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Effect of pH on the Solubility of $CaSO_4 \cdot 2H_2O$ in Aqueous NaCl Solutions and Physicochemical Solution Properties at 35 °C

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The solubility of $CaSO_4 \cdot 2H_2O$ has been determined in aqueous NaCl solutions up to very high salinities in the pH range of 2.5 to 8.5 at 35 °C. The solubility of $CaSO_4 \cdot 2H_2O$ increased slightly with a decrease in the pH of the solution. A nearly 12 % enhancement in the solubility maximum of $CaSO_4 \cdot 2H_2O$ was seen at pH 2.5. Surprisingly, an increase in the solubility of $CaSO_4 \cdot 2H_2O$ was also seen at pH 8.5. Solubility data at various pH values as a function of the concentration of NaCl have been correlated using polynomial fits with a maximum standard deviation of $< 0.002\ \mathrm{mol \cdot kg^{-1}}$. We have also measured the solution density (ρ), viscosity (η), and speed of sound (u) for the system $CaSO_4 \cdot 2H_2O + NaCl + H_2O$ at 35 °C and at different pH values. The isentropic compressibility (κ_S) of the solutions has been derived using the experimental density and speed of sound values. A relatively higher η and κ_S have been observed for the solutions of pH 8.5. All of the experimentally determined physical properties as a function of ionic strength of the solution have been correlated using polynomial or linear fits by the least-squares method. These studies are of relevance in the production of NaCl from natural brines with low impurities of Ca^{2+} and SO_4^{2-} ions.

Introduction

$CaSO_4 \cdot 2H_2O$ is predominantly a sparingly soluble electrolyte present in seawater and industrial water systems. It precipitates and forms scale once its saturation limit exceeds a certain threshold limit. Therefore, solubility data of $CaSO_4 \cdot 2H_2O$ in water or aqueous electrolyte solutions and other physicochemical data of gypsum-containing solutions are important for industries that use brines as a feed material. Such data are also important for understanding ionic equilibrium, ion−solvent and ion−ion interactions in natural waters.[1,2] In the past, a great amount of research work was done on the solubility-related problems of aqueous sodium chloride systems saturated with gypsum.[3−6] A research paper by Atkinson et al. provides a detailed review of the solubility of $CaSO_4 \cdot 2H_2O$ in aqueous and aqueous electrolyte solutions.[7] Despite a number of investigations on the subject, there are certain gaps in the solubility measurements and the physicochemical properties other than solubility data are scarcely available.

We are continuing our research program on the measurement and interpretation of the physical properties of aqueous electrolyte solutions saturated with $CaSO_4 \cdot 2H_2O$. In our earlier papers, we reported accurate physicochemical data of $CaSO_4 \cdot 2H_2O$ in aqueous NaCl,[8,9] $CaCl_2$,[9,10] and ($NaCl + CaCl_2$)[11] solutions. In the present study we investigate the effect of a change in pH on the solubility of $CaSO_4 \cdot 2H_2O$ in aqueous sodium chloride solutions. Other physicochemical properties, such as density, speed of sound, and viscosity of the solutions, have also been measured as a function of composition and pH of the solutions. A broad pH range of 2.5 to 8.5 has been covered in this study. Such studies can be very useful in the recovery of high purity NaCl from natural brines. During the solar evaporation of brines, the ionic impurities of Ca^{2+}, Mg^{2+}, SO_4^{2-}, and so on come along with the precipitation of NaCl. The presence of ionic impurities in NaCl above certain levels is not desired by user industries. The ratio of Ca^{2+} to Mg^{2+} in NaCl is also very critical when feeding the brines for further processing in the chlor-alkali or soda ash industries.[12] Therefore, a motive of the present study is to see whether the number of Ca^{2+} ions can be altered so as to achieve a ratio of Ca^{2+} to Mg^{2+} in sodium chloride in the range of 2:1 to 3:1 by slight adjustment of the pH of the brines. Another objective is to provide accurate physicochemical data of the system, $CaSO_4 \cdot 2H_2O + NaCl + H_2O$, at different pH values that are not available in the literature.

