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DENSIMETRIC INVESTIGATIONS OF WATER-ACETAMIDE SYSTEM WITHIN  
THE TEMPERATURE RANGE 25-85°C

The density of aqueous acetamide solutions was measured over the temperature range 25-85°C.

From the obtained density data apparent molal volume of acetamide in water and volume expansibility coefficient were calculated.

The analysis of changes of these functions in relation to acetamide concentration and temperature allowed to conclude on breaking effect of acetamide on water structure.

The thermodynamic investigations of electrolyte solutions in molten acetamide showed certain similarity of the molten acetamide to water used as electrolyte solvents. It seems possible that this similarity is due to the existence of the partially disturbed three-dimensional network of H-bonds in both solvents [1].

The present paper deals with the densimetric investigations of acetamide aqueous solutions which we intend to use as electrolyte solvents. We have measured the density of water-acetamide system in the temperature range 25-85°C. Density values of the solutions enable the calculation of apparent molal volume of the solute and the volume expansibility coefficient of the investigated solutions. The concentration and temperature effect on the calculated quantities makes it possible to draw certain conclusions on the effect of the dissolved substance on the solvent.

## Experimental

### Reagents

The solutions used for investigations were prepared by mixing the weighted amounts of twice distilled water with acetamide. Analytical grade acetamide "Xenon-Łódź" product previously crystallized from methanol and dried for about 50 hours at the temperature  $60^{\circ}\text{C}$  was used for the investigations. The melting temperature of acetamide was  $81-82^{\circ}\text{C}$  which is in accordance with literature data [2-5].

### Apparatus

The density measurements of the investigated solutions were performed with the use magnetic densimeter constructed in our laboratory, basing on the description included in the works of Millero and Franks [6-8]. The densimeter applied by us consisted of a glass vessel (Pyrex) of 100 ml capacity, glass float with a magnetic core attached to it by means of polyethylene and a support made of brass, containing two solenoids in its lower part. The subsidiary upper solenoid served to move the float into the magnetic field of the lower measuring solenoid. The densimeter was used in a wide temperature range therefore the solenoids were insulated by polystyrene and epoxy resin. The weight of the float was 21.3933 g and its volume at the temp.  $25^{\circ}\text{C}$  was 22.6995 ml. The densimeter was placed in a thermostate bath keeping the desired temperature with accuracy to  $\pm 0.02^{\circ}\text{C}$ .

### The Density Measurements

The densimeter filled with solution was thermostated for about 60 min. By means of subsidiary solenoid the float is moved to the bottom of the glass vessel of densimeter and the current in the measuring solenoid is switched on. The intensity

of the current slowly decreases up to the moment when the float is detached from the bottom of the vessel. The measurement was being repeated till the current intensity at the moment of detachment of the float became reproducible. The densimeter was calibrated using twice distilled water as the standard liquid. The current intensity was determined by measuring the voltage drop on the standard resistance ( $1\Omega$ ) by means of digital voltmeter of the V 534 type by Mera-Tronic.

The density of the solution was calculated from the formula:

$$d = \frac{W + w + fi}{V + w/d_{Pt}} \quad (1)$$

where:

$W$  - the weight of float

$w$  - the weight of platinum rings loading the float

$f$  - solenoid constant

$i$  - current intensity in the measuring solenoid at the moment of departure of the float from the bottom,

$V$  - volume of the float

$d_{Pt}$  - platinum density

The solenoid constant and the float volume are determined by measuring the standard liquid density. The measurements carried out for aqueous NaCl and NaI solutions were in agreement with literature data with accuracy to  $\pm 1 \times 10^{-5} \text{ g/cm}^3$  (NaCl [9], NaI [10]).

### Results

The results of density measurements of acetamide solutions in water at the temperatures  $25^\circ\text{C}$ ,  $40^\circ\text{C}$ ,  $60^\circ\text{C}$ ,  $75^\circ\text{C}$  and  $85^\circ\text{C}$  are given in Table 1.

