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Oligomerization of benzylic alcohols and its mechanism

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Abstract

A number of cyclic (**5a–b**) and linear (**3a–c**; **4b–c**) oligomers arose from benzylic alcohols have been synthesized by treatment of **1a–c** with a bentonitic earth. A mechanism for the formation of both cyclic and linear oligomers is proposed based on the isolation of the key intermediate; the ethers **2a–c**. © 1997 Elsevier Science B.V.

Keywords: Heterogeneous catalysis; Bentonite clay; Benzylic oligomerization mechanism; Electrophilic aromatic substitution

1. Introduction

The acid catalyzed condensation of benzylic alcohols bearing electron donating groups provides a simple route to the cycloveratrylenes (CVTs) and its homologues [1]. Because its strong tendency to self-condensation observed in a number of reports, the veratryl cation has been suggested as the logic intermediate in the formation of CTVs, whatever the way in which the former is generated from a variety of precursors under acidic conditions [2–6]. In a recent report, higher cyclic oligomers, tetra, penta and hexacycloveratrylenes have been prepared and characterized by treatment of veratryl alcohol with trifluoroacetic acid [7]. On the other hand, bentonitic clay has long played a role in organic chemistry, mainly as catalytically active agent [8–10]. Recently, we have described the cyclic and linear oligomerization of [9–11] trimethoxybenzyl alcohol with a bentonitic clay as catalyst to give the corresponding tricycloveratrylene **5b** along with other interesting linear oligomers (**3b**, **4b**) not found before in this type of reaction [11]. Based on the product structures, we inferred that they might be formed through a different pathway that the one previously suggested under acidic conditions [1]. Now, in this paper we wish to give evidences that support the benzylic ethers **2a–c** as the key intermediates in the formation of both cyclic and linear oligomers.

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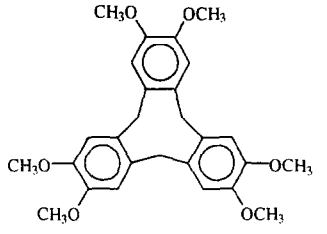
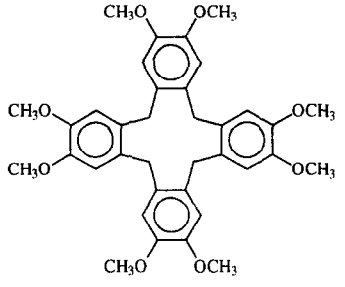
2. Results and discussion

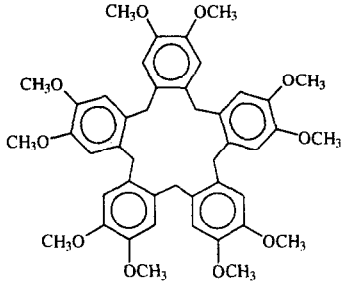
A series of subsequent experiments performed with bentonitic earth and benzylic alcohols led us to the following results. When alcohol **1a** was treated with the clay in 1:4 w/w ratio, in methylene chloride at room temperature for 2 h, the corresponding cyclotriveratrylene (CTV) **5a**, cyclotetraveratrylene (CTTV), cyclopentaveratrylene (CPV) and cyclohexaveratrylene (CHV) were isolated in 44%, 12%, 14% and 11% yield respectively. During the course of this reaction, the formation of a main product was observed by TLC within the first 15 min, however the product vanished with the appearance of the oligomers. In order to isolate and characterize such product, the amount of bentonitic earth was reduced to a 1:1 ratio w/w and stopping the reaction by filtering the mixture before the starting material was totally consumed. Under these milder conditions the ether **2a** was isolated in 44% yield along with CTV **5a**; 15% and unreacted alcohol **1a**; 37%. After this result, we envisaged that the ether **2a** should be the precursor of both linear and cyclic oligomers.



