Scientific paper

Ageing of Water or Dissolution of Glass: An Electrical Conductivity Study

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Received: 19-10-2014

Abstract

Water and water based solutions are often stored in glass vessels. There are many studies in the literature dealing with solubility of glass-strictly speaking leaching of its components-in water. In present work the leaching process was investigated by measuring the electrical resistance/conductivity of water in a gas tight closed cell under nitrogen atmosphere during three weeks in the temperature range from 5 °C to 40 °C. From obtained results it was concluded, that the observed increase in electrical conductivity of water-actually extremely diluted solution of ions released form glass – in the used time period, can be ascribed to the leaching of ions from the glass solely.

Kaywords: Water, glass, dissolution, leaching, silicate, electrical conductivity

1. Introduction

Water is the most common and vital medium for all the biochemical reactions that constitute the living process and takes part in many of these reactions. Despite the chemical simplicity of the water molecule, its bulk properties are very peculiar and have been still investigating. Nevertheless, there is also already a huge amount of literature devoted to water and its unique behaviour demonstrating that recent physico-chemical studies, allied to increasingly sophisticated computer simulations, have reached the stage where many of the old controversies have been resolved. Nowadays structuring in liquid water is being described with increasing confidence, in the pure liquid, at interfaces and in solutions.¹

However, the term »pure liquid« refers to an ideal liquid system without any additions or impurities. In reality, all "pure liquids" are actually extremely diluted solutions containing the impurities in traces. At our laboratory work it is believed that these impurities do not affect the experiments and results, what usually is true. But when glassware is used for the storage of water samples, reagents and standard solutions, dissolution of silicate and other ions from the glass containers can contaminate the samples. Several studies have been carried out to demonstrate that dissolution from glassware can introduce micromolar silicate within a few hours.^{2,3} It was found that the extent of dissolution depends on contact time, salinity and pH of the solution as also on the surface area of the glass-water interface which can be correlated to the size and shape of the containers.²

The kinetics study of dissolution of amorphous silica, SiO₂(am), in deionized water and NaCl solutions in the temperature range from 40 °C to 250 °C⁴ revealed that absolute dissolution rates of SiO₂(am) in deionized water are ~10 times faster compared to quartz. The introduction of NaCl to near-neutral pH solutions (~0.05 M NaCl) enhances rates by ~21 times compared to deionized water.

The degradation behaviour of borosilicate glass with the nominal composition 0.70 SiO₂, 0.039 Na₂O, 0.028 K_2O , 0.21 B₂O₃, and 0.01 Al₂O₃ was investigated at different temperatures in acid and alkaline media by Jagannath et al. in 2006.⁵ They reported that the chemical attack was less expressed in acid medium compared to that in alkaline medium.

The kinetics of corrosion of alkali borosilicate glasses – containing apart from the above mentioned constituents also ZnO, Fe_2O_3 , Cs_2O , BaO and PbO – in water was studied recently using glass samples ground to a powder with a grain size ranging from 60 to 90 µm following the concentration of caesium and silicate ions.⁶ It was found that the corrosion of this system can be treated as two coupled processes, diffusion and dissolution.

During the dissolution of glass in water several processes take place. If the glass contains any alkali or other highly mobile ions, the ion exchange between these ions and the protonic species (H_3O^+) from the solution can occur. Since all commercial silicate glasses contain alkali ions, the initial step in their dissolution usually involves this process, which can be represented – for example – by following reaction⁴

$$\equiv \text{Si-O-Na} (\text{glass}) + \text{H}_3\text{O}^+ (\text{aq}) \rightarrow$$

$$\rightarrow \equiv \text{Si-O-H} + \text{Na}^+ (\text{aq}) + \text{H}_2\text{O} (\text{l}) \qquad (1)$$

and/or3

$$\equiv \text{Si-O-Na} (\text{glass}) + \text{H}_2\text{O} (1) \rightarrow$$

$$\rightarrow \equiv \text{Si-O-H} + \text{Na}^+ (\text{aq}) + \text{OH}^- (\text{aq}) \qquad (2)$$

