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Thermochemical behaviour of straight-chain ethers $CH_3O(CH_2CH_2O)_nCH_3$ ($n = 1$–4) in aqueous and tetrachlormethane solutions

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Abstract

The enthalpies of solution $\Delta_{sol}H_m$ of straight-chain polyethers $CH_3O(CH_2CH_2O)_nCH_3$ ($n = 1$–4) have been measured in water and tetrachlormethane as inert reference solvent at temperature of 298.15 K. The enthalpic coefficients of pairwise interaction and enthalpies of solvation were determined. The interaction between ether molecules in inert solvent are dominated by electrostatic forces resulting in the negative coefficients of interaction. In water the coefficients of interaction are positive. This is because the partial destruction of hydrophobic and hydrophilic shells becomes the predominant contribution to the coefficient. The enthalpy of solvation of one ethane oxide group in water is more negative than in tetrachlormethane by $-7.4\,\mathrm{kJ\,mol^{-1}}$ that was related to the prevailing contribution of hydrogen bonding between ether and water molecules. The comparison of enthalpic characteristics was performed for the open-chain and cyclic ethers. The more exothermic effect of hydration of CH_2CH_2O group is observed for cyclic ethers. The distinction is connected with strengthening of hydrogen-bond bridges between adjacent ether oxygen atoms in cyclic molecule.

Keywords: Enthalpy of solution; Enthalpy of solvation; Enthalpic coefficient of pairwise interaction; Straight-chain ethers; Glymes; Effect of cyclization; Water; Tetrachlormethane

1. Introduction

Straight-chain polyethers $CH_3O(CH_2CH_2O)_nCH_3$, commonly known as glymes, are of interest as good models for study of more complex polymeric systems and bio-structures that have hydrophobic and hydrophilic groups in close proximity [1,2]. These compounds show surfactant ability and a remarkable drag-reducing property in the flow of water through ducts and channels.

The previous investigation of volume characteristics and compressibility, especially their concentration and temperature dependences [3,4], showed that the hydration of straight-chain ethers is dominated by the hydrophobic hydration of the CH_2 groups. The obtained data indicated that the hydration shell of open-chain ethers has lesser density, thermal stability and resistibility to pressure than hydration structure of cyclic ethers. The NMR and Raman results for aqueous solutions of the ethers [5–7] indicated the formation of hydrogen-bond bridges between adjacent ether oxygen atoms. The stabilization of the gauche conformation of the $OCH_2{-}CH_2O$ segment in ether molecule by hydrogen-bonded bridges between the adjacent oxygens was demonstrated by molecular dynamics and Monte Carlo simulations [8,9]. In this work the enthalpies of solution of chain polyethers ($n = 1$–4) in water and tetrachlormethane as inert reference solvent were measured by the calorimetry at 298.15 K. Our aim was the determination of enthalpic characteristics of the solvation of straight-chain ethers in aqueous and non-polar medium and compare them with those of the cyclic analogs [10,11] to gain insight into the effect of cyclization on solute–solute and solute–solvent interactions.

2. Experimental

The purities of 1,2-dimethoxyethane (monoglyme), diethylene glycol dimethyl ether (diglyme), thriethylene glycol

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Table 1
Experimental enthalpies of solution of straight-chain ethers $CH_3O(CH_2CH_2O)_nCH_3$ in tetrachlormethane and water at 298.15 K