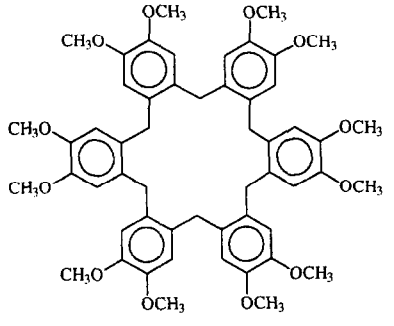
CTV



CTTV



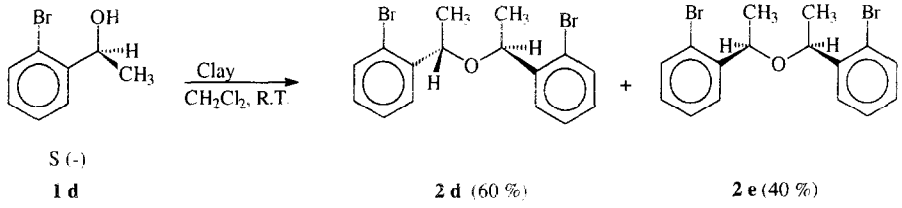
CPV



CHV

Furthermore, a similar compound **2b** had previously been isolated by us [11]. In order to prove the hypothesis, compound 2a was treated upon the same conditions described above and indeed the compound **3a** was isolated along with the corresponding CTV **5a**. Same results were obtained when ethers **2b** and **2c** were treated under the same conditions to afford the corresponding linear **3b–c**, **4c** and cyclic **5b** oligomers.

In a set of similar experiments the alcohol **1c** was treated with bentonite in a 1:1 w/w ratio. After 3 days, the ether **2c** was isolated together with the corresponding linear oligomers **3c** and **4c** in 37%, 33% and 16% yield, respectively. When the amount of catalyst was increased to 1:4 w/w ratio, a new product; **6c** was isolated in addition to the oligomers **3c**, **4c** and ether **2c**.



S (-)
1 d

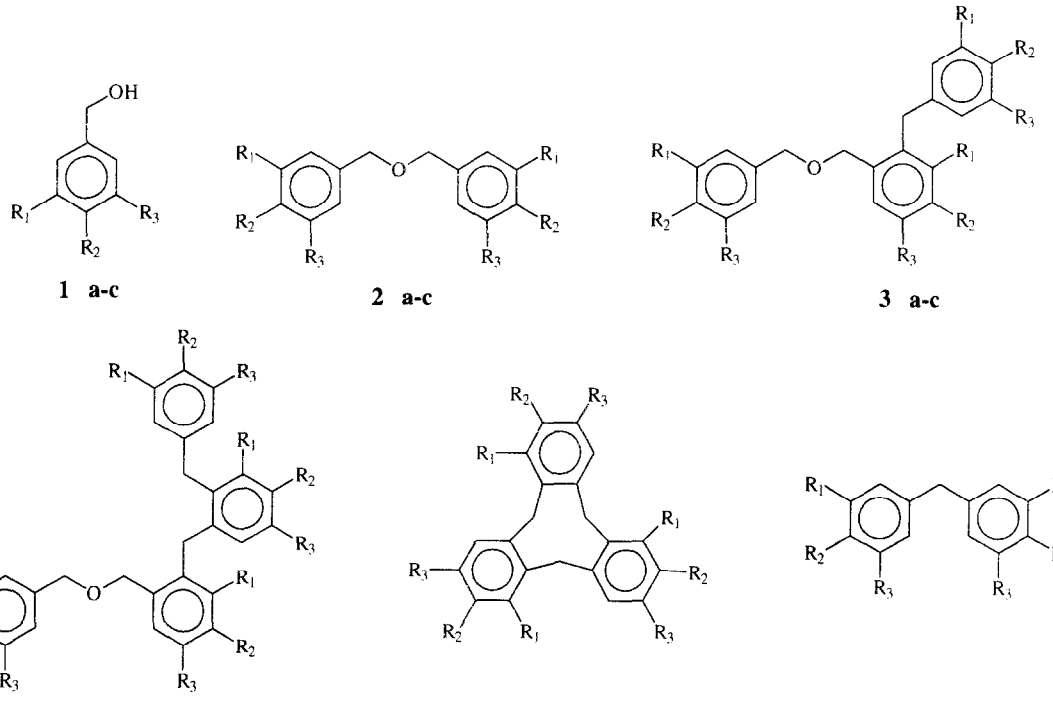
2 d (60 %)

2 e (40 %)

Scheme 1.

