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Synthesis of Poly(aryl ether) Dendritic Structures Based on Calix[4]arenes

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Two new poly(aryl ether) dendritic structures carrying a donor (4-methyl-7-hydroxycoumarin) on the surface as dendrons and calix[4]arenes as a core were synthesized through a convergent synthetic approach. Poly(aryl ether) dendritic structures are connected through ether bonds on the lower rim of calix[4]arene unit. Its structure was determined by ¹H-NMR, ¹³C-NMR and elemental analysis. The photophysical properties of the series of poly(aryl ether) dendrimers have been determined, and the effect of the generation number on the absorption and emission properties of the synthesized dendritic structures was investigated. As the chromophore group number on the surface of the dendritic structure increased, molar absorptivity coefficients and emission intensities of the structures were found to increase.

Keywords: Dendrimer, dendron, calix[4]arene, chromophore, energy transfer, light harvesting

1 Introduction

Over the two past decades dendrimers have attracted considerable attention because of the special properties by their unique physical property and structure, such as highly branched structure, monodispersed molecular weight, globular and symmetrical conformation, and high density of peripheral functionalities (1–6). Despite their first appearance being reported by Vögtle (7), interest in these macromolecules stemmed from reports in the mid 1980's by Tomalia (8), Newkome (9), and Denkewalter (10). They have attracted enormous interest in recent years, as evidenced by the exponential increase in publications and patents concerning them, largely they may be employed in a wide range of applications ranging from physical, chemical, and biological.

There are two ways to build a dendrimer: the divergent approach (7–9) and convergent approach (11, 12). In the divergent approach, the synthetic sequence is from the core to periphery; while the convergent approach is from the periphery to core.

As the field of dendrimers rapidly grows (13, 14), a few key architectures clearly stand out as the most attractive and best documented ones within the highly diverse pool of branched polymers described so far. Poly(aryl ether)

dendrimers belong to this class: because the simplicity, reliability, and flexibility of their convergent synthesis (11,12), together with the commercial availability of the monomer itself, strongly contribute to the prominent role of this structure within the dendrimer literature. Indeed, poly(aryl ether) dendritic macromolecules have been widely used by independent groups for a variety of applications (15–18), and their chemistry (19, 20) and properties (21) are now well established.

Calixarenes (22, 23), well-known macrocyclic compounds of p-substituted phenols and formaldehyde, represent readily available three-dimensional structures widely used as starting materials and useful building blocks in the supramolecular chemistry. Functionalization of their upper and lower rims provides the calixarenes with variable inclusion capability and variable complexation characteristics (23). The exclusive bowl-like structure of calix[4]arene is also conformationally flexible and, as a result, can exist in four possible conformations: cone, partial cone, 1,2-alternate, and 1,3-alternate. Various attempts have been undertaken to incorporate calixarenes into different polymers, some of which are self assembled systems (24–26).

Calixarenes are considered to provide entities of excellent promise for incorporation within dendritic structures to generate new materials (27). To integrate calixarenes and dendrimers is an original idea, as well as their unique properties to form a new structural host, and there have several examples about dendrimers with calixarene as core (27, 29, 30, 31).

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Previously, we reported the synthesized poly(aryl ether) dendritic structures bearing chromophore peripheral groups via convergent methods and to determine their photophysical properties and fluorescence quantum yields. Herein, we report the design, synthesis, characterization, and photophysical properties of two new dendrimers having calix[4]arene as the core with two hydroxyl groups through a simple convergent procedure without protection-deprotection chemistry. We were interested in calix[4]arenes as functionalized core molecules because they are less affected by steric constraints. These kinds of structures are promising candidates for a variety of applications, which range, for example, from catalysis (cleavage and release of a photoactive drug or degradation of a photosensitive toxic substance) to amplification for display or mapping and organic optical media.