The density of the investigated solutions was used to calculate the apparent molal volume  $\Phi_v$  of acetamide in water using the well-known formula:



Densities of aqueous acetamide solutions

wt %	$x_{\text{AcNH}_2}$	25°C		40°C	
		d	$\Phi_V$	d	$\Phi_V$
		$\frac{\text{g}}{\text{cm}^3}$	$\frac{\text{cm}^3}{\text{mole}}$	$\frac{\text{g}}{\text{cm}^3}$	$\frac{\text{cm}^3}{\text{mole}}$
1	0.0031	0.99769	55.56	0.99279	56.23
2	0.0062	0.99831	55.56	0.99336	56.18
5	0.0158	1.00022	55.51	0.99505	56.17
8	0.0258	1.00219	55.46	0.99675	56.16
10	0.0328	1.00355	55.42	0.99792	56.14
15	0.0510	1.00695	55.37	1.00081	56.13
20	0.0708	1.01029	55.37	1.00367	56.14
25	0.0922	1.01374	55.35	1.00651	56.15
30	0.1155	1.01715	55.34	1.00934	56.17
40	0.1688	1.02378	55.38	1.01482	56.22
50	0.2336	1.03024	55.43	1.01995	56.30
60	0.3137	-	-	1.02398	56.44
70	0.4155	-	-	1.02704	56.65
85	0.6333	-	-	-	-
95	0.8527	-	-	-	-
100	1	-	-	-	-

Table 1

in the temperature range 25-85°C (g/cm<sup>3</sup>)

60°C		75°C		85°C	
d	$\Phi_V$	d	$\Phi_V$	d	$\Phi_V$
$\frac{\text{g}}{\text{cm}^3}$	$\frac{\text{cm}^3}{\text{mole}}$	$\frac{\text{g}}{\text{cm}^3}$	$\frac{\text{cm}^3}{\text{mole}}$	$\frac{\text{g}}{\text{cm}^3}$	$\frac{\text{cm}^3}{\text{mole}}$
0.98371	57.21	0.97532	57.92	0.96904	58.53
0.98418	57.21	0.97573	57.98	0.96942	58.56
0.98558	57.22	0.97698	58.00	0.97055	58.59
0.98696	57.24	0.97822	58.01	0.97168	58.60
0.98790	57.24	0.97902	58.04	0.97244	58.60
0.99026	57.24	0.98102	58.07	0.97422	58.66
0.99244	57.29	0.98310	58.06	0.97603	58.67
0.99472	57.30	0.98515	58.07	0.97783	58.69
0.99697	57.32	0.98726	58.06	0.97972	58.68
1.00152	57.34	0.99099	58.13	0.98297	58.76
1.00570	57.39	0.99431	58.22	0.98616	58.82
1.00910	57.51	0.99708	58.34	0.98871	58.92
1.01163	57.67	0.99960	58.45	0.99070	59.04
1.01329	57.98	1.00079	58.75	0.99224	59.28
-	-	-	-	0.99190	59.47
-	-	-	-	0.99072	59.62

$$\Phi_{v, \text{AcNH}_2} = \frac{1000(d_0 - d)}{m d_0} + \frac{M_{\text{AcNH}_2}}{d} \quad (2)$$

where:

$M_{\text{AcNH}_2}$  - the molecular weight of  $\text{AcNH}_2$

$m$  - the concentration of  $\text{AcNH}_2$  in water in moles per 1000 g of water

$d_0$  - the density of water

$d$  - the density of the solution

The obtained values  $\Phi_{v, \text{AcNH}_2}$  are shown in Table 1.

The concentration dependence of  $\Phi_v$  can be described by the polynomial [11]:

$$\Phi_v = \Phi_v^0 + A_v m + B_v \cdot m^2 + \dots \quad (3)$$

where:

$\Phi_v^0$  - apparent molal volume of nonelectrolyte in the infinitely diluted solution.

