# Synthesis and characterization of original [2]rotaxanes including cyclodextrin

Hemat M. Dardeer[1]

## Abstract

Four new [2]rotaxanes (**1**–**4**) comprising β-cyclodextrin (β-CD) threaded on different aliphatic chains have been synthesized and characterized. [2]Rotaxane **1** was formed by threading hexamethylenediamine through β-CD and capping with 1,4,5,6,7,7-hexachloro-5-norbornene-2,3-dicarboxylic anhydride. [2]Rotaxane **2** was synthesized by threading succinic dihydrazide through β-CD then capping with (11*S*,15*R*)-9,10,11,15-tetrahydro-9,10-[3,4]furanoanthracene-12,14-dione. [2]Rotaxane **3** was obtained by threading tetramethylenediamine through β-CD and capping with 2,3-diphenylmaleic anhydride. [2]Rotaxane **4** was formed by threading sebacoyl chloride through β-CD then capping with (11*R*,15*S*)-13-amino-9,10-dihydro-9,10-[3,4]epipyrroloanthracene-12,14-dione. The obtained [2]rotaxanes were characterized in detail by Fourier-transform infrared (FTIR) spectroscopy and $^1$H, $^{13}$C, and 2D correlation spectroscopy (COSY) nuclear magnetic resonance (NMR). Also the morphology of the obtained interlocked compounds was investigated by scanning electron microscopy.

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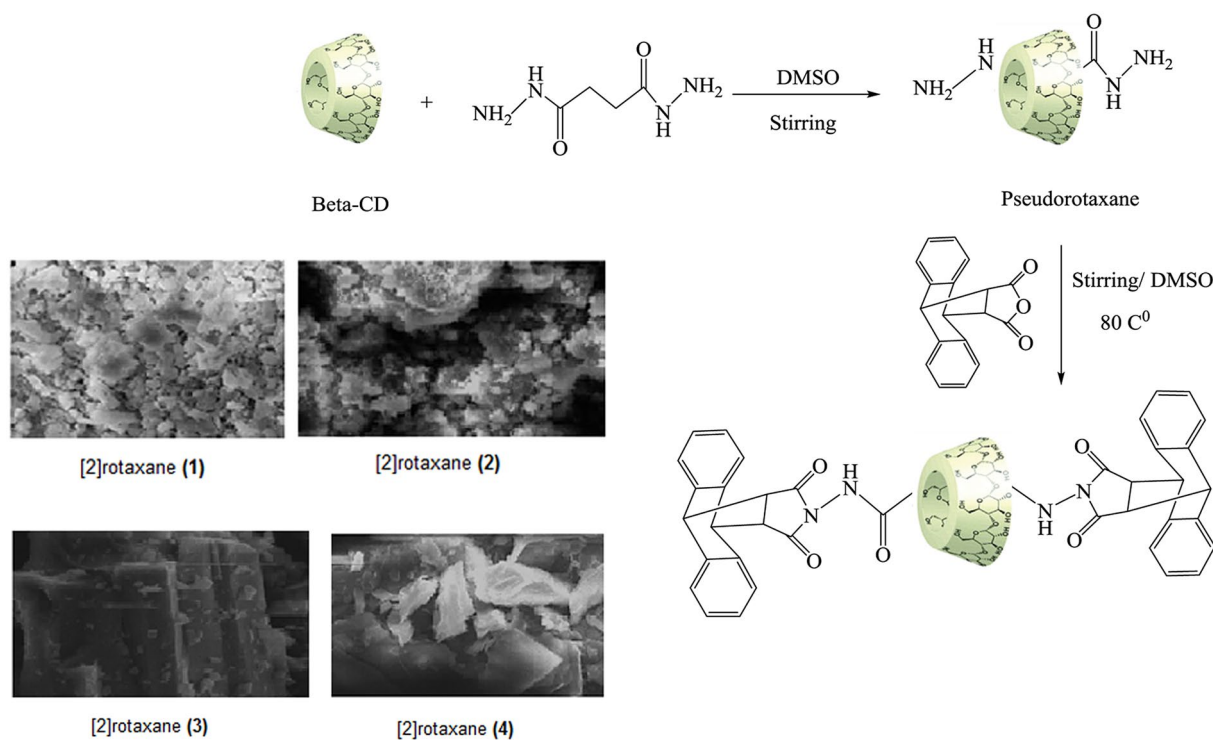
**Graphical abstract**



Beta-CD

Pseudorotaxane



[2]rotaxane **(1)**

[2]rotaxane **(2)**

[2]rotaxane **(3)**

[2]rotaxane **(4)**

**Keywords** Inclusion complex · SEM · Cosy NMR · Cyclodextrins · Rotaxanes

# 1 Introduction

Cyclodextrins are natural products with supramolecular structure. The most widely utilized CDs contain six, seven, or eight glucopyranose units. The unique properties of CDs [1] enhance their applications in everyday life. Various cyclodextrin derivatives have been synthesized [2–5], usually by reaction of primary and secondary hydroxyl groups in the CD moiety with various reagents such as ethers, amines, and acids. CDs play a chief role as the host molecule in the formation of inclusion complexes [6–8] with numerous organic molecules via hydrophobic–hydrophilic interaction to form essential interlocked compounds such as rotaxanes, polyrotaxanes, and catenanes [9–11]. CDs have many applications, including in drugs [12–15] and the food industry [16, 17]. Supramolecular complexes of CDs have been built based on their ability to link together via either covalent or noncovalent bonds. The aim of such derivatizations of cyclodextrins [18, 19] is to improve the solubility of CDs and their inclusion complexes and achieve a connection between the CDs and a guest by reducing the reactivity and movement of the latter. In this way, many of

the physical and chemical properties of hydrophobic guest molecules, e.g., solubility, stability, ultraviolet (UV) light resistance, volatility, taste, and toxicity, can be adjusted. Consequently, CDs have many applications in the food [20], pharmaceutical [21], cosmetic [22], environmental protection [23], and textile [24] industries. Three factors must be considered regarding the creation of such inclusion complexes: the size of the CD relative to that of the guest molecule, the functional groups inside the guest, and the thermodynamics between the CD, guest molecule, and the solvent used. Rotaxanes are supramolecular interlocked molecules with stable properties due to the high activation energy that prevents slippage of the CD ring along the axis. Synthesis of axial inclusion complexes depends on the presence of an axis to insert into a CD ring and the thickness of the guest compared with the interior diameter of the CD ring [25]. The stability of this type of rotaxane is due to intermolecular hydrogen bonds between hydroxyl groups of CDs. The essentially forces that enable the creation of a CD inclusion complex are hydrophobic and van der Waals linkages between the center of the CD ring and hydrophobic sites on the guest [26]. Pseudorotaxanes are

interlocked molecules that may dissociate under different conditions, such as temperature, competitive guests, and change in solvent. Therefore, they should be fixed by capping or macrocyclization [10, 27–29]. The significance of CDs for the preparation of rotaxane compounds is due to their shielding effect on the guest molecule [30] and the effect of light, magnetic field, and pH changes during the synthesis of rotaxanes [31]. Recently, rotaxanes have found vital applications in different fields, e.g., as sensory polymers [32], molecular switches [33], crossbar devices [34], and bioelectronics [35]. Continuing our previous work on synthesis and characterization of [2]rotaxanes [27–29] and inspired by the importance of rotaxane compounds, new [2]rotaxane compounds incorporating β-CD with different caps and threads were synthesized and characterized in this work.

The biological applications of these unique interlocked molecules are under study.

## 2 Experimental

### 2.1 Chemicals and reagents

The chemicals and reagents used in this work were obtained from Sigma Aldrich with 95 % purity, while β-cyclodextrin with 99 % purity was obtained from Acros Organics. The chemicals were used as obtained without any further purification.

### 2.2 Characterization

Melting points were determined using a Stuart SMP11 and are uncorrected. The chemical structure of the obtained compounds was established by $^1$H and $^{13}$C NMR spectroscopy, 2D COSY NMR, and Fourier-transform infrared (FTIR) spectroscopy. $^1$H, $^{13}$C, and COSY NMR spectra were recorded at 25 °C using a Bruker AM-400 NMR (Germany) spectrometer at 400 MHz. The functional groups of the obtained compounds were investigated using a Jasco model 4100 (Japan) infrared spectrometer at room temperature in the wavenumber range from 4000 to 400 cm$^{-1}$. Analytical data were obtained from the Microanalytical Data Unit at Cairo University, Giza, Egypt. Scanning electron microscopy (SEM, model JSM 5500; JEOL, Japan) was used to examine the morphology of the obtained [2]rotaxanes at accelerating voltage of 10 kV.

