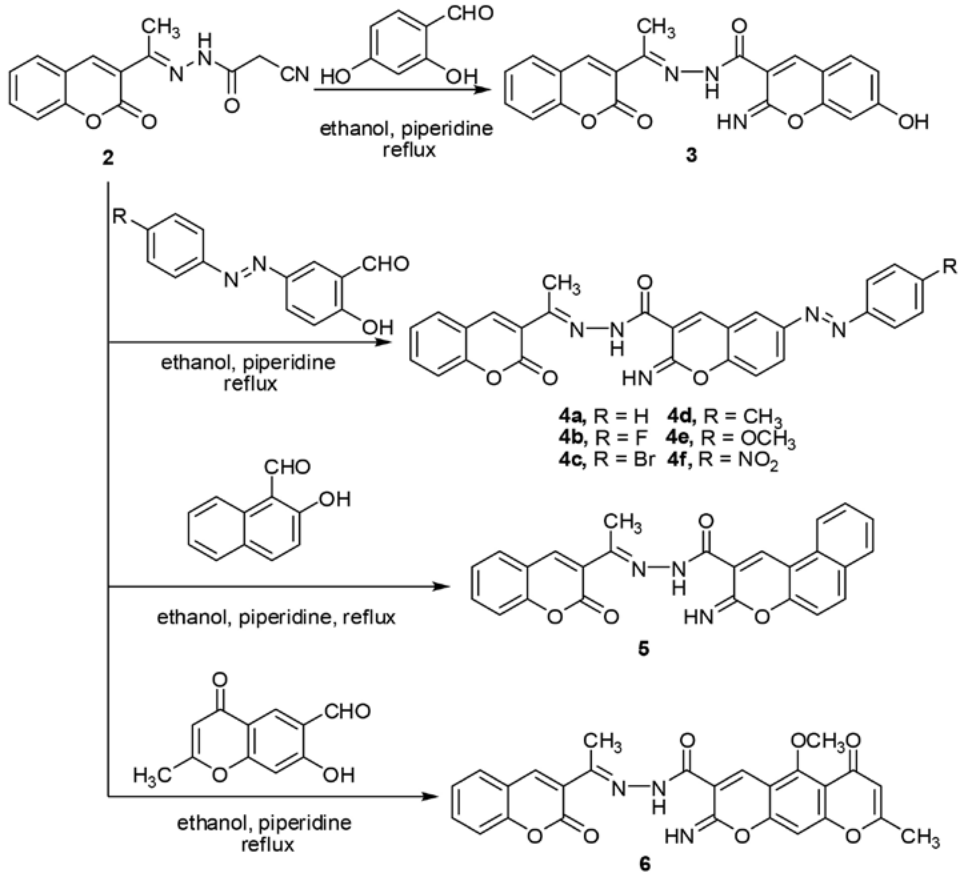
with ethyl acetoacetate. Cyanoacetohydrazone **2** of 3-acetylcoumarin was prepared through the condensation of 3-acetylcoumarin (**1**) with cyanoacetohydrazide.[20]



Scheme 1. Synthesis of the starting material cyanoacetohydrazone **2**

Here, the author aimed to synthesize coumarin scaffold bearing 2-iminochromene moiety hoping to obtain some significant compounds. Thus, as shown in Scheme 2, cyclocondensation of cyanoacetamide **2** with 4-hydroxysalicylaldehyde in ethanol containing piperidine gave 2-iminochromene derivative **3** in excellent yield. ^{1}H NMR spectrum of coumarin derivative **3** was characterized by the presence of one diagnostic singlet signal at δ 2.27 ppm corresponding to methyl protons. The seven protons of two benzene rings were displayed in 7.27–7.96 ppm region. Two diagnostic singlet signals of C_4-H of chromenes appeared at δ 8.29 and 8.64 ppm. Also, ^{1}H NMR spectrum showed three diagnostic singlets, broad and singlet signals at: 9.32, 10.50 and 13.69 ppm with one proton integral value for two imino and hydroxyl protons.