In the case of the average concentrated solutions the equation (3) can be applied [12]:

$$\Phi_v = \Phi_v^0 + A_v \cdot m \quad (4)$$

The value of  $A_v$  slope is interesting because according to Mac Millan-Mayer's solution theory it refers to the interactions among nonelectrolyte molecules [13-16].

The values  $\Phi_v^0$  obtained by extrapolation of the equation (4) up to  $m = 0$  are given in Table 2. For comparison the values of  $\Phi_v^0$  and  $A_v$  for other nonelectrolytes have been also placed in this Table [17-22].

The volume expansibility coefficient of the examined acetamide solutions in water was calculated from the formula:

$$\alpha = -\frac{1}{d} \cdot \left( \frac{\partial d}{\partial T} \right)_{p,x} \quad (5)$$

The derivative  $\partial d / \partial T$  was calculated analytically knowing the values of the coefficients of the equation  $d = a + bt +$

+  $ct^2$  representing the dependence of density on temperature. The obtained values of volume expansibility coefficients of acetamide solutions in water are given in Table 3.

Table 2

Partial molal volumes  $\Phi_V^0$  of nonelectrolytes in water

	$\Phi_V^0$	Av
	$\text{cm}^3 \cdot \text{mole}^{-1}$	$\text{cm}^3 \cdot \text{kg} \cdot \text{mole}^{-2}$
F	38.6	0.06 [17, 18]
NMF	56.87 (25°)	-0.15
	57.45 (35°)	-0.15
	57.95 (45°)	-0.11 [12]
AcNH <sub>2</sub>	55.61 (25°)	-0.123 [11]
	55.6 (25°)	-0.11
	56.2 (40°)	-0.05
	57.2 (60°)	0.01
	58.0 (75°)	0.01
	58.5 (85°)	0.01
CH <sub>3</sub> COCH <sub>3</sub>	66.92 (25°)	-0.370 [11]
	67.0 [17]	
DMSO	68.92 (25°)	-0.262 [11]
CH <sub>3</sub> OH	38.3 [19, 20]	-0.2
CO(NH <sub>2</sub> ) <sub>2</sub>	44.24 (25°)	0.116 [17, 21, 22]
	44.8 (60°)	0.07
	43.8	



Table 3

Volume expansibility coefficient of aqueous acetamide solutions  
( $10^6 \alpha$  [deg<sup>-1</sup>])

wt % AcNH <sub>2</sub>	X AcNH <sub>2</sub>	25°C	40°C	60°C	75°C	85°C
1	0.0031	289	390	519	621	691
2	0.0062	293	391	522	622	694
5	0.0158	308	403	535	634	704
8	0.0258	328	421	547	645	711
10	0.0328	342	432	556	651	715
15	0.0510	372	459	579	672	735
20	0.0708	412	493	602	687	744
25	0.0922	450	524	626	703	756
30	0.1155	491	555	646	714	762
40	0.1638	554	613	694	757	801
50	0.2336	635	680	740	788	821
60	0.3137	-	696	769	825	864
70	0.4155	-	700	783	850	895

### Discussion

Fig. 1 illustrates the dependence of density of aqueous acetamide solutions on concentration and temperature. As it is seen on the plot the density of aqueous acetamide solutions increases with the growth of acetamide (AcNH<sub>2</sub>) concentration in the solution and it decreases with the temperature growth. The range of rectilinear relation  $d = f(\text{wt } \%)$  decreases with the temperature growth. At the temp. 85°C higher than acetamide melting temperature (81-82°C) the curve  $d = f(\text{wt } \%)$  has a maximum for the solutions containing 85 weight % of AcNH<sub>2</sub> i.e. about 63.33 mole % of AcNH<sub>2</sub>. Thus it may be presumed that the solution containing two acetamide molecules falling on one water molecule has maximum packing of molecules.

