Alkanedisulfamic Acid Functionalized Silica-Coated Magnetic Nanoparticles: Preparation and Catalytic Investigation in Synthesis of Mono-, Bis- and Tris[bis(4-hydroxycoumarinyl)methanes]

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Abstract: Alkanedisulfamic acid-functionalized silica-coated magnetic nanoparticles (ADSA-MNPs) were prepared by a simple method and evaluated as efficient catalysts for the preparation of mono-, bis-, and tris[bis(4-hydroxycoumarinyl)methanes] through condensation of 4-hydroxy-2*H*-coumarin-2-one with mono-, di-, or trialdehydes, respectively. The heterogeneous nanocatalyst was readily recovered from the reaction mixture by using external magnet and was reused five times without significant loss of catalytic activity.

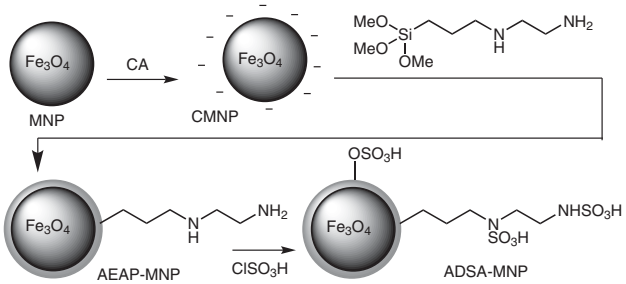
Key words: nanostructures, coumarins, heterogeneous catalysis, condensation, catalyst supports

Acid-functionalized silica-coated magnetic nanoparticles have recently emerged as attractive supports for immobilization of homogeneous catalysts. Various acid-functionalized silica-coated magnetic nanoparticles have been prepared and used as efficient catalysts for a range of organic transformations, such as deprotection of benzaldehyde dimethylacetals[1] and the synthesis of α-amino nitriles;[2] mono-, bis-, and tris[bis(6-aminopyrimidinyl)methanes];[3] quinolines;[4] benzo[*a*]xanthenone derivatives;[5] and aminoimidazopyridine skeletons.[6]

Coumarin derivatives have received considerable attention because of their useful pharmacological activities. 4-Hydroxycoumarin derivatives have attracted particular attention because of their useful biological and pharmacological properties, such as anticoagulant,[7] spasmolytic,[8] and rodenticidal activities.[9] Coumarins in general, and biscoumarins in particular, exhibit antifungal, anti-HIV, anticancer, antithrombotic, antimicrobial, antioxidant[10,11] urease-inhibitory,[12] cytotoxic, and enzyme inhibitory activities.[13] As a result, the synthesis of this class of compounds is very important.

The reaction of 4-hydroxycoumarin with aldehydes affords the corresponding bis(4-hydroxycoumarinyl)methanes, a useful class of organic compounds. This reaction is catalyzed by ruthenium(III) chloride hydrate,[14] piperidine,[12] sulfated titania,[15] iodine,[16] or a poly(4-vinylpyridinium butylsulfonate)-supported catalyst.[17] Although the known procedures for the synthesis of corresponding bis(4-hydroxycoumarinyl)methanes have their merits, they also suffer from some drawbacks, such as low yields, prolonged reaction times, the use of costly reagents or catalysts, or the use of toxic organic solvents. The recovery of the catalyst can also be a problem.

Although the reaction of 4-hydroxycoumarin with monoaldehydes has been investigated,[11–19] there have been no reports on reactions of 4-hydroxycoumarin with di- or trialdehydes for the synthesis of bis- and tris[bis(4-hydroxycoumarinyl)methanes], respectively. As a continuation of our research on the preparation and catalytic investigation of new acid-functionalized magnetic nanoparticles,[3,20] we synthesized new alkanedisulfamic acid magnetic nanoparticles (ADSA-MNPs) by direct reaction of chlorosulfonic acid with diaminoalkylsilica-coated magnetic nanoparticles (Scheme 1), and we used these nanoparticles as a catalyst system for the synthesis of mono-, bis-, and tris[bis(4-hydroxycoumarinyl)methanes] by condensation of 4-hydroxycoumarin with mono-, di-, or trialdehydes, respectively.