When (*s*)-(-)-2-bromo-α-methylbenzyl alcohol was reacted with clay in 1:1 w/w ratio in CH_2Cl_2 at room temperature or at reflux in CS_2 a diasteroisomeric mixture of the optical active ether **2d** (60%) and the meso ether **2e** (40%) was found (Scheme 1).

At this point we thought in the possibility that using strong mineral acids as catalyst for the cyclooligomerization of benzylic alcohols, the key intermediate might be only the corresponding benzylic carbocation as previously claimed [1] and under milder acids, like bentonitic clay, the key intermediate might be the benzylic ether.



1 a-c

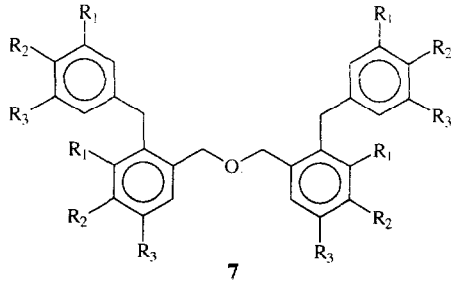
2 a-c

3 a-c

4 a-c

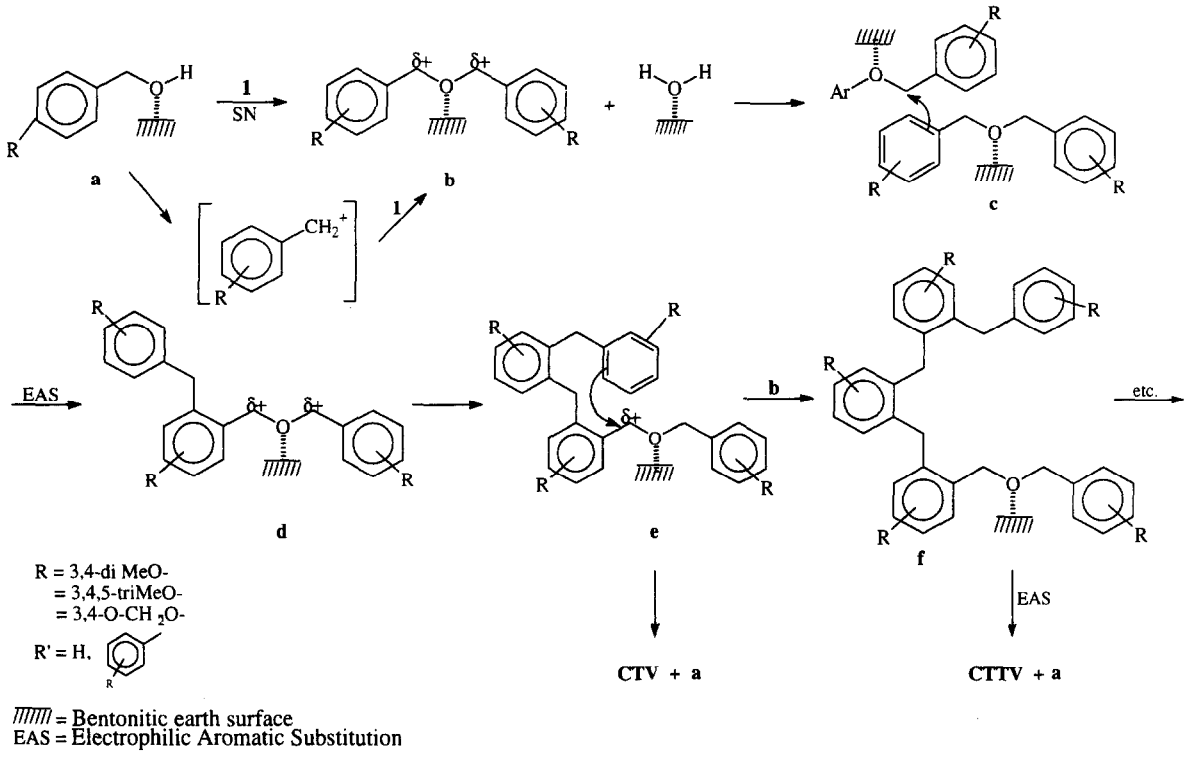
5 a-c

6 c



7

a R_1 = H, R_2 = R_3 = OMe
b R_1 = R_2 = R_3 = OMe
c R_1 = H, R_2, R_3 = -O-CH_2-O-



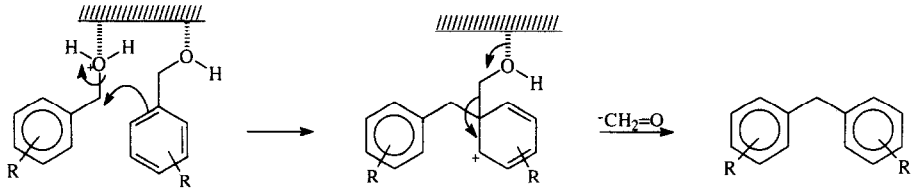
Scheme 2.

In order to clarify this fact, we decided to react the benzylic alcohols **1a–c** with strong mineral acids. Thus, when we repeated the experiment reported by Collet [1] the cyclooligomers, CTV, 23%, CTTV, 39%, CPV, 15% and CHV 5% were isolated. However, upon treatment with more diluted acid (CF_3CO_2H/MeOH 1% w/w at room temperature) we were able to isolate the ether **2a** (9%) and the linear oligomers **3a** (11%) along with the CTV **5a**, 36% as the main product. Furthermore, treatment of alcohols **1b–c** under the same conditions led to the corresponding ethers **2b–c**, linear oligomers **3b–c** and **4b–c** together with the cyclic compound **5b** and the benzyl derivative **6c**. In contrast with alcohols **1a** and **1b**, piperonyl alcohol **1c** did not lead to cyclooligomers at all. Attempts to prepare cyclotriveratrylene **5c** under various acidic conditions proved futile even upon previously experimental conditions described for its preparation [12–14].

According to the results described above, it is possible to suggest a reasonable mechanism for the formation of both linear and cyclic-oligomers.

