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Solubility and scale prevention of gypsum in transportation pipes of well brine with salinities up to 5 M at temperature range of 278–298 K

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ABSTRACT

The results of work performed on reducing gypsum scale formation in transportation pipes of well salt brine are described. Two methods: seeding with calcium sulfate dihydrate and dilution of brine were proposed. With the first method, various mass and size distributions of seeds were introduced into an perspex reactor with well salt brine (NaCl: 300 g/L, Na_2SO_4: 20 g/L) to investigate the factors that influence crystallization of calcium sulfate dihydrate, such as temperature, mass of seed crystals, and size distribution of seed crystals. The kinetics of calcium sulfate dihydrate growth at temperatures ranging between 5 and 25°C were obtained. Results showed that the elimination of supersaturation was enhanced with increasing temperature and the dosage of seed crystal. It was also found that the more efficient in scale prevention was obtained when smaller seed crystals were employed. It was seen that the crystal growth of calcium sulfate dihydrate in brine was proportional to the square of supersaturation at the investigated temperatures. With the second method, results showed that a complete elimination of supersaturation was achieved when 4.3% water was added into the brine with the help of solubility determination of calcium sulfate dihydrate.

Keywords: Scale prevention; Well brine; Calcium sulfate dehydrate; Supersaturation; Seeded crystal growth

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1. Introduction

Calcium sulfate scale formation is widely encountered in many industrial processes, such as production of salt and oil, hydrometallurgical processes [1,2], seawater desalination [3,4] and membrane processes [5–7]. It causes serious technical and economic problems. Scale formation brings about a decrease in the volume capacity of equipment, blocks piping, increases corrosion, and lowers the heat transfer coefficient. Periodic descaling also has an adverse effect on the process economics and the equipment involved. Therefore, methods contributing to prevention of scale formation should be carried out when unit operations are prone to scale formation.

Calcium sulfate precipitates in three main solid phases—calcium sulfate dihydrate, calcium sulfate hemihydrate and calcium sulfate anhydrous, they deposit in turn as temperature increases. The solubility of all calcium sulfate forms decreases with increasing temperature. Prevention of gypsum scaling has been widely investigated. Generally, two methods were used (1) softening method and (2) introduction of scale inhibitor. With the softening method, the feed water is softened by treatment with lime ($Ca(OH)_2$) and soda (Na_2CO_3), CO_2, thermal and ion exchange, so the formation of scaling compounds

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are removed from the system. Linnikov and Podbereznyi [3] studied the prevention of sulfate scale formation in desalination of Aral sea water. It was found that a quick supersaturation removal was obtained with the thermal softening method at a temperature higher than 100 °C, and the supersaturation removal could be accelerated by introducing gypsum crystals at 97.5 °C. Introduction of an inhibitor to the system is another efficient way to prevent scaling. It was implemented by adding very small (ppm) amounts of effective admixtures such as organic polymers, surface active agents or organic phosphonates in the solutions [8–11]. Admixture adsorption onto crystal growth sites has been suggested as a mechanism by which nucleation and crystal growth are reduced. Amjad [12] investigated the inhibition of gypsum scale formation on heat exchanger surfaces by polymeric additives, it was concluded that polymer composition, molecular weight, ionic charge and charge density, and polymer dosage have a significant impact on the performance of the polymeric additive. Shih et al. [13] developed an experimental system to assess and rank the performance of commercial antiscalants designed to inhibit mineral scale formation on reverse osmosis membrane surfaces, and the approach enabled a relatively simple and robust initial screening of candidate antiscalants based on their induction time as a function of the applied dose.

Promotion of precipitation within the bulk solution can also be achieved by introducing a sufficient number of seed crystals. The scale-forming compound is then deposited on the seeds, so reducing the supersaturation below the critical level required for solid nucleation.