## 2.3 Synthesis of [2]rotaxane interlocked compounds

### 2.3.1 Synthesis of [2][hexamethylenediamino]-rota-[β-cyclodextrin] (1)

β-Cyclodextrin (3 g, 2.64 mmol) was dissolved in dimethyl sulfoxide (DMSO, 15 ml) until complete solubility. Hexamethylenediamine (0.3 g, 1.6 mol) was added with stirring for 2 h. 1,4,5,6,7,7-Hexachloro-5-norbornene-2,3-dicarboxylic anhydride (0.98 g, 2.63 mol) was added with continuous stirring for 30 h at 80 °C. The precipitate formed was filtered off and dried to give **1** as white crystals (3.92 g, 2.00 mol) in 76 % yield; m.p. above 350 °C; *Anal*. Calculated for $C_{66}H_{86}N_2O_{39}Cl_{12}$ (1956.11): C, 40.53; H, 4.40; N, 1.43; Cl, 21.75. Found: C, 40.50; H, 4.42; N, 1.45; Cl, 21.72; FTIR (KBr, cm$^{-1}$): $v$(OH) at 3426 cm$^{-1}$, $v$(C=O) in the region 1780–1750 cm$^{-1}$, and glucosydic $v$(C–O–C) at 1159 cm$^{-1}$; $^1$H-NMR (400 MHz/DMSO-$d_6$): δ 1.34 (m, 8H, 4CH$_2$), 2.55 (t, 4H, 2CH$_2$), 3.31–4.02 (CH-protons of β-CD + 4H, CH $sp^3$), 4.84 (H-1 of β-CD), 5.60 (broad s, –OH, OH secondary); $^{13}$C NMR (DMSO-$d_6$): δ 18.85, 25.07, 27.17, 54.62, 56.57, 60.49, 72.53, 72.89, 73.55, 80.01, 82.06, 102.41, 103.60, 131.88, 168.02 ppm.

### 2.3.2 Synthesis of [2][succinic dihydrazide]-rota-[β-cyclodextrin] (2)

β-Cyclodextrin (4 g, 3.53 mmol) was dissolved in DMSO (20 ml). Succinic dihydrazide (0.52 g, 3.52 mol) was added with stirring for 1 h. After that, (11*S*,15*R*)-9,10,11,15-tetrahydro-9,10-[3,4]furanoanthracene-12,14-dione (1.94 g, 7.04 mol) was added with stirring for 26 h at 80 °C. The product was filtered off and dried to give **2** as white crystals (4.55 g, 2.53 mol) in 72 % yield; m.p. above 360 °C; *Anal*. Calculated for $C_{82}H_{100}N_4O_{41}$ (1796.9): C, 54.81; H, 5.57; N, 3.12. Found: C, 54.78; H, 5.60; N, 3.22; FTIR (KBr, cm$^{-1}$): bands due to $v$(OH) at 3401 cm$^{-1}$, $v$(CH-aromatic) at 3012 cm$^{-1}$, $v$(C=O) in the region 1862–1727 cm$^{-1}$, and glucosydic $v$(C–O–C) at 1158 cm$^{-1}$; $^1$H-NMR (400 MHz/DMSO-$d_6$): δ 2.30 (4H, 2CH$_2$), 3.28–3.67 (CH-protons of β-CD + 4H, CH $sp^3$), 4.81 (H-1 of β-CD), 4.87 (4H, CH $sp^3$), 5.62 (broad s, –OH, OH secondary), 7.20–7.48 (m, 16H, arom. H), 10.08 (s, 2H, NH); $^{13}$C NMR (DMSO-$d_6$): δ 18.86, 27.88, 44.81, 46.48, 47.44, 48.36, 60.51, 72.54, 72.90, 73.56, 82.08, 102.43, 123.94, 124.69, 124.88, 125.20, 125.47, 125.76, 126.35, 126.83 127.05, 127.24, 127.55, 141.47, 142.18, 143.75, 172.81, 173.69 ppm.

### 2.3.3 Synthesis of [2][tetramethylenediamino]-rota-[β-cyc lodextrin] (3)

β-Cyclodextrin (3 g, 2.64 mmol) was dissolved in DMSO (15 ml). Tetramethylenediamine (0.47 ml, 2.64 mol) was added for 1 h, then 2,3-diphenylmaleic anhydride (1.32 g, 5.28 mol) was added with stirring for 45 h at 80 °C. The solid formed was filtered off and dried to give **3** as yellow crystals (3.25 g, 1.93 mol) in 73 % yield; m.p. above 350 °C; *Anal*. Calculated for $C_{78}H_{98}N_2O_{39}$ (1686.8): C, 55.54; H, 5.81; N, 1.66. Found: C, 55.50; H, 5.83; N, 1.69; FTIR (KBr, $cm^{-1}$): presence of bands due to $v$(OH) at 3407 $cm^{-1}$, $v$(CH-aromatic) at 2924 $cm^{-1}$, $v$(C=O) in the region 1823–1758 $cm^{-1}$, and glucosydic $v$(C–O–C) at 1159 $cm^{-1}$; $^1$H-NMR (400 MHz/DMSO-$d_6$): δ 1.51 (4H, 2CH$_2$), 3.15 (4H, 2CH$_2$), 3.32–3.67 (CH-protons of β-CD$^)$, 4.31 (broad s, –OH, OH primary), 4.85 (H-1 of β-CD), 5.61 (broad s, –OH, OH secondary), 7.41–7.47 (20H, arom. H); $^{13}$C NMR (DMSO-$d_6$): δ 18.81, 60.49, 72.52, 72.87, 73.55, 82.05, 102.41, 127.95, 128.23, 128.89, 129.15, 129.85, 130.02, 131.11, 136.55, 138.98, 165.53 ppm.

### 2.3.4 Synthesis of [2][sebacoyl amino]-rota-[β-cyclodextrin] (4)

β-Cyclodextrin (4 g, 3.53 mmol) was dissolved in DMSO (20 ml). Sebacoyl chloride (0.84 ml, 3.53 mol) was added with stirring for 2 h. (11*R*,15*S*)-13-amino-9,10-di-hydro-9,10-[3,4]epipyrroloanthracene-12,14-dione (2.04 g, 7.06 mol) was added with continuous stirring for 24 h at 80 °C. The precipitate formed was filtered off and dried to give **4** as white crystals (4.56 g, 2.42 mol) in 67 % yield; m.p. above 360 °C; Anal. Calculated for $C_{88}H_{112}N_4O_{41}$ (1880.96): C, 56.19; H, 5.95; N, 2.98. Found: C, 56.22; H, 5.91; N, 2.96; FTIR (KBr, $cm^{-1}$): presence of bands due to $v$(OH) at 3336 $cm^{-1}$, $v$(NH) at 3260 $cm^{-1}$, $v$(CH-aliphatic) at 2964 $cm^{-1}$, $v$(C=O) in the region 1771–1688 $cm^{-1}$, and glucosydic $v$(C–O–C) at 1157 $cm^{-1}$; $^1$H-NMR (400 MHz/DMSO-$d_6$) δ 1.27 (8H, 4CH$_2$), δ 1.50 (4H, 2CH$_2$), δ 2.19 (4H, 2CH$_2$), 2.55 (4H, CH $sp^3$), 3.16–3.67 (CH-protons of β-CD), 4.58 (broad s, –OH, OH primary), 4.76 (H-1 of β-CD), 4.85 (4H, CH $sp^3$); 5.60 (broad s, –OH, OH secondary), 7.11–7.46 (m, 16H, arom. H); $^{13}$C NMR (DMSO-$d_6$): δ 25.43, 29.02, 43.81, 44.81, 46.48, 47.44, 48.36, 60.51, 72.69, 72.90, 73.36, 82.75, 102.05, 123.43, 124.69, 124.88, 125.20, 125.47, 125.94, 126.35, 126.82, 127.05, 127.41, 127.76, 141.47, 142.18, 143.54, 172.90, 173.81 ppm.
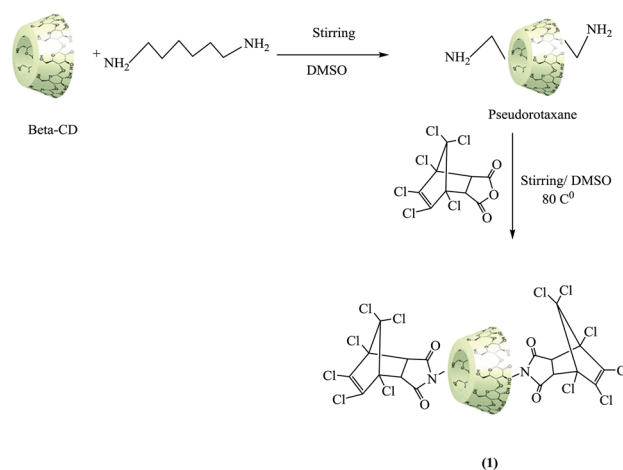