2 Experimental

All commercially available reagents were used without further purification. K_2CO_3 was activated by heating at $150^\circ C$ overnight under vacuum and stored in a desiccator. Column chromatography was carried out with Merck silica gel 70–230 mesh. Preparative TLC plates were Merck aluminum sheets covered with silica gel 60 F₂₅₄. The FTIR spectra were recorded via the KBr pellet method by using a Perkin-Elmer 1605 FTIR spectrophotometer. 1H -NMR and ^{13}C -NMR spectra were recorded on a BRUKER DPX-400 High Performance Digital FT-NMR, with tetramethylsilane (TMS) as the standard. The compounds were characterized by an Elementar Vario CHNS EL III elemental analysis instrument. UV-Vis absorption spectra were measured using a Shimadzu UV-1700 Pharma spectrophotometer. The photoluminescence were recorded on a Varian Cary Eclipse Fluorescence spectrophotometer.

p-*Tert*-butylcalix[4]arene (32), and compound 1–3 (33) were readily obtained according to the literature procedures.

2.1 Compound 1

1,4-bis(bromomethyl) benzene (1 eq., 6 g, 22.73 mmol); 4-methyl-7-hydroxycoumarin (1 eq., 4 g, 22.73 mol.), K_2CO_3 (1.25 eq., 3.924 g, 28.41 mmol) and 18-crown-6 (0.1 eq., 0.6 g, 2.27 mmol) in dry acetone (200 mL) was heated at reflux under N_2 with stirring for 48 h. The cooled solution was poured into water (200 mL), then filtered and washed with 250 mL of water and then dried with vacuum oven. The crude product was purified by column chromatography on silica gel (2:1 dichloromethane/hexane), to give 2.46 g (44%) of product 1 as a white solid. Mp: 116–117°C.

1H -NMR (200 MHz, $CDCl_3$) δ (ppm) = 2.4 (s, 3H, CH_3), 4.51 (s, 2H, $ArCH_2Br$), 5.13 (s, 2H, OCH_2Ar), 6.15 (s, 1H,

$CH=C$), 6.88 (s, 1H, ArH), 6.93 (d, $J = 8.0$ Hz, 1H, ArH), 7.43 (s, 4H, CH_2ArHCH_2), 7.51 (d, $J = 8.0$ Hz, 1H, ArH).

2.2 Compound 2

Compound 1 (2 eq., 2 g, 5.5658 mmol), 3,5-dihydroxybenzyl alcohol (1 eq., 0.39 g, 2.7829 mmol), K_2CO_3 (2.5 eq., 0.975 g, 6.9573 mmol), and 18-crown-6 (0.2 eq., 0.15 g, 0.5566 mmol) in dry acetone (150 mL) was heated at reflux under N_2 with stirring for 48 h. After the solution was cooled, poured into water (75 mL), extracted with dichloromethane (4x50 mL) and then dried over $MgSO_4$. The solvent was evaporated in vacuum. The crude product was purified by column chromatography on silica gel (100:1 CH_2Cl_2 /methanol) to give 1.36 g (68%) of product 2 as a white solid. Mp: $142^\circ C$.

1H NMR (200 MHz, $CDCl_3$) δ (ppm) = 2.39 (s, 6H, CH_3), 4.64 (s, 2H, CH_2OH), 5.05 (s, 4H, $ArCH_2O$), 5.13 (s, 4H, OCH_2Ar), 5.3 (s, 1H, OH), 6.13 (s, 2H, $CH=C$), 6.52 (s, 1H, ArH), 6.62 (s, 2H, ArH), 6.86 (s, 2H, ArH), 6.93 (d, $J = 10.0$ Hz, 2H, ArH), 7.44 (s, 8H, CH_2ArHCH_2), 7.50 (d, $J = 8.0$ Hz, 2H, ArH).

2.3 Compound 3

Compound 2 (1 eq., 1 g, 1.4352 mmol) and NEt_3 (7.5 eq., 1.0872 g, 10.7640 mmol) in dry dichloromethane (100 mL) methanesulphonyl chloride (6 eq., 0.9868 g, 8.6112 mmol) was added in 15 min at $-10^\circ C$ under a nitrogen atmosphere. After stirring for 1 h at $-10^\circ C$, the reaction mixture was poured into a mixture of crushed ice (100 mL). The dichloromethane layer was separated, washed with saturated $NaHCO_3$ solution (100 mL), dried ($MgSO_4$) and evaporated. The crude product was purified by recrystallization in diethyl ether to give 0.78 g (78%) compound 3 as a white solid. M.p: 152.5 – $153^\circ C$.

