Visible Light-Induced Synthesis of Biscoumarin Analogs under Catalyst-Free Conditions

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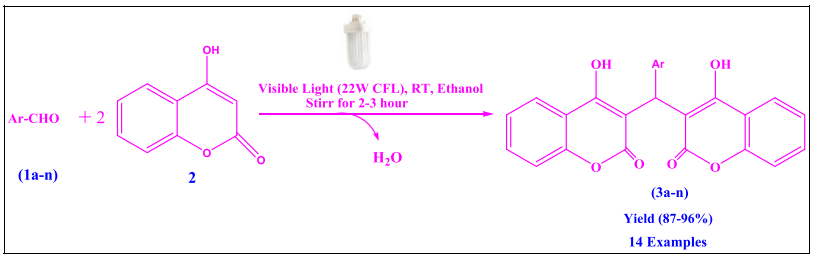

Biscoumarin analogs were synthesized by the reaction between two equivalent of 4-hydroxycoumarin and one equivalent of aryl aldehydes induced by visible light [22 W compact fluorescent lamp (CFL) bulb]. This new method is simple, cost-effective, and furnished excellent yields with broad substrate generality in short reaction time.

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INTRODUCTION

Because of environmental and societal interests, there has been a great demand for the development of the simple and environmental benign synthetic methods that minimizes the use of toxic reagents, catalysts, acids, alkali, and hazardous solvents [1]. The pursuit of greener and facile synthesis to prepare various organic compounds continues to be challenging. Among numerous green methods, the use of visible light [compact fluorescent lamp (CFL) bulb] has received much attention.

Biscoumarins are an important class that often display diverse biological activities [2,3] such as antitumor [4], anti-inflammatory [5], antioxidant [6], and cytotoxicity [7]. These compounds are widely used as ligands [8], reducing and stabilizing agent [9], dyes [10], etc. Up to date, many biscoumarin analogs have been discovered from the natural sources [11] including Dicoumarol (*Melilotus alba*), Gerberinol (*Gerbera lanuginose*), Ismailin (*Diospyros ismaili*), and Bisosthenon (*Citrus funadokoao*) (Fig. 1).

Many synthetic approaches have been reported for the synthesis of biscoumarins [12–15]. The most important protocols are from (i) benzylic alcohols and 4-hydroxycoumarin [16], (ii) 1,2-diols and 4-hydroxycoumarin [17], and (iii) benzaldehydes and 4-hydroxycoumarin [18]. The last method is of particular interest, and much development has been progressed.

In the last decades, use of various catalytic conditions has been established for the preparation of biscoumarins from 4-hydroxycoumarin and aryl aldehydes such as molecular iodine [19], $Zn(proline)_2$ [20], tetramethyl guanidium [21], phosphotungstic acid [22], nano silica chloride [23], propane-1,2,3-triyltris(hydrogen sulfate) [24], choline hydroxide [25], poly(4-vinylpyridine)-supported ionic liquids [26], ruthenium(III) chloride hydrate [27], sulfated titania [28], methane sulfonic acid [29], sodium dodecyl sulfate [30], ruthenium [31], nano TiO_2@KSF [32], silica-bonded *n*-propyl diethylene triamine sulfamic acid [33], and W-doped ZnO nanocomposite [34]. However, these methods suffer from the limitations such as use of expensive, toxic, greater loading of catalysts, incomplete conversion, and lower yields. Prompted by the previous points and our continuous efforts for preparing heterocyclic derivatives [35–37], we envisioned the catalyst-free synthesis of biscoumarin derivatives using visible light (CFL bulb) system at ambient temperature.

To date, no report has described the synthesis of biscoumarins under CFL light irradiation. CFL light intensity is harmless and an easily available energy source. CFL light intensity proved to be a very useful source in the synthesis of unsymmetrical diynes [38], cyclocondensation of pyrrole [39], photocatalytic generation of H_2 [40], carboxylation [41], synthesis of 1,2-diketones [42], trifluromethylated alkenes [43], diaryl disulfides [44], oxysulfonylation of alkenes [45], 3,4-dithiophenes [46], benzothiazoles [47], photochromic molecules [48], iron(III)-based metal organic frame works [49], and photocatalyst [50].