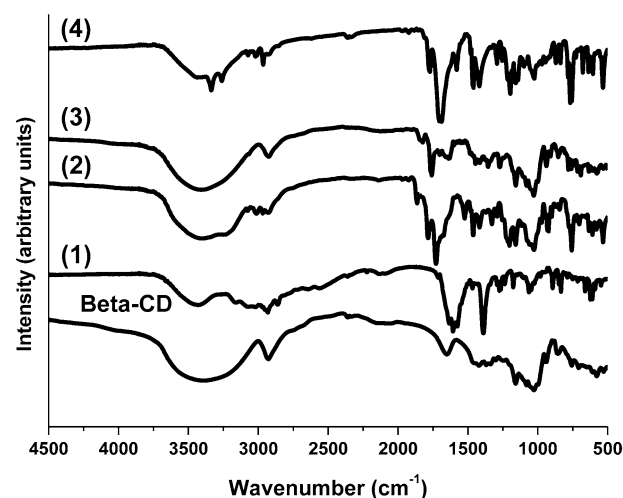Scheme 1 Synthetic route for the preparation of [2]rotaxane **1**



Fig. 1 FTIR spectra of pure β-CD and [2]rotaxanes **1**–**4**

## 3 Results and discussion

### 3.1 Synthesis and spectral analysis

[2][Hexamethylenediamino]-rota-[β-cyclodextrin] (**1**) was obtained by threading hexamethylenediamine (axis) into β-CD (host) to give [2]pseudorotaxane. 1,4,5,6,7,7-Hex-achloro-5-norbornene-2,3-dicarboxylic anhydride was added as cap to prevent slippage of the β-CD ring to form [2]rotaxane **1** (Scheme 1). The FTIR spectrum of [2]rotaxane **1** afforded remarkable confirmation of the structure with the appearance of distinctive absorption bands due

**Fig. 2** $^{1}$H NMR spectrum of [2] rotaxane **1** in DMSO-$d_6$
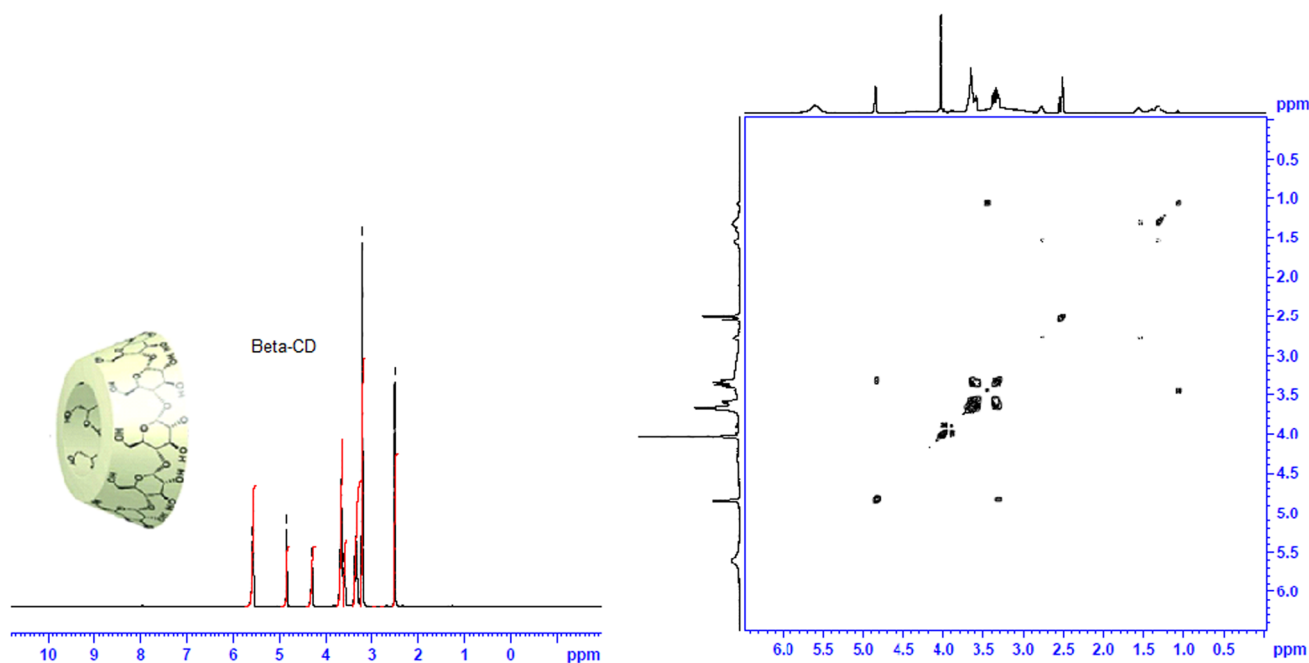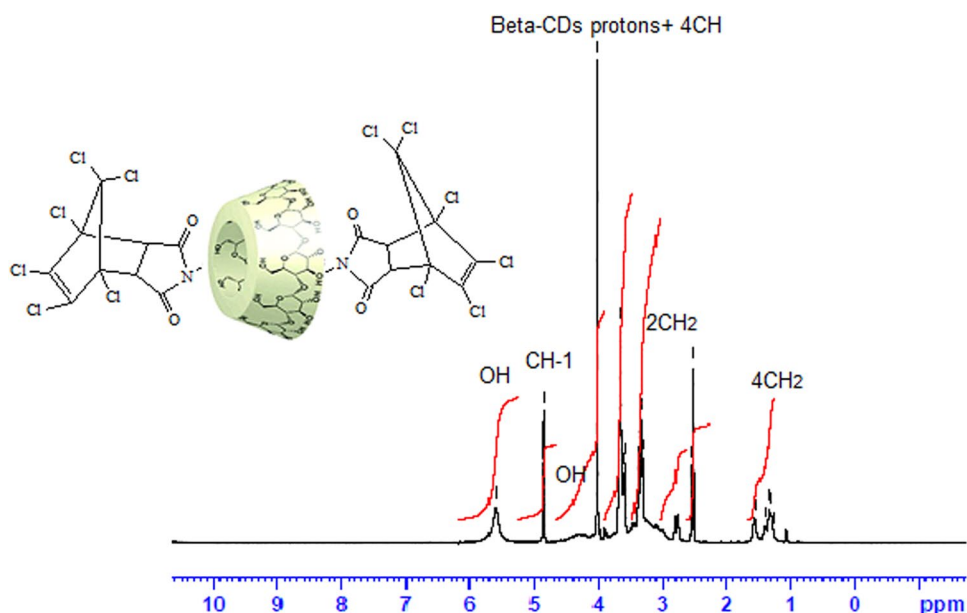


**Fig. 3** $^{1}$H NMR spectrum of β-CD and 2D COSY NMR spectrum of [2]rotaxane **1** in DMSO-$d_6$

to stretching of hydroxyl groups $v$(OH) at 3426 cm$^{-1}$ and characteristic band for glucosydic $v$(C–O–C) at 1159 cm$^{-1}$ (Fig. 1). The $^{1}$H NMR spectrum elucidated the chemical structure of [2]rotaxane **1**, since β-CD protons appeared in the region 3.31–5.60 ppm, in addition to signals due to 12 aliphatic protons (6CH$_2$) at 1.34 and 2.55 ppm (Fig. 2). Also, 2D COSY NMR gave a good indication of the formation of compound **1** (Fig. 3). The stoichiometry ratio for the preparation of the inclusion complex is 1:1. Table 1