The increase in the pH of the solution due to this ion exchange may increase the solubility of the silica in the solution, according to the reaction³

$$\frac{\text{SiO}_2(\text{glass}) + 4 \text{ OH}^-(\text{aq}) \rightarrow}{\rightarrow \text{SiO}_4^{4-}(\text{aq}) + 2 \text{ H}_2\text{O}(\text{l})}$$
(3)

or probably even more likely

$$SiO_2(glass) + 2 OH^-(aq) \rightarrow H_2SiO_4^{2-}(aq)$$
(4)

If these reactions occur in the dissolution process silicate may be released in solution without any significant change in the pH, but "pure water" in a glass vessel must be treated as an extremely diluted solution of different ions. One must be aware that the silicate chemistry is rather complex and here also many other equilibria can take place.

However, there is no kinetic study on the corrosion of the glass from the glass walls in the glassware used in the laboratory every day. In this work the "leaching" of the components of the glass was followed three weeks by measuring the electrical resistance of water in a fully closed borosilicate glass cell under nitrogen in the temperature range between 5 °C and 40 °C. The dissolution of glass was treated as a heterogeneous process occurring on the solid – liquid phase interface controlled by diffusion mainly as it is usual for low soluble solid substances. The rate constants were estimated and – from the reported data on limiting molar conductivities of sodium silicate – a rough assessment of the concentration of silicate ion in water was made.

2. Experimental

2.1. Materials

Demineralised water was distilled in a quartz bidistillation apparatus (DESTAMAT Bi18E, Heraeus). The final product with specific conductivity of less than $5 \cdot 10^{-7}$ S cm⁻¹ was distilled into a flask allowing the storage and the transfer of water in the conductivity cell under a nitrogen atmosphere (Figure 1a).



Figure 1. a) A flask for the collecting and storage of triple distilled water under nitrogen atmosphere.⁷ b) Applied three-electrode conductivity cell (E_1 , E_2 , E_3) with mixing chamber (M) and solvent or stock solution inlet (I), stirrer (S) and stirrer motor (SM) mounted in the assembly lid (A) for immersion in the temperature bath.^{8,9}

2.2. Method

The conductivities of water and solutions were determined with the help of three-electrode flow-through cell, made from borosilicate glass, shown in Figure 1b.^{8,9} By calibration with dilute potassium chloride solutions¹⁰ the cell constant of $B = 0.8114 \pm 0.0001$ cm⁻¹ was determined. To avoid the contact with CO₂, triple distilled water was transferred in the conductivity cell from the flask under nitrogen atmosphere.

The filled cell was blown again with nitrogen, closed with caps and immersed in the high precision thermostat described previously.¹¹ The monoethylene bath was set to each temperature of a temperature programme with reproducibility better than 0.005 °C. The temperature was additionally checked with a calibrated Pt100 resistance thermometer (MPMI 1004/300 Merz) connected to an HP 3458 A multimeter. Resistance was recorded with a PCinterfaced impedance analyzer LCR Meter Agilent 4284 A connected to the electrodes in the measuring cell.

The resistance of water was measured three weeks each day in the temperature range from 5 °C to 40 °C (in steps of 5 °C) at frequencies between 1 and 10 KHz (in steps of 0.2 kHz). At each set temperature the system was kept at least one hour to assure the thermal equilibrium. After finishing the measurement at 40 °C, the system was cooled down to 5 °C and the procedures was repeated. For one temperature cycle about 10 hours was needed, so the experimental error in time (measured in days) can be estimated as \pm 5 hours. By help of the stirrer and stirrer motor mounted in the assembly lid (see Fi-

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gure 1b) water was stirred without interruption with a constant speed, $v = -60 \text{ min}^{-1}$.

A home-developed software package was used for temperature control and acquisition of resistance data.

