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AN HEURISTIC EQUATION CORRELATING SILICA POLYMERIZATION INDUCTION TIMES TO TEMPERATURE AND SILICA CONCENTRATION IN GEOTHERMAL BRINES

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ABSTRACT

Oversaturation is a common phenomenon in various compounds, from physical states like supercooled or superheated water to solubility oversaturation, such as CO_2 or silica in water. In geothermal systems, silica oversaturation is particularly notable. As geothermal brine cools, silica can remain dissolved beyond equilibrium levels, eventually leading to polymerization. This process typically includes a delay known as the "polymerization induction period."

Despite being off-equilibrium, silica oversaturation follows a regular pattern. Several models have been proposed to describe this behavior. In this study, we build on the early empirical work of Rothbaum and Rohde to develop a heuristic expression that fits experimental data and can be refined with further observations. The correlation is given by:

$$\log(\mathrm{It}) = -0.0049([\mathrm{SiO_2}]_0 - [\mathrm{SiO_2}]eq) + 3.6106$$

By incorporating Fournier et al.'s general expression for silica equilibrium, we derive a more comprehensive formula:

$$\log(\mathrm{It}) = 3.6106 - 0.0049\left([\mathrm{SiO_2}]_0 - 10^{\left(4.52 - 731/\mathrm{T(K)}\right)}\right)$$

This expression aligns with polymerization models where initial tetrameric silica rings form or oligomers reach sufficient concentration to start precipitation by oligomer coalescence governed by standard kinetics. The resulting correlation not only fits observed induction times but also provides a theoretical framework for understanding silica polymerization, offering a more robust heuristic model than previous empirical-only approaches.

INTRODUCTION

Silicon dioxide is a chemical species frequently found in the aqueous fractions of endogenous fluids. It forms in water either through the dissolution of quartz or the solubilization of silicate minerals contained in rocks. Its concentration depends on the temperature at which the dissolution equilibrium occurs, and in certain cases, it can be used to infer reservoir temperatures. In this context, silicon dioxide, commonly referred to as silica, was among the first elements employed in the development of so-called geothermometers, which are equations,

often empirical in nature, that estimate the temperature of a reservoir or a fluid based on the chemical composition of the fluid carrying silica. In particular, the concentration of silica in solution is governed by the solubility of quartz, and the geothermometer is constructed from the dissolution equilibrium curve of quartz as a function of temperature. Unlike many substances whose solubility exhibits regular trends with temperature, silica is affected by two distinctive phenomena related to its thermodynamic and kinetic behavior: the solubility equilibrium of silica and the induction time of precipitation. In the first case, experimental observations show that the cooling curve of silica leads to an equilibrium with a solid phase composed of amorphous silica, with quartz often present only in negligible amounts. This results in a hysteresis effect, whereby silica solutions are systematically supersaturated with respect to quartz, and the equilibrium curve corresponds to a completely different species: amorphous and/or microcrystalline silica[1]. The second special behavior is given by the formation of supersaturated solutions whose precipitation occurs only after a certain time interval following the establishment of a metastable supersaturated state. In practice, precipitation does not occur immediately upon reaching saturation but exhibits a latency period, known as the *induction time*, after which precipitation begins (Rothbaum, A.G Rohde 1979, referred as R&R in the rest of text) [2]. The induction time for silica precipitation is strongly influenced by the concentration of silica in solution, particularly its degree of supersaturation, and by the system temperature. Furthermore, induction times and the kinetics of polymerization and precipitation are affected by the presence of chemical species such as aluminum and iron, as well as by pH conditions.

In the present study, the phenomenon of silica induction times was investigated by considering the results of experimental studies by R&R, and an attempt was made to define a law, determined using linear correlation methods, that describes induction times as a function of temperature and the degree of supersaturation of silica solutions. This correlation is potentially of great interest, as it allows an estimation of the practical time available before silica precipitation (scaling) occurs. The ability to control silica scaling is particularly valuable, as it represents one of the few available methods for managing silica deposition in alternative or complementarity to the use chemical additive-based control systems.