Scheme 1 Preparation of alkanedisulfamic acid functionalized silica-coated magnetic nanoparticles (ADSA-MNPs)

To prepare the ADSA-MNPs, magnetic iron(III) oxide nanoparticles were treated with citric acid to give the corresponding charged nanoparticles (CMNPs). The CMNPs were then treated with *N*-[3-(triethoxysilyl)propyl]ethane-1,2-diamine (AEAPS) to give coated magnetic nanoparticles (AEAP-MNPs), which were functionalized by a simple one-step procedure through direct treatment with chlorosulfonic acid to give the ADSA-MNPs. The size of the nanoparticles was determined by scanning electron microscopy (SEM). The SEM micrographs (Figure 1) showed that the ADSA-MNPs were spherical in shape and that their average size was about 38 nm.

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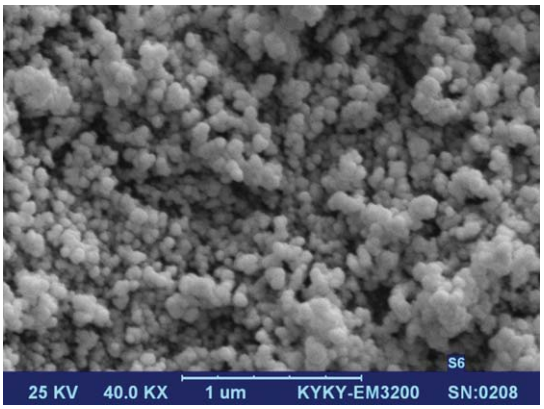


Figure 1 SEM micrograph of ADSA-MNPs

The thermal properties of ADSA-MNPs were analyzed by thermal gravimetric analysis at 20–850 °C under nitrogen. The primary weight loss at up to 160 °C was related to the removal of physically adsorbed solvent. The rate of weight loss between 160 and 500 °C was relatively slow, showing that the ASDA-MNPs have a reasonably high thermal stability at up to 500 °C. The maximum rate of weight loss for these nanoparticles began at 500 °C. There was a well-defined mass loss of 50% at between 160 and 700 °C, related to the breakdown of the sulfuric acid and alkylamine moieties.

The x-ray diffraction patterns of the ADSA-MNPs showed that the cubic structure of the magnetite was well preserved after introduction of the AEAPS and sulfuric acid functionality.[22] The intensity of the 41.6° reflection of the ADSA-MNPs decreased after introduction of the sulfuric acid group.

To explore the use of ADSA-MNPs as a catalyst system, we initially examined the preparation of 3,3′-[(3-chlorophenyl)methylene]bis(4-hydroxy-2*H*-chromen-2-one) (**3a**) by reaction of 3-chlorobenzaldehyde (**2a**; 1 mmol) with 4-hydroxy-2*H*-coumarin-2-one (**1**; 2 mmol) under various conditions (Table 1).

We began by investigating the effects of various amounts of ADSA-MNPs as catalyst in ethanol at 60 ºC under ultrasound irradiation. After 1 h, with 0, 0.03, 0.06, 0.1, and 0.15 g of ADSA-MNPs, yields of 10, 45, 75, 95, and 81%, respectively, of **3a** were obtained (Table 1, entries 1–5). The solvents were also found to play an important role in this reaction. The use of ethanol, water, acetonitrile, *N*,*N*-dimethylformamide, tetrahydrofuran, or dimethyl sulfoxide as the solvent gave poor yields (entries 6–11); the reaction hardly proceeded in tetrahydrofuran. However, the reaction in 4:1 ethanol–water afforded the product in high yield with nearly complete conversion (entry 4). We therefore selected 4:1 ethanol–water as the solvent for subsequent investigations. The use of MNPs, CMNPs, or AEAP-MNPs as heterogeneous catalysts or sulfuric acid as a homogeneous catalyst gave low yields of the product (Table 1, entries 12–16). The best reaction conditions were therefore obtained by using 0.1 g of ADSA-MNPs in 4:1 ethanol–water at 60 °C with ultrasound irradiation.

