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Design and synthesis of chalcone-based macrocyclic polyethers

Subrata Jana^{1*}

Abstract: A series of chalcone-based macrocyclic ethers have been synthesized. These macrocyclic ethers contain polyether as well as extended conjugation to the benzene ring to function as fluorescent sensor for cations. The modeling studies show that the chalcone part of the receptors remains partially out of plane from the polyether part and the keto moiety of the receptors always directed outwardly in the receptor itself. This may be changed during the recognition of metal ions. The tendency of the fluorophore, to be out of plane, increases with increasing the ring size.

Subjects: Chemical Spectroscopy; Computational and Theoretical Chemistry; Organic Chemistry

Keywords: molecular recognition; cation sensor; macrocyclic ether; chalcone

1. Introduction

Macrocyclic polyether compounds are very important class of compounds, which were discovered by Pedersen (1988). Besides the massive applications as phase transfer catalyst (Beer, Gale, & Smith, 1999; Steed & Atwood, 2000) these are the part of different synthetic receptors (Bühlmann, Pretsch, & Bakker, 1998; Hartley, James, & Ward, 2000), which act as metal ion as well as chiral sensors for amino acids (Bühlmann et al., 1998; Mandl & König, 2005). In case of metal ions, macrocyclic polyether binds the metal ions depending upon the size of the macrocyclic cavity of the crown ether and the ionic radius of the metal ions by electrostatic interactions (Steed & Atwood, 2000;

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Subrata Jana obtained his PhD in organic chemistry from Indian Institute of Engineering Science and Technology (IEST), Shibpur, India. His doctoral work was based on design and synthesis of abiotic receptors for the recognition of biologically active neutral molecules and ions along with development synthetic methodologies. After that he moved to University of Victoria, Canada, to work with Dr Fraser Hof on supramolecular and medicinal chemistry as a post-doctoral fellow. He then worked with Dr Kenneth J Woycechowsky at University of Utah, USA, on protein engineering and enzyme catalysis as post-doctoral research associate. Presently, he is working as associate professor at VEC, Sarguja University, Ambikapur, Chhattisgarh, India from 2013 and his current research focuses on design and synthesis of artificial receptors for the recognition of anions and N-methylated protein residue.

PUBLIC INTEREST STATEMENT

The present work focuses on the synthesis of different benzocrown ether-type compounds. These compounds are important for the recognition of alkaline metal ions with different size. The metal ions are the integral part of biological system of leaving organism and play an important role for biotic and abiotic system. So analytical detection of these ions is very crucial to measure the biological condition for living organism as well as environmental condition (water, soil, etc.) surrounds us. These compounds may be used as fluorescence sensor and can detect the metal ions in very low concentrations.

Table 1. Ionic radii (Å) and hydration enthalpies (kJ mol⁻¹) for alkali metal ions (Ichieda, Kasuno, Banu, & Kihara, 2003; Lide, 2005)

	Li(I)	Na(I)	K(I)	Rb(I)	Cs(I)
Hydration enthalpy	531	416	334	308	283
Coordination number	Ionic radius				
4	0.59	0.99	1.37	–	–
6	0.76	1.02	1.38	1.52	1.67
8	0.92	1.18	1.51	1.61	1.74

Table 1). So different types of receptors were designed and synthesized focusing the selectivity for a specific metal ion (Baruah et al., 2005; Haverlock, Mirzadeh, & Moyer, 2003). Simple crown ether or benzocrown ethers are also attached with different receptors which act as sensor for the salt recognition (Mahoney et al., 2005). The other important aspects of the crown ether are that the receptors containing more than one such unit could effectively recognize the guest substrate containing di/poly ammonium moiety (Hansson, Norrby, & Wärnmark, 1998; Manjula & Nagarajan, 1997; Prevot-Halter & Weiss, 1998). The slightly modified crown ether part was also used to synthesize “N” containing macrocycles (azacrown), which are important for the synthesis and studies as synthetic ion channels (Cazacu, Tong, van der Lee, Fyles, & Barboiu, 2006; Fyles, 2007) as the metal ions play crucial role in different biological processes (Liang, Campopiano, & Sadler, 2007).