Silica scale formation is a widespread issue in geothermal power plants, extensively documented in the literature. Some early studies on silica polymerization are from Makrides et al., (1978 and 1980)[3] including silica scaling measurements and observation of induction time; Other authors have studied silica behavior in geothermal brines and their impact in the efficiency in energy extraction for geothermal power plants (Gunnarsson and Arnórsson, 2003, 2005 and 2010[4]).

Experimental studies indicate that silica precipitation kinetics are sensitive to pH variations and temperature changes. These are common phenomena due to the depressurization and cooling of geothermal fluids during extraction, which leads to supersaturation with respect to amorphous silica and in some cases also pH variations. The resulting degassing and temperature drop can trigger either direct silica scaling or polymerization into colloidal silica. Other studies include De Guzman et al., 2015[5] and Dixit 2016[6]. In some cases, extensive experimental studies have been performed to analyze the kinetic behavior of silica polymerization and kinetics as shown by Iler, 1979[7] and Rothbaum and Rohde, 1979[2]. Understanding the kinetics of silica scaling is crucial for modeling polymerization mechanisms, equilibrium states, and reaction rates, which are essential for developing effective mitigation strategies in geothermal systems. Despite decades of research, the mechanisms of silica precipitation from geothermal fluids remain incompletely understood.

SILICA BEHAVIOUR IN GEOTHERMAL POWER PLANTS

Silica polymerization holds significant scientific and operational interest for geothermal operators due to its crucial, often unpredictable, role in silica scaling formation. The phenomenon is governed by silica thermodynamic behavior, which exhibits hysteresis effects in its dissolution equilibrium, typically defined by quartz solubility and the precipitation constant of amorphous silica, which possesses distinct solubility and

equilibrium parameters. Beyond thermodynamics, precipitation is strongly influenced by its kinetics, particularly the "induction times," which are delays extending up to several hundred minutes before precipitation commence, and are modulated by key factors such as supersaturation levels, temperature, pH, and the chemical composition of the geothermal brine. These complex physicochemical interdependencies challenge the management of geothermal fluid evolution during energy extraction. Therefore, a thorough understanding of silica scaling equilibria and polymerization dynamics is indispensable for optimizing plant operations and mitigating scaling risks.

The direct scaling of silica depends on the interaction of siloxane bonds of the silicic acid to the metal surface. This reaction is known to usually occur at high pH levels (pH>8) and is catalyzed by hydroxyl ions. This produces a hard, dense and vitreous layer. On the other hand, colloidal formation occurs *via* a condensation-polymerization mechanism from silicic acid due to increasing silica saturation/supersaturation[5]. This formation pathway forms small molecular weight dimers and trimers prior to forming rings of various sizes, and cross-linked polymeric chains, and ultimately a complex and amorphous product as discussed by Amjad (2010)[8]. According to the study carried out by Gunnarsson et al. [9,10,11], which process takes place in spent high-temperature geothermal waters can also depend to some extent on the water environment. If the waters are in turbulent flow where there is surface available for monomeric deposition onto the surface, silica scaling is likely to take place, but if the waters are placed in quiet environment, silica polymerization is the favorable process.

Silica polymerization may occur when the concentration of monosilicic acid in aqueous solution exceeds the solubility threshold of amorphous silica. Under such conditions, excess monomeric species progressively vanish from solution as they undergo polymerization. The rate of monomer depletion is commonly described by a kinetic expression originally proposed by Chan (1987)[12] and referring to previous authors:

$$-\frac{dC}{dt} = k(C - C_e)^n \qquad [1]$$

where C is the instantaneous monomer concentration, Ce is the equilibrium concentration at the reaction temperature, and k and n represent the reaction rate constant and reaction order, respectively. Rothbaum and Rohde (1979)[2] investigated the polymerization kinetics of silicic acid at initial concentrations exceeding equilibrium values across a temperature range of 5–180 °C. Their findings revealed that the induction period, defined as the latency before observable polymerization, lengthens with increasing temperature and shortens with rising supersaturation. Their experimental data supported a fourth-order polymerization rate, whereas earlier work by Rothbaum[13] suggested higher order kinetics. The same author in NMR studies[14] points out a fast equilibrium between monomeric and dimeric silica that, accordingly, indicates their contribution in the kinetic behavior of silica. Moreover, the presence of an equilibrium between monomeric and dimeric silica indicates that (nda) kinetic equations should include a reverse depolymerization step for dimeric silica. Iacopini et al. (2005)[15] in kinetic studies at room temperature (25°C) have hypothesized that experimental data are compatible with a fourth order polymerization rate expressed as a function of monomeric silica concentration

$$R = k_4[H_4SiO_4]^4 \qquad [2]$$

and with significant consistency of a critical step of the polymerization forming a cyclic tetramer. Makrides (1977, 1980) postulated that particle growth preceding the induction and nucleation phase was linear with time. According to Angcoy Jr (2006)[16], in particle deposition, the starting process is termed by homogenous nucleation since the polymers develop spontaneously in solutions. The decline of monomer is a strong function of initial supersaturation such that it is often instantaneous or very rapid if the ratio is >2.5 but exhibits a plateau called an induction period which can last from minutes to hours if the ratio is <2.5.

In other studies, the rate by which the monomers disappear to form polymers (polysilicic acid) is usually expressed as (Bohlmann et al., 1980[17]; Weres et al., 1982[18]; Fleming, 1986[19]):

$$-\frac{dC}{dt} = k(C - C_e)^n \qquad [3]$$

where C is the monomeric silica concentration; Ce is the concentration corresponding to amorphous silica saturation at the reaction temperature; k is Reaction constant and n is Reaction order. This consideration is consistent with a preliminary phase of particle growth to a critical size or a critical particle concentration with the silica still in solution followed by particle coalescence and precipitation.

INDUCTION TIMES

One of the main experimental studies of silica polymerization is from R&R that pointed out the induction times for silica describing a delay between silica polymerization initial stages and its precipitation.

As shown in table 1, data from the authors are reported together with the expression added in this study of the logarithm of the induction times themselves. As we can see from Figure [1] the ln (It) shows, for each temperature a linear dependence in respect to oversaturation of the type:

$$ln(It) \cong \alpha - \beta \Delta S \qquad [4]$$

Where α and β are contants derived from the correlation curve between $ln(It)$ and oversaturation $\Delta S = [SiO_2]_0 - [SiO_2]_{eq}$ (expressed as difference between initial silica concentration and silica equilibrium concentration).

Table 1: Induction times after Rothbaum and Rohde and the logarithmic dependence added by the author of present work				**Data elaborated in present work**	
T (°C)0	Initial Supersaturation (g/t) SiO2	Max. rate R, g/t/min	Induction period P, min	log10 induction time	natural log induction time
5	590	20	4	0,6021	1,386
5	520	3.08	12	1,0792	2,485
5	450	2.86	20	1,3010	2,996
5	350	0.345	100	2,0000	4,605
5	200	0.091	300	2,4771	5,704
30	520	17.6	4	0,6021	1,386
30	470	2.94	10	1,0000	2,303
30	380	1.82	35	1,5441	3,555
30	255	0.063	300	2,4771	5,704
60	510	12.5	8	0,9031	2,079
60	420	2.78	30	1,4771	3,401
60	340	1.39	50	1,6990	3,912
60	250	0.04	750	2,8751	6,620
90	700	330	0		
90	560	16.7	7	0,8451	1,946
90	470	9.1	15	1,1761	2,708
90	370	2.5	32	1,5051	3,466
90	300	1.33	60	1,7782	4,094
90	220	0.22	500	2,6990	6,215
120	840	330	0		
120	720	33.3	4	0,6021	1,386
120	630	12.5	6	0,7782	1,792
120	470	2.22	25	1,3979	3,219
120	385	0.65	40	1,6021	3,689
120	170	0.128	500	2,6990	6,215

150	710	100	0		
150	525	18.9	7	0,8451	1,946
150	385	1	30	1,4771	3,401
150	320	0.29	130	2,1139	4,868
150	305	0.26	300	2,4771	5,704
180	600	16.7	4	0,6021	1,386
180	520	4.2	17	1,2304	2,833
180	415	0.74	150	2,1761	5,011
180	325	0.125	300	2,4771	5,704

A similar correlation can be observed including all the data at all the experimental temperatures (bottom plot at right of Figure [1].