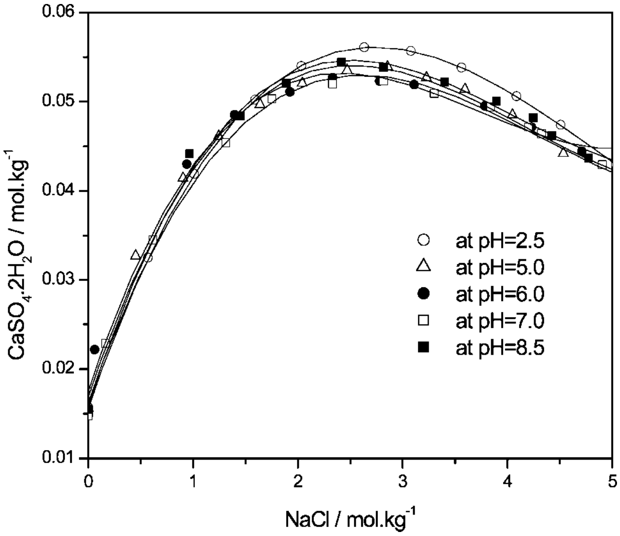

Figure 1. $CaSO_4 \cdot 2H_2O$ solubility in aqueous NaCl solutions at 35 °C and at various pH values.

Experimental Section

Reagent grade $CaSO_4 \cdot 2H_2O$ and NaCl (> 99.8 mol %) obtained from SD Fine Chemicals (Bombay) were used without further purification. Salt solutions were prepared by weight by

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Table 1. Molal Solubilities of $CaSO_4 \cdot 2H_2O$ (m_2) in Aqueous NaCl (m_1) at 35 °C in the pH Range of 2.5 to 8.5

pH = 2.5		pH = 5.0		pH = 6.0		pH = 7.0		pH =8.5	
m_1	m_2	m_1	m_2	m_1	m_2	m_1	m_2	m_1	m_2
0	0.0157	0	0.0156	0	0.0149	0	0.0148	0	0.0155
0.5686	0.0325	0.4506	0.0327	0.0603	0.0222	0.1650	0.0229	0.9635	0.0441
1.0096	0.0419	0.9034	0.0414	0.9390	0.0430	0.6175	0.0345	1.4500	0.0484
1.5915	0.0502	1.2436	0.0461	1.3951	0.0485	1.3110	0.0454	1.8895	0.0521
2.0316	0.0540	1.6384	0.0497	1.9232	0.0511	1.7506	0.0503	2.4135	0.0544
2.6327	0.0561	2.0414	0.0521	2.3298	0.0527	2.3309	0.0520	2.8159	0.0538
3.0769	0.0557	2.4717	0.0535	2.7783	0.0523	2.8165	0.0523	3.4002	0.0522
3.5591	0.0538	2.8549	0.0539	3.1082	0.0519	3.3020	0.0509	3.8930	0.0501
4.0854	0.0506	3.2282	0.0527	3.7841	0.0495	3.8350	0.0494	4.2472	0.0482
4.5064	0.0474	3.5958	0.0514	4.2304	0.0471	4.2002	0.0471	4.4212	0.0462
5.0364	0.0429	4.0483	0.0485	4.7104	0.0444	4.3222	0.0464	4.7711	0.0437
		4.5343	0.0442			4.9037	0.0429		
						5.3408	0.0399		

Table 2. Parameters A_i and the Standard Deviations σ of Equation *1* for the System $CaSO_4 \cdot 2H_2O$ + NaCl + H_2O at 35 °C

	A_1	A_2	A_3	A_4	σ
	solubility/mol·kg^{-1}				
pH = 2.5	0.0158	0.0339	− 0.0086	0.0001	0.0001
pH = 5.0	0.0171	0.0343	− 0.0098	0.0008	0.0012
pH = 6.0	0.0177	0.0350	− 0.0107	0.0010	0.0016
pH = 7.0	0.0165	0.0328	− 0.0090	0.0007	0.0001
pH = 8.5	0.0161	0.0360	− 0.0104	0.0009	0.0011