**Table 1** Interpretation of signal intensities of pure β-CD and [2] rotaxane **1**

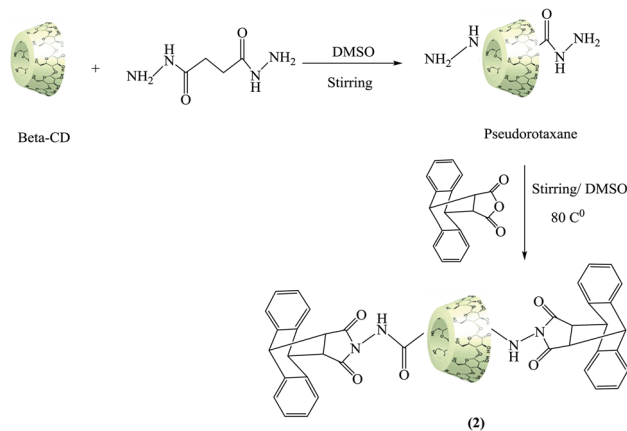| Functional group | Wavenumber (cm$^{-1}$) | | Change |
|---|---|---|---|
| | β-CD | 1 | Δδ |
| $v$(OH) stretching | 3389.28 | 3426.88 | 37.6 |
| $v$(CH$_2$) aliphatic | 2925.48 | 2929.06 | 3.58 |
| $v$(C–O–C) | 1158.55 | 1159.97 | 1.42 |
| $v$(O–H) bending vibration | 1027.87 | 1028.83 | 0.96 |

**Fig. 4** $^{13}$C NMR spectrum of [2] rotaxane **1** in DMSO-$d_6$
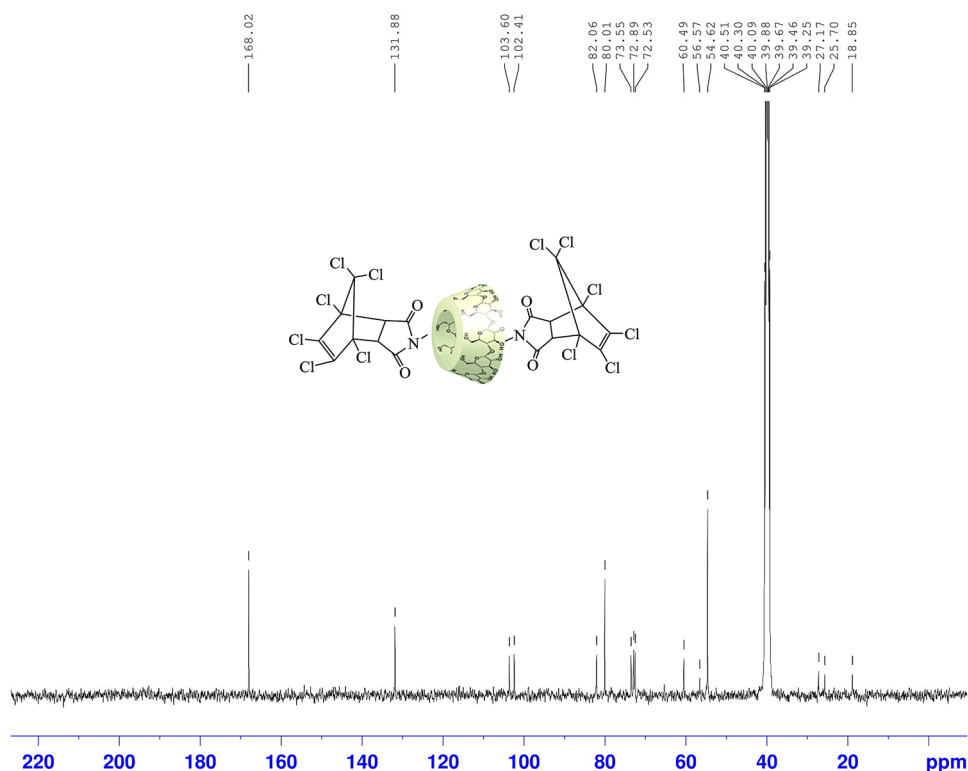


presents the shift in the absorption bands of pure β-CD before complexation and after formation of [2]rotaxane **1** (inclusion complex). This change in the absorption bands results from hydrophobic–hydrophilic and van der Waals interactions between the interior surface of the β-CD and hydrophobic positions on hexamethylenediamine (guest) [26, 27]. The $^{13}$C NMR spectrum of [2]rotaxane **1** provided good evidence for the formation of the inclusion complex **1**, showing three peaks due to aliphatic methylene carbons at 18.85, 25.07, and 27.17 ppm, as well as two signals due to 4CH $sp^3$ at shifts of 54.62 and 56.57 ppm and six different carbon atoms due to β-CD at 60.49, 72.53, 72.89, 73.55, 82.06, and 102.41 ppm, in addition to the appearance of a signal due to two carbons (C=C) at 80.01 ppm, a peak for two C–Cl at 103.60 ppm, a signal due to [C–(Cl)$_2$] at 131.88 ppm, and a signal due to (C=O) groups at 168.02 ppm (Fig. 4). Regarding the formation of amic acid, literature search did not show any sign of opening of the anhydride ring to give the free amic acid [36–40].

Scheme 2 indicates the mechanism for synthesis of [2][succinic dihydrazide]-rota-[β-cyclodextrin] (**2**). The succinic dihydrazide (rod) is inserted into the β-CD macromolecule to form [2]pseudorotaxane, then (11$S$,15$R$)-9,10,11,15-tetrahydro-9,10-[3,4]furanoanthracene-12,14-dione was added as bulky blocking group to attach to the ends of the axis. The FTIR spectrum



**Scheme 2** Synthetic route for the preparation of [2]rotaxane **2**

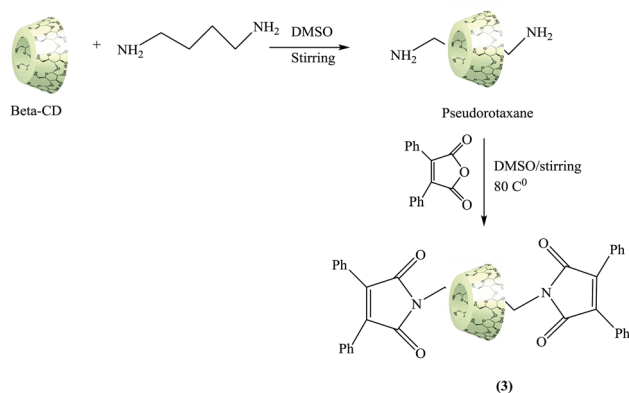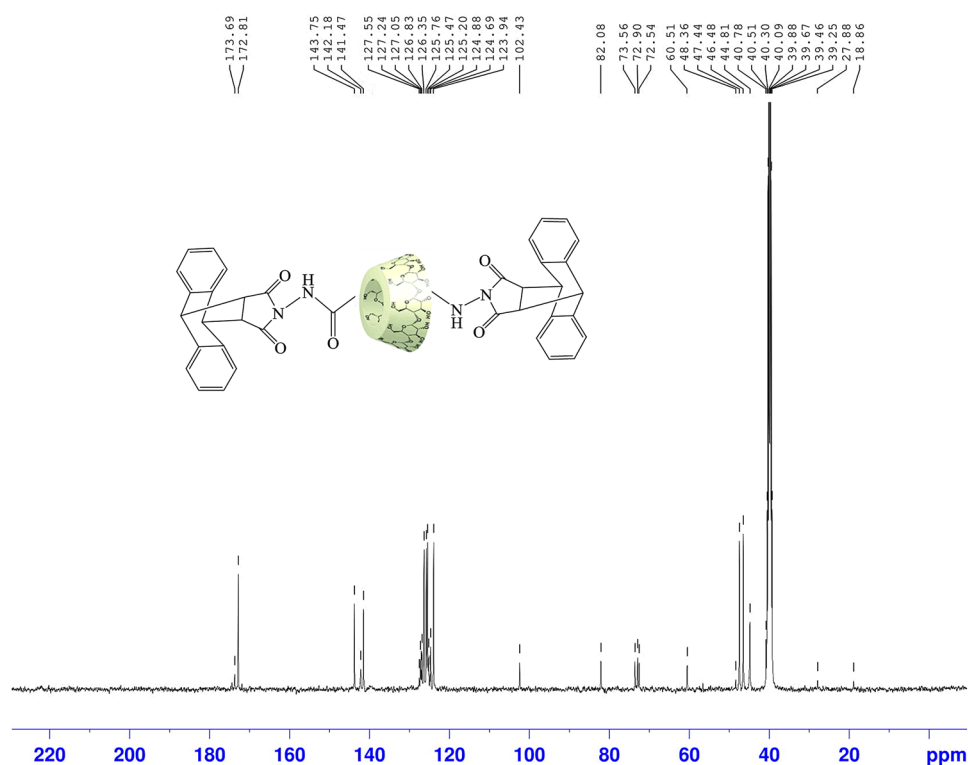**Table 2** Interpretation of signal intensities of pure β-CD and [2] rotaxane **2**