2. Results and discussion

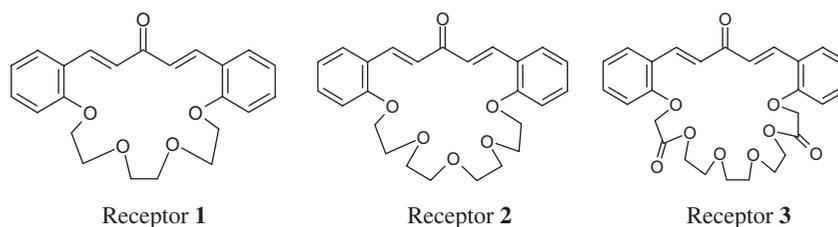
Benzocrown ether compounds containing diversified structural features are designed, synthesized, and studied towards the recognition behavior for alkaline metal ions with different size (Gibson et al., 2005; Huang, Zakharov, Rheingold, Ashraf-Khorassani, & Gibson, 2005). Generally aliphatic ether part is attached with *ortho* position of the benzene rings to form a benzocrown containing receptors for the recognition of metal ions and another guest specific recognition site as well as fluorophore attached with the benzene ring for the sensing of other substances. So for detection of metal ions by the fluorescence sensing, crown ether as well as a fluorophore is an essential part of the receptors, which are linked with suitable spacers. Besides this cyclohexanone and other suitable spacers are included in the macrocyclic structures (Higham, Kreher, Raston, Scott, & Strauss, 2004).

In this work, receptors are designed where fluorophore is the part of macrocyclic crown ether (intrinsic) (Figure 1). The polyether part in these receptors is attached with benzene rings through the ether linkage whereas another part of the macrocycle is the α,β -unsaturated keto moiety which is connected to the benzene rings. This α,β -unsaturated keto moiety is responsible for the fluorescence change upon complexation with metal ions (Bell & Hext, 2004; Marcotte & Fery-Forgues, 2000; Marcotte, Fery-Forgues, & Lavabre, 1999).

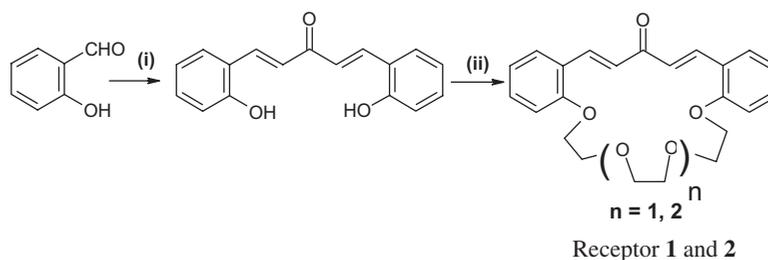
2.1. Synthesis

The synthesis of cyclic ether compounds containing conjugated phenyl and carbonyl compounds drew much attention for their structural diversity and application for the cation recognition process (Higham et al., 2004; Mondal, Mandal, & Mallik, 2012; Mondal, Samanta, Sarkar, & Mallik, 2014; Ovchinnikova et al., 2011). There are different approaches which were reported for the synthesis of

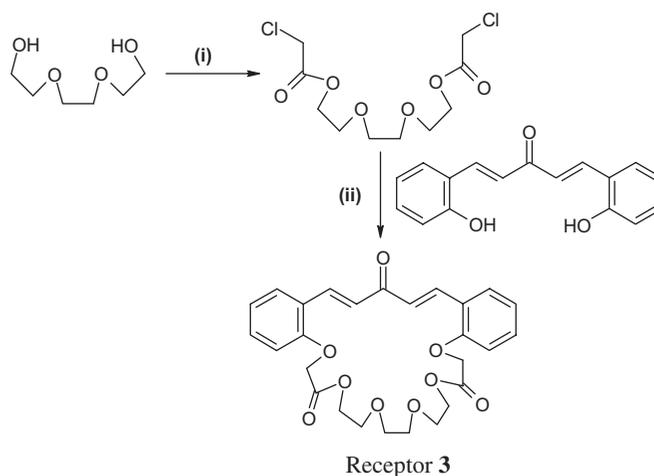
Figure 1. Dibenzylidene acetone containing crown ethers.



Scheme 1. Reagents and conditions: (i) acetone (0.5 eqv.), 1:1 EtOH and 5 M NaOH solution, 15–20°C, 4 h; reaction mixture was acidified with acetic acid when deep red color turns to yellow; (ii) tri/tetra ethylene glycol ditosylate, K₂CO₃, TBAB, Acetone, r.t., 2 days.