the use of an analytical balance with a precision of ± 0.0001 g (Denver Instrument APX-200) in Millipore grade water. Waters of the desired pH values were prepared by the addition of either hydrochloric acid or sodium hydroxide to the Millipore grade water. Stock solutions of NaCl were prepared by the addition of a known amount of oven-dried salt to the waters of various pH values. We then prepared a range of solutions of different NaCl concentrations saturated with $CaSO_4 \cdot 2H_2O$ by diluting the stock solutions of sodium chloride with the waters of various pH values and adding excess $CaSO_4 \cdot 2H_2O$. The resulting solutions were stirred in a thermostatically controlled water bath. After the solutions were stirred with an electrical paddle for about 24 h, liquid samples were periodically withdrawn and analyzed for different ions, as described in our previous paper.[8]

The density ρ of the solutions was measured with an Anton Paar (model DMA 4500) vibrating-tube densimeter with a resolution of $5 \cdot 10^{-5}$ g·cm^{-3}. The densimeter was calibrated with doubly distilled and degassed water, with dry air at atmospheric pressure, and also against the densities of NaCl(aq)[13] with an accuracy of 0.01 %. The temperature of the apparatus was controlled to within ± 0.03 K by a built-in Peltier device.

The viscosity (η) was measured with a Ubbelhode suspended-level viscometer (capillary diameter = 0.46 mm), as described by Pal and Singh.[14] The viscometer was calibrated with KCl(aq) solutions. Viscosity values were accurate to within 1 %. The reproducibility of the viscosity measured in triplicate was 0.25 %.

Speeds of sound (u) in aqueous solutions of electrolytes were measured at 51 600 Hz by the use of a concentration analyzer (model 87, SCM laboratory sonic composition monitor) on the basis of a sing-around technique[15] with a single transducer cell immersed in a water bath with temperature controlled to ± 0.01 K. The analyzer was calibrated by measurements of speed of sound in water as a reference, and the relative error was estimated to be less than ± 0.02 m·s^{-1}.[16]

Results and Discussion

Experimental results of the solubility of $CaSO_4 \cdot 2H_2O$ in aqueous NaCl solutions at different pH values are given in Table 1 and are shown in Figure 1. The composition dependence of $CaSO_4 \cdot 2H_2O$ solubility in the aqueous NaCl solutions of various pH values are well correlated by means of a polynomial type equation

$$F(Q) = A_0 + A_1(m\text{NaCl}) + A_2(m\text{NaCl})^2 + A_3(m\text{NaCl}) \quad (1)$$

where m is the concentration (mol·kg^{-1}) of NaCl in the solution. The values of the parameters A_i (i = 0 to 3) were evaluated by the least-squares method with all points weighted equally. The parameters A_i and standard deviation σ are given in Table 2.

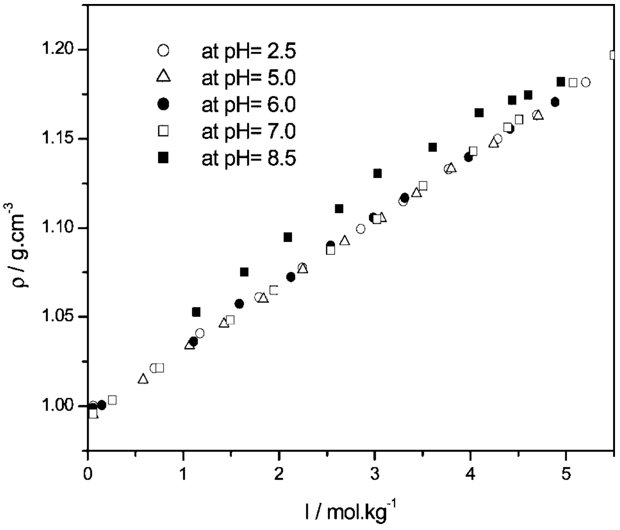

Figure 2. Density (ρ) of aqueous NaCl solutions saturated with $CaSO_4 \cdot 2H_2O$ at 35 °C and at various pH values.

