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A Solubility Model for Amorphous Silica in Concentrated Electrolytes

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ABSTRACT

This paper describes the development of an aqueous thermodynamic model for accurately predicting the solubility of amorphous silica and other silica-containing compounds in the system $\text{Na}^+ - \text{H}^+ - \text{Mg}^{2+} - \text{NO}_3^- - \text{SO}_4^{2-} - \text{Cl}^- - \text{H}_2\text{O}$ to high concentration and across the temperature range 25-100° C. This model, which utilizes the aqueous thermodynamic model of Pitzer, includes only one dissolved silica species, $\text{H}_4\text{SiO}_4(\text{aq})$, and is valid in neutral to very acidic solutions. The model is parametrized from the extensive set of solubility data in the literature as well as from new experimental data on amorphous silica solubility in HNO_3 and HCl developed as part of this study. The accuracy of the model is tested on solutions more complex than those used in model parameterization.

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INTRODUCTION

Silica is one of the major constituents of the earth's crust and is ubiquitously present in most natural materials. The solubility of silica and other silica-containing compounds is, therefore, of primary concern in geochemistry and in chemical processing applications where silica scale formation, resulting from changes in temperature and electrolyte composition, can cause problems in process design and operation. Unfortunately, the solubility of amorphous silica ($\text{SiO}_2(\text{am})$) and other silica-containing compounds is strongly dependent on temperature and electrolyte composition. Hence, accurate experimental and thermodynamic data are required to predict the solubilities of silicate phases for geochemical or chemical engineering application.

In aqueous solutions of $\text{pH} < 9$, the principal dissolved silica species is silicic acid $\text{H}_4\text{SiO}_4(\text{aq})$. It is the interactions of silicic acid with the other bulk chemical species (such as Na^+ , NO_3^- , Cl^-) in aqueous solutions that will determine the solubility of amorphous silica and other silica-containing compounds under neutral and acidic conditions. In this study, we developed a set of temperature-dependent ion interaction parameters for silicic acid with important bulk chemical species in order to accurately predict the solubility of amorphous silica in the system $\text{Na}^+ - \text{H}^+ - \text{Mg}^{2+} - \text{NO}_3^- - \text{SO}_4^{2-} - \text{Cl}^- - \text{H}_2\text{O}$ to high concentration and over the temperature range 25-100°C. Such a model is also necessary for predicting the solubility of more complex silica-containing compounds, such as aluminum silicates. This model was developed from the extensive set of solubility data for amorphous silica present in the literature (Marshall 1980a,b; Marshall and Warakowski 1980; Chen and Marshall 1982; Marshall and Chen 1982a,b; Elmer and Nordberg 1958) as well as solubility data on amorphous silica in HNO_3 and HCl obtained as part of this study.

EXPERIMENTAL PROCEDURES

The experimental procedure used in this study was the same as that used by Marshall and Warakowski (1980). Briefly, the procedure consisted of suspending 4 g of silica gel (Baker analyzed reagent) in 30-mL of either HNO_3 , HCl , or NaNO_3 placed in 50-mL centrifuge tubes. The HNO_3 , HCl , or NaNO_3 ranged in concentration from 0.0001M to 2.0M. Only four NaNO_3 samples were included. These samples were used to check our results against the results of Marshall (1980a). The silica gel had been previously washed with deionized water and air-dried overnight. The silica suspensions were placed on an orbital shaker and continuously shaken. Studies were conducted both at room temperature (22-23°C) and at 47°C. The studies at 47°C were conducted only in HCl because data were available in the literature for HNO_3 at 36°C and 65°C. The room temperature suspensions were sampled after 7 and 37 days equilibration. The 47°C suspensions were sampled after 7 and 38 days equilibration.

Sampling consisted of centrifugation at 2000 g for 7 to 10 minutes followed by filtration through Amicon-type F-25 Centriflo membrane cones with an approximate pore size of 0.0018 μm . The filters were pretreated by soaking and rinsing in deionized water. A small aliquot of the sample was then passed through each filter to saturate any possible adsorption sites (this aliquot was discarded), followed by sufficient solution to analytically determine silica. Silica was analyzed by inductively coupled plasma spectroscopy (ICP).