1H NMR (200 MHz, DMSO) δ (ppm) = 2.39 (s, 6H, CH_3), 2.88 (s, 3H, SO_2CH_3), 5.06 (s, 4H, $ArCH_2O$), 5.14 (s, 4H, OCH_2Ar), 5.15 (s, 2H, $ArCH_2OSO_2$), 6.13 (s, 2H, $CH=C$), 6.64 (s, 1H, ArH), 6.65 (s, 2H, ArH), 6.88 (s, 2H, ArH), 6.91 (d, $J = 4.0$ Hz, 2H, ArH), 7.45 (s, 8H, CH_2ArHCH_2), 7.50 (d, $J = 10.0$ Hz, 2H, ArH).

2.4 Compound 4

Compound 1 (2 eq., 1 g, 2,7860 mmol), *p*-*tert*-butylcalix [4]arene (1 eq., 0.9039 g, 1,3930 mmol), dry K_2CO_3 (2.5 eq., 0.4813 g, 3.4825 mmol) in CH_3CN (100 mL) was heated at reflux under N_2 with stirring for 48 h. After being cooled to room temperature, the reaction mixture was poured into water (100 mL) and extracted with CH_2Cl_2 (3 x 50 mL). The combined organic extracts were dried over $MgSO_4$. The solvent was evaporated in vacuum. The crude product was purified by column chromatography on silica gel (2,5:1 ethyl acetate/hexane) to give 0.7553 g (%48) of product 4 as a white solid. Mp: 296 – $297^\circ C$.

IR (KBr): 3431 (O–H), 1732 (C=O), 1615 (C=C), 1390 (CH₃), 1278, 1144 (COC) cm⁻¹.

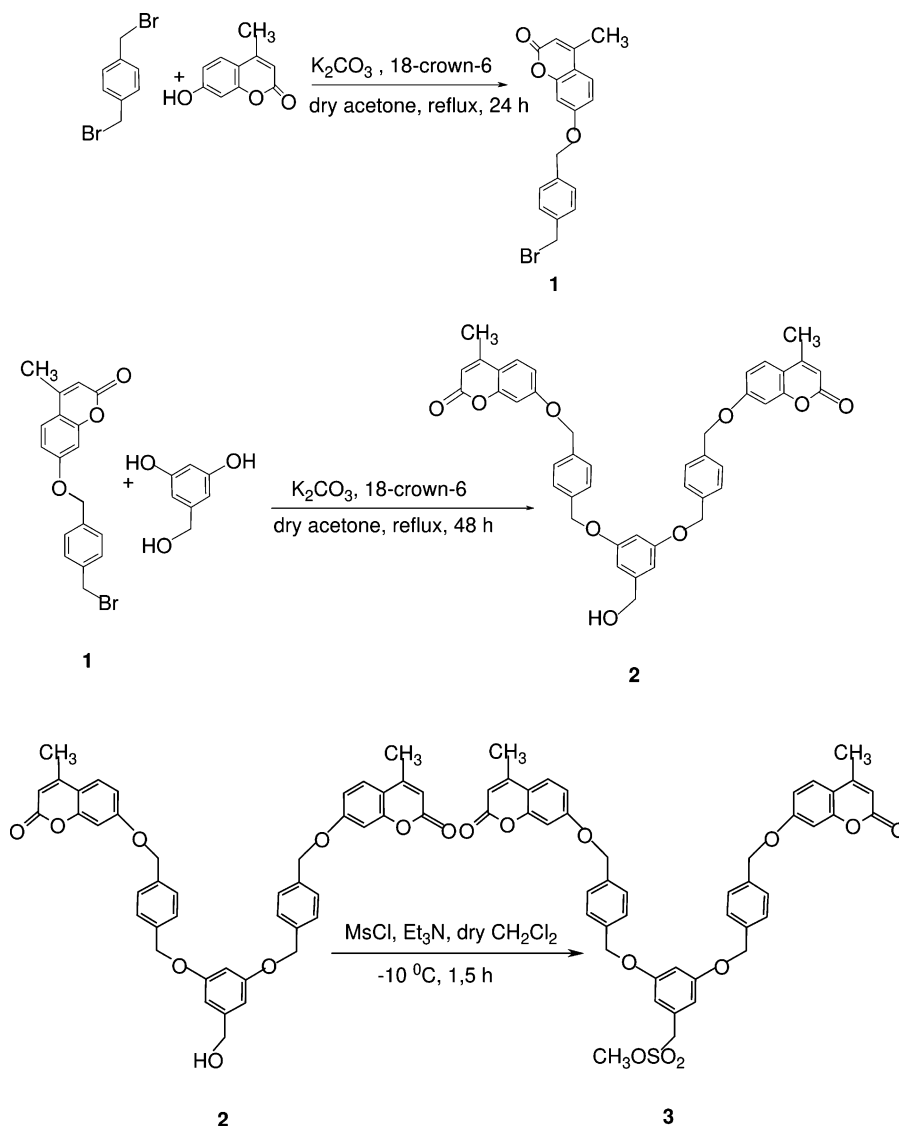
¹H-NMR (400 MHz, CDCl₃) δ (ppm) = 0.97 (s, 18H, Bu^t), 1.32 (s, 18H, Bu^t), 2.39 (s, 6H, CH₃), 3.34 (d, 4H, *J* = 13.12 Hz, ArCH₂Ar), 4.33 (d, 4H, *J* = 13.02 Hz, ArCH₂Ar), 5.07 (s, 4H, ArCH₂Br), 5.11 (s, 4H, OCH₂Ar), 6.09 (d, 2H, *J* = 1.18 Hz, CH=C), 6.71 (d, 2H, *J* = 2.47 Hz, ArH), 6.83 (s, 4H, ArH), 6.90–6.92 (m, 2H, ArH), 7.09 (s, 4H, ArH), 7.35 (s, 2H, OH), 7.47 (d, 8H, *J* = 8.6 Hz, CH₂ArHCH₂), 7.78 (d, 4H, *J* = 8.0 Hz, CH₂ArHCH₂).