Table 1 Optimization of Reaction for the Synthesis of Bis(4-hydroxy-2*H*-chromen-2-one) (**3a**) with Ultrasound Irradiation at 60 °C[a]

Entry	Catalyst	Solvent	Yield (%)
1	–	4:1 EtOH–H_2O	10
2	ADSA-MNPs (0.03 g)	4:1 EtOH–H_2O	45
3	ADSA-MNPs (0.06 g)	4:1 EtOH–H_2O	75
4	ADSA-MNPs (0.10 g)	4:1 EtOH–H_2O	95
5	ADSA-MNPs (0.15 g)	4:1 EtOH–H_2O	81
6	ADSA-MNPs (0.10 g)	EtOH	75
7	ADSA-MNPs (0.10 g)	H_2O	30
8	ADSA-MNPs (0.10 g)	MeCN	85
9	ADSA-MNPs (0.10 g)	DMF	55
10	ADSA-MNPs (0.10 g)	THF	20
11	ADSA-MNPs (0.10 g)	DMSO	40
12	MNPs (0.10 g)	4:1 EtOH–H_2O	20
13	CMNPs (0.10 g)	4:1 EtOH–H_2O	22
14	AEAP-MNPs (0.10 g)	4:1 EtOH–H_2O	38
15	H_2SO_4 (0.03 g)	4:1 EtOH–H_2O	80
16	H_2SO_4 (0.05 g)	4:1 EtOH–H_2O	70

[a] Reaction conditions: 3-ClC_6H_4CHO (**2a**; 1 mmol), 4-hydroxy-2*H*-coumarin-2-one (**1a**; 2 mmol), catalyst, solvent, 1 h, ultrasound.

Table 2 Synthesis of Mono-, Bis-, and Tris[bis(4-hydroxycoumarinyl)methanes] Catalyzed by ADSA-MNPs at 60 °C with Ultrasound Irradiation

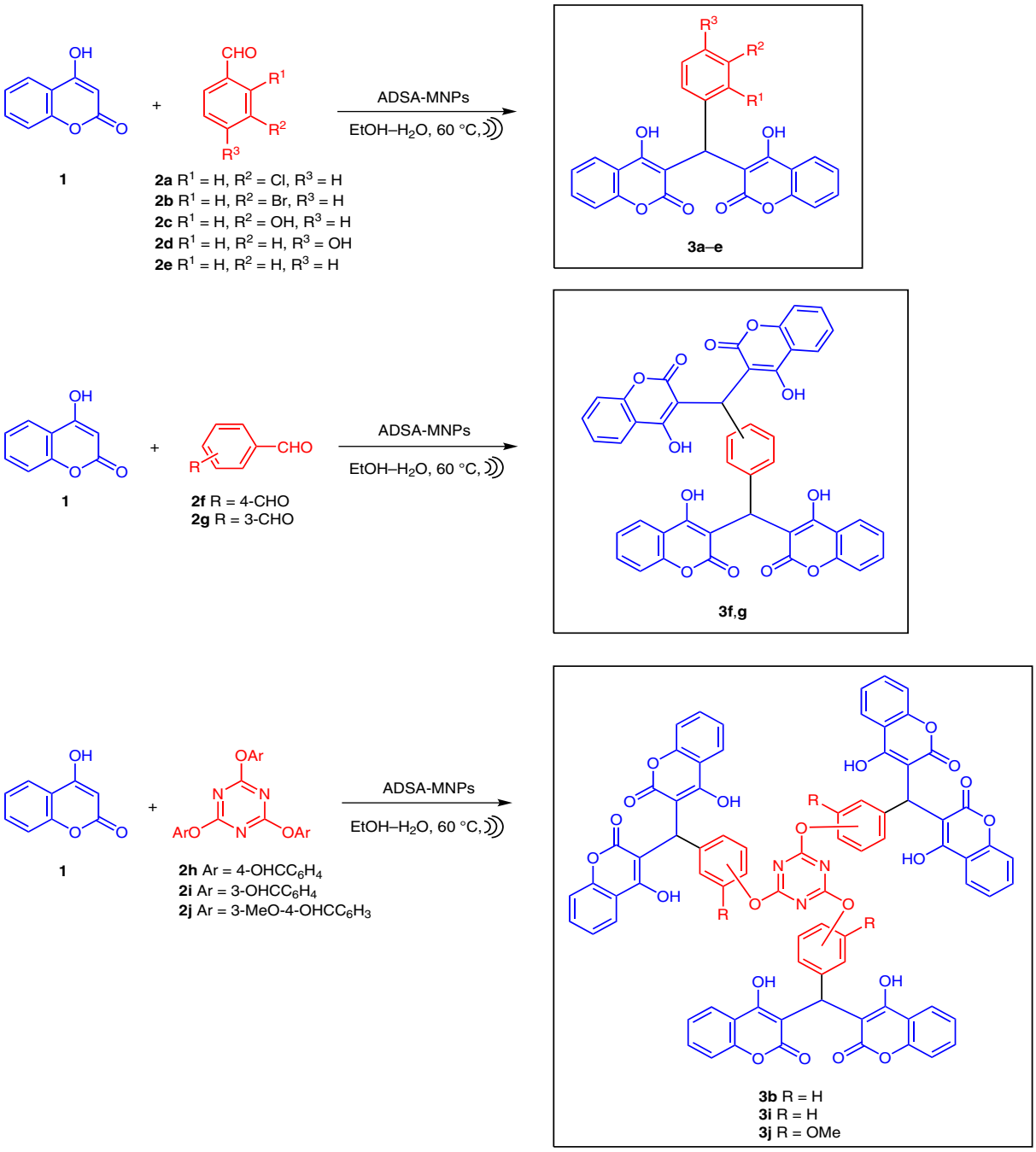
Entry	Aldehyde	Product	Time (h)	Yield (%)	Mp (°C)	Mp (Lit.[12])
1	**2a**	**3a**	1	92[a]	214–216	215
2	**2b**	**3b**	1	95	225–227	–
3	**2c**	**3c**	1	92	212–214	210.5
4	**2d**	**3d**	1	88	200–203	193
5	**2e**	**3e**	1	93	226–228	228
6	**2f**	**3f**	3	83	>300	–
7	**2g**	**3g**	3	80	293–295	–
8	**2h**	**3h**	4	88[b]	255–257	–
9	**2i**	**3i**	4	85	260–262	–
10	**2j**	**3j**	4	83	>300	–