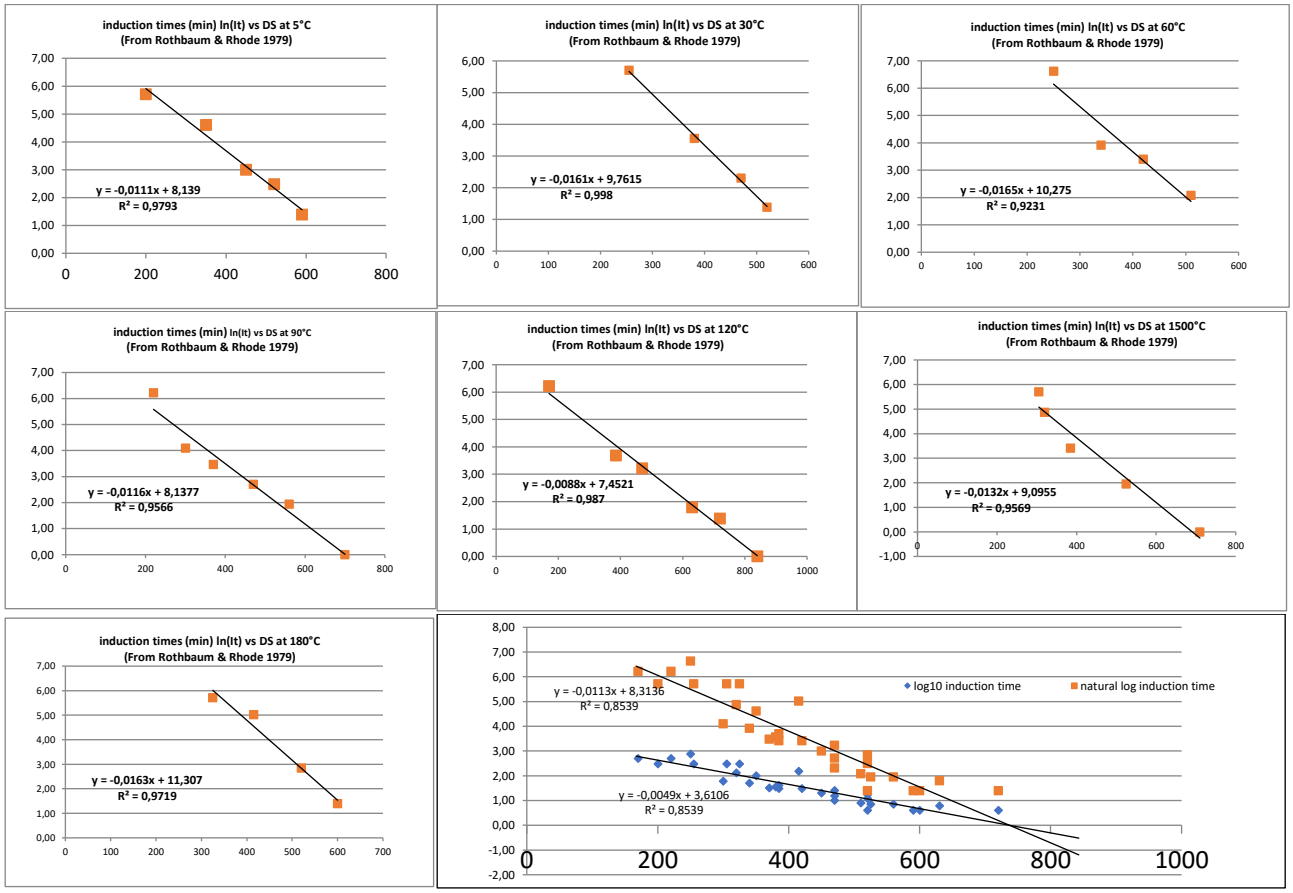


Figure 1: correlation relations of Ln(It) vs silica oversaturation at the experimental temperatures and the all experimental data correlation at all the experimental temperatures, obtained from analysis of Rothbaun and Rohde data[2].

The interpretation of this type of correlation requires some consideration on silica polymerization process, and though a theoretical picture is not provided some consideration between the initial polymerization phases, and an interpretation of the induction times is provided with the intent of describing silica polymerization and precipitation coherently with the observed experimental data.

SILICA POLYMERIZATION

We have hypothesized a first polymerization process described by growth of silica oligomers soluble in solution, not reacting up to a critical concentration of total silica corresponding to the preliminary step of precipitation. Only after reaching a critical size of particles with a distance in solution short enough to coalesce the particles start the precipitation. The critical step could be a tetramer formation as stated by Iacopini et al.[15] or an oligomer with proper size capable of coalescing. These last hypotheses or the first one should possibly agree with R&B observation, based on their experimental data. The phases of the polymerization process which, starting from the supersaturated monomer, lead to large particles and gels or powders, have been schematically represented by Iler[7]. This representation is applicable to aqueous systems, while little is known about the polymerization of silica when mono silicic acid is found in non-aqueous solutions. The various steps of polymerization can be indicated in the form of progressive chain growth. Examples are given the reactions up to the formation of the tetramer and from this the cyclic tetramer. Indicating with the symbol (Si) the various oligomers and, to simplify the notation, also the corresponding concentrations, we have:

$$2S_1 \Leftrightarrow S_2 - \frac{1}{2}\frac{dS_1}{dt} = \frac{dS_2}{dt} = k_1 {S_1}^2 - k_{-1}S_2 \qquad [5]$$

$$S_1 + S_2 \Leftrightarrow S_3 - \frac{dS_1}{dt} = \frac{dS_3}{dt} = k_2 S_1 S_2 - k_{-2}S_3 \qquad [6]$$

$$S_1 + S_3 \Leftrightarrow S_4 - \frac{dS_1}{dt} = \frac{dS_4}{dt} = k_3 S_1 S_3 - k_{-3}S_4 \qquad [7]$$

$$S_4 \Leftrightarrow S_{cy} - \frac{dS_4}{dt} = \frac{dS_{cy}}{dt} = k_{cy} S_4 - k_{-cy}S_{cy} \qquad [8]$$

The reactions can be considered up to the formation of the cyclic tetramer according to Iacopini or more generally the polymerization sequence up to the oligomer Sn:

$$\frac{dS_n}{dt} = k_{n-1} S_1 S_{n-1} - k_n S_1 S_n \qquad [9]$$

Such systems of equations do not allow analytical solutions, so the evolution of polymerization can be obtained with numerical simulations and knowing initial conditions, the kinetic constants of the various steps including the condensation reactions between different oligomers. A simplification of the equations, often used in the general treatment of polymerizations, can be obtained by considering only the chain growth reactions, excluding inverse reactions for each of them, neglecting condensation reactions between oligomers and considering that the kinetic constants for each step can be considered almost identical (ki=k). With these approximations and considering that all reactions contribute to the disappearance of the monomer, we have:

$$-\frac{dS_1}{dt} = 2k{S_1}^2 - kS_1S_2 - \mathrm{k}S_1S_3 \ldots\ldots - \mathrm{k}S_1S_n \qquad [10]$$