Several diazenyl derivatives have been synthesized for their potential activities.[21–25] Thus, the present work describes the preparation of chromene nucleus containing aryldiazo group. Thus, cyclization of cyanoacetamide **2** with 5-aryldiazosalicylaldehydes in the presence of piperidine afforded of 6-(aryldiazenyl)-2-iminochromene derivatives **4a**–**f**. ^{1}H NMR spectrum of iminochromene derivative **4d** showed two singlet signals at δ 2.29 and 2.43 ppm with three protons integral value for two methyl groups. Beside the diagnostic singlet signal for iminochromene-(H-5) at δ 8.44 ppm, the remaining ten benzopyran protons were assigned in δ 7.35–8.10 region. The two singlet signals at δ 8.30 and 8.76 were displayed for two C_4-H of chromene protons. The two imine protons signals were assigned at δ 9.52 and 13.58 ppm. Moreover, cyclocondensation reaction of **2** with 2-hydroxy-1-naphthaldehyde in ethanol containing piperidine smoothly furnished benzo[*f*]chromene derivative **5**. Finally, cyclocondensation reaction of **2** with derivative of 7-hydroxychromone-6-carboxaldehyde in ethanol containing piperidine smoothly furnished pyrano[3,2-*g*]chromene derivative **6**. ^{1}H NMR spectrum of pyrano[3,2-*g*]chromene derivative **6** was characteristic by the presence of three singlet signals at



Scheme 2. Synthesis of 2-iminochromene derivatives **3**–**6**

δ 2.30, 2.36 and 3.96 ppm assignable for two methyl and one methoxy protons. The two singlet signals at δ 10.25 and 11.16 ppm were assigned to the two imino groups.

3. Conclusions

In this study, new functionalized coumarin derivatives bearing 2-iminochromene moiety were synthesized. The new coumarin derivatives were obtained in excellent yields and in high purity through ring closure of cyanoacetohydrazone derivative with 4-hydroxysalicylaldehyde, different derivatives of 5-aryldiazosalicylaldehyde, 2-hydroxy-naphthaldehyde and 7-hydroxychromone-6-carboxaldehyde derivative.

4. Experimental Section

Nuclear magnetic resonance spectra were carried out in deuterated dimethylsulfoxide (DMSO-d_6) by using Bruker spectrometers (^{1}H NMR 400 MHz) with chemical shift in δ from internal TMS. Elemental analyses were carried out on a EuroVector instrument C, H, N analyzer EA3000 Series.

Synthesis of 2-Cyano-*N*'-(1-(2-oxo-2*H*-chromen-3-yl) ethylidene)acetohydrazide (2)

The mixture of equimolar amounts of 3-acetylcoumarin (**1**) (1.88 g, 0.01 mol) and the cyanoacetic acid hydrazide (0.99 g, 0.01 mol) in the mixture of 1 mL AcOH and 50 mL ethanol was heated under reflux for 0.5 h, left to cool, the resultant solid product was collected by filtration. The solid product was crystallized from ethanol. Yield 2.152 g (80%); m.p. 175–176 °C; IR: ν/cm^{-1} 3181 (NH), 3082 (CH-arom.), 2962, 2922 (CH-aliph.), 2265 (C≡N), 1715, 1680 (C=O), 1619, 1604 (C=N); ^{1}H NMR: δ 2.26 (s, 3H, CH_3-C=N), 4.10 (s, 2H, CH_2), 7.40 (t, 1H, *J* = 7.5 Hz, C_6-H of chromene), 7.43 (d, 1H, *J* = 8.2 Hz, C_8-H of chromene), 7.65 (t, 1H, *J* = 7.8 Hz, C_7-H of chromene), 7.79 (dd, 1H, *J* = 7.7, 1.3 Hz, C_5-H of chromene), 8.36 (s, 1H, C_4-H of chromene), 11.44 (s, 1H, NH).