[a] Yields after successive recoveries and recycling of catalyst: 90, 88, 85, and 85%.
[b] Yields for H_2SO_4 catalyst 50% (0.03 g) and 55% (0.06 g).

Next, we examined the reaction of a wide variety of monoaldehydes **2a–e**, dialdehydes **2f** and **2g**, and trialdehydes **2h–j** to establish the scope of this catalytic transformation (Scheme 2).

The mono-, di- and tri-[bis(4-hydroxycoumarinyl) methanes] synthesized by our method are listed in Table 2. ADSA-MNPs as catalyst showed a high activity and could be recovered and recycled four times without significant loss of activity (Table 2, entry 1). The use of sulfuric acid as catalyst in the synthesis of **3h** favored side reactions and gave a low yield of the product (entry 8).

In conclusion, we prepared ADSA-MNPs and examined their catalytic activity. The results showed that this catalyst system has some advantages. ADSA-MNPs were prepared in a simple manner by direct functionalization of AEAP-MNPs with chlorosulfonic acid. The ADSA-MNPs can be dispersed in solvents and can be isolated after use by using a magnet. In condensation reactions of 4-hydroxy-2*H*-coumarin-2-one (**1a**) with aldehydes **2**, ADSA-MNPs showed short reaction times coupled with a simple reaction procedure. The inexpensive and reusable catalyst make this method one of the most efficient methods for the synthesis of mono-, bis-, and tris[(4-hydroxycoumarinyl)methanes].



Scheme 2 Synthesis of mono-, bis- and tris[bis(4-hydroxycoumarinyl)methanes] **3a–i** catalyzed by ADSA-MNPs

4-Hydroxy-2*H*-coumarin-2-one (**1**), monoaldehydes **2a**–**e**, terephthalaldehyde (**2f**), isophthalaldehyde (**2g**), $FeCl_3$, AEAPS, cyanuric chloride, and the various solvents were used without further purification. Fe_3O_4 nanoparticles (MNP) and citric acid-modified nanoparticles (CMNPs) were prepared according to the procedure described in the literature.[23,24] Trialdehydes **2h**–**j** were also prepared according to a procedure described in the literature.[25] Sonication was performed in a Struers Metason 200 HT ultrasonic cleaner at a frequency of 50–60 kHz and an output power of 140 W. The products were characterized by elemental analysis and by IR, ^{1}H NMR, and ^{13}C NMR spectroscopy. FTIR spectra were recorded by using a Unicom Galaxy Series FTIR 5000 spectrophotometer. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance 300 MHz spectrometer at 300 and 75 MHz, respectively. Mass spectra were recorded on a Shimadzu QP 1100 EX mass spectrometer operated in the EI mode. Elemental analyses were performed by using Vario EL III elemental analyzer.