THERMODYNAMIC MODEL

The aqueous thermodynamic model of Pitzer (1979) was utilized throughout this study to account for the variations in the activity coefficients of neutral and dissolved species. The exact equations used have been described in detail elsewhere (see Felmy and Weare 1986; Felmy et al. 1989, Pitzer 1979). In this study the most important ion-interaction parameters involve interactions between the electrolyte ions (i.e. Na^+ , NO_3^- , SO_4^{2-} ...) and the

dominant aqueous silica species under neutral and acidic conditions, $\text{H}_4\text{SiO}_4(\text{aq})$. In terms of the Pitzer model, these ion-interaction parameters are those describing neutral-cation, neutral-anion, and neutral-cation-anion interactions. For example, in NaCl solutions these parameters would be $\lambda(\text{H}_4\text{SiO}_4\text{-Na}^+)$, $\lambda(\text{H}_4\text{SiO}_4\text{-Cl}^-)$, and possibly $\lambda(\text{H}_4\text{SiO}_4\text{-Na}^+\text{-Cl}^-)$. In addition, to develop an accurate thermodynamic model requires knowledge of the important electrolyte ion-interaction parameters (β^0 , β^1 , and C^ϕ) to describe binary ion-interactions like $\text{Na}^+\text{-Cl}^-$ and common-ion ternary ion-interactions (Θ and ψ) like $\text{Na}^+\text{-K}^+\text{-Cl}^-$, as a function of temperature. The electrolyte ion-interaction parameters do not directly affect the calculated activity coefficient of $\text{H}_4\text{SiO}_4(\text{aq})$ but do affect the calculated activity of water. The activity of water can affect the solubility calculations for amorphous silica at high electrolyte concentration via the rxn,

All of the ion-interaction parameters and standard chemical potentials developed or utilized in this study were fit to the following empirical temperature-dependent expression (see Møller 1988),

$$P(T) = a_1 + a_2T + a_3/T + a_4\ln T + a_5/(T-263) + a_6T^2 + a_7/(680.-T) + a_8/(T-227) \quad (2)$$

where $P(T)$ is a temperature-dependent ion-interaction parameter or standard chemical potential. The necessary coefficients in eqn(2) for the Debye-Huckel A^ϕ term and the standard chemical potential of $\text{H}_2\text{O}(l)$ were taken from Greenberg and Møller (1989). The coefficients for eqn(2) for the NaCl and Na_2SO_4 ion-interaction parameters were also taken from Greenberg and Møller (1989). A complete listing of the other coefficients of eqn (2) used in this study is given in Table 1. A brief description of the method used to calculate these coefficients follows.

The coefficients for HCl were determined by calculating the values of β^0 , β^1 , and C^ϕ at 5°C intervals using the temperature- and density-dependent

TABLE 1. Coefficients for the temperature dependent expression for electrolyte ion-interaction parameters.

Parameter	a_1	a_2	a_3	a_4	a_6
HCl (β^0)	-5.109945e+00	-2.73385e-03	1.188690e+02	9.780609e-01	1.469778e-06
(β^1)	9.9847059e+02	5.424992e-01	-2.21692e+04	-1.86027e+02	-2.88597e-04
(C^ϕ)	3.8062945e-01	4.132298e-04	-2.35792e+00	-8.12431e-02	-3.30966e-07
HNO ₃ (β^0)	-5.650010e-01	4.388300e-03	-	-	-6.95000e-06
(β^1)	-1.116200e-01	1.339600e-03	-	-	-
(C^ϕ)	4.4287000e-03	-5.79520e-05	-	-	8.160000e-08
MgSO ₄ (β^0)	4.2672400e-01	-6.90000e-04	-	-	-
(β^1)	-1.218700e+00	1.530000e-02	-	-	-
(β^2)	3.8202000e+01	-2.53000e-01	-	-	-
(C^ϕ)	-1.300380e-01	5.200000e-04	-	-	-
NaNO ₃ (β^0)	-1.315403e-01	4.538667e-04	-	-	-
(β^1)	-1.000309e+00	4.049333e-03	-	-	-
(C^ϕ)	1.1951454e-02	-4.03764e-05	-	-	-
MgCl ₂ (β^0)	5.7606600e-01	-9.31654e-04	-	-	5.939150e-07
(β^1)	2.6013500e+00	-1.09438e-02	-	-	2.601690e-05
(C^ϕ)	6.5786700e-02	-2.89125e-04	-	-	3.018230e-07