There is considerable interest in the mechanism of growth of calcium sulfate crystals. Liu and Nancollas [14] showed that the growth with seed crystal follows a second order reaction in relative supersaturation and the rate constant is independent of surface area but dependent upon the number of active sites. Smith and Sweett [15] used a similar method for analysis of seeded growth experiments with gypsum, and found that the nucleation was heterogeneous. They also indicated that the growth rate was proportional to the surface area and to the square of supersaturation. Both of them confirmed that a surface reaction step was the effective rate controlling step. Christoffersen et al. [16] has shown that the growth rate of calcium sulfate dihydrate crystals in aqueous suspension to be screw dislocation controlled in the supersaturation range $1.03 < C/C_{sat} < 1.15$. Klepetsanis et al. [17] studied the spontaneous precipitation of calcium sulfate in supersaturated solutions over the temperature range between 25 and 80 °C, and found that the linear dependence of the rates of precipitation on the relative solution supersaturation suggested a mechanism according to which the growth units were integrated into the active sites of the supercritical nuclei by surface diffusion. Recently, Sheikholeslami and Ong [18] found that the kinetics of pure $CaSO_4$ precipitation was strongly affected by the level of salinity.

Gypsum scale depositing on the transportation pipes is one of most prominent problem in industrial production. Hoang et al. [19] investigated the effects of temperature on the formation of calcium sulfate scales in pipes, using a pipe flow system. Results indicated that high temperature produced a large increase of scale amounts and a significant decrease of induction periods. The scaling rate is a linear function of time and the scale mass is an exponential function of the inverse absolute temperature.

The present work was performed on reducing calcium sulfate formation in transportation pipes of well salt brine. Jiangsu Jingshen Salt Industry Co., Ltd in China is a salt-making enterprise through vacuum evaporation method. The raw material brine was obtained through brine wells, then transported to factory through steel pipes. During the transport process, scale was formed quickly in the pipeline, much more seriously in winter. The scale was primarily analyzed by X-ray diffraction (XRD) and complex titration with EDTA. The results showed that the scale was mainly composed of gypsum with the purity of more than 95%. By measuring the calcium sulfate solubility in the brine, it was seen that the brine was supersaturated by calcium sulfate. Two methods were proposed to prevent scale formation. (1) seeded with gypsum and (2) dilution with water. In the first method, factors such as temperature, mass of seed crystals, and size distribution of seed crystals were studied. The kinetics growth of gypsum in the high salinity brine was also obtained. In the second method, the amount of water added was determined by measuring solubility of calcium sulfate in diluted brine.

2. Materials and experimental methods

2.1. Materials

Calcium sulfate dihydrate $CaSO_4{\cdot}2H_2O$ (≥99.0% mass fraction) was from Shantou Xilong Chemical Factory, Guang Dong province of China. All the experiments were carried out with well salt brine (Table 1) taken from Jiangsu Jingshen Salt Industry Co., Ltd in November 2007.

Table 1
Chemical composition of well salt brine

Temperature (°C)	Density (g/ml)	Component	C (g/L)	m (mol/kg)
25	1.209	NaCl	300.28	5.794
		Na_2SO_4	19.98	0.159
		$CaSO_4$	1.99	0.0163

2.2. Solubility measurement

A similar experimental procedure with Li and Demopoulos [20] was used to determine the solubility of calcium sulfate in well salt brine at a temperature range from 278 to 348 K. A standard equilibration time of 12 h was used. After the solid–liquid equilibrium was attained, stirring was stopped to allow solids to settle. The supernatant solution was then withdrawn and immediately filtered through 0.22 μm membrane filter (Xingya Purify Material Factory, Shanghai, China). The calcium stock solution was analyzed by titration with standard EDTA using murexide as indicator. The saturated filtrate was also used to determine the density of saturated solutions at the experimental temperature by using a 50 mL volumetric flask. The solid phase was washed with double distilled water three times and dried at 50 °C, then subjected to XRD (X'Pert PRO MPD PANalytical Netherlands) analysis to determine whether the solid phase had been altered by phase transformation. The double distilled water was used in all experiments.

2.3. Kinetics growth of seeded gypsum in well salt brine

The seed was prepared from analytic calcium sulfate dihydrate, thoroughly ground and sifted out, and three types of seed crystals with various diameters were obtained (Table 2). A crystallizer (Fig. 1) was used to carry out the experiments. The crystallizer was a round-bottom vessel of perspex, equipped with an agitator and baffles. The temperature (±0.1 °C) was controlled by water from a thermostat through a jacket surrounding the crystallizer. Normal operating capacity was 1 liter. 1 l brine was added in the crystallizer and allowed to equilibrate thermally before the addition of seed crystals. The stirring rate was maintained at 300 rpm with a Polytetrafluoroethylene stirrer. Three quality levels of 0.5 g, 1.0 g, and 1.5 g of seed crystals were added to the brine. Seeded growth in the brine was detected at 5 °C, 15 °C and 25 °C. Crystal growth was initiated by the addition of seed crystals, about 15 ml suspension was withdrawn at interval times. The suspensions were quickly filtered through 0.22 μm membrane filter and the filtrate was analyzed for calcium concentration by titration with EDTA using murexide as indicator. Scanning electron microscopy was also used to analyze the morphology of the growing crystals during the growth experiments.