{[*N*-(2-Aminoethyl)-3-amino]propyl}silica-Coated Magnetic Nanoparticles (AEAP-MNPs)

CMNPs (1.5 g) were dispersed in EtOH–H_2O (1:1; 250 mL) and sonicated for 30 min to ensure dispersion. AEAPS (2.5 mL) was added with mechanical stirring, and the mixture was agitated at 40 °C for 4 h. The black precipitate was isolated by magnetic decantation, washed with deionized H_2O and EtOH, and dried at r.t.

Alkanedisulfamic Acid-Functionalized Silica-Coated Magnetic Nanoparticles (ADSA-MNPs)

The prepared AEAP-MNPs were placed in a two-necked flask equipped with a constant-pressure dropping funnel and a tube for removing the HCl gas that formed by conducting it to an adsorbent solution. $ClSO_3H$ (1.5 mL) was added dropwise over 30 min at r.t., and the mixture was subjected to slow mechanical stirring. HCl gas immediately evolved from the reaction vessel. The mixture was then shaken well for 30 min. The ADSA-MNPs were washed with acetone and distilled water to remove excess $ClSO_3H$ and then dried in an oven at 60 °C for 6 h.

Mono-, Bis-, and Tris[bis(4-hydroxycoumarinyl)methanes] (3a–j); General Procedure

A mixture of monoaldehyde **2a**–**e**, dialdehyde **2f**–**g**, or trialdehyde **2h**–**j** (1 mmol), 4-hydroxy-2*H*-coumarin-2-one **1** (2.2 mmol for **2a**–**e**, 5 mmol for **2f** and **2g**, or 7 mmol for **2h**–**j**) and ADSA-MNPs (0.1 g for **2a**–**e**, 0.12 g for **2f** or **2g**, or 0.15 g for **2h**–**j**) in 4:1 EtOH–H_2O (10 mL) was exposed to ultrasound irradiation at 60 °C for the appropriate time (see Table 2). When the reaction was complete (TLC), the ADSA-MNPs were removed by using an applied external magnetic field. The solution was concentrated then left to evaporate slowly. An EtOH–H_2O (5:1) mixture was added, and the resulting solid product was collected by filtration and washed with EtOH–H_2O (5:1) to remove excess coumarin **1**.

3,3′-[(3-Chlorophenyl)methylene]bis(4-hydroxy-2*H*-chromen-2-one) (3a)[12]

White solid; yield: 0.41 g (92%); mp 214–216 °C (Lit.[12] 215 °C).

IR (KBr): 3272, 2889, 2825, 1668, 1620, 1562, 1489, 1450, 906 cm^{-1}.

Anal. Calcd for $C_{25}H_{15}ClO_6$: C, 67.20; H, 3.38. Found: C, 67.41; H, 3.52.

3,3′-[(3-Bromophenyl)methylene]bis(4-hydroxy-2*H*-chromen-2-one) (3b)

White solid; yield: 0.465 g (95%); mp 225–227 °C.

IR (KBr): 3267, 2892, 2804, 1662, 1606, 1564, 1452, 910 cm^{-1}.

^{1}H NMR (300 MHz, DMSO-d_6): δ = 5.43 (br s, 2 H, OH), 6.31 (s, 1 H, $CH_{methine}$), 7.17 (q, *J* = 2.0 Hz, 2 H, H_{arom}), 7.27–7.35 (m, 6 H, H_{arom}), 7.59 (t, *J* = 2.0 Hz, 2 H, H_{arom}), 7.9 (d, *J* = 2.0 Hz, 2 H, H_{arom}).