| Functional group | Wavenumber (cm$^{-1}$) | | Change |
|---|---|---|---|
| | β-CD | **2** | $\Delta\delta$ |
| $v$(OH) stretching | 3389.28 | 3401.81 | 12.53 |
| $v$(CH$_2$) aliphatic | 2925.48 | 2927.37 | 1.89 |
| $v$(C–O–C) | 1158.55 | 1158.04 | −0.51 |
| $v$(O–H) bending vibration | 1027.87 | 1029.80 | 1.93 |

**Fig. 5** $^1$H NMR spectrum of [2] rotaxane **2** in DMSO$d_6$
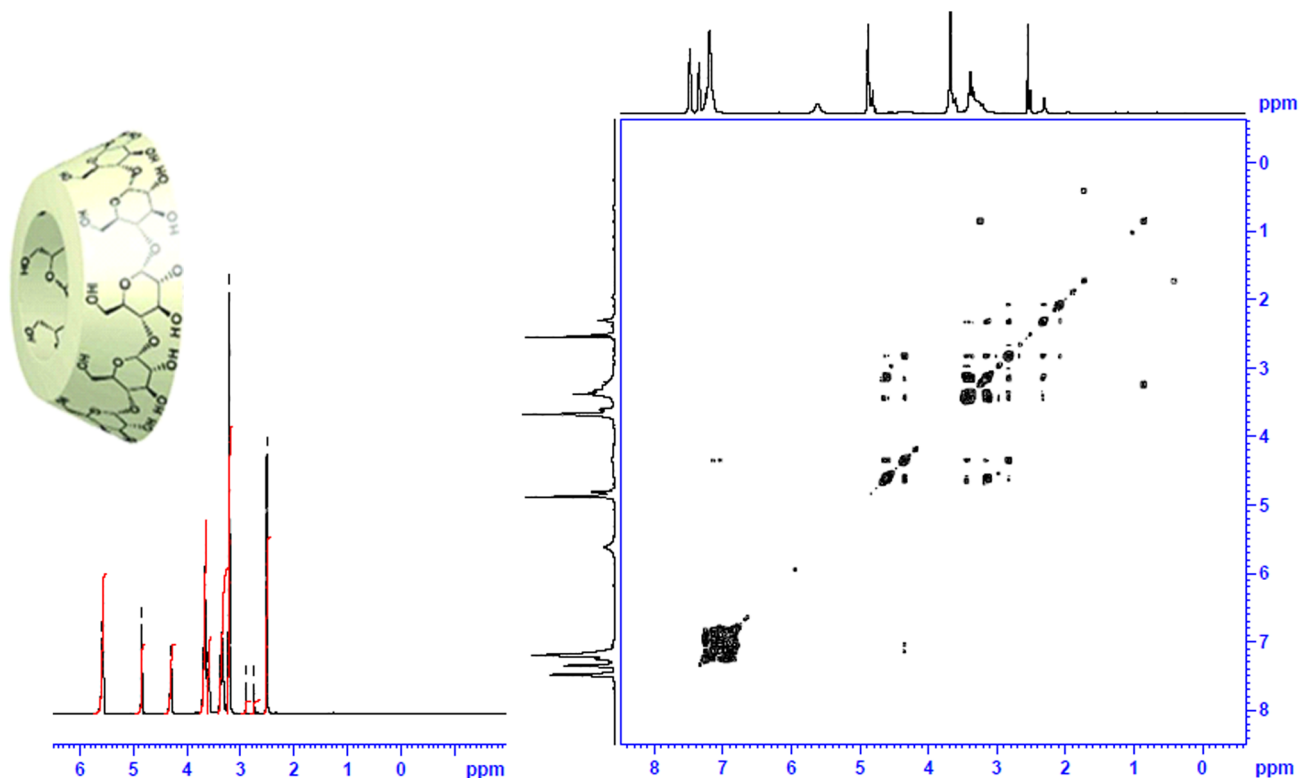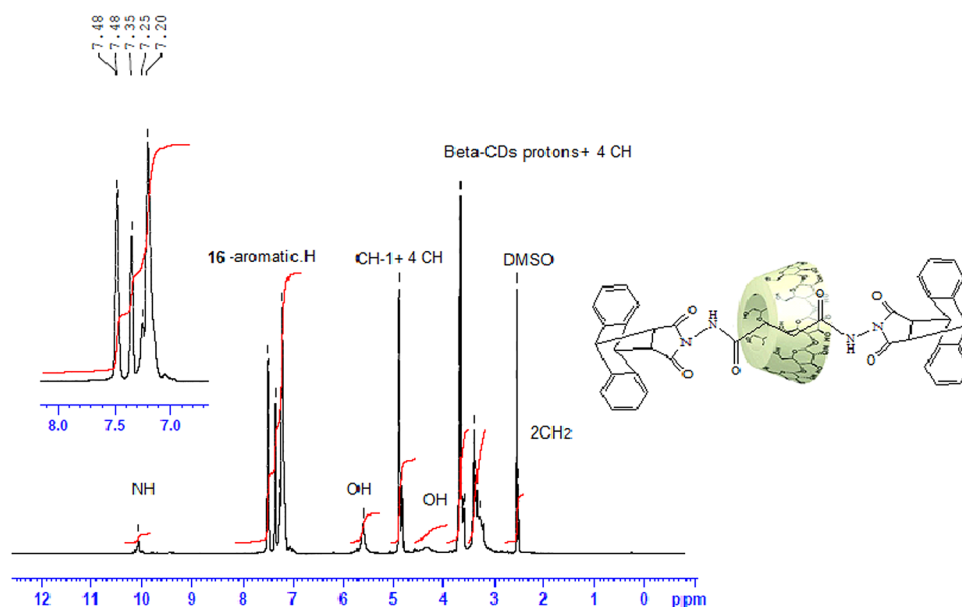


**Fig. 6** $^1$H NMR spectrum of β-CD and 2D COSY NMR spectrum of [2]rotaxane **2** in DMSO-$d_6$

of [2]rotaxane **2** afforded excellent confirmation of the formation of the inclusion complex between the β-CD ring (host) and succinic dihydrazide (guest). Figure 1 shows the characteristic absorption band due to hydroxyl groups of [2]rotaxane **2**, being sharper than

observed for pure β-CD [41]. The FTIR spectrum of [2] rotaxane **2** presented characteristic absorption bands due to aromatic CH at 3012 cm$^{-1}$ and carbonyl groups in the region 1862–1727 cm$^{-1}$. Table 2 describes the shift in the absorption bands after complexation, owing to