3. Results and Discussion

3. 1. Electrical Conductivity of Water

A typical frequency dependence of measured resistance at different temperatures is presented in Figure 2. Evidently, the resistance decreases with increasing temperature, but reaches a maximum value, R_{max} , at a given frequency, v_{max} . Interestingly this maximum appears at 5 °C at low frequency ($v_{max} \approx 2 \text{ kHz}$) and is the most expressed. With increasing temperatures the maximum is shifted towards higher frequencies and is less expressed. Due to the observed maximum in R we decided not to carry out an

extrapolation of the recorded frequency-dependent resistance R(v) = f(1/v), to 1/v = 0, but to read its maximum value, R_{max} .

The time dependence of R_{max} is presented on Figure 3a. At 35 °C and 40 °C it turned out that at later stage of experiment the maximum in resistance cannot be detected in this frequency rang but it should be reached at frequency exceeding the range of the instrument. In these cases the (highest) values at 10 k Ω were read and they are shown with dotted lines in Figure 3.

Measured resistance, R, can be converted to the electrical conductivity, κ , by help of known value of the cell constant, B, by using the relation $\kappa = B/R$. Thus, at R_{max} the minimum values of conductivity, κ_{min} , were estimated and are presented in Figure 3b. From Figure 3b it is evident that the electrical conductivity of water – under nitrogen atmosphere – is increasing despite the fact, that the cell was gas tightly closed and not opened during the



Figure 2. Frequency dependence of measured resistance: a) at different temperatures on the first day of experiment; b) at 25 °C during the whole time period.



Figure 3. Time dependence of a) resistance at the maximum, R_{max} , and b) corresponding conductivity, κ_{min} , of water in the investigated temperature range.

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experiment The results are not surprising, they are well in accordance to the literature data reporting the leaching of the ions from the glassware.^{1–6} Therefore, observed increase in the conductivity can be ascribed to the dissolution of glass in water solely.

3. 2. Kinetics of Glass's Dissolution in Water

Dissolution of glass in water can be regarded as dissolution of any other solid substances where the rate of the process is determined mainly by the diffusion transports the dissolved substance across a thin diffusion layer, δ . Here it can be assumed that the concentration of dissolved substance continuously decreases from the concentration of saturated solution, c_s , at the solid surface to the concentration, c, in the bulk solution. The driving force of diffusion is the concentration gradient according to first Fick's law,

$$\frac{\mathrm{dn}}{\mathrm{dt}} = -\mathrm{DS}\frac{\mathrm{dc}}{\mathrm{dx}} \tag{5}$$

where dn is the amount of the dissolved substance within time interval dt, D is the diffusion coefficient, S represents the total surface (phase interface) of the dissolved solid substance and dc/dx is the mentioned concentration gradient.

When the diffusion layer is very thin, the concentration gradient may be replaced by a single linear approximation and equation (5) can be rewritten in

$$\frac{dc}{dx} = -\frac{c_s - c}{\delta}$$
(6)

dn can be expressed as Vdc, where V is the volume of the solution and equation (5) gets the form

$$\frac{d\mathbf{c}}{d\mathbf{t}} = \frac{DS}{\delta V} (\mathbf{c}_{s} - \mathbf{c}) \tag{7}$$

For the investigated system the expression $DS/\delta V$ can be regarded as constant and we obtain

$$\frac{\mathrm{d}\mathbf{c}}{\mathrm{d}\mathbf{t}} = \mathbf{k} \cdot (\mathbf{c}_{\mathrm{s}} - \mathbf{c}),\tag{8}$$

where k represents the rate constant of dissolution. After separation of variables and integration we obtain

$$c = c_s(1 - e^{-k \cdot t}), \tag{9}$$

which is formally equivalent to the equation for the first order reaction kinetics. By integration in the time range from t = 0 (c = 0) to t, when the concentration of solute in the solution is c it follows

$$\ln \frac{c_{\rm s}}{c_{\rm s} - c} = \mathbf{k} \cdot \mathbf{t} \tag{10}$$

In diluted electrolyte solutions the conductivity is proportional to the concentration of ions what can be applied in equation (10) giving

$$\ln \frac{\kappa_{\rm s} \cdot \kappa_0}{\kappa_{\rm s} \cdot \kappa} = \mathbf{k} \cdot \mathbf{t} \tag{11}$$