Anal. Calcd for $C_{25}H_{15}BrO_6$: C, 61.12; H, 3.08. Found: C, 61.45; H, 3.42.

3,3′-[(3-Hydroxyphenyl)methylene]bis(4-hydroxy-2*H*-chromen-2-one) (3c)[12]

White solid; yield: 0.393 g (92%); mp 212–214 °C (Lit.[12] 210.5 °C).

IR (KBr): 3200, 2870, 2800, 1666, 1620, 1560, 1502, 1478 cm^{-1}.

^{1}H NMR (300 MHz, DMSO-d_6): δ = 5.45 (br s, 3 H, OH), 6.28 (s, 1 H, $CH_{methine}$), 7.13–7.15 (m, 2 H, H_{arom}), 7.24–7.33 (m, 6 H, H_{arom}), 7.57 (t, *J* = 2.0 Hz, 2 H, H_{arom}), 7.85 (d, *J* = 2.0 Hz, 2 H, H_{arom}).

3,3′-[(4-Hydroxyphenyl)methylene]bis(4-hydroxy-2*H*-chromen-2-one) (3d)[12]

White solid; yield: 0.376 g (88%); mp 200–203 °C (Lit.[12] 193 °C).

IR (KBr): 3272, 2860, 2801, 1662, 1614, 1580, 1452, 954 cm^{-1}.

Anal. Calcd for $C_{25}H_{16}O_7$: C, 70.09; H, 3.76. Found: C, 70.42; H, 4.05.

3,3′-(Phenylmethylene)bis(4-hydroxy-2*H*-chromen-2-one) (3e)[12]

White solid; yield: 0.383 g (93%); mp 226–228 °C (Lit.[12] 228 °C).

IR (KBr): 3228, 2885, 2807, 1672, 1604, 1560, 1491, 748 cm^{-1}.

Anal. Calcd for $C_{25}H_{16}O_6$: C, 72.81; H, 3.91. Found: C, 73.12; H, 4.26.

3,3′,3″,3‴-[1,4-Phenylenedi(methanetriyl)]tetrakis(4-hydroxy-2*H*-chromen-2-one) (3f)

White solid; yield: 0.619 g (83%); mp >300 °C.

IR (KBr): 3274, 2889, 2857, 1660, 1618, 1586, 1520, 1483 cm^{-1}.

^{1}H NMR (300 MHz, DMSO-d_6): δ = 5.65 (br s, 4 H, OH), 6.36 (s, 2 H, $CH_{methine}$), 7.07 (s, 4 H, H_{arom}) 7.34–7.41 (m, 8 H, H_{arom}), 7.63 (t, *J* = 2.0 Hz, 4 H, H_{arom}), 7.94 (d, *J* = 2.0 Hz, 4 H, H_{arom}).

MS (EI): *m/z* = 746 [M^+].

Anal. Calcd for $C_{44}H_{26}O_{12}$: C, 70.78; H, 3.51. Found: C, 71.04; H, 3.86.

3,3′,3″,3‴-[1,3-Phenylenedi(methanetriyl)]tetrakis(4-hydroxy-2*H*-chromen-2-one) (3g)

White solid; yield: 0.597 g (80%); mp 293–295 °C.

IR (KBr): 3280, 2982, 2800, 1660, 1620, 1602, 1566, 1494, 914 cm^{-1}.

^{1}H NMR (300 MHz, DMSO-d_6): δ = 4.46 (br s, 4 H, OH), 6.27 (s, 2 H, $CH_{methine}$), 6.92–6.94 (m, 3 H, H_{arom}), 7.09 (t, *J* = 2.0 Hz, 1 H, H_{arom}), 7.19–7.23 (m, 8 H, *J* = 2.0 Hz, H_{arom}), 7.53 (t, *J* = 2.0 Hz, 4 H, H_{arom}), 7.67 (d, *J* = 1.8 Hz, 4 H, H_{arom}).

^{13}C NMR (75 MHz, DMSO-d_6): δ = 35.78, 115.78, 116.34, 117.22, 118.81, 123.57, 123.73, 125.11, 127.99, 128.79, 131.82, 132.69, 138.88, 151.85, 164.75.

Anal. Calcd for $C_{44}H_{26}O_{12}$: C, 70.78; H, 3.51. Found: C, 71.11; H, 3.89.