¹³C-NMR (100 MHz, CDCl₃) δ (ppm) = 18.46, 31.00, 31.66, 31.73, 33.84, 33.96, 70.44, 101.50, 111.94, 112.75, 113.67, 125.03, 125.58, 125.66, 127.41, 127.60, 128.18, 132.52, 135.06, 137.75, 141.47, 147.19, 149.61, 150.72, 152.73, 155.02, 161.14, 161.61. Anal. Calcd. for C₇₉H₈₄O₁₀: C, 78.74; H, 7.02. Found: C, 79.09; H, 6.88.

2.5 Compound 5

Compound 3 (2 eq., 0.2518 g, 0.3082 mmol), *p*-*tert*-butylcalix[4]arene (1 eq., 0.1 g, 1.5410 mmol), dry K₂CO₃ (2.5 eq., 0.0449 g, 3.8525 mmol), KI (1 eq., 0.0256 g, 1.5410 mmol) in CH₃CN (50 mL) was heated at reflux under N₂ with stirring for 96 h. After being cooled to room temperature, the reaction mixture was poured into water (50 mL) and extracted with CH₂Cl₂ (3 × 50 mL). The combined organic extracts were dried over MgSO₄. The solvent was evaporated in vacuum. The crude product was purified by column chromatography on silica gel (100:2 CH₂Cl₂/MeOH) to give 0.147 g (%51) of product 5 as a white solid. Mp: 142°C.

IR (KBr): 3427 (O–H), 1728 (C=O), 1388 (CH₃), 1278, 1145 (COC) cm⁻¹.