 κ_0 is the conductivity of (pure) water. Values of κ_s can be estimated by help of extrapolation of $R_{max} = f(1/t)$ to 1/t = 0 as shown in Figure 4a. At extrapolation only values of R_{max} measured in last 10 days over 1 M Ω were taken into account, to avoid the experimental error in time and resistance. Extrapolated values R_{∞} are listed in Table 1.



Figure 4. a) Estimation of the resistance of the "glass saturated" solution, R_{ω} , at different temperatures. b) Plot of $\ln(1/R_{\omega}-1/R)$ as a function of time for the whole time period at different temperatures. From the slopes of the straight lines the rate constants of dissolution of glass were determined.

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The expression $\kappa_s - \kappa_0$ is constant at given temperature, thus the rate constant, k, can be evaluated from the slope of the diagram $\ln(1/R_{\infty}-1/R) = f(t)$, as it is presented on Figure 4b.

Estimated values of rate constants, k, are listed in Table 1. Evidently, the process is only slightly dependent on the temperature. The reason for the relatively small increase of k from 5 °C to 35 °C could be also the execution of the experiment, where every day temperature dependent conductance data were obtained but not only at one temperature during the course of the experiment (for example three weeks at 25 °C only). In this context, the rate constants given in Table 1 would signify the high internal consistence of the measured temperature dependent conductivity data also. Despite this hesitation, the observed systematic increase of electrical conductivity during the experiment at all temperatures can be ascribed to the release of ions from glass and thus obtained data reflect the kinetics of glass corrosion process, which is obviously more expressed at higher temperatures.

Table 1. Values of extrapolated resistance to $t = \infty$, R_{∞} , and estimated rate constants, k, in units day⁻¹ and s⁻¹.

| T/ °C | R _ω /MΩ | k/day ⁻¹ | $k/10^{-7} \cdot s^{-1}$ |
|-------|--------------------|---------------------|--------------------------|
| 5 | 1.320 | 0.0416 ± 0.0006 | 4.815 |
| 10 | 1.149 | 0.0437 ± 0.0006 | 5.058 |
| 15 | 0.9931 | 0.0448 ± 0.0005 | 5.185 |
| 20 | 0.8818 | 0.0437 ± 0.0006 | 5.058 |
| 25 | 0.8125 | 0.0451 ± 0.0006 | 5.220 |
| 30 | 0.7467 | 0.0454 ± 0.0006 | 5.255 |
| 35 | 0.7003 | 0.0474 ± 0.0005 | 5.486 |
| 40 | 0.6841 | 0.0544 ± 0.0007 | 6.296 |
| | | | |

From temperature dependence of rate constant k the activation energy, E_a , of the process can be obtained by help of Arrhenius relationship

$$\mathbf{k} = \mathbf{A} \cdot \mathbf{e}^{-\mathbf{E}_{a}/\mathbf{R}T} \tag{12}$$

where A is the Arrhenius parameter. From Arrhenius plot (Figure 5) values of $E_a = 2.6 \pm 0.4$ kJ/mol and A = 0.13 \pm 0.02 day⁻¹ were estimated for the temperature range between 5 °C and 35 °C.

From Table 1 and Figure 5 it is evident, that the process is considerable faster at 40 °C than at lower temperatures. This finding confirms also the previous assumption that the performed experiment does not signify only the high internal consistence of the measured temperature dependent conductivity data but provides an insight in the kinetics of glass corrosion.