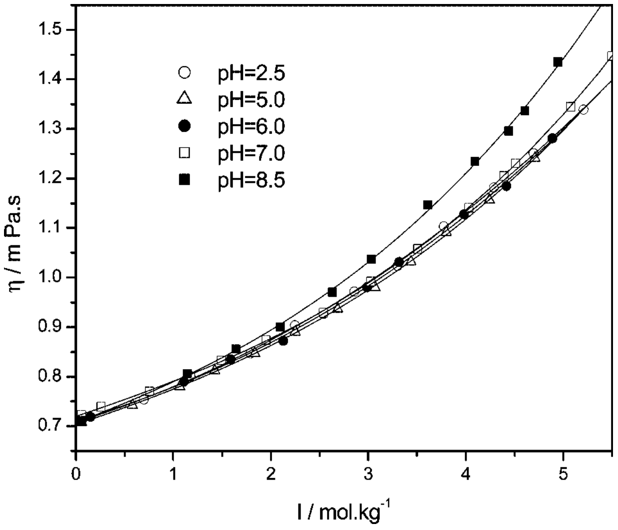

Figure 3. Viscosity (η) of aqueous NaCl solutions saturated with $CaSO_4 \cdot 2H_2O$ at 35 °C and at various pH values.

Table 3. Density (ρ), Viscosity (η), Speed of Sound (u), and Isentropic Compressibility (κ_S) of the $CaSO_4 \cdot 2H_2O$ + NaCl + H_2O System at 35 °C as a Function of Ionic Strength in the pH Range of 2.5 to 8.5

I / $mol \cdot kg^{-1}$	ρ / $g \cdot cm^{-3}$	η / m $Pa \cdot s$	u / $m \cdot s^{-1}$	κ_S / $T \cdot Pa^{-1}$
		pH = 2.5		
0.0628	1.0003	0.711	1521.3	432.0
0.69874	1.0213	0.754	1553.7	405.6
1.17727	1.0409	0.801	1583.8	383.0
1.79217	1.0612	0.847	1612.0	362.6
2.24768	1.0777	0.904	1640.3	344.9
2.85722	1.0995	0.972	1668.4	326.7
3.29984	1.1149	1.024	1692.6	313.1
3.77445	1.1329	1.103	1719.0	298.7
4.28761	1.1499	1.182	1741.1	286.9
4.69582	1.1631	1.251	1763.0	276.6
5.20792	1.1818	1.339	1787.7	264.8
		pH = 5.0		
0.0624	0.9953	0.708	1521.7	433.9
0.58144	1.0148	0.742	1546.3	412.1
1.06895	1.0339	0.780	1570.3	392.3
1.42786	1.0461	0.812	1592.1	377.1
1.83735	1.0601	0.847	1614.6	361.8
2.24982	1.0766	0.890	1636.3	346.9
2.68567	1.0923	0.936	1658.1	333.0
3.07032	1.1053	0.980	1677.0	321.7
3.43928	1.1193	1.033	1697.1	310.2
3.80122	1.1332	1.091	1716.2	299.6
4.24232	1.1471	1.157	1737.6	288.7
4.71097	1.1628	1.241	1757.8	278.3
		pH = 6.0		
0.0596	0.9992	0.714	1521.3	432.5
0.14917	1.0007	0.719	1527.2	428.5
1.11105	1.0364	0.790	1577.2	387.9
1.58914	1.0576	0.835	1603.3	367.9
2.12764	1.0726	0.872	1628.1	351.7
2.54047	1.0902	0.927	1652.9	335.7
2.98728	1.1058	0.980	1675.3	322.2
3.31567	1.1168	1.032	1690.4	313.4
3.9822	1.1398	1.128	1722.0	295.9
4.41872	1.1555	1.185	1743.5	284.7
4.88792	1.1707	1.281	1770.0	272.7
		pH = 7.0		
0.0592	0.9959	0.723	1521.4	433.8
0.25651	1.0036	0.739	1533.6	423.7
0.75574	1.0215	0.771	1557.5	403.6
1.49239	1.0484	0.833	1594.2	375.3
1.95171	1.0651	0.874	1617.5	358.9
2.53883	1.0874	0.93	1648.4	338.4
3.02581	1.1049	0.992	1673.6	323.1
3.50544	1.1235	1.058	1699.4	308.3
4.03238	1.1429	1.141	1727.4	293.2
4.38838	1.1564	1.205	1747.0	283.3
4.5076	1.1608	1.231	1753.5	280.2
5.07518	1.1815	1.345	1783.9	266.0
5.50031	1.1970	1.447	1807.2	255.8
		pH = 8.5		
0.062	0.9962	0.711	1521.3	433.8
1.14002	1.0528	0.807	1583.5	378.8
1.64364	1.0754	0.856	1612.4	357.7
2.09781	1.0949	0.901	1637.2	340.7
2.63098	1.1107	0.970	1665.1	324.7
3.03127	1.1306	1.037	1686.1	311.1
3.60892	1.1451	1.147	1714.9	296.9
4.0933	1.1646	1.234	1738.1	284.2
4.43994	1.1717	1.296	1754.2	277.3
4.60615	1.1745	1.337	1761.9	274.3
4.94574	1.1819	1.435	1777.1	267.9