Therefore, the present synthesis of biscoumarins is novel, highly efficient, cost-effective, environmentally sustainable, and compatible for catalytic, biomedical, and industrial applications.

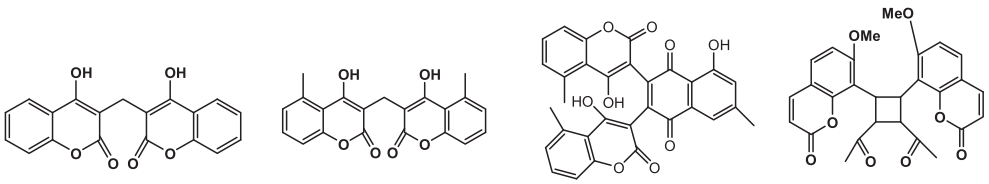


Figure 1. Some naturally occurring biscoumarins.

RESULTS AND DISCUSSION

In order to establish the optimal conditions for the synthesis of biscoumarins, 4-hydroxycoumarin (**2**) and benzaldehyde (**1g**) were chosen as a model substrate and allowed to react in the presence of visible light (20 W, CFL) intensity in DMF at room temperature for 4 h (Scheme 1). The presence of visible light intensity was crucial because, in its absence, the reaction did not proceed. The initial examination, to our delight, showed that CFL light intensity can catalyze the model reaction to afford the (**3g**) in 46 % yield.

A survey on intensities of light and solvent suggests that ethanol at 22 W is optimum because its reaction time and yields are better than those of others (Table 1). When the temperature was increased from room temperature to 78°C (reflux), the time and yield of the reaction was not improved. Therefore, room temperature, ethanol solvent, and 22 W CFL light intensity were selected as the optimal reaction condition for further experiments. When taking 2:1 molar ratio of 4-hydroxycoumarin (**2**) and benzaldehyde (**1g**), which are condensed under visible light intensity, the target compound (**3g**) was obtained in 95% yield.

Encouraged by the remarkable optimization results, we explored the generality and scope of the protocol by reacting 4-hydroxycoumarin with structurally diverse aldehydes (Table 2) under optimized conditions. The reaction effectively proceeded with both electron donating and electron withdrawing precursors. Interestingly, hetero aromatic aldehydes such as indole and thiophene were found to be equally efficient at yielding the expected products in excellent yields.

In accordance with previous literature reports [50–54], a plausible radical mechanism is proposed (Scheme 2). It is proposed that the aldehydes undergo Norrish-type

Scheme 1. Synthesis of biscoumarin (**3g**) under visible light. [Color figure can be viewed at wileyonlinelibrary.com]

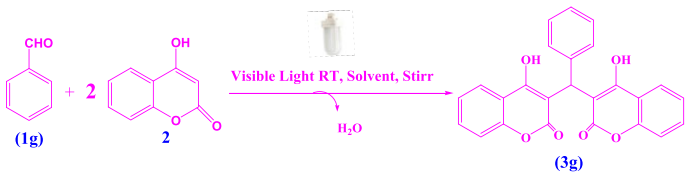


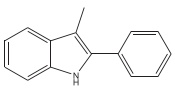
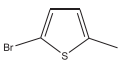
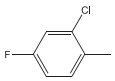
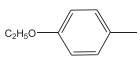
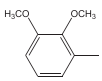
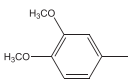
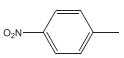
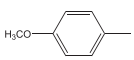
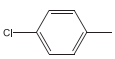
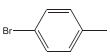
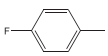
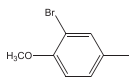
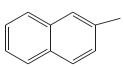
Table 1

Optimization of solvent and visible light intensity for the synthesis of (**3g**).