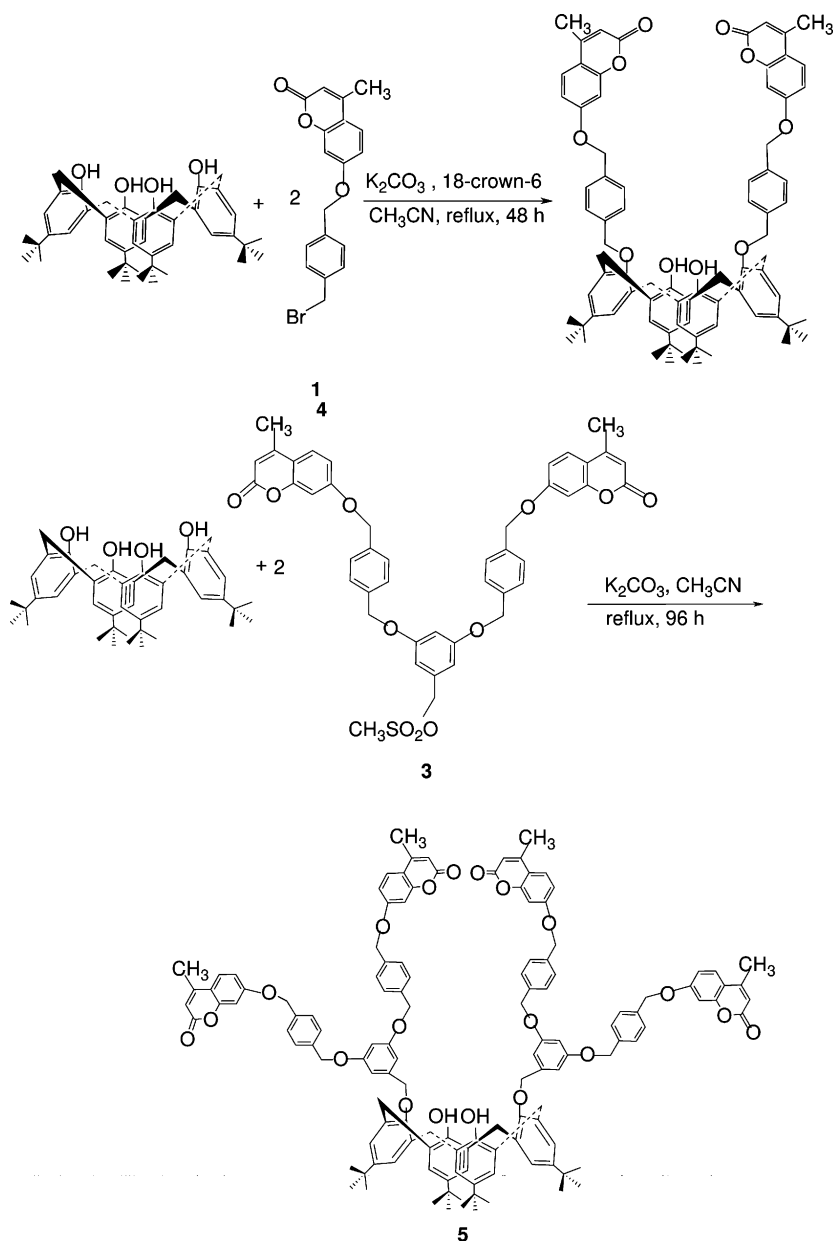
Sch. 1. Synthesis of dendritic structures 1, 2 and 3.

^1H NMR (400 MHz, CDCl_3) δ (ppm) = 0.97 (s, 18H, Bu^t), 1.33 (s, 18H, Bu^t), 2.39 (s, 12H, CH_3), 3.32 (d, 4H, $J=13.0$ Hz, ArCH_2Ar), 4.31 (d, 4H, $J=13.0$ Hz, ArCH_2Ar), 4.85 (s, 8H, ArCH_2O), 4.97 (s, 4H, ArCH_2O), 5.01 (s, 8H, OCH_2Ar), 6.13 (s, 4H, $\text{CH}=\text{C}$), 6.56 (s, 2H, OH), 6.83 (s, 8H, ArH) 6.89 (d, 2H, $J=1.89$ Hz, ArH), 6.92 (d, 2H, $J=1.89$ Hz, ArH), 6.99 (s, 4H, ArH), 7.11 (s, 4H, OArHCH_2), 7.33 (s, 16H, $\text{CH}_2\text{ArHCH}_2$), 7.36 (s, 2H, OArHCH_2), 7.49 (d, 4H, $J=8.8$ Hz, ArH). ^{13}C -NMR (100 MHz, CDCl_3) δ (ppm) = 18.68, 31.00, 31.31, 31.59, 31.75, 33.89, 33.98, 69.30, 70.16, 101.87, 102.15, 105.28, 112.05, 112.81, 113.77, 125.09, 125.61, 127.57, 127.59,

127.83, 132.50, 135.18, 137.37, 139.58, 141.75, 147.29, 149.49, 150.67, 152.56, 155.16, 160.06, 161.23, 161.63. Anal. Calcd. for $\text{C}_{129}\text{H}_{124}\text{O}_{20}$: C, 76.05; H, 6.13. Found: C, 75.23; H, 5.99.