$CH_3O(CH_2CH_2O)CH_3$		$CH_3O(CH_2CH_2O)_2CH_3$		$CH_3O(CH_2CH_2O)_3CH_3$		$CH_3O(CH_2CH_2O)_4CH_3$	
m (mol kg^{-1})	$-\Delta_{sol}H_m$ (kJ mol^{-1})	m (mol kg^{-1})	$-\Delta_{sol}H_m$ (kJ mol^{-1})	m (mol kg^{-1})	$-\Delta_{sol}H_m$ (kJ mol^{-1})	m (mol kg^{-1})	$-\Delta_{sol}H_m$ (kJ mol^{-1})
+Tetrachlormethane							
0.03596	1.32(0.02)	0.01083	2.13(0.02)	0.01475	2.66(0.02)	0.00314	3.02(0.02)
0.05015	1.30(0.02)	0.01421	2.14(0.02)	0.01772	2.67(0.02)	0.01291	3.06(0.02)
0.06177	1.32(0.02)	0.01521	2.14(0.02)	0.03037	2.72(0.01)	0.01774	3.10(0.02)
0.07126	1.32(0.02)	0.01594	2.14(0.02)	0.03203	2.73(0.01)	0.02442	3.12(0.02)
0.07876	1.30(0.01)	0.02048	2.15(0.02)	0.03688	2.74(0.01)	0.03298	3.16(0.01)
		0.02323	2.15(0.01)	0.03748	2.74(0.01)	0.04985	3.22(0.01)
		0.04460	2.17(0.01)	0.04661	2.76(0.01)		
				0.04850	2.77(0.01)		
				0.05540	2.78(0.01)		
+Water							
0.09712	22.21(0.02)	0.00536	30.04(0.02)	0.00104	38.48(0.02)	0.00144	46.21(0.02)
0.10528	22.24(0.02)	0.01242	30.01(0.02)	0.00289	38.43(0.02)	0.00215	46.20(0.02)
0.16725	22.32(0.02)	0.01731	30.01(0.02)	0.00600	38.40(0.02)	0.00490	46.19(0.02)
0.23632	22.30(0.01)	0.02381	29.98(0.02)	0.01069	38.40(0.02)	0.01027	46.14(0.02)
0.31158	22.23(0.01)	0.02713	29.94(0.02)	0.02000	38.33(0.01)	0.01921	46.08(0.01)
		0.03367	29.92(0.01)	0.02508	38.33(0.01)	0.02296	46.07(0.01)
		0.03672	29.95(0.01)	0.03482	38.28(0.01)	0.02360	46.06(0.01)
		0.03834	29.93(0.01)	0.09620	38.11(0.01)		
		0.04311	29.92(0.01)				
		0.07749	29.89(0.01)				
		0.23250	29.54(0.01)				

Instrumental uncertainties of solution enthalpy are given in parentheses.

dimethyl ether (thriglyme), and tetraethylene glycol dimethyl ether (tetraglyme) were 99.4, 99.5, 99.5, and 98%, respectively. All chemicals obtained from Fluka were used without further purification. To prepare the aqueous solutions doubly distilled water was used. The tetrachlormethane was purified according to standard procedure [12], and dried by molecular sieves.

Enthalpies of solution were measured with an isoperibol calorimeter at a temperature of (298.15 ± 0.005) K as was described earlier [11].

3. Results and discussion

Results of the calorimetric experiments are summarized in Table 1. The final solute molalities were within the concentration range of 0.003–0.055 mol kg^{-1} for the tetrachlormethane solutions, and of 0.001–0.2 mol kg^{-1} for the aqueous solutions. A variation of the measured solution enthalpies with molality was observed for nearly all of the systems studied. Figs. 1 and 2 demonstrate linear concentration dependence of enthalpy of solution in this range. To evaluate limiting enthalpy of solution at infinite dilution, $\Delta_{sol}H_m^\circ$, and enthalpic coefficients of solute–solute pairwise interaction, h_{22}, experimental data were expressed by using a virial expansion. For systems under the investigation, the weighted molar enthalpies of solution were fitted as a satisfactory linear function of molality over the concentration range studied:

$$\Delta_{sol}H_m = \Delta_{sol}H_m^\circ + h_{22}m$$

The standard deviation at this comes up to 0.07 kJ mol^{-1}. The weight of each value of $\Delta_{sol}H_m$ equated to reciprocal value of instrumental uncertainty is varied over the concentration range studied. The h_{22} and $\Delta_{sol}H_m^\circ$ values are changed up to 100 J kg mol^{-2} and up to 0.01 kJ mol^{-1}, respectively, when the weighting of $\Delta_{sol}H_m$ experimental results is taken into account in regression analysis. Obtained values of $\Delta_{sol}H_m^\circ$ and h_{22} are listed in Table 2. In literature, there are values of limiting partial molar excess enthalpies for monoglyme (−22.36), diglyme (−29.91), triglyme (−38.59) and tetraglyme (−46.25 kJ mol^{-1}) in water [13] determined by flow calorimetry as averaged experimental magnitudes at concentration from 0.028 to 0.3 mol kg^{-1}, which are close to our data. In the present work the integral enthalpies of solution have been measured at lower concentra-