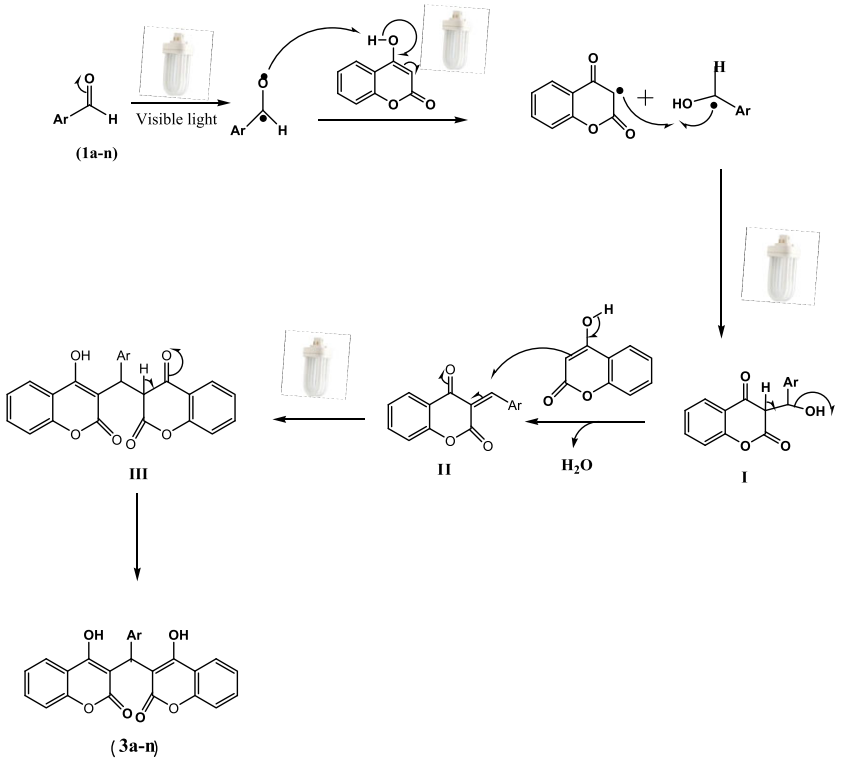
Entry	Solvent	Visible light intensity (W)	Time (h)	Yield (%)
1	CH_2Cl_2	20	5.5	41
2	Dioxane	20	6.5	78
3	Acetonitrile	20	4	67
4	Toluene	20	4.5	70
5	THF	20	5	72
6	Methanol	20	3.5	82
7	DMF	20	4.0	46
8	DMSO	20	5.5	52
9	Ethanol	20	2.3	88
10	**Ethanol**	22	2	**95**
11	Ethanol	15	3.1	77
12	Ethanol	8	4.2	69
13	Ethanol	32	2	95

Table 2

Visible light-induced synthesis of biscoumarin analogs under optimized condition.

Entry	Ar	Product	Time (h)	Yield (%)	Obs.Mp (°C)	Lit.Mp (°C)
1		**3a**	2	92	144–146	–
2		**3b**	2.2	91	199–201	–
3		**3c**	2.7	91	255–257	–
4		**3d**	2.2	90	212–214	–
5		**3e**	1.9	88	255–257	–
6		**3f**	2.8	87	245–247	264–266[27]
7		**3g**	2.3	95	228–230	230–231[12]
8		**3h**	2.5	96	234–236	232–233[12]
9		**3i**	2.6	89	250–252	251–252[12]
10		**3j**	2.3	92	259–261	259–260[12]
11		**3k**	2.5	96	265–267	266–268[12]
12		**3l**	2.6	89	215–217	214–216[12]
13		**3m**	2.5	91	242–244	241–243[12]
14		**3n**	2.8	90	264–266	263–265[12]

Scheme 2. Plausible free radical mechanism for the synthesis of biscoumarins. [Color figure can be viewed at wileyonlinelibrary.com]



cleavage and phenolic hydrogen of the 4-hydroxycoumarin breaks symmetrically to form the radical. These radicals further react to form the intermediates I, II, and III and finally gives the stable biscoumarin analogs. To verify whether the reaction proceeds through radical pathway or ionic pathway, the same model reaction was performed in the presence of hydroquinone, which is a radical inhibitor [55]. Only a trace amount of product (**3g**) was produced. This experiment clearly exhibited that the reaction proceeded via a radical pathway.