**Fig. 7** $^{13}$C NMR spectrum of [2]
rotaxane **2** in DMSO$d$-$_6$



**Scheme 3** Synthetic route for the preparation of [2]rotaxane **3**

**Table 3** Interpretation of signal intensities of pure β-CD and [2] rotaxane **3**

| Functional group | Wavenumber (cm$^{-1}$) | | Change |
| --- | --- | --- | --- |
| | β-CD | **3** | Δδ |
| $v$(OH) stretching | 3389.28 | 3407.60 | 18.32 |
| $v$(CH$_2$) aliphatic | 2925.48 | 2930.30 | 4.82 |
| $v$(C–O–C) | 1158.55 | 1159.79 | 1.24 |
| $v$(O–H) bending vibration | 1027.87 | 1030.76 | 2.89 |

intermolecular hydrogen bonding between the NH group of the guest molecule and the hydroxyl groups of the β-CD ring [27, 41, 42]. The $^1$H NMR spectrum in DMSO indicates a triplet at 2.55 ppm for two methylene protons, a singlet due to 4CH$sp^3$ at 3.60 ppm, a signal due to aliphatic protons of β-CD in the region 3.28–3.76 ppm, a signal due to 4CH$sp^3$ at 4.87 ppm, a multiplet due to aromatic protons at 7.20–7.48 ppm, and a singlet for NH protons at 10.08 ppm (Fig. 5). COSY NMR of compound **2** provided excellent indication of the formation of [2]

rotaxane **2** (Fig. 6). The $^{13}$C NMR spectrum of [2]rotaxane **2** provided great evidence of the suggested structure **2**, showing two peaks due to two carbons (CH$_2$) at 18.86 and 27.88 ppm and the appearance of four signals due to 8CH $sp^3$ at chemical shifts of 44.81, 46.48, 47.44, and 48.36 ppm. Also, six signals due to different carbon atoms of β-CD appeared at 60.51, 72.54, 72.90, 73.56, 82.08, and 102.43 ppm, in addition to signals for different aromatic carbons and due to two amidic carbonyl (CO–NH) at 142.18 and 143.75 ppm. Finally, signals due to C=O groups at 172.81 and 173.69 ppm were observed (Fig. 7).

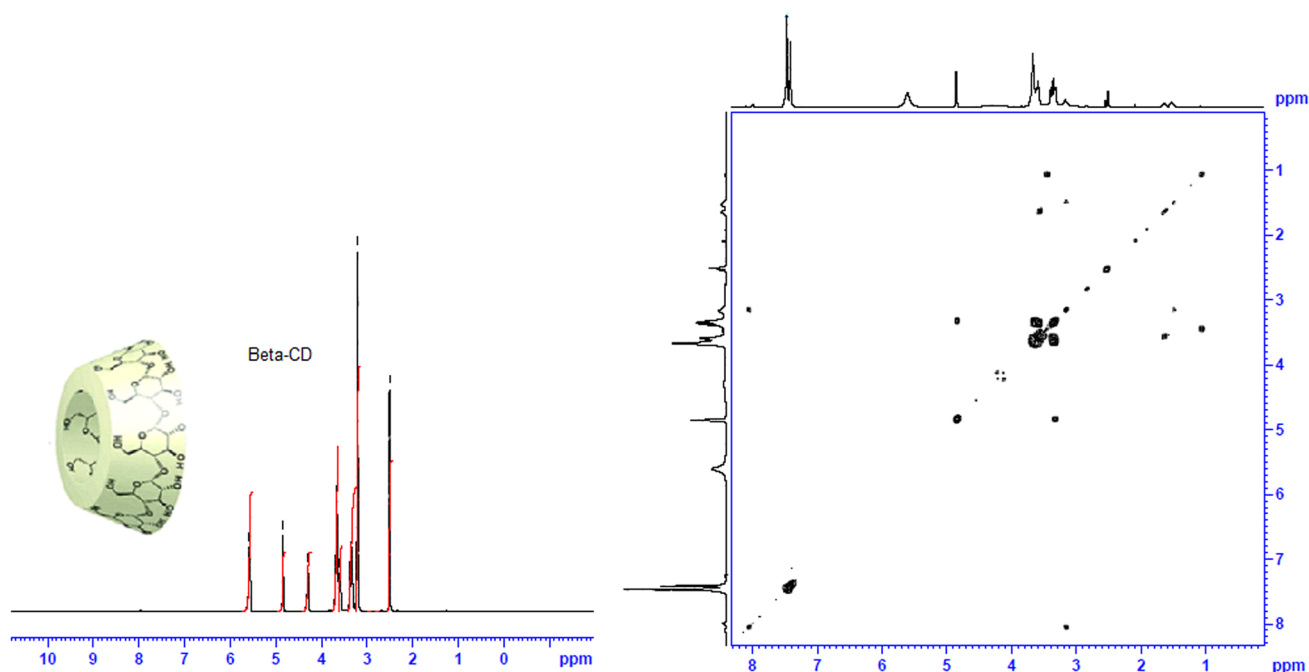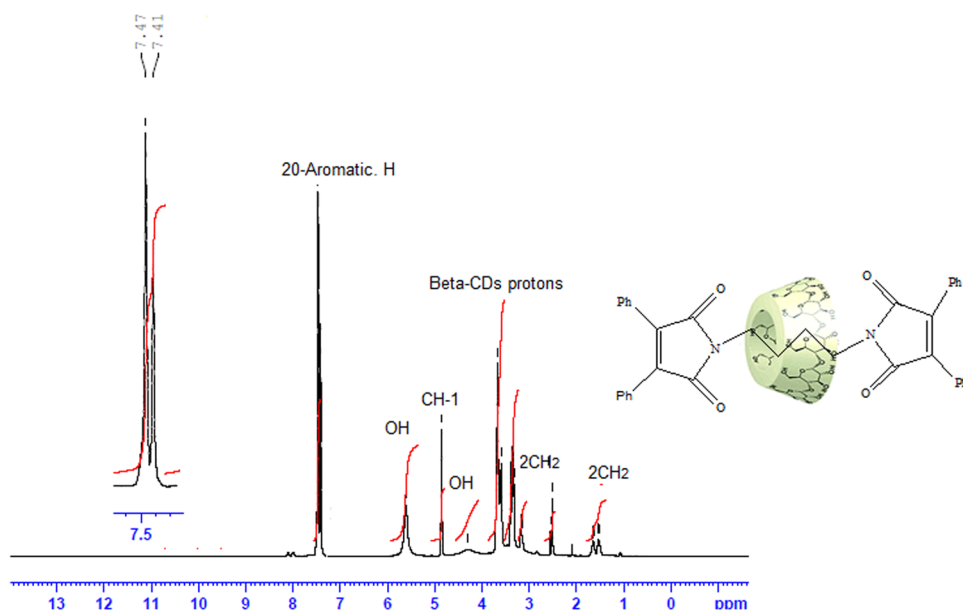[2][Tetramethylenediamino]-rota-[β-cyclodextrin] (**3**) was synthesized in two steps according to Scheme 3. The

**Fig. 8** $^1$H NMR spectrum of β-CD and 2D COSY NMR spectrum of [2]rotaxane **3** in DMSO-$d_6$

**Fig. 9** $^1$H NMR spectrum of [2]
rotaxane **3** in DMSO-$d_6$



aliphatic chain (tetramethylenediamine) was threaded into β-CD to give the [2]pseudorotaxane, then capped using 2,3-diphenylmaleic anhydride to afford the [2]rotaxane **3**. The FTIR spectrum revealed the presence of characteristic absorption bands due to hydroxyl groups at

3407 cm$^{-1}$, CH-aromatic at 2924 cm$^{-1}$, absorption bands due to carbonyl groups in the region 1823–1758 cm$^{-1}$, and glucosydic group at 1159 cm$^{-1}$ (Fig. 1; Table 3). Figure 8 shows the $^1$H NMR spectrum of β-CD and the 2D COSY NMR spectrum of [2]rotaxane **3** in DMSO. The $^1$H