Synthesis of 7-Hydroxy-2-imino-*N*'-(1-(2-oxo-2*H*-chromen-3-yl)ethylidene)-2*H*-chromene-3-carbohydrazide (3)

To a hot solution of the mixture of equimolar amounts of cyanoacetamide **2** (269 mg, 1 mmol) and 4-hydroxysalicylaldehyde (138 mg, 1 mmol) in ethanol (100 mL), piperidine (8.5 mg, 0.1 mmol) was added. The reaction mixture was heated under reflux for 0.5 h. The solid product formed while hot was collected by filtration and recrystallized from dioxane to give **3**. Yield 350 mg (90%); m.p. 273–275°C; IR: ν/cm^{-1} 3327 (NH), 3070 (CH-arom.), 2957 (CH-aliph.), 1719, 1694, 1640 (C=O); ^{1}H NMR: δ 2.27 (s, 3H, CH_3), 7.27–7.52 (m, 3H, Ar-H), 7.57–7.73 (m, 2H, Ar-H), 7.80–7.96 (m, 2H, Ar-H), 8.29 (s, 1H, C_4-H of chromene), 8.64 (s, 1H, C_4-H of chromene), 9.32 (s, 1H, NH), 10.50 (br, 1H, OH), 13.69 (s, 1H, NH); MS: *m/z* (%) 389 (M^+; 52.5). Anal. calcd for $C_{21}H_{15}N_3O_5$ (389.36): C, 64.78; H, 3.88; N, 10.79. Found: C, 64.82; H, 3.86; N, 10.83%.

Synthesis of 2-Imino-*N*'-(1-(2-oxo-2*H*-chromen-3-yl) ethylidene)-6-(aryldiazenyl)-2*H*-chromene-3-carbohydrazides 4a–f

To a hot solution of the mixture of equimolar amounts of cyanoacetamide **2** (269 mg, 1 mmol) and 5-aryldiazenylsalicyaldehyde (namely, 5-((phenyl)diazenyl)salicyaldehyde (226 mg, 1 mmol), 5-((4-fluorophenyl) diazenyl)salicyaldehyde (244 mg, 1 mmol), 5-((4-bromophenyl)diazenyl)salicyaldehyde (305 mg, 1 mmol), 5-((4-methylphenyl)diazenyl)salicyaldehyde (240 mg, 1 mmol), 5-((4-methoxyphenyl)diazenyl)salicyaldehyde (256 mg, 1 mmol) and 5-((4-nitrophenyl)diazenyl)salicyaldehyde (271 mg, 1 mmol)) in ethanol (100 mL), piperidine (8.5 mg, 0.1 mmol) was added. The reaction mixture was heated under reflux for 0.5 h. The solid product formed while hot was collected by filtration and recrystallized from dioxane to give products **4a–f**.

2-Imino-*N*'-(1-(2-oxo-2*H*-chromen-3-yl)ethylidene) -6-(phenyldiazenyl)-2*H*-chromene-3-carbohydrazide (4a): Yield 453 mg (95%); m.p. 208–210 °C; IR: ν/cm^{-1} 3327 (NH), 3041 (CH-arom.), 2963 (CH-aliph.), 1694, 1641 (C=O), 1607 (C=N); ^{1}H NMR: δ 2.27 (s, 3H, CH_3), 7.33–8.46 (m, 12H, Ar-H), 8.78 (s, 1H, C_4-H of chromene), 9.10 (s, 1H, C_4-H of chromene), 9.54 (s, 1H, NH), 13.59 (s, 1H, NH); MS: *m/z* (%) 477 (M^+; 55.4). Anal. calcd for $C_{27}H_{19}N_5O_4$ (477.47): C, 67.92; H, 4.01; N, 14.67. Found: C, 67.87; H, 3.99; N, 14.72%.

6-((4-Fluorophenyl)diazenyl)-2-imino-*N*'-(1-(2-oxo-2*H*-chromen-3-yl)ethylidene)-2*H*-chromene-3-carbohydrazide (4b): Yield 446 mg (90%); m.p. 193–195 °C; IR: ν/cm^{-1} 3324 (NH), 3007 (CH-arom.), 1723, 1702 (C=O); ^{1}H NMR: δ 2.27 (s, 3H, CH_3), 7.38–8.45 (m, 11H, Ar-H), 8.78 (s, 1H, C_4-H of chromene), 9.09 (s, 1H, C_4-H of chromene), 9.54 (s, 1H, NH), 13.59 (s, 1H, NH); MS: *m/z* (%) 495 (M^+; 31.2). Anal. calcd for $C_{27}H_{18}FN_5O_4$ (495.46): C, 65.45; H, 3.66; N, 14.14. Found: C, 65.41; H, 3.65; N, 14.11%.