CONCLUSION

Eventually, we can conclude that visible light (CFL bulb, 22 W) can induce reaction between two equivalent of 4-hydroxy coumarin and one equivalent of aryl aldehydes to produce biscoumarin derivatives. The excellent yield, broad substrate profile, short reaction time, and catalyst free are the merits of this protocol.

GENERAL INFORMATION

The melting points were determined using electric melting point apparatus and are uncorrected. NMR spectra were recorded on a Jeol spectrometer (400 MHz for H^1-NMR and 100 MHz for C^{13}-NMR) using TMS as an internal standard in $CDCl_3$. Chemical shifts (δ) are measured in ppm. The progress of the reaction and purity of the compounds were checked by TLC. The elemental analyses were performed on Elemental Vario Micro Cube CHN Rapid Analyzer. Philips CFL bulbs are used as a visible light source.

General experimental procedure for the synthesis of biscoumarin derivatives (3a–n). A mixture of arylaldehydes (1 mmol), 4-hydroxycoumarin (2 mmol) A), and ethanol (10 mL) was taken in a 100-mL beaker. A glowing CFL bulb (22 W) was placed inside the beaker but just above (2-cm height) the reaction mixture. The reaction mixture was stirred at RT for the time shown in Table 2. The progress of the reaction was monitored by TLC (Eluent: EtOAc and *n*-hexane). The evaporation of solvent in vacuo afforded the crude product, which was recrystallized from ethanol to obtain biscoumarins**.**

SPECTRAL DETAILS

4-Hydroxy-3-((4-hydroxy-2-oxo-2*H*-chromen-3-yl)(2-phenyl-1*H*-indol-3-yl)methyl)-*2H*-chromen-2-one (3a). A purple solid; ^{1}H NMR ($CDCl_3$ 400 MHz): δ 5.82 (s, 1H, CH), 7.08–7.67 (m, 17H, Ar-H), 8.40 (s, 1H, OH), 8.42

(s, 1H, OH), 8.87 (s, 1H, NH) ppm. ^{13}C NMR ($CDCl_3$ 100 MHz): δ 36.0, 110.8, 111.0, 116.7, 122.3, 123.3, 123.5, 124.1, 124.5, 125.1, 129.0, 129.2, 129.5, 130.1, 132.6, 138.2, 144.0, 144.2, 144.5, 144.7, 155.7, 155.9, 165.7, 165.9 ppm. HRMS: [M + H] = *m*/*z* 528.6214; *Anal*. Calcd for $C_{33}H_{21}NO_6$: C, 75.13; H, 4.01; N, 2.66; found: C, 75.10; H, 4.00; N, 2.63%.

3-((5-Bromothiophen-2-yl)(4-hydroxy-2-oxo-*2H*-chromen-3-yl)methyl)-4-hydroxy-*2H*-chromen-2-one (3b). A light yellow solid; ^{1}H NMR ($CDCl_3$ 400 MHz): δ 6.09 (s, 1H, CH) 6.60–7.64 (m, 10H, Ar-H), 8.02 (s, 1H, OH), 8.04 (s, 1H, OH) ppm. ^{13}C NMR ($CDCl_3$ 100 MHz): δ 51.7, 110.7, 116.7, 124.4, 125.0, 125.7, 141.5, 147.0, 147.4, 148.2, 159.3, 159.6, 160.9, 164.9 ppm. HRMS: [M] = *m*/*z* 497.3352; *Anal*. Calcd for $C_{23}H_{13}BrO_6S$; C, 55.55; H, 2.63; found: C, 55.53; H, 2.62%.