Table 2
Various conditions investigated in experiments of kinetics growth of seeded gypsum

Mass of seeds (g)	Particle size of seeds (μm)	Temperature (°C)
0.5	25	5
1.0	48	15
1.5	75	25

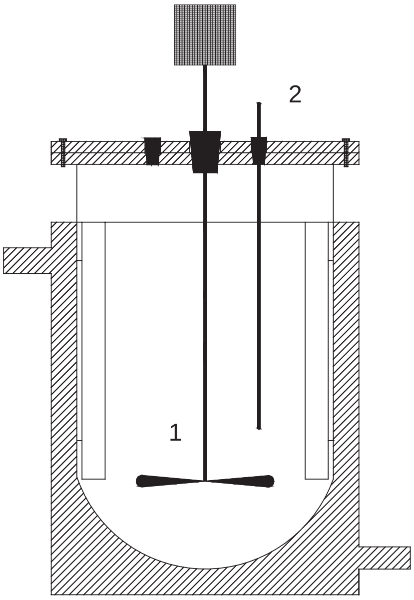

Fig. 1. Experimental apparatus. 1–Stirrer; 2–Thermometer; 3–Water circulator.

3. Results and discussion

3.1. Solubility of calcium sulfate in the brine ($NaCl–Na_2SO_4–H_2O$)

The solubility of calcium sulfate in NaCl solution at low temperature has been extensively studied. Raju and Atkinson [21] presented a comprehensive review of $CaSO_4$ solubility data in $NaCl–H_2O$ system. Ostroff and Metler [22] determined the solubility of gypsum in sodium chloride solutions at 28, 38, 50, 70, 90 °C. The results indicated that the solubility of gypsum increased with increas in NaCl concentration to 2.5 M, then decreased at higher concentrations, and remained constant with temperature for NaCl concentration up to 2.5 M. Block and Waters [23] measured the solubilities of gypsum in Na_2SO_4-$NaCl–H_2O$ system at salinities of 0.0, 0.5, 1.0, 2.0, 4.0 M NaCl over the temperature range from 25 to 100 ºC. Yeatts and Marshall [24] reported on the solubility of gypsum in $NaCl–Na_2SO_4–H_2O$ system at constant ionic strength of 0.5, 2, 6 over a wide range of ratio at 25 °C. In this study, however, the brine with composition given in Table 1 has such a high salinity of 5.8 m NaCl, and the solubility of calcium sulfate in the brine was carried out at temperatures up to 75 °C

Table 3
The solubility of calcium sulfate in the brine

Temperature (°C)	Density (g/ml)	C (g/l)	m (mol/kg)
5	1.219	1.65	0.0137
15	1.214	1.69	0.0141
25	1.209	1.74	0.0144
35	1.203	1.86	0.0154
45	1.198	1.97	0.0163
55	1.194	2.10	0.0174
60	1.191	2.29	0.0190
65	1.188	2.48	0.0205
70	1.184	1.77	0.0147
75	1.182	1.73	0.0143

to determine the supersaturation since literature was unavailable for solubility of gypsum. The solubility data is listed in Table 3 and presented in Fig. 2. From Fig. 2, it is seen that the solubility of calcium sulfate increases with increasing temperature, after passing a maximum at 65 °C, then the solubility declines sharply. The XRD analysis of equilibrated solid phases are presented in Fig. 3, which showed gypsum in brine to be stable at the temperature range of 5 to 65 °C, while the solid phase changed to be calcium sulfate hemihydrate above 70 °C.