6-((4-Bromophenyl)diazenyl)-2-imino-*N*'-(1-(2-oxo-2*H*-chromen-3-yl)ethylidene)-2*H*-chromene-3-carbohydrazide (4c): Yield 528 mg (95%); m.p. 199–201 °C; IR: ν/cm^{-1} 3275 (NH), 3045 (CH-arom.), 1703 (C=O), 1607 (C=N); ^{1}H NMR: δ 2.26 (s, 3H, CH_3), 6.88 (t, 1H, *J* = 7.3 Hz, Ar-H), 6.97 (d, 1H, *J* = 8.1 Hz, Ar-H), 7.30–7.48 (m, 2H, Ar-H), 7.70 (d, 1H, *J* = 8.9 Hz, Ar-H), 7.74–7.94 (m, 5H, Ar-H), 8.27 (d, 1H, *J* = 8.8 Hz, Ar-H), 8.34 (s,1H, C_4-H of chromene), 9.07 (s, 1H, C_4-H of chromene), 11.16 (s,

1H, NH), 13.57 (s, 1H, NH); MS: *m/z* (%) 556.37 (M^+; 28.7). Anal. calcd for $C_{27}H_{18}BrN_5O_4$ (556.37): C, 58.29; H, 3.26; N, 12.59. Found: C, 58.32; H, 3.25; N, 12.63%.

2-Imino-*N*'-(1-(2-oxo-2*H*-chromen-3-yl)ethylidene)-6-(*para*-tolyldiazenyl)-2*H*-chromene-3-carbohydrazide (4d): Yield 441 mg (90%); m.p. 205–206 °C; IR: ν/cm^{-1} 3318 (NH), 3047 (CH-arom.), 1705 (C=O); ^{1}H NMR: δ 2.29 (s, 3H, CH_3), 2.43 (s, 3H, CH_3), 7.35–8.10 (m, 10H, Ar-H), 8.30 (s, 1H, C_4-H of chromene), 8.44 (s, 1H, Ar-H), 8.76 (s, 1H, C_4-H of chromene), 9.52 (s, 1H, NH), 13.58 (s, 1H, NH); MS: *m/z* (%) 491.50 (M^+; 42.2). Anal. calcd for $C_{28}H_{21}N_5O_4$ (491.50): C, 68.42; H, 4.31; N, 14.25. Found: C, 68.37; H, 4.30; N, 14.31%.

2-Imino-6-((4-methoxyphenyl)diazenyl)-*N*'-(1-(2-oxo-2*H*-chromen-3-yl)ethylidene)-2*H*-chromene-3-carbohydrazide (4e): Yield 464 mg (90%); m.p. 214–215 °C; IR: ν/cm^{-1} 3327 (NH), 3070, 3035 (CH-arom.), 2954 (CH-aliph.), 1719, 1693 (C=O); ^{1}H NMR: δ 2.29 (s, 3H, CH_3), 3.85 (s, 3H, OCH_3), 7.35–8.10 (m, 10H, Ar-H), 8.30 (s, 1H, Ar-H), 8.44 (s, 1H, Ar-H), 8.76 (s, 1H, C_4-H of chromene), 9.52 (s, 1H, NH), 13.58 (s, 1H, NH); MS: *m/z* (%) 507 (M^+; 33.8). Anal. calcd for $C_{28}H_{21}N_5O$ (507.50): C, 66.27; H, 4.17; N, 13.80. Found: C, 66.32; H, 4.16; N, 13.78%.

2-Imino-6-((4-nitrophenyl)diazenyl)-*N*'-(1-(2-oxo-2H-chromen-3-yl)ethylidene)-2*H*-chromene-3-carbohydrazide (4f): Yield 496 g (95%); m.p. 217–219 °C; IR: ν/cm^{-1} 3307 (NH), 3062 (CH-arom.), 1682 (C=O), 1606 (C=N); MS: *m/z* (%) 522 (M^+; 62.3). Anal. calcd for $C_{27}H_{18}N_6O_6$ (522.47): C, 62.07; H, 3.47; N, 16.09. Found: C, 62.12; H, 3.46; N, 16.11%.