3. 3. Solubility of Glass in Water

The electric conductivity of sodium silicate in aqueous solutions was investigated thoroughly by Ukihashi in



Figure 5. Arrhenius plot for the investigated dissolution of glass. From the slope of the straight line the activation energy, E_a , was estimated.

fifties.^{12,13} His measurements were performed for the aqueous solutions of sodium silicate prepared by SiO_2 and Na_2O with different molar ratio. The data analysis based on the assumption of the presence of sodium silicate in the form $Na_2(H_2SiO_4)$ dissociating in solution in

$$Na_2(H_2SiO_4) \rightarrow 2 Na^+(aq) + H_2SiO_4^{2-}(aq)$$
 (13)

accompanying by hydrolysis which can take place in two steps,

$$H_2SiO_4^{2-}(aq) + H_2O(l) \rightarrow H_3SiO_4^{2-}(aq) + OH^{-}(aq)$$
 (14)

$$\mathrm{H}_{3}\mathrm{SiO}_{4}^{-}(\mathrm{aq}) + \mathrm{H}_{2}\mathrm{O}(\mathrm{l}) \rightarrow \mathrm{H}_{4}\mathrm{SiO}_{4} + \mathrm{OH}^{-}(\mathrm{aq}) \tag{15}$$

Limiting molar conductivities, Λ_0 , for Na₂(H₂SiO₄) in water are reported for different SiO₂/Na₂O ratios and it turned out that at molar ratio 3.95 the hydrolysis can be neglected and thus only the contributions of two kind of ions (Na⁺, H₂SiO₄²⁻) to the conductivity can be assumed.

According to reactions (2) and (4) the same can be supposed also for our experiment. In extremely diluted solution an approximation of $\Lambda \approx \Lambda_0$ is acceptable and thus the concentration of silicate solutions, c, can be obtained as $c = (\kappa - \kappa_0)/\Lambda$.

As mentioned before, κ_0 is the conductivity of (pure) water, which was estimated from intercept of plots in Figure 4b. In Table 2 the extrapolated values of resistance, R_0 together with published data for Λ_0 of $Na_2(H_2SiO_4)$ in water and the estimated concentration of silicate ion $H_2SiO_4^{2-}$ in water at $t = \infty$, c_{∞} , obtained as $c_{\infty} = (\kappa_{\infty} - \kappa_0)/\Lambda_0$.

Values of c_{∞} in Table 2 are lower than reported concentration for saturated aqueous solution of quartz and amorphous silica as reported by Lier at al.,¹⁴ but comparable to those obtained by Icenhower and Dove at 40 °C.⁴ But it must be stressed, that this experiment was carried

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Table 2. Values of extrapolated resistance for pure water, R_0 , as obtained from the intercept in Figure 4b, reported values for limiting molar conductivity, Λ_0 of Na₂(H₂SiO₃) in water, estimated concentration of silicate ion in water at $t = \infty$, c_{∞} , and values for the concentration of saturated solutions of amorphous silica and quartz in water, c_s , taken form the literature.

| T/ °C | R ₀ /MΩ | $\begin{array}{c} \Lambda_0 \ (\mathrm{Na_2} (\mathrm{H_2SiO_4}))^{\mathrm{a}} / \\ \mathrm{cm}^2 \cdot \mathrm{S} \cdot \mathrm{mol}^{-1} \end{array}$ | c_/ 10 ⁻⁵ ∙ mol ∙ L ⁻¹ | c _s (silica)∕ mol ∙ kg ⁻¹ | c _s (quartz)/ mol⋅kg ⁻¹ |
|-------|--------------------|--|---|--|--|
| 5 | 5.314 | 224 | 1.07 | | |
| 10 | 4.473 | | | | |
| 15 | 3.679 | 296 | 1.04 | | |
| 20 | 3.381 | | | | |
| 25 | 2.987 | 384 | 0.98 | $2 \cdot 10^{-3b}$ | $1.8 \cdot 10^{-4b}$ |
| 30 | 2.701 | | | | |
| 35 | 2.548 | 476 | 0.91 | | |
| 40 | 2.512 | | | $(7.6 \cdot 10^{-6} - 1.4 \cdot 10^{-5})^{c}$ | |