Scheme 2. Reagents and conditions: (i) chloroacetyl chloride (2.2 eqv.), dry CH₂Cl₂, Et₃N, r.t., 10 h; (ii) dihydroxy dibenzylidene acetone (1 eqv.), K₂CO₃ (5 eqv.), TBAB, acetone, r.t., 36 h.



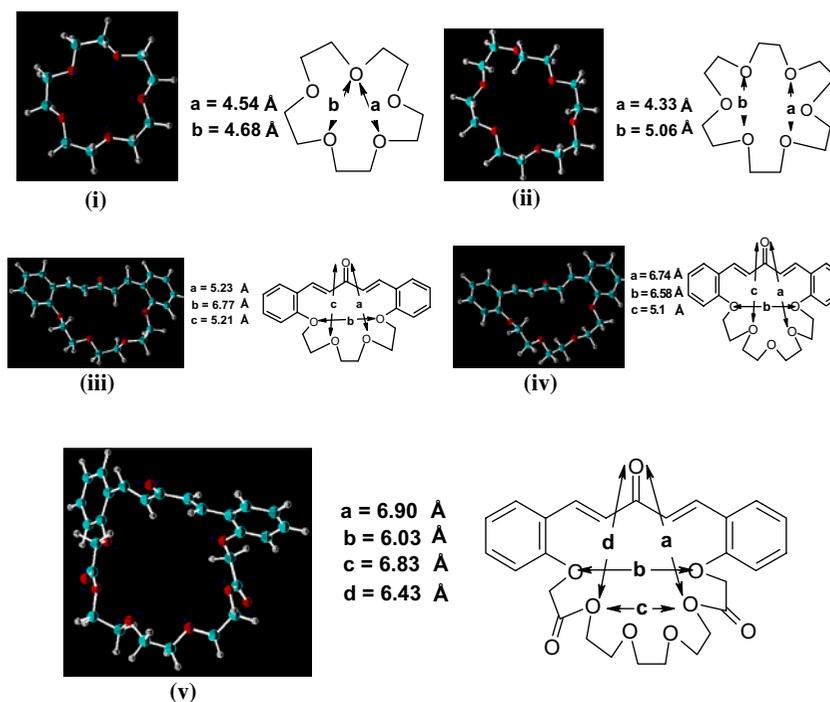
macrocyclic polyether compounds. Among these, most convenient approach was followed according to Scheme 1. The hydroxy-substituted dibenzylidene acetone was synthesized initially from the direct condensation of salicylaldehyde and acetone under alkaline alcoholic condition at low temperature (15–20°C) (Shriner & Shriner, 1976). After condensation dihydroxy dibenzylidene acetone remains in deep red colored quinoid form of the compound in solution. The dihydroxy dibenzylidene acetone was obtained with acidification by slow addition of cold glacial acetic acid. The macrocyclic polyether compounds were synthesized from di(2-hydroxybenzylidene)acetone and tri/tetra ethylene glycol ditosylate (Receptors 1 and 2) with K₂CO₃ and acetone at room temperature. The structures of the macrocyclic receptors (1 and 2) were confirmed by the spectral data. The HRMS spectra of the receptors 1 and 2 are interesting. In case of receptor 1, base peak was found at 783.3133 [(2M + Na)⁺] whereas in case of receptor 2, it was 447.1782 [(M + Na)⁺]. Besides this other peak at 1163.4798 [(3M + Na)⁺] was observed in case of receptor 1 but no such peak was observed in case of receptor 2.

The receptor 3 was synthesized from the dichloro polyether ester and dibenzylidene acetone (Scheme 2). The dichloro polyether ester was synthesized initially from chloroacetyl chloride and triethylene glycol in dry dichloromethane and triethyl amine. The receptor 3 has some special features than the receptors 1 and 2. The receptors 1 and 2 remain stable in purified form whereas receptor 3 turns brown after a few days and finally turns black. This probably happens due to the trapping of the solvent acetone inside the hydrated macrocyclic cavity of the receptor 3, which is removed in keeping at the room temperature for few weeks. This result may be drawn from the HRMS of the receptor 3 where 3·H₂O·(CH₃)CO is the base peak with 100% intensity.