3 Results and Discussion

In order to obtain poly(aryl ether) dendritic structures based on calix[4]arenes our studies began with the synthesis of *p*-*tert*-butylcalix[4]arene (32), compound 1 (33), and compound 3 (33) to the literature procedures.



Sch. 2. Synthesis of dendritic structures based on calix[4]arenes 4, 5.

3.1 Synthesis of Dendritic Structures

The synthesis of dendritic structures are shown in Scheme 1. Under typical Williamson ether synthesis conditions in the presence of K_2CO_3 , 1,4-bis-(bromomethyl)benzene spacer group was reacted with 4-methyl-7-hydroxycoumarin to synthesize compound **1** in a yield of 41% (29). Following a standard convergent strategy, the first generation of benzyl alcohol (compound **2**) was prepared in 68% yield through a coupling reaction of compound **1** and 3,5-dihydroxybenzyl alcohol monomer (Scheme 1) (33). Compound **2** was converted to the compound **3** in 78% yield using methanesulphonyl chloride in the presence of excess NEt_3 at $-10^\circ C$ (Scheme 1) (33).

3.2 Synthesis of Dendritic Structures Based on Calix[4]arene

Synthesis of dendritic structures based on calix[4]arene is soluble in common organic solvents such as dichloromethane, chloroform, and acetonitrile, which are helpful for separation and purification. All dendritic structures based on calix[4]arenes were characterized by FTIR, elemental analysis, 1H -NMR and ^{13}C -NMR spectroscopy.

The synthesis of dendritic structures based on calix[4]arene is shown in Scheme 2. Under typical Williamson ether synthesis conditions in the presence of K_2CO_3 , *p*-*tert*-calix[4]arene was reacted 4-(4-methylcoumarine-7-yl-oxymethyl) benzylbromide **1** in acetonitrile at reflux temperature to give the narrow-rim 1,3-disubstituted compound **4** in good yields. Compound **4** show the expected NMR pattern, with two signals being obtained for both the aromatic protons of the calix[4]arene and the *tert*-butyl groups, indicative of 1,3-disubstitution on the narrow rim of calix[4]arene. Furthermore, the cone conformation of the calix[4]arenes is also confirmed by the presence of two doublets at 3.34 and 4.33 ppm.

The Fréchet type convergent approach in dendrimer synthesis is the most frequently used procedure. In this method the activation of benzylic alcohols to prepare poly(aryl ether) bromides utilizes PPh_3/CBr_4 (11). The advantages of the mesylate route compared to the bromide route at the synthesis are reduced reaction times and simple purification of products by crystallization; hence, chromatographic separations can be avoided. An extra benefit is the higher reactivity of the mesylates (33, 34). Therefore previously we used the mesylate activation route to synthesis of compound **3** (33).

Following a standard convergent strategy, in the presence of K_2CO_3 and KI, *p*-*tert*-butylcalix[4]arene **1** was reacted compound **3** in acetonitrile at reflux temperature to give the narrow-rim 1,3-disubstituted compound **5** in good yields (Scheme 2). Compound **5** was easily identified 1H -NMR spectrum, with two signals being obtained for both the aromatic protons of the calix[4]arene and the *tert*-butyl groups, indicative of 1,3-disubstitution

on the narrow rim of calix[4]arene. Additionally, the cone conformation of the calix[4]arenes is also confirmed by the presence of two doublets at 3.32 and 4.31 ppm.

3.3 Photophysical Properties

The investigated dendritic structures **4**, **5** consist of a calix[4]arene core appended with two 4-methyl-7-hydroxycoumarin units (compound **4**), four 4-methyl-7-hydroxycoumarin units (compound **5**), respectively. In these measurement 1×10^{-5} M solutions of each compound was used. Absorption spectra for the dendritic structures based on calix[4]arenes are shown in Figure 1. Accordingly, the spectra of these compounds show absorption maximum in the 288 nm and 320 nm. As expected, an increase in the generation number leads an increase in the number of the peripheral chromophores and doubles the absorption from one to the next. Hence, with increasing generation number, the amount of light that the peripheral antenna is capable of harvesting is dramatically enhanced. We can conclude that the absorption spectra of the examined compounds are those expected from the spectra of the component chromophoric units.