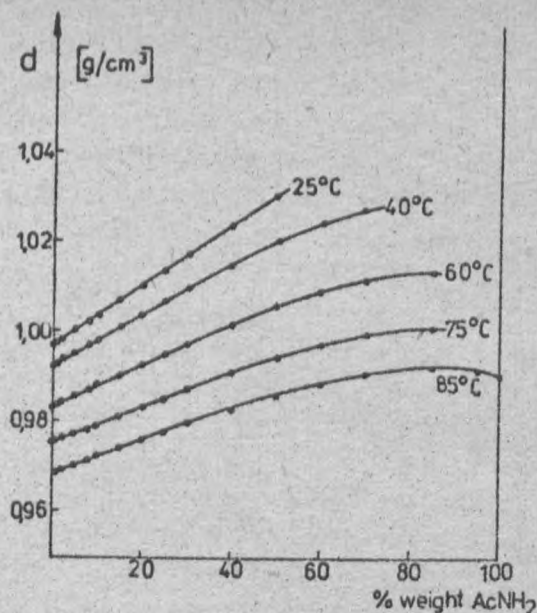


Fig. 1. The dependence of density of aqueous acetamide solutions on concentration and temperature

The X-ray [23-25] and spectroscopic [26-27] investigations showed that in solid state acetamide three-dimensional network of  $\text{NH}\dots\text{O}$  hydrogen bonds occurs. In liquid acetamide a strong association was observed [28-30]. It is justifiable, because each acetamide molecule can form hydrogen bond both as donor and acceptor of proton. It follows from the spectroscopic investigations [26-27] that energy of hydrogen bonding in acetamide is close to the bonding energy in water. The length of bondings is also similar:  $2.76 \text{ \AA}$  in water and  $2.86 \text{ \AA}$  in acetamide. So it is probable that the bond between water and acetamide molecules differs only a little from the bondings in pure components.

Assuming that at the temp.  $85^\circ\text{C}$ , near the melting point of  $\text{AcNH}_2$  ( $81\text{--}82^\circ\text{C}$ ) its three-dimensional structure will be only slightly disturbed it may be presumed that water molecules can built in acetamide structure interstitials.

If all the vacancy of acetamide structure were filled with



water molecules it follows from the crystallographic analysis [31] that one water molecule should fall on two acetamide molecules. It corresponds with the solution with the composition 66.6 mole % of  $\text{AcNH}_2$ , which is in agreement with the placement of maximum on the  $d = f(x)$  curve obtained in this work. Apparent molal volumes of some nonelectrolytes in water have been calculated, using the data on the density of the examined so-