3,3′,3″,3‴,3⁗,3⁗′-[1,3,5-Triazine-2,4,6-triyltris(oxy-4,1-phenylenemethanetriyl)]hexakis(4-hydroxy-2*H*-chromen-2-one) (3h)

White solid; yield: 1.19 g (88%); mp 255–257 °C.

IR (KBr): 3270, 2980, 2878, 1660, 1620, 1562, 1475, 910 cm^{-1}.

^{1}H NMR (300 MHz, DMSO-d_6): δ = 4.80 (br s, 6 H, OH), 6.33 (s, 3 H, $CH_{methine}$), 6.92–7.03 (m, 9 H, H_{arom}), 7.24–7.34 (m, 15 H, H_{arom}), 7.58 (t, *J* = 1.8 Hz, 6 H, H_{arom}), 7.86 (d, *J* = 1.7 Hz, 6 H, H_{arom}).

^{13}C NMR (75 MHz, DMSO-d_6): δ = 35.94, 115.67, 116.35, 118.58, 123.17, 123.93, 128.78, 130.51, 131.41, 132.71, 140.56, 142.59, 144.59, 152.353, 164.59.

Anal. Calcd for $C_{78}H_{45}N_3O_{21}$: C, 68.87; H, 3.33; N, 3.09. Found: C, 69.09; H, 3.65; N, 3.41.

3,3′,3″,3‴,3⁗,3⁗′-[1,3,5-Triazine-2,4,6-triyltris(oxy-3,1-phenylenemethanetriyl)]hexakis(4-hydroxy-2*H*-chromen-2-one) (3i)
White solid; yield: 1.15 g (85%); mp 260–262 °C.

IR (KBr): 3378, 2933, 2887, 1664, 1618, 1566, 1487, 950 cm^{-1}.

^1H NMR (300 MHz, DMSO-d_6): δ = 5.35 (br s, 6 H, OH), 6.33 (s, 3 H, $CH_{methine}$), 7.07–7.09 (m, 6 H, H_{arom}), 7.17–7.20 (m, 6 H, H_{arom}), 7.27–7.33 (m, 12 H, H_{arom}), 7.56 (t, *J* = 2.0 Hz, 6 H, H_{arom}), 7.89 (d, *J* = 2.0 Hz, 6 H, H_{arom}).

^3C NMR (75 MHz, DMSO-d_6): δ = 35.69, 115.70, 116.35, 118.72, 118.92, 120.92, 122.33, 123.35, 124.05, 127.76, 131.00, 131.48, 132.71, 138.75, 149.10, 152.31, 166.59.

Anal. Calcd for $C_{78}H_{45}N_3O_{21}$: C, 68.87; H, 3.33; N, 3.09. Found: C, 69.12; H, 3.62; N, 3.44.

3,3′,3″,3‴,3⁗,3⁗′-{1,3,5-Triazine-2,4,6-triyltris[oxy(3-methoxy-4,1-phenylene)methanetriyl]}hexakis(4-hydroxy-2*H*-chromen-2-one) (3j)
White solid; yield: 1.20 g (83%); mp >300 °C.

IR (KBr): 3416, 3078, 2935, 1664, 1614, 1566, 1454, 927 cm^{-1}.

^1H NMR (300 MHz, DMSO-d_6): δ = 3.55 (s, 9 H, H_{OCH3}), 4.3 (br s, 6 H, OH), 6.31 (s, 3 H, $CH_{methine}$), 6.74–6.88 (m, 6 H, H_{arom}), 7.27–7.32 (m, 15 H, H_{arom}), 7.54 (t, *J* = 2.0 Hz, 6 H, H_{arom}), 7.88 (d, *J* = 2.0 Hz, 6 H, H_{arom}).

^3C NMR (75 MHz, DMSO-d_6): δ = 36.10, 55.99, 103.67, 111.65, 115.67, 116.33, 118.98, 123.16, 124.07, 131.33, 132.66, 140.02, 141.32, 152.33, 152.35, 158.32, 161.32, 164.55.

Anal. Calcd for $C_{81}H_{51}N_3O_{24}$: C, 67.08; H, 3.54; N, 2.90. Found: C, 67.35; H, 3.86; N, 3.23.

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