^a reference¹³, ^b reference¹⁴, ^d reference⁴

out in a different way as usual "solubility" experiments, where the investigated material was grounded. In this context the concentrations c_{∞} seem to be reasonable. This study reveals that one has to be very careful at measuring the conductivity of extremely diluted solutions in glass vessels. Conductivity is an overall quantity and reflects the contribution of all ionic species in solutions namelyalso those leached from glass are far to be negligible.

In addition, any rate constant for dissolution of glass, reported in Table 1 is valid only for this cell under the same condition. It depends on the contact surface between water and glass and thickness of the diffusion layer (equations 7 and 8), which depends on the speed of stirring. According to Icenhower and Dove⁴ the kinetics of dissolution of borosilicate glass is not understood yet. Nevertheless, obtained results could be helpful at estimating the contribution of water to the measured (overall) conductivity of solutions if needed. At higher concentration of ions in the electrolyte solutions (over ~0.001 mol $\cdot L^{-1}$) the contribution of the ions leached from the glass vessel could be neglected.

Finally, silicate is present in all aqueous solutions stored in glass containers. Although it is not toxic, pharmacists and physicians must be aware that silicate might be present in large amounts in parenteral preparations stored in glass containers, as warned also by Bohrer et al. in 2008.³

4. Conclusions

The dissolution of glass from the glass's wall was followed by measuring the electrical resistance of water in a gas tight closed cell in a nitrogen atmosphere during three weeks in the temperature range from 5 °C to 40 °C. It was found that the resistance was decreasing (and consequently the electrical conductivity increasing) simultaneously over the whole time period of experiment. This increase in conductivity was ascribed to the release of silicate and alkali ions (Na⁺) from the glass as reported in the literature.^{3,13} The process was regarded as dissolution of any other solid substances where the rate of the process is determined mainly by the diffusion transports and the rate constants at all temperatures were evaluated. It turned out that the process is slightly dependent on the temperature-the process is faster at higher temperatures. From literature data on limiting conductivity of sodium silicate in water, the concentration of "saturated" solution of glass in water was estimated to be about 10^{-5} mol \cdot L⁻¹. Thus at the electrical conductivity measurements of extremely diluted aqueous solutions in glass cells the dissolution of the components from the glass vessel has to be taken into account.

However, it would be worth to repeat the experiment in another way by carrying out the conductivity measurements at each temperature separately.

Nevertheless, observed increase in electrical conductivity of water during time can be ascribed to the leaching of glass solely. Strictly speaking, "pure" water in glass vessel does not exist, but it is rather an extremely diluted aqueous solution of glass's components.

5. Acknowledgement

The author is grateful to Prof. Dr. Josef Barthel and Dipl. Ing. Herbert Hilbinger from University of Regensburg (Germany) for providing the flask for the storage of solvents under nitrogen atmosphere and the conductivity cell. The work was supported by SRA Grant No. P1-0201.

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Povzetek

Vodo in vodne raztopine pogosto hranimo v steklenih posodah. Objavljenih je kar nekaj raziskav, ki se ukvarjajo s topnostjo stekla v vodi, oz. bolj pravilno izluževanjem komponent iz stekla v vodo. V tem delu smo ta proces zasledovali tri tedne s pomočjo merjenja električne upornosti/prevodnosti vode v zaprti posodi v atmosferi dušika v temperaturnem območju med 5 °C in 40 °C. Na podlagi dobljenih podatkov lahko sklepamo, da opažen porast električne prevodnosti vode – oz. dejansko ekstremno razredčene raztopine iz stekla sproščenih ionov – lahko pripišemo izključno izluževanju komponent iz stekla.