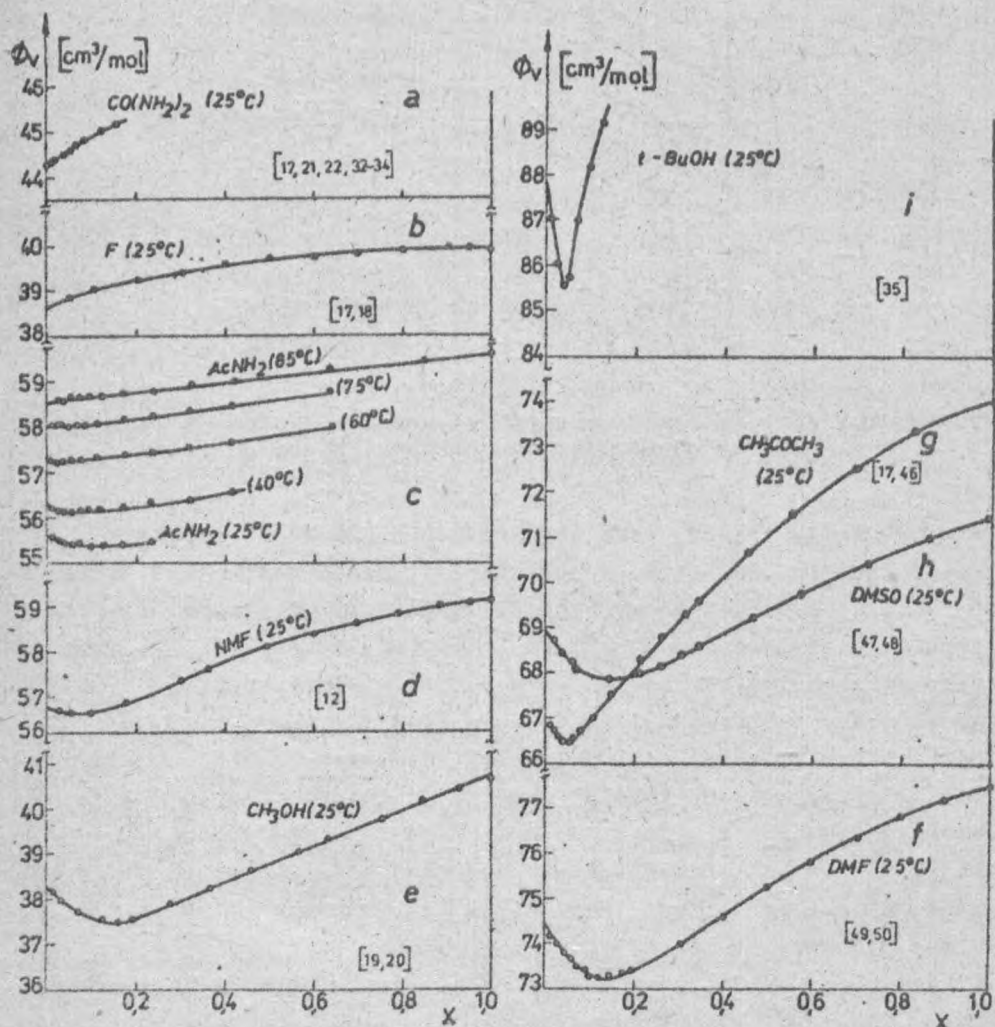


Fig. 2. The apparent molar volume of nonelectrolytes as a function of the mole fraction of nonelectrolyte in water

lutions of acetamide in water and literature data concerning other nonelectrolytes. The obtained results are presented as the function  $\Phi_v = f(x)$  on Fig. 2.

As it is seen from Table 1 and from Fig. 2c the course of  $\Phi_v$ ,  $\text{AcNH}_2$  concentration relation changes with the growth of temperature. At the temp.  $25^\circ\text{C}$  this relation shows small minimum in the solution with concentration  $\sim 10$  mole %  $\text{AcNH}_2$ . The minimum decreases with the growth of temperature and starting from  $60^\circ\text{C}$  it does not exist any more.

In case of aqueous solutions of urea (U) [17, 21, 22, 32-34] and formamide (F) [17, 18] (Fig. 2a, 2b)  $\Phi_v$  increases with the growth of nonelectrolyte concentration minimum does not occur. In the remaining systems presented on Fig. 2 minimum appears on curve  $\Phi_v = f(x)$ .

According to many authors [11, 35-36] minimum appearing on curve  $\Phi_v = f(x)$  is characteristic for hydrophobic substances. It seems that the depth of minimum may be treated as a measure of hydrophobic character of nonelectrolyte. It is seen from Fig. 2 that methanol (MeOH), dimethylsulphoxide (DMSO) and dimethylformamide (DMF) show almost analogous minima at about 15 mole % of nonelectrolyte content in water. Thus it can be presumed that these substances display similar hydrophobic character and have similar influence on water structure.

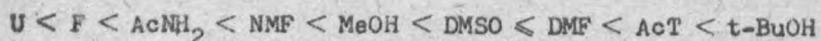
According to Avédikian, Perron and Desnoyers [35] these substances probably order water structure within the concentration range 0-15 mole % of nonelectrolyte while in more concentrated solutions they disorder it, because of the formation of mixed hetero-associates. The greatest minimum can be observed in case of aqueous *t*-BuOH solutions. Strong hydrophobic interactions between *t*-butanol and water is undoubtedly connected with greater number of methyl groups in a molecule of this alcohol in comparison to the above mentioned substances.

