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Enthalpies of solution of glycylglycine in water–organic solvent media at 298.15 K

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ABSTRACT

The enthalpies of solution of glycylglycine in aqueous solution of 1,4-dioxane, acetone, formamide, *N*-methylformamide and *N*,*N*-dimethylformamide, with the co-solvent content up to 0.4 mole fractions, have been measured calorimetrically at $T = 298.15$ K. The results obtained were used to calculate the standard enthalpies of solution ($\Delta_{sol}H^{\circ}$) and transfer ($\Delta_{tr}H^{\circ}$) of the glycylglycine from water into the mixtures as well as the enthalpy coefficients of pair-wise interaction h_{xy} of the solute with the organic co-solvents in aqueous media. The h_{xy} values were correlated with the properties of organic solvents using the Kamlet–Taft equation. The effect of the structure properties of the mixed solvent on the specified enthalpy characteristics of glycylglycine is discussed.

1. Introduction

Development and designing of new separation and purification processes for bioactive compounds dissolved in organic media require the accurate knowledge of thermodynamic properties of such solutions. Both peptides and organic solvents are widely used in medicine, pharmacology, cosmetics and other manufactures. Therefore, much attention is being paid to studying thermodynamic properties of peptides in the mixtures of water with organic co-solvents [1–6]. Thermodynamic characteristics of peptides in such mixtures have been already the subject of our previous researches [7–14].

In these works, we have shown that the interactions between the molecules of simple peptides, such as glycylglycine [7,8,12,13], DL-alanyl-DL-glycine [10], L-alanyl-L-alanine [11] or DL-alanyl-DL-valine [14], and various organic solvents can strongly influence their thermodynamic properties in the mixed aqueous media. The given paper is an extension of our previous works on studying the enthalpy effects of interaction between glycylglycine and alcohols [12], acetonitrile (AN) [8] and dimethylsulfoxide (DMSO) [7] in aqueous media.

We report here the enthalpies of solution, $\Delta_{sol}H^{m}$, for glycylglycine in aqueous solution of 1,4-dioxane (1,4-DO), acetone (AC), formamide (FA), *N*-methylformamide (MFA) and *N*,*N*-dimethylformamide (DMF) with a view to gaining information on energy-related changes in the glycylglycine–organic solvent intermolecular interaction under the influence of structure properties of the latter. The content of organic co-solvent in the studied mixtures ranged up to 0.4 mol fractions. All the chosen organic solvents differ from each other by the molecular shape and size as well as donor–acceptor ability and the exerted effect on the water structure. The mixed solvents used in this work are of doubtless interest for both practice and theory of solutions.

2. Experimental

Chromatographically homogeneous glycylglycine (Reanal Co., Hungary, Assay: ≥98.5%) were recrystallized twice from (water + ethanol) mixture, dried in a vacuum chamber at 333 K for 48 h, and kept over P_2O_5 in vacuum desiccators. The molal concentration (m) of the glycylglycine solutions was varied in the range of $0.005 < m < 0.015$ mol per 1 kg of mixed solvent. FA, MFA and DMF were distilled twice under reduced pressure from NaOH according to [15]. AC and 1,4-DO were purified as recommended earlier [16]. Water content determined by Karl Fisher titration [17] did not exceed 0.03 wt % for all organic solvents. Water was purified by deionization and double distillation until a specific conductivity of *ca.* 1.0×10^{-5} S m^{-1}. All the mixtures were prepared by weight.

The values of $\Delta_{sol}H^{m}$ for glycylglycine were measured at (298.15 ± 0.005) K using an isoperibol hermetic calorimeter fitted with a 60 cm^3 reaction vessels and electrical calibration. The calorimeter setup and experimental procedure were described in detail previously [18–20]. The relative random error of measurements did not exceed 0.5%. The calorimeter was tested by measuring (10 experiments) the enthalpy of solution of potassium chloride (KCl) in water at 298.15 K according to [18,19,21].