**Fig. 10** $^{13}$C NMR spectrum of [2]rotaxane **3** in DMSO-$d_6$
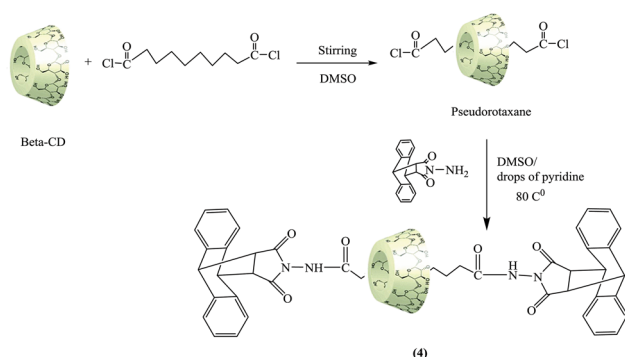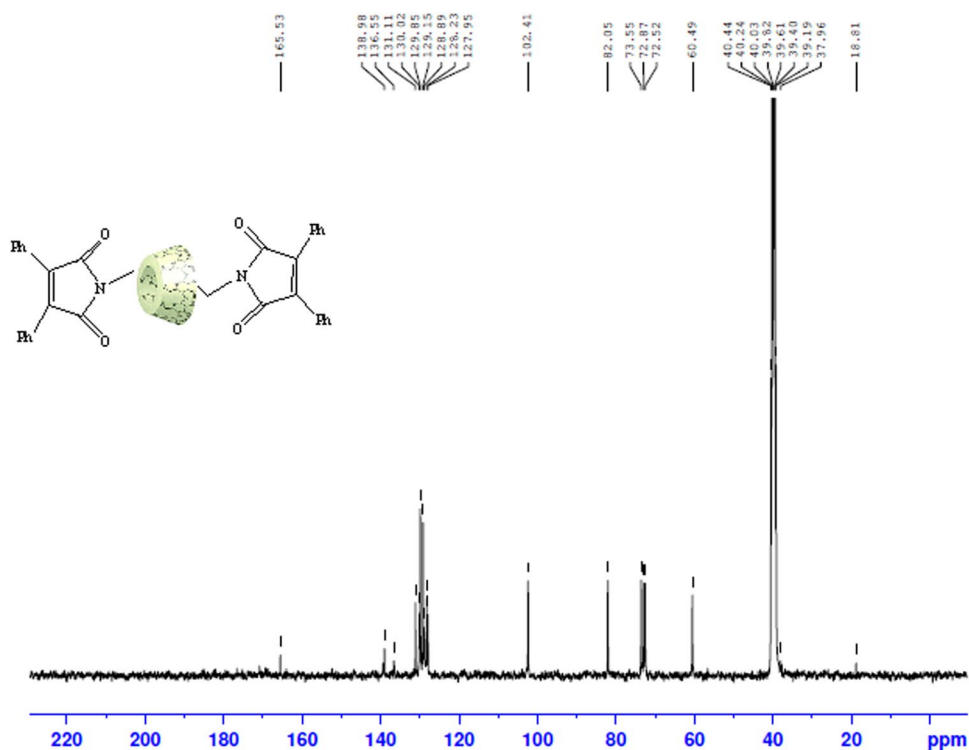




**Scheme 4** Synthetic route for the preparation of [2]rotaxane **4**

**Table 4** Interpretation of signal intensities of pure β-CD and [2]rotaxane **4**

| Functional group | Wavenumber (cm$^{-1}$) | | Change |
| --- | --- | --- | --- |
| | β-CD | **4** | Δδ |
| ν(OH) stretching | 3389.28 | 3336.24 | −53.04 |
| ν(CH$_2$) aliphatic | 2925.48 | 2928.37 | 2.89 |
| ν(C–O–C) | 1158.55 | 1159.9 | 1.35 |
| ν(O–H) bending vibration | 1027.87 | 1029.8 | 1.93 |

NMR spectrum of [2]rotaxane **3** confirms its chemical structure (Fig. 9), showing a triplet due to two methylene protons at δ 1.51 ppm, a triplet due to two methylene protons at 3.15 ppm, and C–H protons of β-CD at 3.32–3.67 ppm. Furthermore, the hydroxyl groups appear at δ 4.31 (OH primary) and δ 5.61 (OH secondary), and 20 aromatic protons at chemical shifts of 7.41–7.47 ppm. The $^{13}$C NMR spectrum of [2]rotaxane **3** in DMSO established its chemical structure, showing a signal due to four CH$_2$ carbons at 18.81 ppm and peaks for six different carbon atoms of β-CD at 60.49, 72.52, 72.87, 73.55, 82.05, and 102.41 ppm. Also, signals due to different aromatic carbons appeared, plus a signal due to C=O groups at 165.53 ppm (Fig. 10).

[2][Sebacoyl amino]-rota-[β-cyclodextrin] (**4**) was formed by insertion of sebacoyl chloride (thread) through β-CD to afford the [2]pseudorotaxane, which was capped by adding (11*R*,15*S*)-13-amino-9,10-dihydro-9,10-[3,4]epipyrroloanthracene-12,14-dione to form [2]rotaxane **4** (Scheme 4). The chemical structure of **4** was rigorously confirmed by microanalytical, FTIR, $^1$H, $^{13}$C, and 2D COSY NMR spectral analyses. The

**Fig. 11** $^1$H NMR spectrum of [2] rotaxane **4** in DMSO-$d_6$
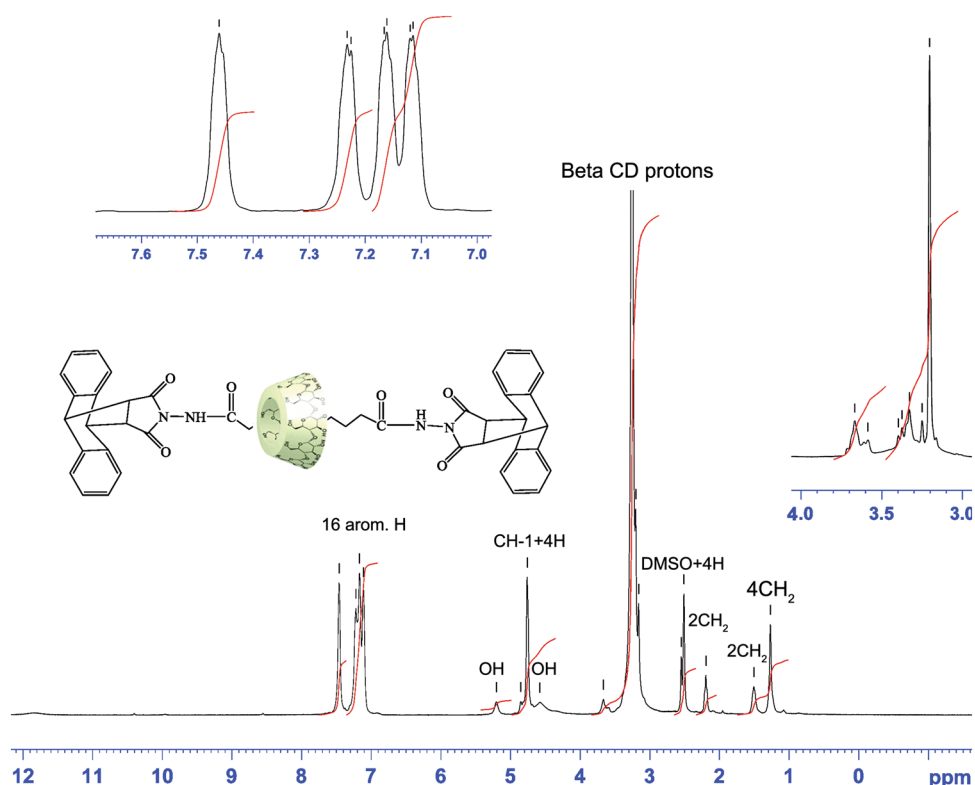


FTIR spectrum revealed typical absorption bands at 3336 cm$^{-1}$ for hydroxyl groups, 3260 cm$^{-1}$ for NH groups, 2964 cm$^{-1}$ due to aliphatic protons, absorption peaks due to carbonyl groups in the region of 1771–1688 cm$^{-1}$, and glucosydic group at 1157 cm$^{-1}$ (Fig. 1). Table 4