Applying the above criterion of hydrophobicity it can be easily observed that acetone (AcT) displays hydrophobic character intermediate between *t*-BuOH and DMSO, DMF and MeOH. Stronger hydrophobic character of AcT in comparison to MeOH is probably caused by the presence of two methyl groups in AcT. The weaker hydrophobic character of DMSO and DMF in comparison to

AcT is probably caused by higher dipole moment of these substances (DMSO - 3.96, DMF - 3.86, AcT - 2.76) [11].

In case of NMF-H<sub>2</sub>O system (NMF-N-methylformamide) [12] and AcNH<sub>2</sub>-H<sub>2</sub>O (25°, 40°C) a small minimum occurs, which may be the proof of a very weak hydrophobic substances of these compounds. With the temperature growth the hydrophobic character of AcNH<sub>2</sub> and NMF decreases. Above 60°C in case of AcNH<sub>2</sub> the hydrophilic character begins to dominate. Similarly, on the basis of course of  $\Phi_v = f(x)$  curve of aqueous formamide solutions it can be observed that the hydrophilic character of this compounds prevails. The replacement of hydrogen in amine group by methyl group in amine group (F i NMF) increases, as it can be presumed the hydrophobic character of nonelectrolyte. The presence of methyl group at carboxyl carbone (F and AcNH<sub>2</sub>) has a similar effect but it is slightly smaller ( $\Phi_{v,NMF}$  grows more the solution concentration growth than  $\Phi_{v,AcNH_2}$ ). The last of the discussed nonelectrolytes - urea(U) shows a strong hydrophilic character. Urea introduced to water causes water structure disordering [37].

Because of the hydrophobic character the discussed nonelectrolytes can be arranged in the following way:



The examined AcNH<sub>2</sub> exhibits very small hydrophobic and hydrophilic interactions with water. Comparing the results of the analysis of  $\Phi_v$  and thermodynamic values both of binary mixtures and electrolyte solutions in these mixtures we can observe some discrepancies. As for as DMF is concerned it follows from the thermodynamic analysis [18] that this substance disturbs water structure. A similar order of the hydrophobic character may be observed when the values  $A_v$  given in Table 2 are discussed. The smallest absolute values  $A_v$  were obtained for AcNH<sub>2</sub> and F which according to literature [19-22], points to the greater capability of hydrogen bond formation among AcNH<sub>2</sub> and F molecules in comparison to the remaining substances. Certain information about the effect of the dissolved substance on the solvent structure can be obtained from the analysis of the dependence of volume expansibility coefficient  $\alpha$  on the

concentration of the solution and on the temperature. It follows from works of Krumgalz [38, 39] and Baron [40] that the increase of temperature or introduction of some electrolytes breaking the solvent structure cause the increase of the values of the above mentioned coefficient. Solvent structure ordering electrolytes, on the other hand, cause the lowering of the coefficient  $\alpha$  value.

In order to get additional information about acetamide effect on water structure the values of aqueous acetamide solution coefficient  $\alpha$  were calculated over the temperature range 25-85°C (formula 5). As it is seen from Fig. 3 the volume expansibility coefficient  $\alpha$  of aqueous acetamide solutions grows with the increase of the concentration of the solution and its temperature.

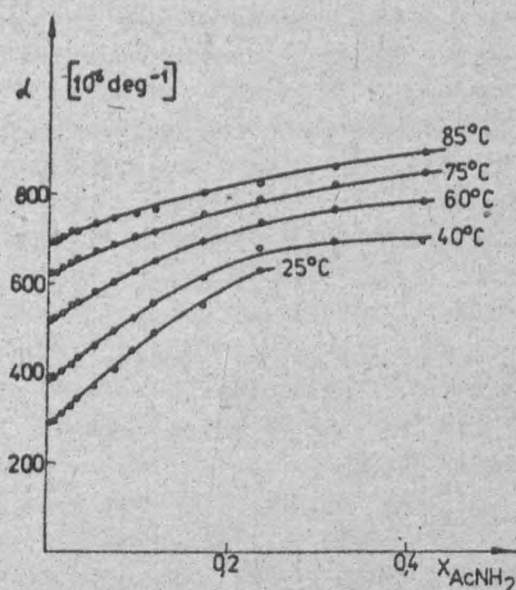


Fig. 3. The volume expansibility coefficient as function of the concentration and temperature

