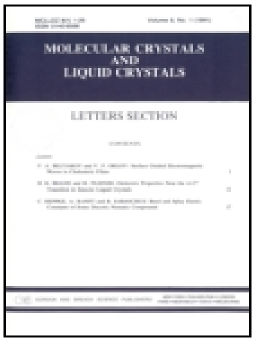

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The Effects of Molecular Conformation and Chain Length in Complexes of Tetra-*n*-alkylammonium Halide with Nonplanar Aromatic Molecule

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The crystal structures of tetrabutyl- and tetrapentylammionium bromide $(C_4H_9)_4N^+Br^-$, *and* $(C_5H_{11})_4N^+Br^-$ *complexes with rac-1,1′-bi-2-naphthol* $(C_{20}H_{14}O_2)$*; TBAB/BNP and TPAB/BNP, respectively, have been solved by X-ray diffraction techniques. The molecules of TBAB/BNP and TPAB/BNP are held in their aggregates by strong hydrogen bond (O-H... Br) and weak hydrogen bonds; C-H... O, C-H... Br, and C-H...* π*. The packing structures are different from each other, as well as those observed in tetrabutylammonium bromide with planar aromatic molecules. The tetrabutylammonium cation exhibits conformational and orientational disorder in TBAB/BNP complex, while in TPAB/BNP the alkyl chains of the tetrapentylammonium cation adopt biradial conformation in which there are two sets of paired chains. The tetrapentylammonium cations in TPAB/BNP completely entrap the BNP molecules in a manner that is attributed to the effect of increase in chain length as well as influence of biradial conformation in the tetrapentylammonium cation. However, in TBAB/BNP the naphthol planes of BNP form a void and sandwiches the tetrabutylammonium cation. The patterns of hydrogen bonding network as well as conformation of the tetra-*n*-akylammonium cations contribute to the formation of the shape of the voids that entrap either BNP or tetrabutylammonium cation. The molecular conformation of the tetra-*n*-alkylammonium bromides does affect the packing fashion of the nonplanar-shaped BNP molecule and vice versa.*

Keywords Conformation; complexes; rac-1,1′-bi-2-naphthol; tetrabutylammonium bromide; tetrapentylammonium bromide; chain length

Introduction

Tetraalkylammonium halides ($R_4N^+.X^-$) are widely used as reagents for the cleavage of silyl ethers or other silyl protecting groups [1], as catalysts for various reactions with silicon compounds [2], in the synthesis of ethers and as bases in organic synthesis [3]. They are also of interest due to different combinations of conformational and orientational disorder possible, depending on the lengths of the alkyl chains. The molecular aggregation between alkylammonium salts and organic molecules is of importance in the mechanistic study of

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the role of membranes [4], and separation of phenol derivatives [5]. Since, the interactions between alkylammonium halides and organic molecules play important roles in biological systems, separation science and technology, the complexes of alkylammonium halides with organic molecules are being studied by many supramolecular chemists.

Thus far, many hydrogen bonding patterns and packing fashions in complexes of *di-,tri-* alkylammonium halides with planar and nonplanar aromatic molecules that can be used for manipulation of the spatial arrangement of molecules in the solid-state have been identified and reported [6–10]. However, in respect to research studies on complexes of tetra-*n*-alkylammonium halides ($n = 4, 5, \ldots$) with nonplanar aromatic molecules, no studies have ever been done as per revealed by *Cambridge Structural Database version 5.34, 2013.* A further search of the Cambridge Structural Database for tetrabutylammonium halides ($R_4N^+.X^-$, where $R =$ butyl and X = Br or Cl) with phenol derivatives and other planar or nonplanar aromatic molecules revealed that only two complexes viz; tetrabutylammonium bromide complex with phenol [11] and catechol [12] have been reported.