**Fig. 12** 2D COSY NMR spectrum of [2]rotaxane **3** in DMSO-$d_6$

presents the difference in the absorption bands after formation of the inclusion complex. The $^1$H NMR spectrum established the presence of β-CD protons at chemical shifts of 3.16–3.67 ppm, in addition to signals due to eight CH $sp^3$ at 2.55 and 4.85 ppm and signals for 16 aromatic protons at 7.11–7.46 ppm (Fig. 11). The COSY NMR spectrum provided additional confirmation of the formation of [2]rotaxane **4** (Fig. 12). The $^{13}$C NMR spectrum of [2]rotaxane **4** in DMSO demonstrated formation of the interlocked compound, showing three signals due to aliphatic carbons (8CH$_2$) at 25.43, 29.02, and 43.81 ppm, four signals due to eight CH $sp^3$, six signals due to different carbon atoms of β-CD at 60.51, 72.69, 72.90, 73.36, 82.75, and 102.05 ppm, and signals due to different aromatic carbons. In addition to the peaks due to amidic carbonyl at 142.18 and 143.54 ppm, signals due to C=O carbons were observed at 172.90 and 173.81 ppm (Fig. 13).
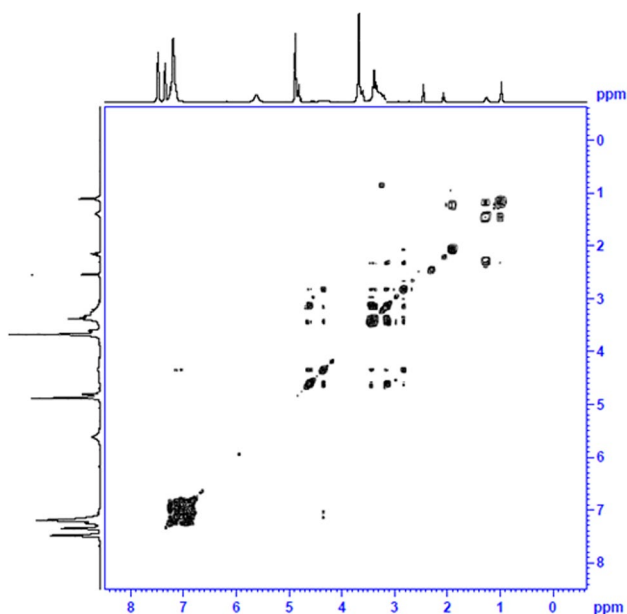
### 3.2 Surface morphology

The surface morphology of the obtained [2]rotaxanes **1**–**4** was examined by SEM. Figure 14 shows SEM images of pure β-CD and the [2]rotaxanes **1**–**4**, revealing a clear change in surface morphology. [2]Rotaxane **1** showed regular shape looking like crusts, [2]rotaxane **2** showed small circular shape, [2]rotaxane **3** had characteristic polygonal

**Fig. 13** $^{13}$C NMR spectrum of [2]rotaxane **4** in DMSO-$d_6$





Beta-CD



[2]rotaxane **(1)**



[2]rotaxane **(2)**



[2]rotaxane **(3)**
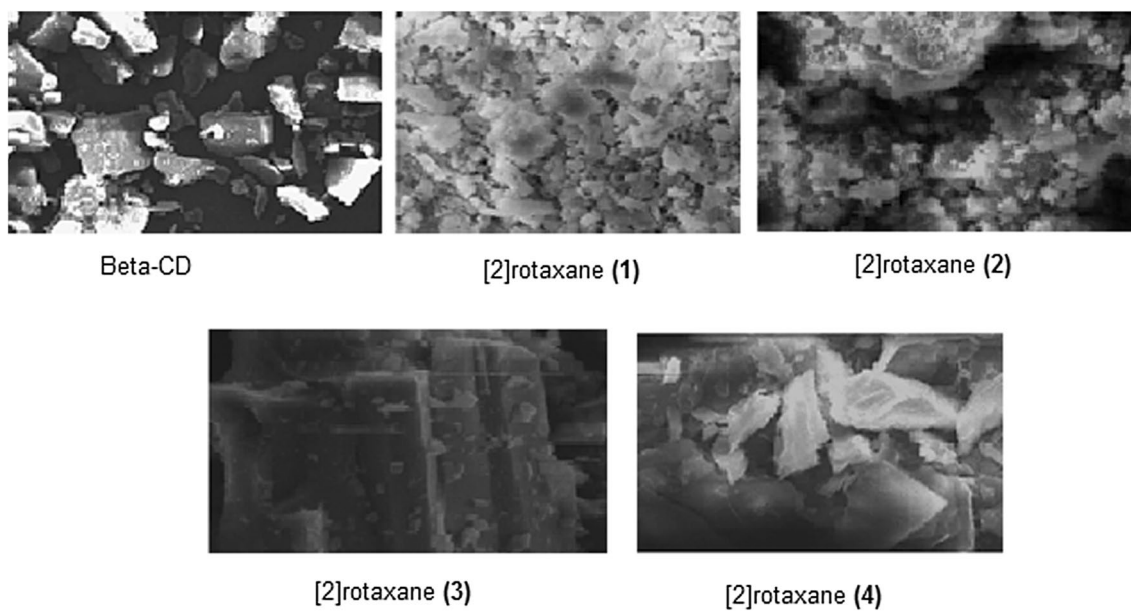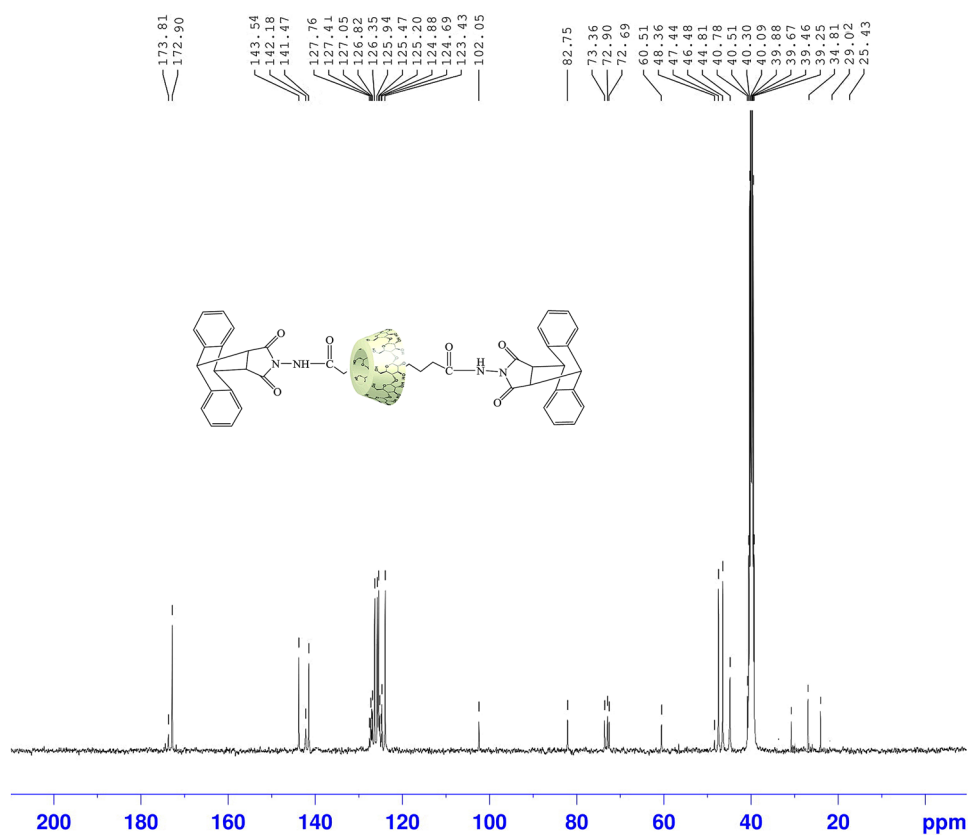


[2]rotaxane **(4)**

**Fig. 14** SEM images of β-CD and [2]rotaxanes **1**–**4**

shape, while the surface morphology of [2]rotaxane **4** was similar to cut glass. These differences in the morphology of the prepared [2]rotaxanes compared with β-CD afford additional evidence for the formation of the inclusion complexes.

## 4 Conclusions

Four new [2]rotaxanes **1**–**4** including β-cyclodextrin (β-CD) were synthesized and characterized. The main contribution of this study is the identification of a new end-capping reaction exploiting imidation reaction between the terminal amines of the axle and succinic anhydride derivatives as capping agents. The formation of these compounds depends on host–guest interaction, hydrogen bonding, and van der Waals forces. This type of compounds are well known for their unique structure consisting of mechanically interlocked threads and macrocycles. The chemical structures of the prepared [2]rotaxanes were confirmed by spectroscopic analysis, FTIR spectroscopy, and $^1$H, $^{13}$C, and 2D COSY NMR spectra. The stoichiometry for the formation of the inclusion complexes is 1:1 molar ratio. The morphology of the obtained [2]rotaxanes **1**–**4** was investigated to further elucidate their synthesis.

## Compliance with ethical standards

**Conflicts of interest** The author declares that she has no conflicts of interest.

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