It follows that the more concentrated solutions expands more than the diluted ones, which may point to the disordering effect of the introduced acetamide on water structure. At a higher temperature the values of the coefficient  $\alpha$  are also greater, since the solution expands more easily because of more inten-



sive thermal motions of the molecules. At the same time the effect of the dissolved substance on the value of coefficient  $\alpha$  decreases, as the  $\alpha = f(x)$  curve slope indicates.

Quite obviously there exists a temperature at which the coefficient  $\alpha$  does not depend on the concentration of the solution, whereas above this temperature the coefficient  $\alpha$  will decrease with the growth of concentration of the solution. This temperature may be called the inversion temperature. At this temperature the effect of disordering the solvent structure compensate with the effects of solvation, association etc. From the course of the relation  $\alpha = f(T)$  of aqueous acetamide solutions it follows that the inversion temperature is in this case  $123^{\circ}\text{C}$ , so it is above water boiling temperature. It also follows that over the temperature range  $0-100^{\circ}\text{C}$  acetamide weakly disorders water structure.

The above opinion is in agreement with the conclusions arising from the analysis of the course of relation of the excess of the relative partial molal entropy of water in aqueous acetamide solutions on concentration and temperature [41] and is compliant with the conclusion from the dielectric permittivity of  $\text{AcNH}_2\text{-H}_2\text{O}$  system [42-44]. Gonczarow et al. [42, 43] claim that  $\text{AcNH}_2$  molecule can without greater difficulty built in water structure if this molecule occupies one interstitial hole and three lattice nodes. The acetamide molecule forms hydrogen bonds with water molecules slightly changing the length of hydrogen bonds and the angles between them. According to the above mentioned authors [42, 43] these bondings are a bit weaker than the hydrogen bondings in pure water. Unlike water molecules, the polar groups of  $\text{AcNH}_2$  molecules cannot form four hydrogen bonds, so in the new structure of  $\text{H}_2\text{O-AcNH}_2$  system there will be much more broken hydrogen bonds than in pure water.

Then tetrahedric structure occurs constructed from water and acetamide, which is gradually destructed at higher  $\text{AcNH}_2$  content in the solution. The opinion about breaking water structure by  $\text{AcNH}_2$  molecules is also expressed by Christoffers and Kegeles who examined the diffusion coefficient of acetamide in water at the temp.  $25^{\circ}\text{C}$  [45].

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DENSYMETRYCZNE BADANIA UKŁADU WODA-ACETAMID W ZAKRESIE  
TEMPERATURY 25-85°C

Zmierzono gęstość wodnych roztworów acetamidu w zakresie temp. 25-85 C. Na podstawie otrzymanych badanych gęstości obliczono wartości pozornej molowej objętości acetamidu w wodzie oraz współczynnik rozszerzalności objętościowej danych roztworów. Analiza zmian wymienionych wielkości w zależności od stężenia acetamidu i temperatury pozwoliła wysnuć wniosek o niszczącym wpływie acetamidu na strukturę wody.

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ДЕНСИТОМЕТРИЧЕСКИЕ ИССЛЕДОВАНИЯ СИСТЕМЫ ВОДА-АЦЕТАМИД  
В ИНТЕРВАЛЕ ТЕМПЕРАТУРЫ 25-85°C

Определена плотность системы вода-ацетамид в интервале температуры 25-85 C. Рассчитано кажущийся и парциальный молярный объём, а также коэффициент термического расширения  $\alpha$ . Анализ полученных данных в зависимости от концентрации и температуры позволил сделать вывод, что ацетамид разрушает структуру воды.