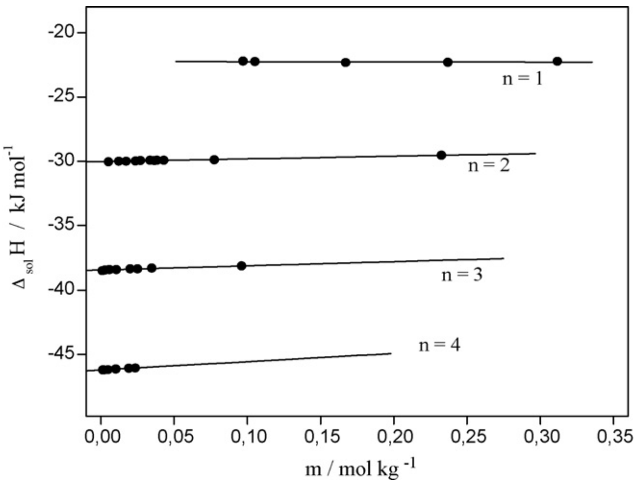

Fig. 1. Concentration dependence of enthalpy of solution for straight-chain ethers in water.

Table 2
Enthalpies of solution and enthalpic coefficients of pairwise interaction for open-chain and cyclic ethers in water and tetrachlormethane at infinitive dilution and at 298.15 K

Solute	+Tetrachlormethane		+Water	
	$\Delta_{sol}H_m^\circ$	h_{22}	$\Delta_{sol}H_m^\circ$	h_{22}
$CH_3O(CH_2CH_2O)CH_3$	−1.32 (0.02)	0	−22.24 (0.06) −22.95 (0.03)[a]	−96 (296)
$CH_3O(CH_2CH_2O)_2CH_3$	−2.12 (0.01)	−1060 (95)	−30.02 (0.01)	2065 (98)
$CH_3O(CH_2CH_2O)_3CH_3$	−2.63 (0.01)	−2867 (209)	−38.44 (0.01)	3724 (340)
$CH_3O(CH_2CH_2O)_4CH_3$	−3.01 (0.01)	−4251 (221)	−46.22 (0.01)	6654 (297)
$(CH_2CH_2O)_2$	−0.41 (0.02)		−9.34 (0.1)[b]	3034[c]
$(CH_2CH_2O)_4$	1.8 (0.2)		−28.95 (0.1)[d]	4095[c]
$(CH_2CH_2O)_5$	2.7 (0.1)[e]		−39.75 (0.1)[e]	5611[c]
$(CH_2CH_2O)_6$	32.4 (0.3)[f]		−21.5 (0.2)[f]	

Units: $\Delta_{sol}H^\circ$ in kJ mol^{-1} and h_{22} in J kg mol^{-2}.
[a] Ref. [14].
[b] Ref. [13].
[c] Ref. [18].
[d] Ref. [17].
[e] Ref. [10].
[f] Ref. [11].

tions and the concentration dependence of enthalpy of solution has been found. The obtained enthalpy of solution for 1,2-dimetoxyethane in water is in good agreement with value reported in [14].

3.1. Enthalpic characteristic of solute–solute interaction

Data on h_{22} reflect enthalpic effects of interaction between solvated solute molecules. Three effects are commonly considered to describe the interaction between the polar non-electrolyte molecules in aqueous solution [15]: (1) dipole–dipole interaction between polar groups (exothermic contribution), (2) hydrophobic–hydrophobic interaction of the alkyl groups (endothermic contribution), (3) "thermochemical repulsion" between the solvated apolar alkyl groups and solvated polar groups (endothermic contribution). The latter two effects are related to the hydrophobic hydration.