Recently, we have succeeded in obtaining crystal complexes formed between tetrabutylammonium bromide (TBAB), and rac-1,1′-bi-2-naphthol (BNP); TBAB/BNP, as well as tetrapentylammonium bromide (TPAB) with BNP; TPAB/BNP, and investigated how the effects of increase in alkyl chain length and molecular conformation of the tetra-*n*-alkylammonium cation can change the packing fashion of the naphthol moieties of the BNP molecule as well as how the shape of BNP can influence a change in conformation of the alkyl chains of alkylammonium cations. Furthermore, how the H-bond network contributes significantly to the formation of voids in which BNP or the tetra-*n*-alklyammonium halide may be caged is investigated. A comparison of both crystal complexes with those reported on tetrabutylammonium bromide with phenol and catechol are also considered to understand the molecular assembly phenomenon of nonplanar aromatic molecules with tetra-*n*-alkylammonium halides. Thus, in this manuscript the aforementioned interest in the crystal structures of TBAB/BNP and TPAB/BNP complexes are discussed.

Experimental

Preparation of Complexes

All reagents were purchased from Aldrich and used without further purification. X-ray quality crystal complexes of TBAB/BNP, and TPAB/BNP were grown by dissolving mixed TBAB and BNP as well as TPAB and BNP in 1:1 stoichiometry ratio in acetone solution, warmed and left for slow evaporation. Suitable crystals were obtained after 3 days and characterized through single crystals X-ray diffraction at 150 K.

Data Collection and Structure Determination

The single crystal X-ray diffraction data of both complexes were collected on Oxford Diffraction (Agilent) SuperNova single crystal diffractometer using Cu K^α radiation ($\lambda = 1.5418$ Å), an Atlas detector, and at 150 K with a Cryostream N_2 open-flow cooling device [13]. Data collection strategy was calculated with the program CrysAlis PRO [14]. Series of ω-scans were generally performed to provide sufficient data to a maximum resolution of 0.77 Å. Data reduction and cell refinement were carried out using CrysAlis PRO Agilent [14]. Intensity data were processed and corrected for absorption effects by the multi scan method. In respect to TBAB/BNP) complex, a total of 17,864 reflections were measured out of which 6669 were independent and 6172 were observed, and used in structure solution and refinement, while in TPAB/BNP, a total of 21,657 reflections were measured out of which 7640 were independent and 6821 were observed, and used in structure solution and refinement. In both cases, structure solution was carried out with direct methods using the programs SIR 92 [15] or superflip within the CRYSTALS software suite [16], and refined by full-matrix least-squares ‘methods based on F^2. In general, coordinates and anisotropic displacement parameters of all nonhydrogen atoms were refined separately. The hydroxyl hydrogen atom of BNP were found in the difference Fourier map and coordinates were refined. The coordinates of all the other H atoms were also visible in the difference map and included in a refinement with isotropic parameters. Molecular geometry calculations were prepared using Mercury [17]. Details of data collection and structure refinement are given in Table 1. Final fractional atomic coordinates, anisotropic thermal parameters, bond lengths and angles and tables of observed and calculated structure factors of TBAB/BNP and TPAB/BNP have been deposited at Cambridge Crystallographic Database Center (CCDC). The hydrogen bond geometry and short contacts for both complexes are shown in Tables 2 and 3.