2.2. Model study

The energy minimization of the macrocyclic receptors was carried out to study the actual orientation of the oxygen atoms of the receptors. The minimum energy (E_{\min}) of the receptors 1, 2, and 3 are

Figure 2. Energy minimized forms and the probable distances: (i) 15-crown-5, (ii) 18-crown-6, (iii) receptor 1, (iv) receptor 2, and (v) receptor 3.



47.98, 47.06, and 50.83 kJ mol⁻¹, respectively. From similar calculation, 15-crown-5 and 18-crown-6 ethers show the minimum energy (E_{\min}) of 18.74 and 20.4 kJ mol⁻¹, respectively, which are lower than the receptors. This is probably due to the strain of the α,β -unsaturation in the macrocyclic ring. In all the cases, carbonyl oxygen atom of the receptors is directed outwardly which may be moved inward during complexation with metal cations. The distances between the first oxygen atom and the third oxygen atom of the macrocyclic receptors were calculated by PC Model software (PCMODEL Serena Software 1993).

We have compared the approximate distances (Figure 2) of the receptors with the 15-crown-5 and 18-crown-6 ethers to get the idea of the selectivity for particular metal ions as the 15-crown-5 selectively binds Na⁺ and 18-crown-6 shows better selectivity towards K⁺ (Steed & Atwood, 2000). In case of the receptor 1, the distances are close to the 15-crown-5 but the structural dissimilarity may force the receptor 1 for different modes of binding. This may be preliminarily inferred from the HRMS spectra of the receptor 1 where one Na⁺ ion may form a sandwich-type complex with two molecules of receptor 1. But the distances in case of receptor 2 is comparatively higher than 18-crown-6. Though the presence of same structural dissimilarity due to the presence of α,β -unsaturated keto moiety force the receptor 2 to show greater selectivity for Na⁺ than the receptor 1.

3. Conclusion

In the present work, three receptors were synthesized by common protocol. The common features of these receptors are the presence of fluorophore due to the extended conjugation with benzene ring. The preliminary results related to the binding behavior might be guessed from the model study and HRMS data. In all the cases, α,β -unsaturations are in trans-trans (*E, E*) form which were observed in the complex in one case (Receptor 3 and NaClO₄). So the receptors will bind metal ions in its present form.

4. Experimental

4.1. General procedure for the synthesis of polyether ditosylate

Tri/tetra ethylene glycol (0.1 mol) was stirred and cooled with triethyl amine (50 mL) in ice-salt bath. After cooling, the reaction mixture tosylchloride (0.22 mol) was added slowly with vigorous stirring.

The reaction was continued for 5–6 h and kept in the refrigerator overnight. The reaction mixture was then poured in ice water and the precipitate (for triethylene glycol ditosylate) was filtered out with repeated washing by cold water to remove the excess triethyl amine and triethyl ammonium hydrochloride. The solid was dried and recrystallized from hot ethanol. The product was obtained in very good yield (85%) and melting point matched with reported data (76–78°C). But in case of tetraethylene glycol ditosylate, the reaction crude was in dense liquid and washed with ice water and purified with column chromatography (silica gel: 100–200 mesh and 8% EtOAc in pet ether). These ditosylates were used for the synthesis of macrocyclic compounds (Receptors **1** and **2**) with synthesized chalcone.

4.2. General procedure for the synthesis of chalcone-based macrocyclic polyether compounds (receptors **1** and **2**)

One equivalent of chalcone and ditosylate of tri/tetra ethylene glycol were stirred at room temperature in acetone in presence of K_2CO_3 (5.0 eqv.) and TBAB (0.1 eqv.) for two days. Reaction was monitored from time to time. After completion of the reaction, acetone was distilled off and extracted with $CHCl_3$ (20 mL \times 4) after washing with water and the solvent was dried in presence of anhydrous Na_2SO_4 and concentrated under reduced pressure to afford yellow colored solid crude product. This crude product was purified by column chromatography using silica gel (100–200 mesh) and EtOAc/pet ether (1:5 v/v) mixture of required polarity as eluant. The spectral data are as follows and the yield of the compounds is mentioned with the spectral data.

4.3. 13,16,19,22-Tetraoxa-tricyclo[21.4.0.0^{7,12}]heptacos-1(23),2,5,7(12),8,10,24,26-octaen-4-one (receptor **1**)

Yield: 42%.

Mp.: 84–86°C.