The reaction proceeds via the key ether **2**, formed through a dehydration process. In the case of using mineral acids, the ether is formed through a carbocation intermediate, generated in the usual fashion. In a second stage, the ether can reversibly be fragmented to afford a carbocation which in turn can react with other molecule of ether by an electrophilic aromatic substitution process to lead the linear and cyclic oligomers.

However, in the case of using clay as catalyst, the key ether seems to be formed through two different pathways: first generating the corresponding carbocation **b** which in turn is attacked by a molecule of alcohol and/or by a nucleophilic substitution reaction on alcohol **a** by other molecule of alcohol (Scheme 2). These processes are supported by the amazing results observed from alcohol **1d** (Scheme 1). Indeed, the meso ether **2e** may come from a carbocation and/or by a nucleophilic



Scheme 3.

displacement SN^2-like on alcohol **a** (Scheme 2). The major optical active ether **2d** might be formed through the carbocation and/or by a SNi-like displacement.

The role of the bentonitic earth is to provide the acidic media to carry out the intermolecular substitution reaction in which an alcohol molecule on the clay surface is attacked by a molecule of alcohol to form the ether (a → b, Scheme 2). The clay may provide active sites in the micropore surface or interlayer to have in close contact the two molecules of alcohol at the reaction site. Thus, in a second stage two molecules of ether can react on the lattice surface through an electrophilic aromatic substitution mechanism to displace a molecule of **1** leading to compound **3** (c → d). A similar process should follow in order to give compound **4** (d → e), which in turn can incorporate stepwise units of 'alcohol' to form linear oligomers (e → f). These intermediates (e, f, etc.) should be the precursors of the corresponding cyclooligomers CTV, CTTV, CPV and CHV, which are formed through an intramolecular electrophilic aromatic substitution.