Results and Discussion

The TBAB/BNP and TPAB/BNP crystal complexes crystallize in monoclinic crystal system (space *group* $P2_1/n$). The asymmetric unit of TBAB/BNP complex consists of a tetrabutylammonium cation, BNP and bromide anion (Fig. 1a), while that of TPAB/BNP consists of tetrapentylammonium cation, BNP, and bromide anion (Fig. 1b). The packing structure of TBAB/BNP (Fig. 2a) has revealed that the rigid naphthol planes form a void and traps TBAB in a fashion that is different from the packing structure in TPAB/BNP, in which the tetrapentylammonium cations trap the BNP molecules in a manner that may be attributed to the effect of increase in chain length from $n = 4$ to 5 ($n =$ number of carbon atoms in alkyl chain), as well as the influence of the biradial [18] conformation (Fig. 3a) in which there are two sets of paired alkyl chains in the tetrapentylammonium cation. The packing structures observed in both cases are completely different from each other, and that observed in complexes of tetrabutylammonium bromide with phenol and catechol (both planar) in which the phenol or catechol molecules are located in zigzag channels formed by the tetragonal-shaped tetrabutylammonium cation. This suggests that the molecular conformation of tetrabutylammonium bromide and tetrapentylammonium bromide do affect the packing fashion of the nonplanar-shaped BNP molecule and vice versa significantly. The BNP molecule adopts the *cisoid* conformation [19]. The dihedral angle between the two naphthol planes in TBAB/BNP defined by O1, C1, C2 . . . , C10 and O2, C1I, . . . , C20 is $-88.9\,(17)^\circ$, while in TPAB/BNP the value is $71.8\,(2)^\circ$, and suggest that the naphthol planes in TBAB/BNP are almost normal to each other and agree with the value obtained by Mori *et al,* 1993 and others [20–22], while those in TPAB/BNP are not normal to each other and could be attributed to the effect of packing fashion of alkyl chains on the naphthol planes in BNP. The bond lengths and angles of the two naphthol groups in both complexes are substantially the same and normal, as well as not significantly different from those obtained in other naphthol derivatives complexes [20, 22]. The bond lengths and angles of BNP in TBAB/BNP are in the range from 1.358 (2) to 1.430 (18) Å for C-C, from 117.7 (13) to 121.3 (13)° for C-C-C , from 1.358 (17) to 1.360 (17) Å for both O-C bonds, from 118.0 (12) to 120.7 (13)° for O-C-C angle, while those of BNP in TPAB/BNP are in the range from 1.356 (3) to 1.430 (2) Å for C-C, from 117.6 (16) to 122.3 (15)° for C-C-C, from 1.358 (2) to 1.365 (2) Å for both O-C bonds, from 118.6 (15) to 120.2 (15)° for O-C-C angle.

Table 1. Crystallographic data for TBAB/BNP and TPAB/BNP

	TBAB/BNP	TPAB/BNP
Molecular formula	$C_{16}H_{36}BrN.C_{20}.H_{14}O_2$	$C_{20}H_{44}BrN.\ C_{20}.H_{14}O_2$
Molecular mass	607.90	663.9
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/n$
a/Å	10.571	10.637
b/Å	26.221	20.832
c/ Å	11.787	16.529
$\beta/^\circ$	100.529	92.65
V/ Å^3	3212.53	3659.7
D_c/g cm^{-3}	1.258	1.207
Z	4	4
Crystal dimensions/mm	0.41 × 0.43 × 0.45	0.15 × 0.20 × 0.40
$F(000)$	1296	1424
μ/(CuKα) cm^{-1}	1.974	1.772
T/K	150	150
Absorption correction type	Multi scan	Multi scan
	(CrysAlis, Oxford diffraction, 2006)	*(CrysAlis, Oxford diffraction, 2006)*
$\theta_{\min}, \theta_{\max}$ /°	3.371, 76.521	3.415, 76.780
H, k, l	$-11\rightarrow13, -32\rightarrow32, -11\rightarrow14$	$-13\rightarrow10, -26\rightarrow25, -14\rightarrow20$
$T_{\min}, T_{\max}$	0.36, 0.45	0.54, 0.77
No. of reflections measured	6669	7640
No. of observed reflections used	6172 [$I > 2\sigma$ (I)]	6821[$I > 2\sigma$ (I)]
No. of parameters	361	397
Restraints	0	0
R	0.029	0.0376
R_w	0.073	0.104
$\Delta\rho_{min}, \Delta\rho_{\max}$, /e A^{-3}	−0.56, 0.29	−0.87, 0.61
Goodness-of-fit	0.986	1.00

The C-C bond joining the two naphthol rings in TBAB/BNP and TPAB/BNP are 1.494 (18), 1.497 (2) Å, respectively, which are not significantly longer than the corresponding racemate conformation (1.475) [20, 22].