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Table 1
The standard enthalpies (kJ/mol) of dissolution ($\Delta_{sol}H^{\circ}$) of glycylglycine in aqueous solution of some organic solvents at 298.15 K.

m_2[a]	1,4-DO	m_2[a]	AC	m_2[a]	FA	m_2[a]	MFA	m_2[a]	DMF
0.525	11.99 ± 0.02	0.703	11.81 ± 0.02	1.242	11.07 ± 0.02	0.721	11.89 ± 0.02	0.717	12.09 ± 0.02
1.086	12.37 ± 0.02	1.484	12.16 ± 0.02	2.345	10.61 ± 0.02	1.536	12.28 ± 0.02	1.193	12.65 ± 0.02
1.693	12.65 ± 0.02	2.358	12.41 ± 0.02	3.748	10.27 ± 0.02	2.415	12.53 ± 0.02	2.102	13.12 ± 0.02
2.353	12.96 ± 0.02	3.339	12.69 ± 0.02	5.079	9.96 ± 0.02	3.396	12.88 ± 0.02	2.598	13.89 ± 0.02
3.112	13.34 ± 0.02	4.453	13.19 ± 0.03	7.521	9.21 ± 0.02	4.457	13.16 ± 0.03	3.528	14.48 ± 0.03
3.905	13.57 ± 0.03	5.725	13.62 ± 0.03	8.477	8.68 ± 0.03	5.599	13.34 ± 0.03	4.372	14.99 ± 0.03
4.845	13.89 ± 0.03	7.193	13.84 ± 0.03	10.867	8.47 ± 0.03	6.914	13.53 ± 0.02	5.586	15.38 ± 0.02
5.836	13.99 ± 0.03	8.906	14.55 ± 0.02	12.756	8.15 ± 0.03	8.409	13.69 ± 0.02	6.503	16.01 ± 0.02
7.029	14.08 ± 0.02	10.93	14.91 ± 0.02	17.652	7.51 ± 0.03	10.087	13.85 ± 0.03	9.282	16.49 ± 0.03
8.328	13.94 ± 0.03	13.359	15.46 ± 0.02	25.445	6.91 ± 0.03	12.006	13.81 ± 0.03	12.996	16.97 ± 0.03
11.597	13.58 ± 0.03	16.328	15.18 ± 0.03	35.557	6.72 ± 0.03	16.781	13.57 ± 0.03	18.183	17.37 ± 0.02
16.306	13.01 ± 0.02	20.038	14.83 ± 0.03	–	–	23.518	13.34 ± 0.03	25.926	16.84 ± 0.03

[a] The molal concentration of organic solvents, mol/kg.

Our values of ($\Delta_{sol}H^{m}$ ($m = 0.111$ mol kg^{-1}) = 17.60 ± 0.04 kJ mol^{-1} and $\Delta_{sol}H^{\circ}$ = 17.23 ± 0.07 kJ mol^{-1}) agree with the recommended literature values (17.56 ± 0.02 [22]/17.58 ± 0.02 [23] kJ mol^{-1} and 17.22 ± 0.04 kJ mol^{-1} [18,20], respectively).

3. Results

The standard enthalpies of solution $\Delta_{sol}H^{\circ}$ were calculated by averaging the results of five independent measurements of $\Delta_{sol}H^{m}$ for each composition of water + organic co-solvent, because no dependences of $\Delta_{sol}H^{m}$ versus m for glycylglycine were observed in the mixed solvents studied. The values $\Delta_{sol}H^{\circ}$ for glycylglycine in the mixed aqueous solutions with different molal concentrations are given in Table 1. The transfer enthalpy $\Delta_{tr}H^{\circ}$ is derived from the difference between the enthalpy of solution in each of mixed solvents under comparison ($\Delta_{sol}H^{\circ}(w+y)$) and that in pure water ($\Delta_{sol}H^{\circ}(w)$) respectively

$$\Delta_{tr}H^{\circ} = \Delta_{sol}H^{\circ}(w+y) - \Delta_{sol}H^{\circ}(w) \quad (1)$$

Enthalpy of dissolution of glycylglycine in water ($\Delta_{sol}H^{\circ}(w)$ = 11.56 ± 0.17 kJ mol^{-1}) was taken from [24].

4. Discussion

From the inspection of Fig. 1 one can see that $\Delta_{tr}H^{\circ}$ versus x_2 for glycylglycine depends considerably on the structure of organic solvent as well as from its content in the mixed aqueous solution.