For open-chain ethers h_{22} are negative (Table 2) in tetrachlormethane in which H-bonding and (2) and (3) effects resided in aqueous solution are absent. Their absolute values increase with lengthening of the ether straight-chain (Fig. 2). This means that the long-range electrostatic interaction between solute molecules is dominant in apolar solvent resulting in the exothermic contribution to h_{22}. This interaction is accompanied by the partial desolvation of the molecules, thus weakening the exothermic effect. The increase of the negative h_{22} values agrees with increasing of dipole moment in the series of studied ethers from 1.61 D for monoglyme to 2.16 D for triglyme [16].

In contrast, in case of aqueous solution h_{22} have positive values with the exception of monoglyme. They increase remarkably with lengthening of chain. It can be concluded that electrostatic forces are prevailing for interaction between shortest ether chains only and do not play a determinative role for long ether chains. In latter case the interaction can be described in view of effects (2) and (3). As all alkyl groups, CH_2CH_2 unit in the ether molecule has the hydrophobic hydration shell. At overlapping of hydrophobic shells some water molecules may be released to the bulk. This effect gives a positive contribution to h_{22}. The overlapping of hydrophilic shells, which are structured by hydrogen bond with ether, leads to a positive contribution also. The "thermochemical repulsion" breaks the hydrophobic hydration structure of alkyl groups resulting in a positive contribution to h_{22}. Thus, h_{22} data indicate that the hydrophobic hydration of ethane groups plays the appreciable role in the hydration process of glymes. The analogous conclusion has been made from volume and compressibility results [3,4].

3.2. Enthalpic characteristic of solute–solvent interaction

The data on $\Delta_{sol}H_m^\circ$ (Table 2) and enthalpy of evaporation of the solutes [14,18–20] were used to calculate the limiting enthalpy of solvation of glymes in tetrachlormethane and water, $\Delta_{solv}H^\circ$. These data are presented in Table 3.

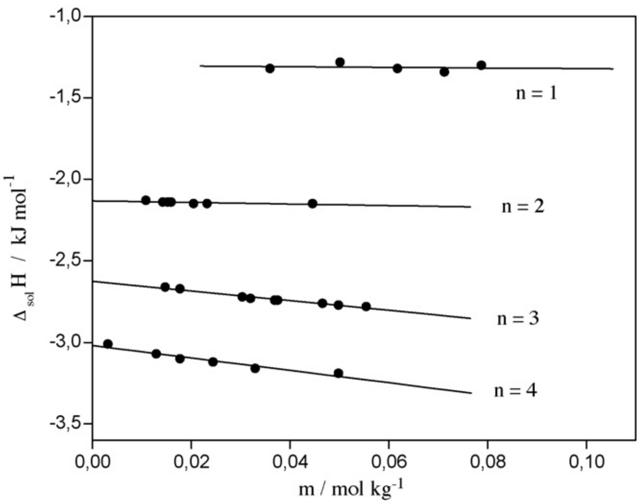

Fig. 2. Concentration dependence of enthalpy of solution for straight-chain ethers in tetrachlormethane.

Table 3
Enthalpies of evaporation and solvation for open-chain and cyclic ethers in water and tetraclormethane at 298.15 K

Solute	$\Delta_{\mathrm{ev}}H$	$-\Delta_{\mathrm{solv}}H^{\circ}_{\mathrm{m}}$	
		In water	In tetrachlormethane
$CH_3O(CH_2CH_2O)CH_3$	36.4 (0.03)[a]	58.8 (0.07)	37.7 (0.04)
$CH_3O(CH_2CH_2O)_2CH_3$	48.0 (0.6)[b]	78.0 (0.6)	50.1 (0.6)
$CH_3O(CH_2CH_2O)_3CH_3$	63.7 (3.3)[b]	102.15 (3.3)	66.3 (3.3)
$CH_3O(CH_2CH_2O)_4CH_3$	76.9 (2.6)[b]	123.2 (2.6)	79.9 (2.6)
$(CH_2CH_2O)_2$	37.49 (0.4)[c]	46.8 (0.4)	37.9 (0.4)
$(CH_2CH_2O)_4$	65.67 (0.37)[d]	94.6 (0.4)	63.9 (0.4)
$(CH_2CH_2O)_5$	79.57 (0.29)[d]	119.3 (0.3)	76.9 (0.3)
$(CH_2CH_2O)_6$	133.16 (0.27)[d]	154.7 (0.3)	100.8 (0.4)
	128.1 (2.3)[b]	149.6 (2.3)	95.7 (2.3)

Units: $\Delta_{\mathrm{sol}}H^{\circ}$ in kJ mol^{-1} and h_{22} in J kg mol^{-2}.