The bond lengths and angles in tetrabutylammonium and tetrapentylammonium cations are within the accepted limits and not significantly different from those reported on tetrabutylammonium bromide [23] and crystal complexes of tetrabutylammonium bromide with phenol [11], tetrabutylammonium bromide with catechol [12]. The bond lengths and angles in tetrabutylammonium cation are in the range from 1.514 (2) to 1.531 (2) Å for C-C, from 108.2 (13) to 113.9 (13)° for C-C-C, from 1.518 (18) to 1.530 (17) Å for N-C, and from 107.9 (11) to 111.3 (11) ° for C-N-C. The average bond lengths and angles are;1.522 Å

Table 2. Hydrogen bonding geometry and short contacts (Å, °) for TBAB/BNP complex

D-H... A	D-H	H... A	D... A	D-H... A
O1-H1... Br^{i}	0.83	2.41	3.237(11)	171 (4)
O2-H8... Br	0.82	2.39	3.205(11)	177 (4)
C2-H2... Br^{i}	0.93	2.94	3.697(15)	140
C12-H9... Br	0.94	2.93	3.693(15)	139
C13-H10... Br^{ii}	0.96	3.02	3.829(15)	157
C21-H16... Br	0.96	2.88	3.831(15)	173
C26′-H44... O1^{ii}	0.96	2.58	3.462(19)	153
C26-H36... O2^{iii}	0.96	2.64	3.459(15)	141

Symmetry codes: (i) $1/2 + x$, $3/2 - y$, $-1/2 + z$ (ii) $1/2 + x$, $3/2 - y$, $1/2 + z$ (iii) $-1/2 + x$, $3/2 - y$, $1/2 + z$.

for C-C, 111.8° for the C-C-C angle, 1.523 Å for the N-C, 109.9° for C-N-C angle. The C-C-C-C torsion angles are in the range 180 ± 20° with exception of C11-C12-C13-C14 (−67.5°) which depicts a *gauche* conformation about C12-C13. The tetrabutylammonium cation in TBAB/BNP complex also exhibits conformational and orientational disorder (Fig. 3b), and is evident in one of the alkyl chains that shows *gauche* bend conformation, and in the packing structure of TBAB/BNP (Figure 2a) in which one of the unpaired alkyl chains of tetrabutylammonium cation seems to be oriented in the direction of one of the naphthol planes. The distorted alkyl chains contributes to poorer packing in the bromide anion. The mean distance from a bromide anion to the nearest nitrogen atom is about 4.8 Å, and the shortest distance between N^+ and Br- in TBAB/BNP and TPAB/BNP complexes are 4.5 and 5.2 Å, respectively. This suggests that the Br− anion lies closer to N^+ in TBAB than in TPAB, and may be linked to influencing the creation of space available for the biradial conformation in tetrapentylammonium cation when the alkyl chain length is increased from $n = 4$ to 5.

In regard to tetrapentylammonium cation, the bond lengths and angles are in the range from 1.505(3) to 1.531(2) Å for C-C, from 106.8 (15) to 114.3(2)° for C-C-C, from 1.515 (2) to 1.522 (2) Å for N-C, and from 104.8 (13) to 112.1° for CN-C. The C-N-C bond angle of 104.8 (13)° in the tetrapentylammonium cation deviates significantly from the usual

Table 3. Hydrogen bonding geometry and short contacts (Å,°) for TPAB/BNP complex

D-H... A	D-H	H... A	D... A	D-H... A
O1-H1... Br^{i}	0.82	2.41	3.231(13)	173
O2-H8... Br^{ii}	0.82	2.53	3.341(12)	172
C22-H17... Br^{ii}	0.97	2.90	3.840(2)	164
C23-H18... Br	0.96	2.96	3.801(2)	147
C21′-H26... O2	0.97	2.46	3.418(2)	168
C22-H17... O1^{iii}	0.97	2.52	3.442(2)	160
C23′-H31... O1^{iii}	0.97	2.67	3.251(2)	119

Symmetry codes: (i) $\frac{1}{2} + x$, $\frac{1}{2} - y$, $1/2 + z$ (ii) 1 + x,y,z (iii) $-1/2 + x$, $1/2 - y$, $-1/2 + z$.