We would like to point out the remarkably selectivity in the electrophilic aromatic substitution on compound **3** to give **4**, which takes place only on one of the two possible aromatic rings. In fact we were not able to isolate any compound with general structure like **7**. This selectivity might be probably due to a stronger interaction between the oxygen of the benzylic ether moiety and the Lewis acid center of the bentonitic clay leaving the other aromatic ring more 'free' to react.

Finally, the formation of compound **6c** can be rationalized by an ipso aromatic nucleophilic substitution (Scheme 3) between two molecules of benzylic alcohols and subsequent elimination of formaldehyde, [15] this last suggests the laminar surface participation which supports an internal attack.

In conclusion, oligomerization of benzylic alcohols occurs through a key intermediate ether **2**. When mineral acids are used as catalyst the formation of the ether proceeds through a carbocation intermediate. Later, the ether can reversibly be fragmented to afford a carbocation which leads to the linear and cyclic oligomers, while using clay, additionally to the carbocation a SN^2 versus SN^i type pathway could be involved.

3. Experimental

General remarks: All melting points were determined with a Fisher–Johns melting point apparatus and are uncorrected. Infrared (IR) spectra were recorded on a Perkin Elmer 552 and a Nicolet FTIR-Magna 700 spectrometer. ^{1}H-NMR spectra were measured with a Varian Gemini (200 MHz) and a Varian Unity Plus (300 MHz). The chemical shifts are expressed in ppm downfield from tetramethylsilane, using tetramethylsilane ($\delta = 0$) as an internal standard. Splitting patterns are indicated as s, singlet; d, doublet; dd, double–double; t, triplet; q, quartet; m, multiplet. Mass spectra were taken with a JEOL JMS AX505HA mass spectrometer. For thin layer chromatographic (TLC) analyses, Merck precoated TLC plates (silica gel 60 F254, 0.25 mm, Art 5715) were used.

4. Oligomerization of benzylic alcohols with bentonitic earth [1]

General procedure: A suspension of 3 g of benzylic alcohol **1a**, 80 ml of methylene chloride and 12 g (1:4 w/w ratio) of bentonitic earth were stirred vigorously at room temperature, until disappearance of the starting material $\simeq 2$ h. The reaction was conveniently monitored by TLC. The clay was eliminated by filtration through celite and washed with ethylacetate (3×10 ml). The combinated filtrates were dried on anhydrous Na_2SO_4 and solvent evaporated under reduced pressure. From the residue, cyclotetraveratrilene CTTV, 246 mg (12%) was separated by crystallization from $CHCl_3$/AcOEt. The filtrate was chromatographed on a silica gel column and using a mixture of hexane:ethylacetate 7:3 as eluent to afford cyclotriveratrilene [2] CTV **5a** (44%), cyclopentraveratrilene [2] CPV (14%) and cyclohexaveratrilene [2] CHV (11%).

4.1. Treatment of benzylic alcohols (1a–c) with bentonitic earth 1:1 w/w ratio and stopping the reaction after 1 h

The following products were isolated: From alcohol **1a**; ether **2a** (44%), CTV **5a** (15%), alcohol **1a** (recovered 37%). From alcohol **1b**; ether **2b** (13%), oligomer 3b (7%), CTV **5b** (41%) and alcohol **1b** (recovered 5%). From alcohol **1c**; ether **2c** (17%) and alcohol **1c** (recovered 53%).

4.2. Treatment of benzylic alcohols (1a–c) with bentonite earth 1:1 w/w ratio until starting material had dissapeared

From alcohol **1a**; ether **2a**, (9%), oligomer **3a** (23%) and CTV **5a** (47%). From alcohol **1b**; ether **2b** (4%), oligomers **3b** (18%), **4b** (15%) and CTV **5b** (27%). From alcohol **1c**; ether **2c** (19%), oligomers **3c** (16%) and **4c** (6%). This alcohol **1c** with 1:4 w/w ratio: ether **2c** (8%), oligomers **3c** (13%), **4c** (6%) and biphenylmethane **6c** (4%).