equation given by Holmes et al. (1987) and then refitting these calculated values to eqn(2). The coefficients for HNO₃ were calculated from the first and second derivatives with respect to temperature given for β^0 , β^1 , and C^ϕ by Clegg and Brimblecombe (1990). In a similar fashion the coefficients for MgSO₄ were calculated from the first derivative with respect to temperature expression given by Pitzer (1979). The coefficients for MgCl₂ were taken from Delima and Pitzer (1983). The values of NaNO₃ were calculated at 25°C from the osmotic data of Wu and Hamer (1980) extending to NaNO₃ saturation (i.e. 10.83m) and at 100°C from the osmotic data of Voigt et al. (1990) extending to 12.2m. These two sets of values were then used to determine the coefficients for NaNO₃ given in Table 1.

The coefficients in eqn(2) for the parameters for silicic acid ion-interactions are summarized in Table 2. In determining these parameters the solubility data in the various salt solutions were determined isothermally and

TABLE 2. Coefficients for the temperature-dependent expression for H_4SiO_4 ion-interaction parameters.

Parameter	a_1	a_2	a_4	a_6
$\lambda(H_4SiO_4-NO_3^-)$	9.2844972e+01	1.197600e-01	-2.12175e+01	-8.46934e-05
$\lambda(H_4SiO_4-Na^+)$	-1.142800e-01	1.186670e-04	-	-
$\lambda(H_4SiO_4-Cl^-)$	-4.394000e-02	1.295300e-03	-	-2.24930e-06
$\lambda(H_4SiO_4-SO_4^{2-})$	-8.737600e-02	5.533300e-04	-	-
$\lambda(H_4SiO_4-Mg^{2+})$	6.2274500e-01	-1.81330e-03	-	-
$\zeta(H_4SiO_4-H^+-NO_3^-)$	-0.0033	-	-	-
$\zeta(H_4SiO_4-Na^+-Cl^-)$	5.9630e-02	-2.00000e-04	-	-
$\zeta(H_4SiO_4-Mg^{2+}-Cl^-)$	-2.2861e-01	5.94133e-04	-	-

the coefficients for eqn(2) determined by fitting the calculated parameter values at different temperatures.

The most difficult parameter to determine was the standard chemical potential of $SiO_2(am)$. This parameter is primarily determined by the dilute solution solubility. Determining the dilute solution solubility of amorphous silica from different batches of starting material has always been difficult and has been discussed in length by Marshall (1980a). Briefly, differences in particle size and possibly lack of attainment of equilibrium at lower temperatures appear to be part of the problem. The net result of this difficulty is that different dilute solution solubilities and, hence, different calculated standard chemical potentials of $SiO_2(am)$ are determined or extrapolated from different electrolyte solutions. The tabulated best fit values for the standard chemical potential of $SiO_2(am)$ at various temperatures are given in Table 3. These values are used in all subsequent calculations except for the case of Na_2SO_4 solutions at 25°C where the standard chemical potential of $SiO_2(am)$ was independently determined. The values shown in Table 3 all assume μ^0/RT for $H_4SiO_4(aq) = -531.121$, the value at 25°C given by Wagman et al. (1982).

The following section presents a detailed comparison of the calculated and experimental amorphous silica solubilities over the temperature range 25-100°C.

TABLE 3. Calculated values of the standard chemical potential of $\text{SiO}_2(\text{am})$ at different temperatures.

Temperature ($^{\circ}\text{C}$)	μ°/RT
22-23	-346.184
25	-345.900
36	-347.410
47	-348.645
65	-350.250
95	-352.396
100	-352.800

RESULTS AND DISCUSSION

A comparison of our experimental results for NaNO_3 with those of Marshall (1980a) were presented in Silva et al. (1993) and is reproduced here (see Figure 1) for the convenience of the reader and to demonstrate the consistency between the two sets of experimental measurements.