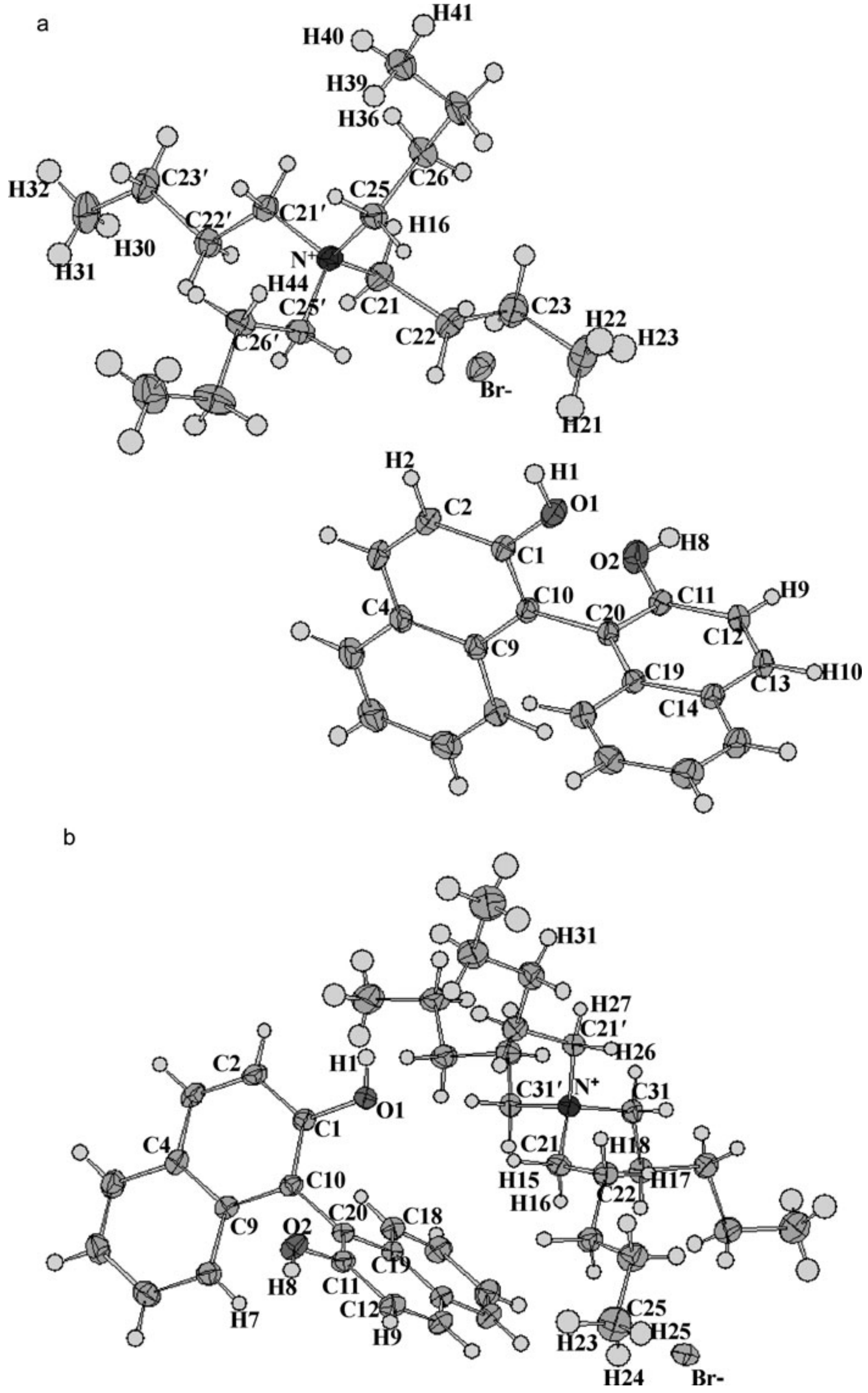

Figure 1. (a) ORTEP view of the TBAB/BNP complex with the atom labeling scheme. Displacement parameters are drawn at the 50% probability level. (b) ORTEP view of the TPAB/BNP with the atom labeling scheme. Displacement parameters are drawn at the 50% probability level.