4.3. Treatment of the ethers 2a–c with bentonitic earth 1:1 w/w ratio

Products isolated by column chromatography: From ether **2a**; oligomers **3a** (27%) and CTV **5a** (39%). From ether **2b**; oligomers **3b** (15%), **4b** (8%) and CTV **5b** (35%). From ether **2c**; oligomers **3c** (17%), **4c** (11%) and biphenylmethane **6c** (3%).

4.4. Treatment of benzylic alcohols (1a–c) with diluted TFAA (CF_3CO_2H/MeOH 1% v/v).

From alcohol **1a**; ether **2a** (9%), oligomer **3a** (11%) and CTV **5a** (36%). From alcohol **1b**; ether **2b** (3%), oligomer **3b** (9%) and CTV **5b** 29%). From alcohol **1c**; ether **2c** (10%), oligomers **3c** (17%) and **4c** (7%), and biphenylmethane **6c** (7%).

[1] Bentonite–Clay. On examination by X-ray fluorescence, the montmorillonite type clay used in this work proved to have the following composition (%); SiO_2, 75.4; Al_2O_3, 9.3; MgO, 0.4; Fe_2O_3, 1.3; CaO, 4.0; K_2O, 0.4; TiO_2, 0.4; H_2O (110°), 9.5 The commercial acid-activated material was obtained from Tonsil Mexicana S.A. and analyzed with a Phillips Spectrometer using Cr primary radiation. The measured specific suface area was 300 m^2/g (B.E.T. N_2) and the pore volume was 0.4789 cm^3/g. The acidity by NH_3 thermodesorption was 0.099 meq/g. The particle size was 325 mesh.

[2] The spectra and physical properties were found to be identical with those reported for all these compounds. See Ref. [3].

4.5. Treatment of benzylic alcohol **1d** with bentonitic earth 1:1 w / w ratio

The reaction was stopped as soon as the ethers **2d** and **2c** were formed starting material almost disappeared ca. 20 min.

A 3:2 ratio mixture of ethers **2d** and **2c** was obtained. The products were separated by preparative thin layer chromatography; SiO_2 and using a 7:3 mixture of hexane-ethylacetate as eluent.

Compound **2a**. White solid mp 69–70°C. IR ($CHCl_3$): 2958, 2936, 2837, 1594, 1515, 1463, 1360, 1262, 1154, 1028 cm^{-1}. ^{1}H-NMR ($CDCl_3$ 200 MHz) δ: 6.92–6.87 (3H, m, ArH), 4.47 (2H, s, $ArCH_2$–O), 3.89 (6H, s, CH_3–O). MS *m/z*: 318 (M^+, 14%), 152 (100), 151 (70), 137 (24), 121 (31).

Compound **2b**. White solid mp 74–75°C. IR (film): 2994, 2939, 2838, 1592, 1506, 1460, 1421, 1330, 1128, 1007, 828 cm^{-1}. ^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 6.61 (2H, s, Ar–H), 4.51 (2H, s, $ArCH_2$–O), 3.88 (6H, s, CH_3O), 3.86 (3H, s, CH_3–O). MS *m/z*: 378 (M^+, 6%), 182 (100), 167 (23), 151 (25).

Compound **2c**. White solid mp 41–42°C. IR ($CHCl_3$): 3076, 2889, 2775, 1608, 1489, 1371, 1250, 1189, 1070, 935 cm^{-1}. ^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 6.86 (1H, s, ArH), 6.78 (2H, s, Ar–H), 5.95 (2H, s, O–CH_2–O), 4.41 (2H, s, $ArCH_2$–O). MS *m/z*: 286 (M^+, 2%), 135 (100), 77 (60), 149 (39), 121 (5), 65 (35).