Fluorescence emission spectra for 1×10^{-5} M solutions of the dendritic structures based on calix[4]arenes are shown in Figure 2. Excitation of the dendritic structure series at $\lambda_{max} = 320$ nm (maximum absorption wavelength for donor chromophore) resulted in emissions at 387 nm (compound **4**), 384 nm (compound **5**).

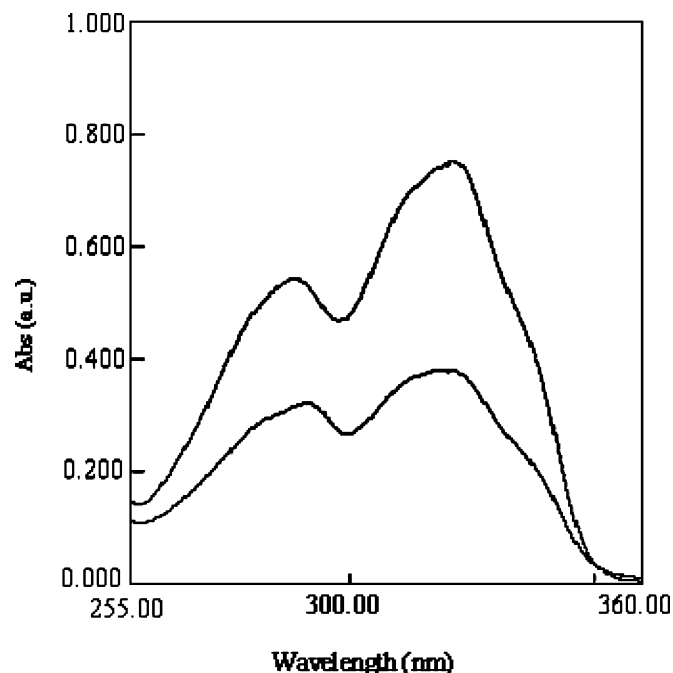


Fig. 1. Absorption spectra of dendritic structures based on calix[4]arenes **4**, **5** in CH_2Cl_2 .

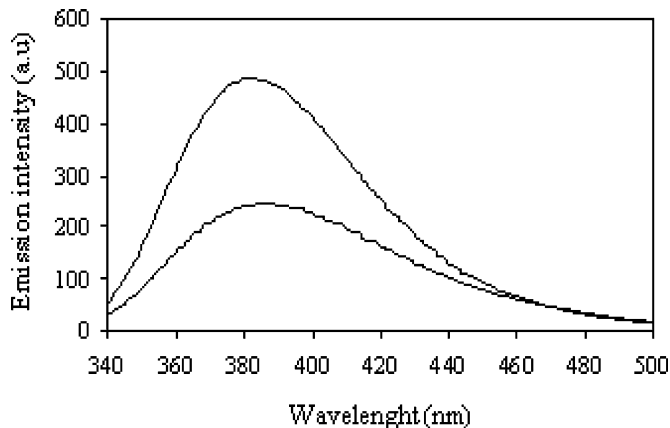


Fig. 2. Emission spectra of dendritic structures based on calix[4]arenes **4**, **5** in CH_2Cl_2 , $\lambda_{\text{ex}} = 320 \text{ nm}$.

4 Conclusions

The potential use of light harvesting and emitting materials in a wide variety of research and application areas increases the importance of chromophore group containing poly(aryl ether) dendritic structures as one of the major building blocks for the synthesis of dendrimers and hyperbranched polymers. We have synthesized two new dendritic structures bearing poly(aryl ether) moiety as dendrons and calix[4]arene as a core through a convergent synthetic strategy. It can be seen from the absorption and emission spectra (Figs. 1, 2) that as the number of peripheral chromophores double from one dendrimer generation to the next, the amount of absorbed and emitted light also nearly doubles (33). Highly functionalized core molecules are very effective for synthesis of dendrimers. Macrocyclic compounds like *p*-*tert*-butylcalix[4]arene is suitable as core molecules because of easy synthesis and adequate space between functional groups (30). Also, these dendrimers obtained from calix[4]arene core units have potential as molecular recognition materials.

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