1H NMR ($CDCl_3$, 300 MHz): δ (ppm): 7.79 (d, 2H, $J = 15.9$ Hz), 7.54 (d, 2H, $J = 15.9$ Hz), 7.47 (d, 2H, $J = 7.7$ Hz), 7.31 (t, 2H, $J = 7.8$ Hz), 6.99 (t, 2H, $J = 7.5$ Hz), 6.90 (d, 2H, $J = 8.3$ Hz), 4.25 (t, 4H, $J = 4.4$ Hz), 3.98 (t, 4H, $J = 4.4$ Hz), 3.81 (s, 4H).

^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm): 191.75, 158.7, 140.31, 133.96, 131.4, 129.3, 124.61, 121.43, 112.02, 71.29, 69.71, 67.84.

FT-IR (KBr): 3446, 2924, 2871, 1667, 1615, 1569, 1493, 1339, 1259, 1122, 992, 943, 745 cm^{-1} .

MS (HRMS-ESI): m/z (%): Calculated for $C_{23}H_{24}O_5Na$ is 403.1516, found 403.1525 (10), 783.3133 [(2M + Na)⁺, 100], 800.2985 (8), 1163.4798 [(3M + Na)⁺, 20].

Anal. Calcd for $C_{23}H_{24}O_5$; C, 72.61; H, 6.36. Found: C, 72.81; H, 6.33.

4.4. 13,16,19,22,25-Pentaoxa-tricyclo[24.4.0.0^{7,12}]triaconta-1(26),2,5,7(12),8,10,27,29-octaen-4-one (receptor **2**)

Yield: 29%.

Mp.: 83–85°C.

1H NMR ($CDCl_3$, 500 MHz): δ (ppm): 7.89 (d, 2H, $J = 16.1$ Hz), 7.51 (dd, 2H, $J = 7.7$ Hz), 7.33 (t, 2H, $J =$), 7.25 (d, 2H, $J = 16.1$ Hz), 6.99 (t, 2H, $J =$), 6.94 (d, 2H, $J = 8.3$ Hz), 4.46 (t, 4H, $J = 4.7$ Hz), 3.94 (t, 4H, $J = 4.7$ Hz), 3.68 (dd, 4H, $J = 4.2, 2.5$ Hz), 3.62 (dd, 4H, $J = 2.5, 4.0$ Hz).

^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm): 191.74, 158.37, 139.83, 131.57, 131.15, 128.26, 125.21, 121.61, 113.21, 71.67, 71.23, 70.14, 68.96.

FT-IR (KBr): 3433, 2924, 1653, 1617, 1486, 1448, 1334, 1249, 1104, 749 cm^{-1} .

MS (HRMS-ESI): m/z (%): Calculated for $C_{25}H_{28}O_6Na$ is 447.1783, found 447.1782 (100), 425.2064 (M + H⁺, 66), 463.1650 (M + K, 32).

Anal. Calcd for C₂₅H₂₈O₆: C, 70.74; H, 6.65. Found: C, 70.81; H, 6.62.

4.5. 13,16,19,22,25,28-Hexaoxa-tricyclo[27.4.0.0^{7,12}]trtriaconta-1(29),2,5,7(12),8,10,30,32-octaene-4,15,26-trione (receptor 3)

Yield: 32%.

Mp.: 86–88°C.

¹H NMR (CDCl₃, 300 MHz): δ (ppm): 7.85 (d, 2H, J = 16.4 Hz), 7.49 (d, 2H, J = 7.5 Hz), 7.27 (t, 2H, J = 7.4 Hz), 7.03 (d, 2H, J = 16.5 Hz), 6.98 (d, 2H, J = 7.9 Hz), 6.94 (t, 2H, J = 8.5 Hz), 4.75 (s, 4H), 4.39 (s, 4H), 3.74 (s, 4H), 3.63 (s, 4H), 2.44 (s, 6H, trapped solvent acetone).

¹³C NMR (CDCl₃, 125 MHz): δ (ppm): 201.50, 156.45, 141.15, 132.31, 130.06, 128.14, 121.93, 121.08, 116.99, 70.93, 69.33, 65.94, 27.23.

MS (HRMS-FAB): m/z (%): 514 [(M + H₂O)⁺, 5], 577 [(M + acetone + Na)⁺, 100], 578 (32), 593 (14).

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Note

The minimization of energy was carried out by MMX (PCMODEL Serena Software 1993). Molecular modeling was performed using standard constants and the dielectric constant was maintained at 1.5.

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