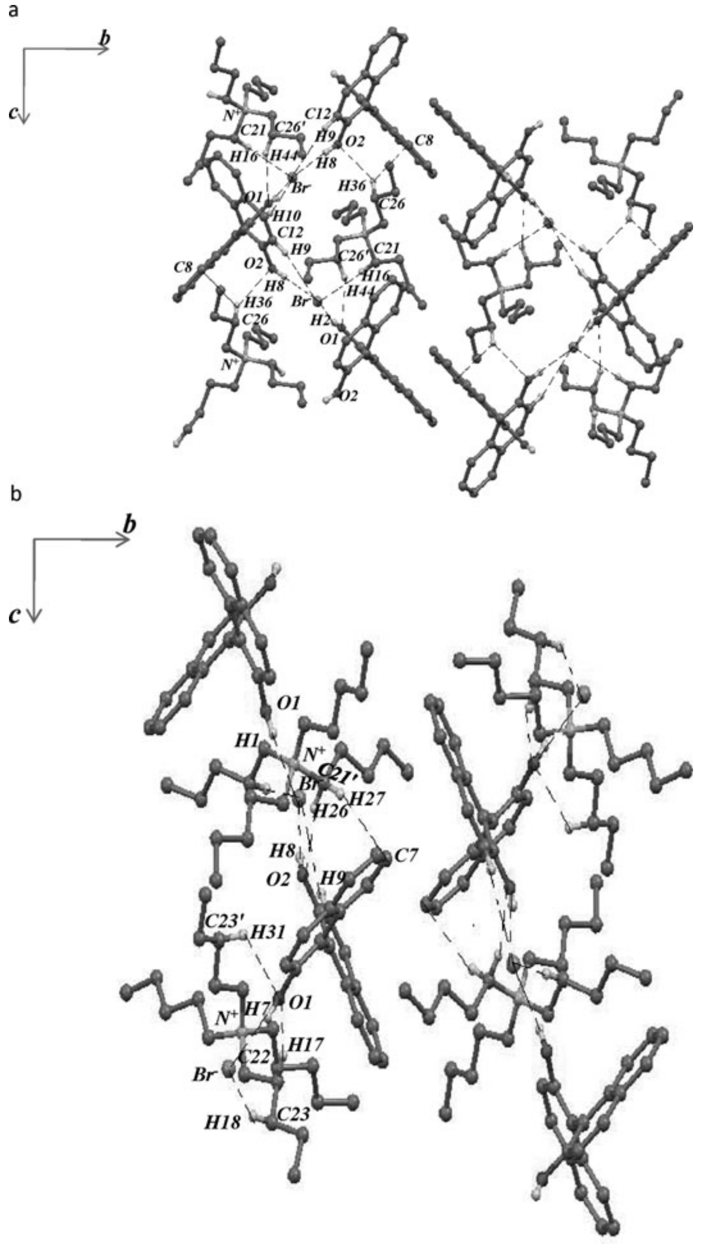

Figure 2. (a) Packing structure of TBAB/BNP complex showing the intermolecular interactions in the *bc*-plane. (b) Packing structure of TPAB/BNP complex showing the intermolecular interactions in the *bc*-plane.

tetrahedral bond angle (109.5°) and the overall conformation of the tetrapentylammonium cation is not tetrahedral, because there is a twist about the N-C bond at the cationic center extended chains at angles that do not correspond to 109.5°. However, in the case of tetrabutylammonium cation the angles about N-C bond do not vary significantly from