$$\frac{dS_2}{dt} = k{S_1}^2 - kS_1S_2 \qquad [11]$$

$$\frac{dS_3}{dt} = kS_1S_2 - kS_1S_3 \qquad [12]$$

$$\frac{dS_n}{dt} = kS_1S_{n-1} - kS_1S_n \qquad [13]$$

If a limit dimension corresponding to the oligomer Sn is admitted, the last term becomes:

$$\frac{dS_n}{dt} = kS_1S_{n-1} \qquad [14]$$

Once again, the above equations admit analytical solutions for special conditions. In the case of the early stages of polymerization or under conditions where the concentration of the monomer S1 is >> of Si. The equations are simplified:

$$-\frac{dS_1}{dt} \cong 2k{S_1}^2 \Rightarrow S_1 = S_1(0)\frac{1}{1+2kS_1(0)t} \qquad [15]$$

Summing up the expressions of oligomer formation kinetics yields:

$$\frac{dS_2}{dt} + \frac{dS_3}{dt} + \cdots \frac{dS_n}{dt} = \frac{d\sum_{i=1}^{n} S_n}{dt} = 2k{S_1}^2 - kS_1S_n = kS_1(2S_1 - S_n) \qquad [16]$$

In which the term could be set null if we assume a limit stage of polymerization before the beginning of precipitation, we have:

$$-\frac{dS_1}{dt} = 2k{S_1}^2 + kS_1\sum_{i=2}^{n} S_n \qquad [17]$$

If we indicate by A the total concentration of the polymers formed

$$A(t) = \sum_{i=2}^{n} S_n \qquad [18]$$

we have

$$-\frac{dS_1}{dt} = 2k{S_1}^2 - kS_1(A(t) - S_n) \qquad [19]$$

$$\frac{dA}{dt} = 2k{S_1}^2 - kS_1S_n = kS_1(2S_1 - S_n) \qquad [20]$$

In which again the term could be null if we stop the polymerization at the term nkS_1S_n

$$\frac{dA}{dt} = 2k{S_1}^2 \qquad [21]$$

And the term in S_1, considering the initial conditions in which A(t) almost zero

$$-\frac{dS_1}{dt} = 2k{S_1}^2 \qquad [22]$$

This last equation has a solution for a short time and therefore neglecting the contribution of oligomer formation reactions:

$$S_1 = S_1(0)\frac{1}{1+2kS_1(0)t} \qquad [23]$$

And for the term A(t):

$$A(t) = \frac{1}{4k}ln(1 + 2kS_1(0)t) \qquad [24]$$

Finally, considering the reaction in the phases in which high concentrations of the critical polymer Sn are reached, the following simplification can be obtained:

$$\frac{dS_n}{dt} = kS_1S_{n-1} \cong kS_1S_n \qquad [25]$$

And considering that the achievement of the critical dimensions of the oligomer occurs in conditions of still strong monomer excess, it can be obtained

$$\frac{dS_n}{dt} \cong kS_1(0)\frac{1}{1+2kS_1(0)t}S_n \qquad [26]$$

With solutions such of the type:

$$S_n \cong S_n(t_{cr})\sqrt{\frac{1+2kS_1(0)t}{1+2kS_1(0)t_{cr}}} \qquad [27]$$

In which the critical time represents a characteristic time to reach the concentration $S_n(t_{cr})$ of Sn oligomers that can initiate the silica precipitation phase. In a real situation, these equations, with all the approximations mentioned above, can represent the first stages of polymerization and the stages for which it can be admitted that, when a concentration of oligomers is reached (actually with a value of terms not so much equal to n but in the neighborhood of n described by a distribution of values around n), it can also represent the conditions in which silica precipitation is observed in conditions of strong supersaturation. This condition, according to the author of the present study, is well represented by the experimental R&R observations in which the temporal evolution of silica solubility as a function of strong supersaturation is experimentally studied and in which induction times have been observed that could be interpreted with a critical time such as the one introduced in the discussion so far.