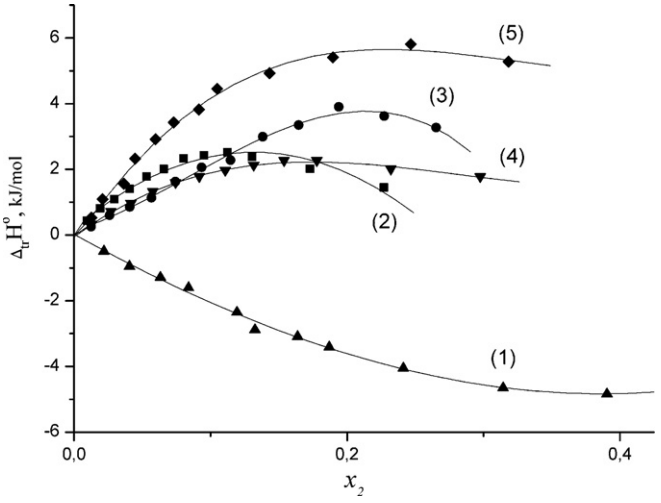

Fig. 1. Enthalpies of transfer $\Delta_{tr}H^{\circ}$ of glycylglycine from water into the H_2O + FA (1), H_2O + 1,4-DO (2), H_2O +A C (3), H_2O + MFA (4) and H_2O + DMF (5) mixed solvent as functions of the alcohol mole fraction (x_2) at 298.15 K.

In the cases of H_2O + AC, H_2O + 1,4-DO, H_2O + MFA and H_2O + DMF mixtures, the dissolution of glycylglycine in a mixed solvent is the endothermic process within the investigated concentration range. On the contrary, the $\Delta_{tr}H^{\circ}$ ($H_2O \rightarrow H_2O$ + FA) value for glycylglycine is negative by sign over the studied range of the mixture composition and smoothly decreases (in magnitude) as the content of FA is rising. In this system, H_2O and FA molecules form H-bonded network [25,26]. Therefore, the $\Delta_{tr}H^{\circ}$ value for glycylglycine in the specified mixed solvent is determined by the superposition between the energy consumed for the dissociation of H-bonded self-associates, the energy of dehydration of the glycylglycine (amide) and the energy released in the formation of heterocomponent H-bonds. The exothermicity of transfer process testifies to the preferential role of a direct intermolecular FA–glycylglycine interaction. In our opinion, the negative sign at h_{xy} for the peptide solution in (H_2O + FA) points to following. The H_2O–H_2O, H_2O–FA, and FA–FA hydrogen bonds are comparable in energy [27] that results in the slightly endothermic effect of mixing of H_2O with FA over the whole range of compositions [28]. Therefore, the endothermic contribution from dehydration of FA molecules will be the least among the studied organic solvents. Note that the enthalpy coefficients of pair-wise interaction between a zwitterion of glycine and a molecule of FA ($h_{xy} = -224$ J kg mol^{-2}) are also negative in sign [29]. It is result of a predominant role of the highly exothermic effect of the direct interaction between a zwitterion of glycine and a molecule of FA, as compared to the endothermic effect of dehydration of both glycine and FA in the aqueous medium. Transferring from the (H_2O + FA) to the (H_2O + MFA) ones causes the increase in the endothermicity process of glycylglycine dissolution (transfer) as the concentration of the MFA in the mixture is rising. The given effect is induced by strengthening of the hydrophobic properties of MFA. In addition, the energy of intramolecular H-bonds in a MFA medium are lower than that in a FA medium because of the greater flexibility of the *N*-site-methylated chain structure of the former [30,31]. As a consequence, the energy-related contribution in $\Delta_{tr}H^{\circ}$ from the destruction of MFA self-associates will be considerably lower than in the case of FA, and the MFA hydration is stronger. DMF, being a typical aprotic polyfunctional solvent, do not form the intramolecular hydrogen bonds. An increase in the endothermic effect of dissolution (transfer) of glycylglycine in the H_2O + DMF mixture is likely caused by the strengthening of its hydrophobic properties [32].