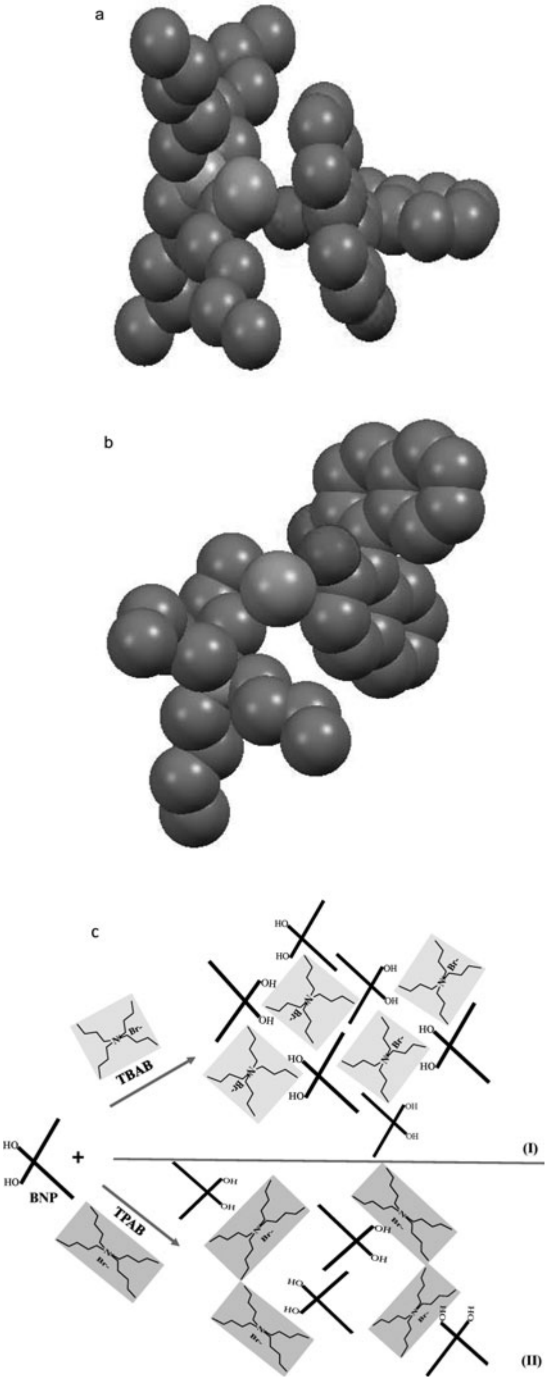

Figure 3. (a) Space filling model showing the biradial conformation in tetrapentylammonium cation i. (b) Space filling model showing the conformation and orientational disorder in tetrabutyl ammonium cation in TBAB/BNP complex. (c) A schematic illustration showing the entrapment of **(I)**: TBAB by BNP molecules and **(II)**: BNP by TPAB, in different created voids.

the tetrahedral value (109.5°). The average bond lengths and angles are;1.518 Å for C-C, 111.5° for the C-C-C angle, 1.519 Å for the N-C, 108.6° for C-N-C angle. The C-C-C-C torsion angles are in the range 180 ± 10° and exhibits no *gauche* conformation. All the alkyl chains adopt fully extended *zigzag* and *transoid* conformation. However, in complexes involving tetrabutylammonium bromide with phenol or catechol, the tetrabutylammonium cation adopts a tetragonal shape [23, 24], conformational and orientational disorder.