1(*S*), 1′(*R*), 1,1′ di-(2″-bromophenyl)diethyl ether (**2e**). Less polar liquid. IR ($CHCl_3$): 3065, 2980, 2931, 1569, 1468, 1438, 1373, 1341, 1265, 1090, 1023 cm^{-1}. ^{1}H NMR ($CDCl_3$, 300 MHz) d: 7.61 (1H, dd, $J = 7.9$, $J = 1.8$), 7.49 (1H, td, $J = 7.9$, $J = 1.3$), 7.37 (1H, td, $J = 7.9$, $J = 1.3$), 7.13 (1H, dd, $J = 7.9$, $J = 1.8$), 4.63 (1H, q, $J = 6.4$), 1.36 (3H, d, $J = 6.4$). ^{13}C NMR ($CDCl_3$) d: 143.05, 132.66, 128.73, 127.86, 127.23, 122.81, 74.15, 23.02. MS *m/z*: 386 ($M^+ + 4$, 1), 384 ($M^+ + 2$, 2), 382 (M^+, 1), 371 (3), 369 (6), 367 (5), 201 (25), 199 (26), 185 (100), 183 (97), 101 (15), 104 (46).

1(*S*), 1′(*S*), 1,1′ di-(2″-bromophenyl)diethyl ether (**2d**). Liquid, $[\alpha]_D = -86.46$ (c 1.04, Me_2CO), IR ($CHCl_3$): 3065, 2980, 2931, 1568, 1470, 1437, 1371, 1341, 1266, 1094, 1022 cm^{-1}. ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.47 (1H, dd, $J = 7.9$, $J = 1.8$), 7.44 (1H, td, $J = 7.9$, $J = 1.3$), 7.23 (1H, td, $J = 7.9$, $J = 1.3$), 7.05 (1H, dd, $J = 7.9$, $J = 1.8$), 4.89 (1H, q, $J = 6.4$), 1.44 (3H, d, $J = 6.4$). ^{13}C NMR ($CDCl_3$) δ: 143.24, 132.32, 128.44, 127.70, 127.23, 121.88, 73.83, 22.22. MS *m/z*: 386 ($M^+ + 4$, 1), 384 ($M^+ + 2$, 2), 382 (M^+, 1), 371 (12), 369 (23), 367 (15), 201 (31), 199 (26), 185 (100), 183 (98), 105 (35), 104 (93).

Compound **3a**. Oil, IR (film): 2999, 2933, 2835, 1606, 1514, 1463, 1425, 1262, 1236, 1028 cm^{-1}. ^{1}H-NMR ($CDCl_3$ 200 MHz) δ: 6.91 (2H, m, ArH), 6.84 (2H, m, ArH), 6.75 (1H, m, ArH), 6.62 (2H, m, ArH), 4.46 (2H, s, Ar–CH_2–O), 4.43 (2H, s, Ar–CH_2–O) 3.93 (2H, s, Ar–CH_2–Ar), 3.85 (6H, s, CH_3–O), 3.80 (6H, s, CH_3–O), 3.80 (3H, s, CH_3–O), 3.77 (3H, s, CH_3–O). MS *m/z* 468 (M^+, 2%), 300 (23), 260 (100), 179 (46), 151 (43). Anal. calcd. for $C_{27}H_{32}O_7$: C, 69.21; H, 6.88%. Found: C, 69.33; H, 6.71%.

Compound **3b**. White powder mp 90–93°C. IR (film) 2996, 2938, 2837, 1591, 1506, 1456, 1421, 1330, 1236, 1124, 1008, 830 cm^{-1}. ^{1}H-NMR ($CDCl_3$ 300 MHz). 6.77 (1H, s, Ar–H), 6.55 (2H, s, Ar–H), 6.29 (2H, s, Ar–H), 4.45 (2H, s, Ar–CH_2–O), 4.41 (2H, s, $ArCH_2$–O), 3.95 (2H, s, Ar–CH_2–Ar), 3.86 (3H, s, CH_3–O), 3.85 (3H, s, CH_3–O), 3.82 (3H, s, CH_3O), 3.81 (6H, s, CH_3O), 3.77 (3H, s, CH_3O), 3.71 (9H, s, CH_3–O). MS *m/z* 558 (M^+, 11%), 377 (8), 361 (23), 329 (100), 298 (31), 209 (71), 181 (90) [11]. Anal. calcd. for $C_{30}H_{38}O_{10}$: C, 64.50; H, 6.86%. Found: C, 64.27; H, 6.75%.