Thus, the effect of transferring the water molecules into the hydration shells of both zwitterion and peptide group strengthens their hydration, as a whole. In other words, the endothermic effect of partial dehydration of the "reinforced" hydration layers of both zwitterion and peptide group becomes the more pronounced. Besides, an increase in the size of glycylglycine molecule (in comparison with glycine one) results in the greater destruction of a spatial (tetrahedral-coordinated) H-bond network in the sur-

rounding aqueous medium. Herewith the total effect induced by interaction of glycylglycine with components of the mixed solvent becomes more endothermic. The weakening of solvation of glycylglycine in going from (H_2O + AN) to aqueous FA, 1,4-DO, DMSO or DMF is associated with strengthening the intermolecular interactions in these mixtures, as it follows from the enthalpies of mixing [33]. The given circumstance leads to an increase in the endothermic contribution from the dehydration of the co-solvent molecules. Taking into account the fact that the hydrophobicity of methyl groups in AC are stronger than those of methylene groups in 1,4-DO, one can understand why the curve 3 (AC) in Fig. 1 lies higher than the curve 2 (1,4-DO) in the same figure.

The interparticle interactions in the ternary aqueous system with small concentration of co-solvent can be characterized in terms of the McMillan–Mayer theory [34] adapted by Kauzmann, Friedman and Desnoyers [35–37] for calculating the enthalpy coefficients of pair-wise interactions h_{xy}. For this purpose, the $\Delta_{sol}H^{\circ}$ versus m_2 functions were approximated by the third-order polynomial equation

$$\Delta_{sol}H^{\circ} = a_0 + a_1 m_2 + a_2 m_2^2 + a_3 m_2^3 \quad (2)$$

where m_2 is the molal concentration of the alcohol, and a_i are coefficients calculated by a least-squares method. The correlation coefficient, R, and the Student criterion value, t_α, ranged from 0.993 to 0.998 and from 0.170 to 0.312, respectively. The h_{xy} value was calculated from the a_1 coefficient related to the coefficient of pair-wise interactions as $h_{xy} = a_1/2$ [37]. The calculated enthalpy coefficients of pair-wise interaction for the glycylglycine–co-solvent pair are presented in Table 2. The analogous data concerning the interactions of EtOH, PrOH, i-PrOH [12], DMSO [7] and AN [8] with glycylglycine are also given in Table 2. The negative values of h_{xy} for (H_2O + FA) and (H_2O + AN) suggest that the interactions between the co-solvent and glycylglycine molecules dominate over the effects of their dehydration. On the contrary, a positive sign at the value of h_{xy} in the case of (H_2O + 1,4-DO), (H_2O + AC), (H_2O + MFA), (H_2O + DMF), (H_2O + DMSO) and (H_2O + alcohol) mixtures suggest that the effects of dehydration of glycylglycine and co-solvent molecules dominate over the direct interactions between them. The obtained results show that the interaction between the co-solvent and glycylglycine molecules depends on the structure properties of the former.

In the previous works [9–11], we have showed that the enthalpy coefficients of pair-wise intermolecular interaction of amino acids or peptides with organic co-solvents depend on the properties of these solvents and can be quantitatively expressed in the form of the multi-parameter equation (by the LSER principle). For these purposes, we have used the Kamlet–Taft modified correlation [38,39]. Herewith the influence of various co-solvent properties on its interaction with glycylglycine have been estimated using the following equation:

$$h_{xy} = A_0 + A_1\left(\frac{\delta^2}{1000}\right)_1\left(\frac{V_2}{100}\right) + A_2(\pi_1^*\pi_2^*) + A_3(\alpha_1\beta_2) + A_4(\beta_1\alpha_2) \quad (3)$$

where π_1^* and π_2^* are the parameters characterizing the dipolarity/polarizability of H_2O and organic co-solvent, respectively; α_1, α_2 and β_1, β_2 are the parameters corresponding to the solvent acidity and basicity, respectively; $\delta^2/1000$ and $V_2/100$ are the structural contributions of both water and the co-solvent to the formation of cavities (these parameters are decreased respectively by factors of 1000 and 100 to simplify the estimation of the relative contributions of various parameters to h_{xy}); δ^2 is the solubility parameter according to Hildebrandt conception (δ^2 is proportional to the cohesion energy density) and V_2 is the molar volume of the organic solvent being the ratio between its molecular weight and density. The solvent parameters necessary for the calculations are listed in Table 2. As a result, we obtained the equation:

$$h_{xy} = 510.08 + 5.44\left(\frac{\delta^2}{1000}\right)_1\left(\frac{V_2}{100}\right) - 1289.42(\pi_1^*\pi_2^*) - 2786.93(\alpha_1\beta_2) + 1463.15(\beta_1\alpha_2),$$
$$R = 0.947,\quad N = 10,\quad SD = 125.80 \quad (4)$$

From Eq. (4) follows that a simultaneous increase in polarity, polarizability and electron–donor ability of the organic solvent enhances its interparticle interaction with the glycylglycine molecules. On the other hand, an increase in the molar volume and electrophilic properties of the organic co-solvent weakens the above-specified pair-wise interactions.