In both complexes, the BNP molecules and the tetra-*n*-alkylammonium bromides are held in their aggregates by strong and weak hydrogen bonds. The strong H-bond network exists between the oxygen of the naphthol groups and the bromide of the tetra-*n*-alkylammonium cation. In TBAB/BNP complex (Fig. 2a), the O-H... Br intermolecular interactions (O1... Br; 3.236 (11), O2... Br; 3.206 (11) Å) form a chain network to entrap the tetrabutylammonium cations, while in TPAB/BNP (Fig. 2b), the pattern of the O-H... Br interactions (O1... Br; 3.231 (13) Å, O2... Br; 3.340 (12) Å) are different and attributed to the effect of the distorted alkyl chains and orientation of the naphthol planes. The patterns of hydrogen bonding network as well as conformation of the tetra-*n*-alkylammonium cation do contribute to the formation of the shape of the voids that entrap either BNP or tetrabutylammonium cation.

In respect to weak hydrogen bonds, both complexes are stabilized by C-H... O, C-H... Br and C-H... π intermolecular interactions (Fig. 2a and Fig. 2b). However, in tetrabutylammonium bromide complexes with phenol or catechol, the crystal structures were stabilized only O... Br hydrogen bonding. . The C-H... O interactions arise from an interaction between a H-atom of a methylene group of a tetra-*n*-akylammonium cation and oxygen atom of hydroxyl moiety of BNP. The interactions are reasonably within the accepted limits (H44... O1; 2.58 Å and C26′... O1; 3.463(11) Å, H36... O2; 2.64, and C26... O2; 3.439 (15) Å in TBAB/BNP), while in TPAB/BNP, the distances are (H17... O1; 2.52 Å and C22... O1; 3.442 (2) Å, H26... O2; 2.46 and C21′... O2; 3.416 (2), H31... O1; 2.67 and C23′-O1; 3.251 (2) Å). The C-H... Br contacts observed in both complexes exist between the C-H of a naphthol moiety and bromide anion (H9... Br; 2.93, and C2... Br; 3.693 (15), H10... Br; 3.02 Å, and C13... Br; 3.829 (15), Å for TBAB/BNP, and H9... Br; 2.95 Å, and C12... Br; 3.716 (2), and H18... Br; 2.96 and C22... Br; 3.801(2) Å, for TPAB/BNP) as well as those between C-H of an alkyl chain and Br anion (H16... Br; 2.88 and C... Br; 3.829 (2) Å for TBAB/BNP, and H17... Br; 2.90 and C... Br; 3.840 Å for TPAB/BNP. The interactions between an H-atom of a methylene group with carbon in a phenyl moiety of BNP suggests C-H... π interactions, because the H... C and C... C contact distances observed were found to be less than sum of the *van der* Waals radius (2.97 and 3.40 Å, respectively) [25]. The corresponding values are; H36... C8 (2.82 Å), C26... C8 (3.564 (2) Å) in TBAB/BNP, and H27... C17 (2.71 Å), C21′... C17 (3.567 (12) Å) in TPAB/BNP. Depending on the direction in which the H-atom is oriented and the angle of inclination of the naphthol moieties, an optimal C-H... π interaction is observed in both complexes.

Conclusions

The TBAB/BNP and TPAB/BNP complexes are held in their aggregates by strong and weak hydrogen bonds. The packing structures in both complexes are different from those observed in tetrabutylammonium bromide with planar aromatic molecules (phenol and catechol). The tetrabutylammonium cation exhibits conformational and orientational disorder in TBAB/BNP complex (Fig. 3b). However, in TPAB/BNP the alkyl chains of the tetrapenthylammonium cation adopt biradial conformation (Fig. 3a). The tetrapentylammonium cations in TPAB/BNP completely entrap the BNP molecules in a manner that is attributed to the effect of increase in chain length (Figs. 2b and 3c-II) as well as influence of biradial conformation in the tetrapentylammonium cation (Fig. 3a). However, in TBAB/BNP the naphthol planes of BNP form a void and sandwiches the tetrabutylammonium cation (Figs. 2a and 3c-I). The patterns of hydrogen bonding network and conformation of the tetra-*n*-akylammonium cation contribute to the formation of the shape of the voids that entrap either BNP or tetrabutylammonium cation. The molecular conformation of the tetra-n-alkylammonium bromide does affect the packing fashion of the nonplanar shaped BNP molecule and vice versa significantly.

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