To proceed further in the interpretation of the experimental R&R data, an observation can be made in the analysis of the experimental data obtained by the authors. First, it can be observed that the induction times depend on the degree of supersaturation of the solution and that the variation of the same between one experiment and the next at a certain temperature depends on the degree of supersaturation itself. Take as an example the graph of concentrations as a function of temperature taken from R&R (Figure 2), for example for the temperature of 180°C t_{cr}.

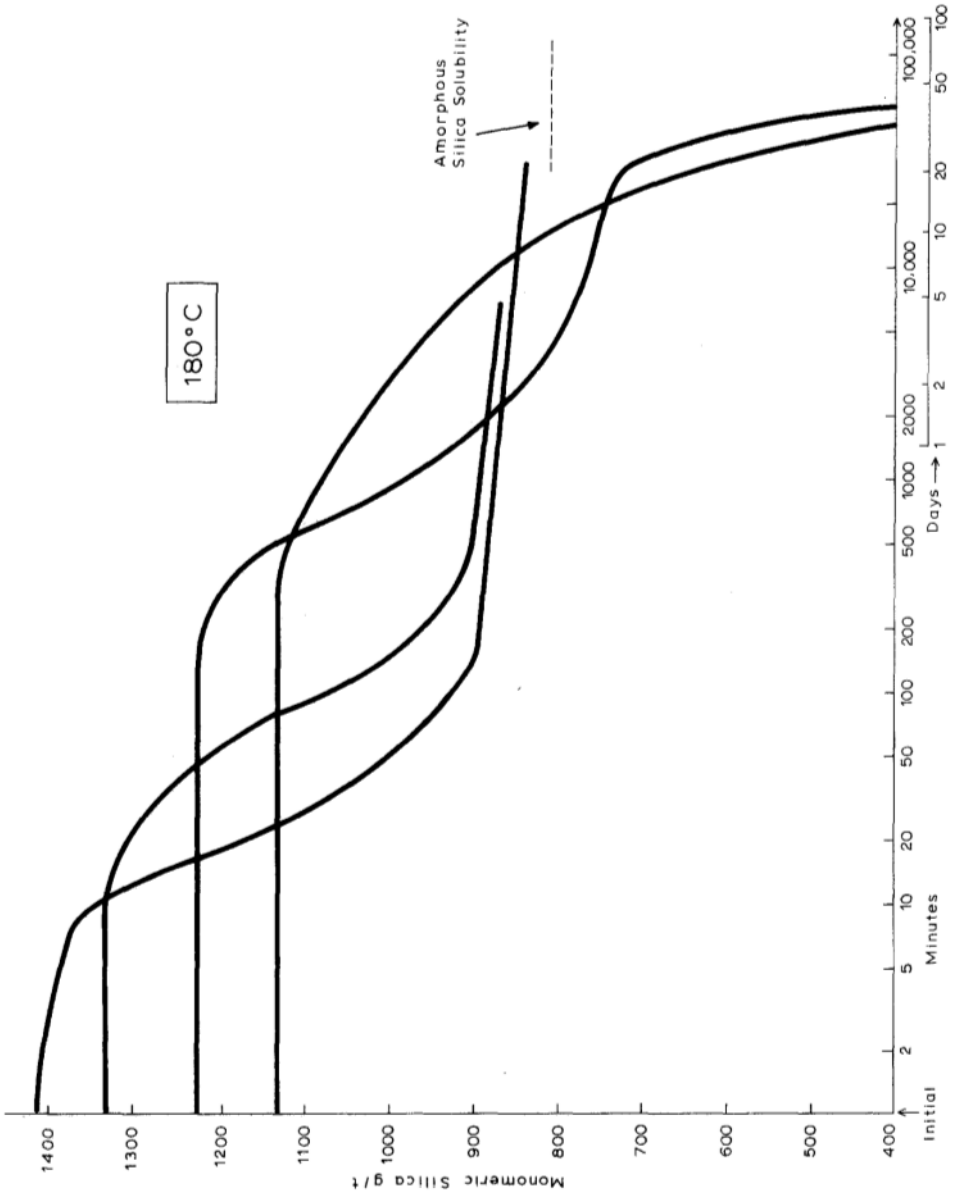


Figure2: induction times and silica precipitation curves after Rothbaun&Rohde[2]