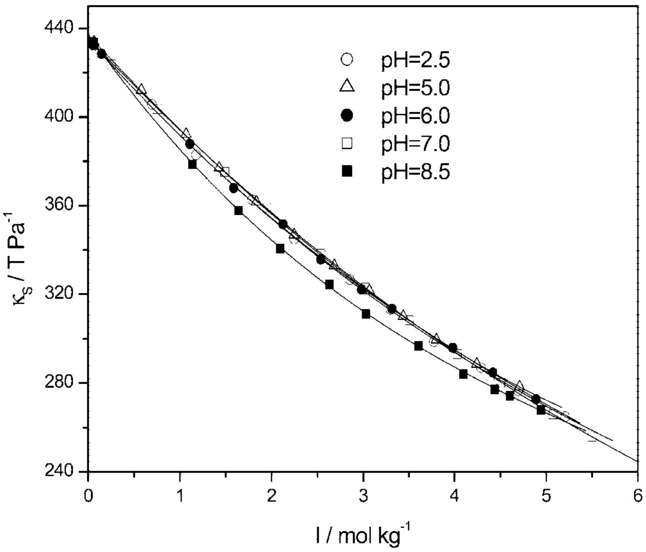

Figure 4. Derived isentropic compressibility (κ_S) of aqueous NaCl solutions saturated with $CaSO_4 \cdot 2H_2O$ at 35 °C and at various pH values.

Table 4. Parameters A_i and the Standard Deviations σ of Equation *2* for the System $CaSO_4 \cdot 2H_2O$ + NaCl + H_2O at 35 °C

	A_1	A_2	A_3	A_4	σ
		$\rho/10^{-3} \cdot kg \cdot m^{-3}$			
pH = 2.5	0.9981	0.0354			0.0009
pH = 5.0	0.9943	0.0362			0.0010
pH = 6.0	0.9974	0.0358			0.0017
pH = 7.0	0.9935	0.0370			0.0004
pH = 8.5	0.9931	0.0560	− 0.0040	0.0001	0.0023
		$\eta/m \cdot Pa \cdot s$			
pH = 2.5	0.710	0.057	0.012		0.005
pH = 5.0	0.704	0.065	0.005	0.001	0.003
pH = 6.0	0.712	0.052	0.013		0.006
pH = 7.0	0.720	0.067	0.002	0.002	0.002
pH = 8.5	0.705	0.075	0.006	0.002	0.008
		$u/m \cdot s^{-1}$			
pH = 2.5	1520.12	51.88			2.42
pH = 5.0	1518.00	51.72			1.99
pH = 6.0	1520.29	51.08			1.91
pH = 7.0	1517.31	52.29			1.64
pH = 8.5	1517.27	60.43	− 1.59		0.28

The solubility of $CaSO_4 \cdot 2H_2O$ increases with an increase in the concentration of NaCl in solution at all studied pH values (i.e., in the range of 2.5 to 8.5). A maximum in solubility is reached at around 2.5 $mol \cdot kg^{-1}$ of NaCl concentration in the solution at all pH values except at a pH of 8.5, where the maximum appears to shift toward a slightly higher NaCl concentration. The solubility of $CaSO_4 \cdot 2H_2O$ also increases with a decrease in the pH value of the solution. An increase in the solubility of $CaSO_4 \cdot 2H_2O$ at low pH values may be because H^+ ions provide localized acidification to dissolve the mineral and also because of the higher hydration of ions.