3-((4-Fluoro-2-chlorophenyl)(4-hydroxy-2-oxo-*2H*-chromen-3-yl)methyl)-4-hydroxy-*2H*-chromen-2-one (3c). A white solid; ^{1}H NMR ($CDCl_3$ 400 MHz): δ 6.02 (s, 1H, CH) 6.82–7.64 (m, 11H, Ar-H), 7.99 (s, 1H, OH), 8.05 (s, 1H, OH) ppm. ^{13}C NMR ($CDCl_3$ 100 MHz): δ 55.2, 114.0, 116.6, 124.3, 124.8, 126.7, 127.6, 132.7, 146.9, 147.6, 152.2, 158.4, 163.9, 164.2 ppm. HRMS: [M + H] = *m*/*z* 464.1414; *Anal*. Calcd for $C_{25}H_{14}ClFO_6$: C, 64.60; H, 3.04; found: C, 64.59; H, 3.03%.

3-((4-Ethoxyphenyl)(4-hydroxy-2-oxo-*2H*-chromen-3-yl) methyl)-4-hydroxy-*2H*-chromen-2-one (3d). A white solid; ^{1}H NMR ($CDCl_3$ 400 MHz): δ 1.37 (t, 3H, CH_3) 3.98 (q, 2H, CH_2), 6.03 (s, 1H, CH), 6.82–7.63 (m, 12H, Ar-H), 8.00 (s, 1H, OH), 8.04 (s, 1H, OH) ppm. ^{13}C NMR ($CDCl_3$ 100 MHz): δ 19.0, 38.0, 63.4, 114.5, 116.6, 124.3, 124.8, 126.7, 127.5, 132.7, 139.9, 140.2, 140.5, 157.8, 163.9, 164.1 ppm. HRMS: [M + H] = *m*/*z* 457.0342; *Anal*. Calcd for $C_{27}H_{20}O_7$: C, 71.05; H, 4.42; found: C, 71.03; H, 4.40%.

4-Hydroxy-3-((4-hydroxy-2-oxo-*2H*-chromen-3-yl)(2,3-dimethoxyphenyl)methyl)-*2H*-chromen-2-one (3e). A white solid; ^{1}H NMR ($CDCl_3$ 400 MHz): δ 3.72 (s, 3H, OCH_3), 3.85 (s, 3H, OCH_3), 6.17 (s, 1H, CH), 6.86–7.61 (m, 11H, Ar -H), 8.00 (s, 1H, OH), 8.01 (s, 1H, OH) ppm. ^{13}C NMR ($CDCl_3$ 100 MHz): δ 33.4, 55.7, 59.9, 110.0, 110.2, 110.3, 110.7, 111.0, 111.7, 116.5, 116.8, 120.1, 123.5, 124.3, 124.7, 129.3, 132.4, 139.3, 139.3, 143.2, 143.4, 144.2, 147.2, 152.1, 159.1, 160.9, 163.8 ppm. HRMS: [M + H] = *m*/*z* 473.1945; *Anal*. Calcd for $C_{27}H_{20}O_8$: C, 68.64; H, 4.27; found: C, 68.60; H, 4.24%.

4-Hydroxy-3-((4-hydroxy-2-oxo-*2H*-chromen-3-yl)(3,4-dimethoxyphenyl)methyl)-*2H*-chromen-2-one (3f). A white solid; ^{1}H NMR ($CDCl_3$ 400 MHz): δ 3.72 (s, 3H, OCH_3), 3.85 (s, 3H, OCH_3), 6.06 (s, 1H, CH) 6.70–7.63 (m, 11H, Ar-H), 8.00 (s, 1H, OH), 8.05 (s, 1H, OH) ppm. ^{13}C NMR ($CDCl_3$ 100 MHz): δ 35.2, 55.7, 56.0, 110.3, 111.2, 116.6, 118.9, 124.3, 124.8, 127.5, 132.8, 139.2, 139.5, 139.7, 148.0, 149.1, 161.8, 162.1 ppm. HRMS: [M + H] = *m*/*z* 473.6214; *Anal*. Calcd for $C_{27}H_{20}O_8$: C, 68.64; H, 4.27; found: C, 68.60; H, 4.24%.