In addition to having a critical time (induction time according to R&B notation) that depends on the ΔS supersaturation, expressed as a first-order development is:

$$t_{cr} = F(\Delta S) = F_0 - F_1 \Delta S \qquad [28]$$

In which represents the supersaturation achieved in the experiment, it is observed that even between one experiment and another critical time increases as the supersaturation decreases and therefore, we will also have:

$$\frac{dt_{cr}}{d\Delta S} = A(\Delta S) = A_0 - A_1 \Delta S \qquad [29]$$

By collecting ΔS in the two expressions and considering the first-order development valid for both, we can deduce a relation of the type:

$$\frac{dt_{cr}}{d\Delta S} - \frac{A_1}{F_1} t_{cr} + A_1 \left(\frac{F_0}{F_1} - \frac{A_0}{A}\right) = 0 \quad [30]$$

That has a general solution of the type:

$$t_{cr} = \alpha e^{-\beta \Delta S} + \gamma \quad [31]$$

And by putting

$$t_{cr}(\Delta S = \infty) = 0 \; and \; t_{cr}(\Delta S = 0) = t_{max} \quad [32]$$

It takes the form:

$$t_{cr} = \alpha e^{-\beta \Delta S} + \gamma \quad [33]$$

that can be expressed, by putting tcr(0)=0 $\Leftrightarrow \gamma = 0$, and tcr=tmax for $\Delta S = 0$. Finally we have

$$t_{cr} = t_{max} e^{-\beta \Delta S} \; or \; \ln(t_{cr}) = \ln(t_{max}) - \beta \Delta S \quad [34]$$

Which is coherent with expression derived from correlation of experimental data that shows a condition of linearity between ln(t) and DS. Finally, to obtain a simple and practical expression of the correlation function we have used the correlation function arising by collection of all the experimental Induction times assuming arbitrarily that the temperature dependence is contained only in the DS function in which the equilibrium silica concentration is assumed to be expression by Fournier et al.

RESULTS AND DISCUSSION

With this assumption we have (taking the 10 base logarithm):

$$\log(\mathrm{It}) = -0.0049([\mathrm{SiO2}] - [\mathrm{SiO2}]eq) + 3.6106 \quad [35]$$

And taking account of the silica equilibrium concentration by Forunier et al.[1]:

$$log([\mathrm{SiO2}]eq) = 4.52 - 731/\mathrm{T(K)} \quad [36]$$

we obtain the final expression

$$\log(\mathrm{It}) = 3.6106 - 0.0049\left([\mathrm{SiO2}]_0 - 10^{\left(4.52 - 731/\mathrm{T(K)}\right)}\right) \quad [37]$$

This expression must be considered as derived from the analysis of experimental data and hence its validity is to be considered in experimental conditions measured by R&R authors and considered in the present analysis, namely:

$$0.5 < \log(\mathrm{It}) < 3 \quad [38]$$

$$170\ mg/kg < \Delta S = [\mathrm{SiO2}] - [\mathrm{SiO2}]eq < 840\ mg/kg \quad [39]$$

The expression is partially described by general silica polymerization equations, in turn strongly dependent on approximations, and the final expression, although coherent with the expressions showing a nearly limiting value for silica accretion oligomers before their coalescence and precipitation, must be considered an expression carrying towards a practical formula derived from theoretical considerations and description of experimental behavior of silica. For this reason, we have defined the expression as a heuristic formula.

We consider anyway necessary to consider the expression as a practical expression giving an idea of the induction times that we can expect in silica handling. Given the strong dependence od silica polymerization, including induction times on critical parameters like temperature and pH over the strong effect that some ions like Aluminum and iron on the complex kinetics that can be observed in actual geothermal power plants we recommend to use this expression as guideline to evaluate the order of magnitude of the induction times and confirm by experimental data the actual behavior of silica in scale polymerization and scale formation.

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