For comparison, we present the equation relating the enthalpy coefficients of pair-wise interactions of glycine with the same organic co-solvents

$$h_{xy} = -21.20 + 547.99\left(\frac{\delta^2}{1000}\right)_1\left(\frac{V_2}{100}\right) - 966.91(\pi_1^*\pi_2^*) - 139.35(\alpha_1\beta_2) + 461.75(\beta_1\alpha_2),\quad R = 0.989,\quad N = 9 \quad (5)$$

It is noteworthy that all the variables in Eqs. (4) and (5) coincide by sign. This fact means that the interparticle interactions of both glycine and glycylglycine with the same co-solvents have a similar nature. However, the values of coefficients at the variables in Eq. (4) are higher in magnitude than the similar values including in Eq. (5). Increasing coefficients at the variables in the case glycylglycine, compared to amino acid, is connected with rising specific solvation of the peptide group with increasing the molar volume of glycylglycine as well as with strengthening its donor–acceptor properties. The greatest contribution to the strengthening of interparticle interaction between glycylglycine and organic co-solvent is caused by the polarity/polarizability ($\pi_1^*\,\pi_2^*$-parameter) being a measure of the co-solvent ability to stabilize a charge or a dipole by virtue of its dielectric effect. On the contrary, the greatest contribution to the weakening of interparticle interaction between glycylglycine and co-solvent is caused by the electrophilicity ($\beta_1\alpha_2$-parameter), which describes the ability of the co-solvent to H-bonding formation.

Comparison of the results obtained in the present work and those that we have been reported earlier in [6–9] led us to conclude that the main factors affecting intermolecular interaction between glycylglycine and organic co-solvent in the mixed aqueous solutions considered are following:

(a) The energy of forming the mixed solvent {weak intermolecular interactions in the mixtures (H_2O + FA) and (H_2O + AN)

Table 2
Enthalpic coefficients of pair-wise interactions (h_{xy}, J kg mol^{-2}) between glycylglycine and organic solvents in aqueous solutions at 298.15 K and parameters of organic solvents [27].

Solvent	h_{xy}	$(\delta^2/1000)_1(V_2/100)$ (J/mol)	π^*	α	β
FA	−197 ± 16	0.912	0.97	0.71	0.55
AN	−17 [8]	1.203	0.75	0.19	0.31
1,4-DO	400 ± 14	1.955	0.55	0.00	0.37
AC	175 ± 27	1.684	0.71	0.08	0.48
DMSO	427 [7]	1.627	1.00	0.00	0.76
MFA	238 ± 6	1.340	0.90	0.62	0.80
DMF	479 ± 34	1.775	0.88	0.00	0.69
EtOH	515 [12]	1.339	0.54	0.83	0.77
n-PrOH	795 [12]	1.714	0.52	0.78	0.82
i-PrOH	1245 [12]	1.756	0.48	0.76	0.95
H_2O	–	–	1.09	1.17	0.18

promoting the solvation of glycylglycine and strong intermolecular interactions in the mixtures (H_2O + 1,4-DO), (H_2O + AC), (H_2O + MFA), and (H_2O + DMF) weakening the peptide solvation}.

(b) The nature and content of the organic co-solvent (increase in the mole fraction of organic co-solvent can gradually increase the exothermicity of solvation of glycine and its oligomers, as it takes place in the case of aqueous AN and FA, and result in the appearance of extreme concentration dependences, for example, in the case of aqueous alcohols, 1,4-DO, AC, MFA, DMF and DMSO).

(c) The presence in a glycylglycine molecule the peptide group promoting a specific salvation of this molecule, but this contribution to the solvation energy is not always exceed the consumption from dehydration of the peptide molecule.

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