Compound **3c**. White solid mp 100–102°C. IR ($CHCl_3$): 3085, 3042, 2890, 2774, 1484, 1441, 1371, 1187, 1097, 933 cm^{-1}. ^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 6.87–6.50 (8H, m, Ar–H), 5.82 (2H, s, O–CH_2–O), 5.79 (2H, s, O–CH_2–O), 5.77 (2H, s, O–CH_2–O), 4.27 (2H, s, Ar–CH_2–O), 4.25 (2H,

s, $ArCH_2$–O), 3.72 (2H, s, Ar–CH_2). MS *m/z*: 420 (M^+, 10%), 285 (13), 268 (100), 238 (48), 210 (15), 163 (53), 152 (34), 135 (38). Anal. calcd. for $C_{24}H_{20}O_7$: C, 68.56, H, 4.79%. Found: C, 68.22; H, 4.62%.

Compound **4b**. Oil, IR (film): 2997, 2937, 2837, 1589, 1506, 1455, 1330, 1238, 1123, 1039, 754 cm^{-1}. ^{1}H-NMR ($CDCl_3$, 300 MHz), 6.79 (1H, s, Ar–H), 6.50 (2H, s, Ar–H), 6.42 (2H, s, Ar–H), 6.30 (1H, s, Ar–H) 4.80 (2H, s, Ar–CH_2–O), 4.24 (2H, s, Ar–CH_2–O–), 4.09 (2H, s, Ar–CH_2–Ar), 3.96 (2H, s, Ar–CH_2–Ar), 3.88–3.54 (36H, MeO). MS *m/z* 738 (M^+, 2%), 557 (5), 441 (10), 509 (22), 377 (10), 361 (15), 329 (45), 209 (38), 181 (100) [11].

Compound **4c**. White solid mp 76–79°C. IR ($CHCl_3$): 3044, 3008, 2889, 2772, 1611, 1503, 1485, 1370, 1249, 1183, 1041, 933 cm^{-1}. ^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 6.87–6.38 (10H, m, ArH), 5.94 (2H, s, O–CH_2–O), 5.91 (2H, s, O–CH_2–O), 5.90 (2H, s, O–CH_2–O), 5.89 (2H, s, O–CH_2–O), 4.33 (2H, s, Ar–CH_2O), 4.27 (2H, s, Ar–CH_2–O), 3.77 (2H, s, Ar–CH_2–O), 3.73 (2H, s, Ar–CH_2). MS (FAB^+) *m/z* 554.18 (M^+14%), 553.17 (10), 154 (100). Anal. calcd. for $C_{32}H_{26}O_9$: C, 69.32; H, 4.73%. Found: C, 69.55, H, 4.51%.

Compound **6c**. White needles mp 140–142°C. IR ($CHCl_3$): 3061, 2887, 2774, 1608, 1503, 1486, 1442, 1359, 1299, 1242, 1189, 1041, 932 cm^{-1}. ^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 6.73 (2H, s, Ar–H), 6.64 (4H, m, Ar–H), 5.91 (4H, s, O–CH_2–O), 3.79 (2H, s, Ar–CH_2). ^{13}C-NMR ($CDCl_3$) δ: 121.58 (d), 109.2 (d), 108.1 (d), 100.8 (5), 41.3 (t). MS *m/z*: 256 (M^+, 100%), 225 (22), 168 (31), 139 (3), 135 (5). Anal. calcd. for $C_{15}H_{12}O_2$: C, 70.30; H, 4.62%. Found: C, 70.43; H, 4.69%.

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