The experimental results of solution density (ρ) viscosity (η), speed of sound (u), and derived isentropic compressibility (κ_S) as a function of ionic strength are presented in Table 3. The composition dependence of ρ, η, and u has been correlated using a polynomial fit of type

$$F(Q) = \sum_{i=1}^{n} A_i I_m^{i-1} \tag{2}$$

where Q represents a general measured property (density, viscosity, or speed of sound) and I_m represents the ionic strength of the solution $\left(I_m = 1/2\sum_{i-1}^{n} c_i z_i^2\right)$. The values of the parameters A_i were evaluated by the least-squares method with all points being weighted equally. The parameters A_i and standard deviation σ are given in Table 4. The dependence of density, viscosity, and derived compressibility as a function of ionic strength in the solutions of different pH values is shown in Figures 2, 3, and 4. Density increases linearly with an increase in concentration except in basic solutions, where the increase is monotonic. No significant change was observed

Table 5. Viscosity Coefficients *B* and *D* of Equation *3* for $CaSO_4 \cdot 2H_2O + NaCl + H_2O$ System of Different pH values at 35 °C

pH	*B*	*D*
2.5	0.088	0.016
5.0	0.094	0.011
6.0	0.091	0.014
7.0	0.089	0.014
8.5	0.111	0.009

in the density of the solutions with the change in pH in the acidic medium. The viscosity of the solutions at all pH values, as shown in Figure 3, also increases monotonically with the increase in concentration of the solution. Solutions in the basic medium are comparatively more viscous as compared with the solutions in the acidic medium. The Jones−Dole equation[17] has been widely used to represent the concentration dependence of viscosity

$$\eta_r = \eta/\eta_o = 1 + A\sqrt{c} + Bc \qquad (3)$$

where η_r is the relative viscosity, η and η_o are the viscosities of the solution and water, respectively, c is the concentration, and A and B are empirical constants. At higher concentrations, eq 3 can be modified to eq 4 by ignoring the contribution of A and adding a c^2 term

$$\eta_r = \eta/\eta_0 = 1 + Bc + Dc^2 \qquad (4)$$

The coefficients, B and D, being empirical and qualitative, are considered to reflect the effect of solute−solvent and solute−solute interactions, respectively, on the viscosity.[18] The B and D coefficients thus calculated using the least-squares method by fitting the parameters of eq 3 to different pH values are given in Table 5. Appreciable positive B coefficient values, which increase slightly with the increase in the pH of the solution, suggest the presence of strong ion−solvent interactions. The D coefficient decreases with an increase in the pH of the solution, indicating the decrease in ion−ion interactions from acidic to basic medium.

The speed of sound increases linearly with an increase in the concentration of NaCl in the solutions saturated with gypsum at all studied pH values. This behavior is consistent with the density measurements. Assuming the ultrasonic absorption under the experimental conditions to be negligible, we derived the isentropic compressibility (κ_S) from u and ρ values by using the Newton−Laplace equation

$$\kappa_S = 1/u^2\rho \qquad (5)$$

Figure 4 shows a comparison of κ_S values as a function of ionic strength of the solution at different pH values. κ_S decreases monotonically with an increase in concentration as well as with an increase in the pH of the medium. This behavior is well supported by the viscosity results wherein the more viscous solutions are less compressible, which may be due to the breaking of the 3D water structure, thereby making the solutions more rigid with the increase in both the concentration and pH of the medium.

Conclusions

The solubility of $CaSO_4 \cdot 2H_2O$ increases with an increase in NaCl concentration up to ∼ 2.5 $mol \cdot kg^{-1}$ and then starts to decrease with a further increase in NaCl concentration in the solution. A similar pattern is maintained in the investigated pH range from 2.5 to 8.5. A decrease in the pH of the solution slightly increases the solubility of $CaSO_4 \cdot 2H_2O$. Solution properties such as density, viscosity, and speed of sound in solution do not significantly change with the reduction in the pH of the solution; however, the solution properties are appreciably altered in the basic medium.

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