4-Hydroxy-3-((4-hydroxy-2-oxo-2*H*-chromen-3-yl)(phenyl) methyl)-*2H*-chromen-2-one (3g). A white solid; ^{1}H NMR ($CDCl_3$ 400 MHz): δ 6.09 (s, 1H, CH), 7.20–7.64 (m, 13H, Ar-H), 7.98 (s, 1H, OH), 8.05 (s, 1H, OH) ppm. ^{13}C NMR ($CDCl_3$ 100 MHz): δ 36.1, 105.6, 116.4, 116.6, 124.4, 124.8, 126.4, 126.8, 128.6, 128.9, 132.8, 135.1, 145.1, 162.8 ppm. HRMS: [M + H] = *m*/*z* 413.1645; *Anal*. Calcd for $C_{25}H_{16}O_6$: C, 72.81; H, 3.91; found: C, 72.76; H, 3.87%.

4-Hydroxy-3-((4-hydroxy-2-oxo-*2H*-chromen-3-yl)(4-nitrophenyl)methyl)-*2H*-chromen-2-one (3h). A light yellow solid; ^{1}H NMR ($CDCl_3$ 400 MHz): δ 6.10 (s, 1H, CH), 7.24–8.09 (m, 12H, Ar-H), 8.16 (s, 1H, OH), 8.18 (s, 1H, OH) ppm. ^{13}C NMR ($CDCl_3$ 100 MHz): δ 48.3, 116.2, 116.7, 116.8, 123.8, 124.4, 125.1, 125.2, 127.5, 133.3, 143.3, 146.9, 164.8, 169.1 ppm. HRMS: [M + H] = *m*/*z* 458.0342; *Anal*. Calcd for $C_{25}H_{15}NO_8$: C, 65.65; H, 3.31; N, 3.06; found: C, 65.60; H, 3.30; N, 3.29%.

4-Hydroxy-3-((4-hydroxy-2-oxo-*2H*-chromen-3-yl)(4-methoxyphenyl)methyl)-*2H*-chromen-2-one (3i). A white solid; ^{1}H NMR ($CDCl_3$ 400 MHz): δ 3.78 (s, 3H, OCH_3), 6.03 (s, 1H, CH), 6.82–7.63 (m, 12H, Ar-H), 7.99 (s, 1H, OH), 8.04 (s, 1H, OH) ppm. ^{13}C NMR ($CDCl_3$ 100 MHz): δ 35.5, 55.2, 114.0, 116.6, 124.3, 124.8, 126.9, 127.6, 132.7, 146.9, 147.6, 152.2, 158.4, 163.9, 164.2 ppm. HRMS: [M + H] = *m*/*z* 443.1645; *Anal*. Calcd for $C_{26}H_{18}O_7$: C, 70.58; H, 4.10; found: C, 70.55; H, 4.07%.

3-((4-Chlorophenyl)(4-hydroxy-2-oxo-*2H*-chromen-3-yl) methyl)-4-hydroxy-*2H*-chromen-2-one (3j). A white solid; ^{1}H NMR ($CDCl_3$ 400 MHz): δ 6.09 (s, 1H, CH), 7.20–7.64 (m, 12H, Ar-H), 7.98 (s, 1H, OH), 8.05 (s, 1H, OH) ppm. ^{13}C NMR ($CDCl_3$ 100 MHz): δ 55.2, 114.0, 116.6, 124.3, 124.8, 126.9, 127.6, 132.7, 147.0, 147.6, 152.2, 158.4, 163.9, 164.2 ppm. HRMS: [M] = *m*/*z* 460.0814. *Anal*. Calcd for $C_{25}H_{15}ClO_6$: C, 67.20; H, 3.38; found: C, 67.17; H, 3.87%.