[a] Ref. [14].

[b] Ref. [19].

[c] Ref. [20].

[d] Ref. [17].

The limiting enthalpy of solvation for studied series of glymes $CH_3O(CH_2CH_2O)_nCH_3$ (where $n = 1$–4) in both solvents is a linear function of number of CH_2CH_2O groups in the solute molecule. The contribution of one these groups to solvation enthalpy determined from the linear correlation between the $\Delta_{\mathrm{solv}}H^{\circ}$ and n was found equal to -14.3 ± 0.5 in tetrachlormethane and -21.7 ± 0.6 kJ mol^{-1} in water. From CH_2CH_2O group contributions obtained in present work and literature results for CH_2 group (-4.7 ± 0.1 kJ mol^{-1} in tetrachlormethane [21] and -3.1 ± 0.1 kJ mol^{-1} in water as it was obtained from data [22] for n-alkanes) the contribution of ether oxygen to $\Delta_{\mathrm{solv}}H^{\circ}$ was assessed as -15.5 kJ mol^{-1} in water and -4.9 kJ mol^{-1} in tetrachlormethane in accordance with sample additive method.

As seen, the $\Delta_{\mathrm{solv}}H^{\circ}$ values, the contributions of one CH_2CH_2O group and ether oxygen atom to the enthalpic effect of solvation in water are much more exothermic than in tetrachlormethane. It can be connected with appearance of two contributions, which reside in aqueous solution: the hydrophobic hydration of solutes and H-bonding between water molecules and oxygen atoms of ether molecules. As shown above, the hydrophobic hydration of glymes is reflected on concentration dependence of solution enthalpy, apparent volume and compressibility. However, a sign and magnitude of contribution of the hydrophobic hydration to $\Delta_{\mathrm{solv}}H^{\circ}$ are debatable.

Extrapolating the dependence of $\Delta_{\mathrm{solv}}H^{\circ}$ values for cyclic ethers on composition of water-organic solvent Jozwiak [23] estimated the enthalpy of hydrophobic hydration for CH_2CH_2O group in pure water as large negative value (-12 or -9.5 kJ mol^{-1}). According to our data, the difference between contributions of ethane oxide group in inert tetrachlormethane and water to $\Delta_{\mathrm{solv}}H^{\circ}$ for open-chain ethers is close to this value. However, from data obtained by Fuchs and Stephenson [21] it follows that solvation enthalpies of alkanes with large molecular size are lesser exothermic in water than those in tetrachlormethane. The contribution of shortest hydrophobic group CH_2 to solvation enthalphy for alkane series is also lesser exothermic by 1.6 kJ mol^{-1} in water than in tetrachlormethane solvent. Abraham [24] found that the contribution of hydrophobic hydration to $\Delta_{\mathrm{solv}}H^{\circ}$ is ranged from 1.3 to 1.8 kJ mol^{-1} for CH_2 group and amounts to negative value for CH_3 group. It is in agreement with decrease of exothermic effect of solvation for alkanes with large molecular size (starting from heptane) in water as compared with that in inert solvents [21].

Taking into account the aforesaid facts, we suppose that the hydrophobic hydration make a slight contribution to enthalpic effect of hydration of CH_2CH_2O group. Therefore, the difference in the increments of ethane oxide group in inert tetrachlomethane and water should be related to exothermic contribution of H-bonding between ether and water molecules. More exothermic contribution of ether oxygen in water confirms this conclusion. The results of NMR [5] and Raman [6,7] studies are a direct evidence of hydrogen-bond bridges between adjacent ether oxygen atoms.