Figure 1. Comparison of Amorphous Silica Solubilities in NaNO_3 .

The experimental and calculated solubilities of amorphous silica in HNO_3 solutions are shown in Figure 2. The difficulty in determining a definitive value for the dilute solution solubility of amorphous silica is clearly exhibited in Figure 2a where the experimental solubilities can vary by approximately 15%. The standard chemical potential of $\text{SiO}_2(\text{am})$ as well as the ion-interaction parameters for $\text{H}_4\text{SiO}_4(\text{aq})$ (i.e., $\lambda_{\text{H}_4\text{SiO}_4-\text{NO}_3^-}$ and $\lambda_{\text{H}_4\text{SiO}_4-\text{NO}_3^--\text{H}^+}$) were determined in each data set.

In determining these parameters, the value of $\lambda(\text{H}_4\text{SiO}_4-\text{NO}_3^-)$ was first fit, along with the standard chemical potential of $\text{SiO}_2(\text{am})$ from the data at HNO_3 concentrations $< 5\text{m}$. The value of $\lambda(\text{H}_4\text{SiO}_4-\text{NO}_3^--\text{H}^+)$ was then adjusted to fit the data at higher concentration. The value of $\lambda(\text{H}_4\text{SiO}_4-\text{H}^+)$ was set to zero at all temperatures by convention.

The experimental and calculated amorphous silica solubilities in NaNO_3 solutions are shown in Figure 3. The values of $\lambda(\text{H}_4\text{SiO}_4-\text{NO}_3^-)$ calculated from the HNO_3 data were used in these calculations. Only the standard chemical potential of $\text{SiO}_2(\text{am})$ and $\lambda(\text{H}_4\text{SiO}_4-\text{Na}^+)$ was adjusted. No higher order $\lambda(\text{H}_4\text{SiO}_4-\text{Na}^+-\text{NO}_3^-)$ parameter was required. The calculated and experimental values are in good agreement.

The experimental and calculated amorphous silica solubilities in HCl solutions are shown in Figure 4. In these calculations the standard chemical potential of $\text{SiO}_2(\text{am})$ was fixed at the values obtained from analysis of the HNO_3 data. The only adjusted parameter was $\lambda(\text{H}_4\text{SiO}_4-\text{Cl}^-)$. No higher order $\lambda(\text{H}_4\text{SiO}_4-\text{Cl}^--\text{H}^+)$ term was required.

The experimental and calculated amorphous silica solubilities in NaCl solutions are shown in Figure 5. In these calculations the values of $\lambda(\text{H}_4\text{SiO}_4-\text{Na}^+)$ were fixed at the values determined from the data in NaNO_3 solutions. In the 25°C calculations only the standard chemical potential of $\text{SiO}_2(\text{am})$ was adjusted. All ion-interaction parameters had previously been determined. The 25°C NaCl solubility data, therefore, represents a partial validation of the aqueous model developed from the HNO_3 , NaNO_3 , and HCl data. In the 100°C calculations the standard chemical potential of $\text{SiO}_2(\text{am})$, $\lambda(\text{H}_4\text{SiO}_4-\text{Cl}^-)$, and $\lambda(\text{H}_4\text{SiO}_4-\text{Na}^+-\text{Cl}^-)$ was adjusted. It was necessary to

Figure 2. Experimental and Calculated Solubilities of Amorphous Silica in HNO_3 solutions. (a) room temperature (22-23°C), (b) 36°C, (c) 65°C, (d) 95°C. Experimental data at room temperature determined in this study. Experimental data at other temperatures from Elmer and Nordberg (1958).

Figure 3. Experimental and Calculated Solubilities of Amorphous Silica in NaNO_3 solutions. (a) 25°C. (b) 100°C. Experimental Data of Marshall (1980).

Figure 4. Experimental and Calculated Amorphous Silica Solubilities in HCl Solutions. Experimental data obtained as part of this study. (a) Room Temperature (22-23°C). (b) 47°C.