Synthesis of 3-Imino-*N*'-(1-(2-oxo-2*H*-chromen-3-yl) ethylidene)-3*H*-benzo[*f*]chromene-2-carbohydrazide (5)

To a hot solution of the mixture of equimolar amounts of cyanoacetamide **2** (269 mg, 0.01 mol) and 2-hydroxy-1-naphthaldehyde (172 mg, 0.01 mol) in ethanol (100 mL), piperidine (8.5 mg, 0.1 mmol) was added. The reaction mixture was heated under reflux for 0.5 h. The solid product formed while hot was collected by filtration and recrystallized from dioxane to give **5**. Yield 402 mg (95%); m.p. 240–242 °C; IR: ν/cm^{-1} 3306 (NH), 2908 (CH-aliph.), 1713, 1687 (C=O); ^{1}H NMR: δ 2.26 (s, 3H, CH_3), 7.21–8.74 (m, 11H, Ar-H), 9.81 (s,1H, C_4-H of chromene), 11.16 (s, 1H, NH), 13.74 (s, 1H, NH); MS: *m/z* (%) 423.42 (M^+; 55.2). Anal. calcd for $C_{25}H_{17}N_3O_4$ (423.42): C, 70.91; H, 4.05; N, 9.92. Found: C, 70.87; H, 4.04; N, 9.88%.

Synthesis of 2-Imino-5-methoxy-8-methyl-6-oxo-*N*'-(1-(2-oxo-2*H*-chromen-3-yl)ethylidene)-2,6-dihydropyrano[3,2-*g*]chromene-3-carbohydrazide (6)

To a hot solution of the mixture of equimolar amounts of cyanoacetamide **2** (269 mg, 1 mmol) and 7-hydroxy-5-methoxy-2-methyl-4-oxo-4*H*-chromone-6-carboxaldehyde (204 mg, 1 mmol) in ethanol (100 mL), piperidine (8.5 mg, 0.1 mmol) was added. The reaction mixture was heated under reflux for 0.5 h. The solid product formed while hot was collected by filtration and recrystallized from dioxane to give **6**. Yield 446 mg (98%); m.p. 243–245 °C; IR: ν/cm^{-1} 3327 (NH), 3009 (CH-arom.), 1699, 1664 (C=O), 1605 (C=N); ^{1}H NMR: δ 2.30 (s, 3H, CH_3), 2.36 (s, 3H, CH_3), 3.96 (s, 3H, OCH_3), 6.22 (s, 1H, Ar-H), 6.80–7.80 (m, 6H, Ar-H), 9.02 (s, 1H, C_4-H of chromene), 10.25 (s, 1H, NH); 11.16 (s, 1H, NH); MS: *m/z* (%) 485 (M^+; 46.5). Anal. calcd for $C_{26}H_{19}N_3O_7$ (485.44): C, 64.33; H, 3.95; N, 8.66. Found: C, 64.29; H, 3.94; N, 8.62%.

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Povzetek

Kumarin je terapevtsko zanimiva spojina, ki predstavlja izhodišče za pripravo mnogih pomembnih spojin z opaznimi učinki. V predstavljeni raziskavi sem sintetiziral nove 2-iminokromenske derivate, ki vsebujejo kumarinski fragment. Najprej sem kot izhodno spojino pripravil cianoacetohidrazon iz 3-acetilkumarina. 2-Iminokromenske derivate sem pripravil s sintezo s pomočjo ciklizacije med cianoacetohidrazonskimi derivati in ustreznimi aldehidi (4-hidroksisalicilaldehid, 5-arildiazosalicilaldehid, 2-hidroksinaftaldehid in 7-hidroksikromon-6-karboksaldehid). Vsi novi pripravljeni kumarinski derivati so bili izolirani v visokih izkoristkih. Novi kumarini tako predstavljajo obogatitev kemijskih knjižnic.