3.3. Effect of cyclization on solute–solute and solute–solvent interaction

In order to compare the enthalpic characteristics of open-chain $CH_3O(CH_2CH_2O)_nCH_3$ and cyclic ethers $(CH_2CH_2O)_{n+1}$, the previously obtained h_{22} and $\Delta_{\mathrm{solv}}H^{\circ}$ values for cyclic compounds [10,11,13,17,18] are also given in Tables 2 and 3. The considered series of cyclic ethers $(CH_2CH_2O)_{n+1}$ includes crown ethers and 1,4-dioxane. It is seen from Table 2, h_{22} value for any open-chain ether in water is more positive as compared with that for analogous cyclic ether (at same number of ether oxygen atoms). The contribution of CH_2CH_2O group to h_{22} is 2190 ± 174 J kg mol^{-2} for open-chain and 1288 ± 131 J kg mol^{-2} for cyclic ethers as found from the linear regression of h_{22} values versus number of ether groups. Thus, the interaction between open-chain ether molecules in water is more endothermic. This may be connected with larger contribution of their desolvation. The analogous trend has appeared in a higher slope of concentration dependence of apparent molar volume and compressibility for open-chain ethers as compared with cyclic ethers [3,4]. In tetrachlormethane the contribution of CH_2CH_2O group to h_{22} was found be equal to $-1456 \pm 97\,\mathrm{J\,kg\,mol^{-2}}$ for open-chain ethers. Data on h_{22} for cyclic ethers in tetrachlormethane are absent in literature.

The values of $\Delta_{\mathrm{solv}}H^\circ$ given in Table 3 for cyclic ethers in water and tetrachlormethane linearly depend on number of CH_2CH_2O groups with exception of 18-crown-6. The significant deviation from additivity is observed for 18-crown-6 with using either of the available values for sublimation enthalpy given in Table 3. At $1 \le n \le 4$ the increment of the ethane oxide group is $-24.1 \pm 0.2\,\mathrm{kJ\,mol^{-1}}$ in water and $-13.00 \pm 0.01\,\mathrm{kJ\,mol^{-1}}$ in tetrachlormethane. As can be seen, the addition of an ethane oxide group to cyclic ether results in more exothermic change of $\Delta_{\mathrm{solv}}H^\circ$ in water and less exothermic change in tetrachlormethane as compared with a similar addition to an open-chain compound. The observed distinction in solvation of the same group located in open-chain or cyclic molecule can be connected with a modification of conformation of the group. The prevalence of the gauche conformation along OC–CO bond is needed for closure of ether ring. The balance of gauche and trans-conformations is characteristic for the open-chain ethers [25]. Thus, the experimental findings indicate that the enthalpic effect of interaction with inert solvent molecules is more favorable for CH_2CH_2O group in the trans-conformation which has higher population in open-chain structure.

In aqueous solution the difference in solvation of CH_2CH_2O group becomes more considerable because of specific interactions. As it has been indicated earlier [8,9], hydrogen-bonded bridges between the adjacent ether oxygens stabilize the gauche conformation of the $O–CH_2–CH_2–O$ segment. In open-chain molecule the cooperative hydrogen bonds between oxygen atoms are substantially broken by the rotation of the aliphatic chain. In cyclic molecule the degree of freedom of oxygen atoms is lesser and they interact freely with water molecules. Therefore, the exothermic effect of hydration of CH_2CH_2O group increases in going from open-chain to cyclic ethers.

4. Conclusion

The solvation of the straight-chain ethers in water is enthalpically more favorable in respect to inert solvent. It can be connected with formation of hydrogen-bond bridges between the adjacent atoms of oxygen in ether chain. The contribution of hydrophobic hydration to enthalpy of hydration of ether moiety CH_2CH_2O may be estimated as slight. Effects of hydrophobic hydration and hydrogen bonding are also exhibited through the concentration dependence of the enthalpy of solution.

The comparison of enthalpic characteristics for open-chain and cyclic ethers leads to the conclusion that solvation of CH_2CH_2O group is conformationally dependent. The exothermic effect of hydration of CH_2CH_2O group increases in going from open-chain to cyclic ethers. The decrease of the degree of freedom of oxygen atoms in cyclic molecule results in stronger specific interaction of gauche conformer with water molecules. The rotation of aliphatic chain in open-chain molecule reduces the population of gauche conformer that leads to decrease of exothermic effect of specific interaction. In an inert solvent the solvation of CH_2CH_2O group is enthalpically more favorable for open-chain ethers for which the population of trans conformer is higher.

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