Figure 5. Experimental and Calculated Solubilities of Amorphous Silica in NaCl Solutions. (a) 25°C (Data of Marshall and Warakowski 1980). (b) 100°C (Data of Chen and Marshall 1982).

determine $\lambda(\text{H}_4\text{SiO}_4\text{-Cl}^-)$ in the 100°C calculations since the HCl solubility data extended to only 47°C.

The experimental and calculated amorphous silica solubilities in Na_2SO_4 solutions is shown in Figure 6. In these calculations the value of $\lambda(\text{H}_4\text{SiO}_4\text{-Na}^+)$ determined from the NaNO_3 data was used. The only adjusted parameters were the standard chemical potential of $\text{SiO}_2(\text{am})$ at 25°C and the values for $\lambda(\text{H}_4\text{SiO}_4\text{-SO}_4^{2-})$ at 25°C and 100°C. The standard chemical potential of $\text{SiO}_2(\text{am})$ at 25°C was refit because the extrapolated dilute solution solubility of amorphous silica in Na_2SO_4 solution does not agree with the extrapolated dilute solution solubility in NaCl solution. This is the only case where a model parameter was used which is different from those given in Tables 1, 2, or 3.

The experimental and calculated amorphous silica solubilities in MgSO_4 solutions are shown in Figure 7. In these calculations the standard chemical potential of $\text{SiO}_2(\text{am})$ was fixed at the corresponding values determined from the data in NaCl solutions. The values for $\lambda(\text{H}_4\text{SiO}_4\text{-SO}_4^{2-})$ were fixed at the values determined from the data in Na_2SO_4 solutions. The only adjusted parameter was $\lambda(\text{H}_4\text{SiO}_4\text{-Mg}^{2+})$.

The experimental and calculated amorphous silica solubilities in MgCl_2 solutions are shown in Figure 8. Two sets of calculations are shown at 25°C. The dashed curve represents the model predictions with no parameters adjusted. Although this curve gives a reasonable overall representation of the data, the fit can be improved by adjusting the higher order $\lambda(\text{H}_4\text{SiO}_4\text{-Mg}^{2+}\text{-Cl}^-)$ term. This final model accurately represents all of the experimental data.

As a final test of our model, the solubilities of amorphous silica were calculated in mixed electrolyte solutions (Table 4, Figure 9). These results, with the possible exception of one data point in mixed NaCl- MgCl_2 , are all predicted well within the experimental error in the measurements. These final predictions represent an excellent validation of our final thermodynamic model.

Figure 6. Experimental and Calculated Solubilities of Amorphous Silica in Na_2SO_4 Solutions. (a) 25°C (Data of Marshall and Warakowski 1980). (b) 100°C (Data of Chen and Marshall 1982).

Figure 7. Experimental and Calculated Solubilities of Amorphous Silica in MgSO_4 Solutions. (a) 25°C (Data of Marshall and Warakowski 1980). (b) 100°C (Data of Chen and Marshall 1982).

Figure 8. Experimental and Calculated Solubilities of Amorphous Silica in MgCl_2 Solutions. (a) 25°C (Data of Marshall and Warakomski 1980). (b) 100°C (Data of Chen and Marshall 1982).

TABLE 4. Experimental and calculated amorphous silica solubilities in mixed electrolyte solutions at 100°C. Experimental data of Marshall and Chen (1982b).

Electrolytes		Experimental	Calculated
NaCl	MgCl ₂		
0.82	1.23	0.0046	0.0031
1.23	0.66	0.0038	0.0039
NaCl	MgSO ₄		
0.97	0.96	0.0045	0.0047
Na ₂ SO ₄	MgCl ₂		
0.71	1.38	0.0031	0.0033
Na ₂ SO ₄	MgSO ₄		
0.75	0.78	0.0055	0.0055
0.90	0.40	0.0055	0.0059
MgCl ₂	MgSO ₄		
1.11	0.38	0.0036	0.0035

Figure 9. Experimental and Calculated Solubilities of Amorphous Silica in mixed NaCl and Na₂SO₄ solutions at 100°C. Experimental Data of Marshall and Chen (1982b).

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