3-((4-Bromophenyl)(4-hydroxy-2-oxo-*2H*-chromen-3-yl) methyl)-4-hydroxy-*2H*-chromen-2-one (3k). A white solid; ^{1}H NMR ($CDCl_3$ 400 MHz): δ 6.10 (s, 1H, CH), 7.24–8.09 (m, 12H, Ar-H), 8.16 (s, 1H, OH), 8.18 (s, 1H, OH) ppm. ^{13}C NMR ($CDCl_3$ 100 MHz): δ 55.2, 114.0, 116.6, 124.3, 124.8, 126.9, 127.6, 132.7, 147.0, 147.6, 152.2, 158.4, 163.9, 164.2 ppm. HRMS: [M] = *m*/*z* 504.0209. *Anal*. Calcd for $C_{25}H_{15}BrO_6$: C, 61.12; H, 3.08; found: C, 61.10; H, 3.06%.

3-((4-Fluorophenyl)(4-hydroxy-2-oxo-*2H*-chromen-3-yl) methyl)-4-hydroxy-*2H*-chromen-2-one (3l). A white solid; ^{1}H NMR ($CDCl_3$ 400 MHz): δ 6.02 (s, 1H, CH), 6.82–7.64 (m, 12H, Ar-H), 7.99 (s, 1H, OH), 8.04 (s, 1H, OH) ppm. ^{13}C NMR ($CDCl_3$ 100 MHz): δ 36.1, 105.7, 116.4, 116.6, 124.4, 124.8, 126.4, 126.8, 128.6, 128.9,

132.8, 135.1, 145.1, 162.9 ppm. HRMS: [M] = *m*/*z* 521.3130; *Anal*. Calcd for $C_{25}H_{15}FO_6$: C, 69.77; H, 3.51; found: C, 69.75; H, 3.50%.

3-((3-Bromo-4-methoxyphenyl)(4-hydroxy-2-oxo-2*H*-chromen-3-yl)methyl)-4-hydroxy-2*H*-chromen-2-one (3m). A white solid; ^{1}H NMR ($CDCl_3$ 400 MHz): δ 3.87 (s, 3H, OCH_3), 6.02 (s, 1H, CH) 6.82–7.64 (m, 11H, Ar-H), 7.99 (s, 1H, OH), 8.05 (s, 1H, OH) ppm. ^{13}C NMR ($CDCl_3$ 100 MHz): δ 36.1, 53.8, 105.6, 116.4, 116.6, 124.4, 124.8, 126.4, 126.8, 128.6, 128.9, 132.8, 135.1, 145.1, 162.8 ppm. HRMS: [M] = *m*/*z* 521.3130; *Anal*. Calcd for $C_{26}H_{17}BrO_{7:}$ C, 59.90; H, 3.29; found: C, 59.87; H, 3.28%.

4-Hydroxy-3-((4-hydroxy-2-oxo-*2H*-chromen-3-yl)(naphthalen-2-yl)methyl)-*2H*-chromen-2-one (3n). A white solid; ^{1}H NMR ($CDCl_3$ 400 MHz): δ 6.24 (s, 1H, CH), 7.24–8.00 (m, 15H, Ar-H), 8.02 (s, 1H, OH) 8.08 (s, 1H, OH) ppm. ^{13}C NMR ($CDCl_3$ 100 MHz): δ 36.4, 116.6, 124.4, 124.6, 124.9, 125.2, 125.8, 126.2, 127.5, 127.8, 128.4, 132.3, 132.6, 132.8, 133.3, 152.1, 152.9, 156.2, 160.9, 163.8 ppm. HRMS: [M + H] = *m*/*z* 463.6214; *Anal*. Calcd for $C_{29}H_{18}O_6$: C, 75.32; H, 3.92; found: C, 75.30; H, 3.89%.

CONFLICTS OF INTEREST

There are no conflicts of interest to declare.

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