The solubility equilibrium for calcium sulfate dihydrate solids in electrolyte aqueous solutions is expressed as follows:

$$Ca^{2+} + SO_4^{2-} + 2H_2O = CaSO_4{\cdot}2H_2O \quad (1)$$

Supersaturation ratio (S) of the solution with respect to calcium sulfate dihydrate is calculated by the below equation:

$$S = C/C_{sat} \quad (2)$$

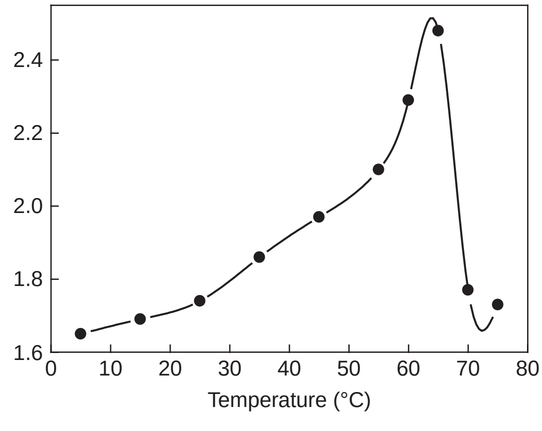

Fig. 2. The solubility of calcium sulfate in the brine.

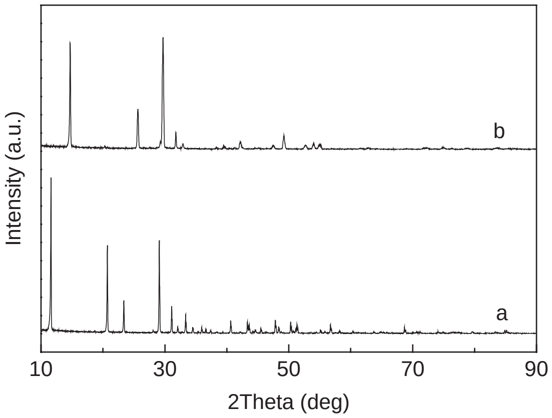

Fig. 3. XRD patterns of calcium sulfate equilibrated in brine (a) 65 °C; (b) 70 °C.

The experimental results show that the brine is supersaturated with calcium sulfate below 45 °C. The precipitation of calcium sulfate dihydrate in pipeline was formed at low operation temperature range of 5~35 °C. The mechanism of scaling in pipes is considered as that, the wall of pipe acts as nucleating site similar to foreign substances rendering heterogeneous nucleation, which requires less energy because the foreign substances reduce the surface energy of the nucleus. It is clear that the driving force for crystallization depends on the supersaturation level of the solution. The brine has a supersaturation of 1.14 at 25 °C while it has much higher value at low temperatures. This leads to more serious scaling in winter.

3.2. Effect of temperature, crystal mass and size distribution—Seeded with calcium sulfate dihydrate in high salinity brine

When gypsum crystallizes out of solutions, heat is absorbed. On the other hand, high temperature speeds up the transport of scale components from the bulk solution to the surface, therefore the crystallization can be accelerated by increasing the temperature. Nevertheless, the brine has a lower supersaturation with increasing temperature. As a result, the driving force for crystallization is relatively smaller at high temperatures. On the other hand, the quantity of seed introduced in the supersaturated solution is also important to the crystal growth. Besides, it is also known that the growth rate of gypsum is controlled by a surface reaction, hence the surface area plays an important role on the crystallization of gypsum. Obviously, the smaller seed crystals have a bigger specific surface area. Thus, in this work, to determine the effect of temperature, seed crystal mass and size distribution on the elimination of supersaturation,

Table 4
Experimental results for the supersaturation (*S*) elimination of $CaSO_4$ under various conditions

Parameters	0.5 g, 25 µm			0.5 g, 48 µm			0.5 g, 75 µm		
	5°C	15°C	25°C	5°C	15°C	25°C	5°C	15°C	25°C
T(min)	*S*	*S*	*S*	*S*	*S*	*S*	*S*	*S*	*S*
0	1.30	1.27	1.23	1.30	1.28	1.23	1.30	1.26	1.26
2	1.25	1.22	1.19	1.25	1.24	1.18	1.27	1.23	1.23
4	1.22	1.21	1.16	1.23	1.24	1.18	1.24	1.23	1.23
6	1.23	1.21	1.16	1.25	1.23	1.17	1.23	1.21	1.22
8	1.22	1.21	1.14	1.24	1.22	1.17	1.22	1.20	1.21
11	1.20	1.20	1.13	1.23	1.22	1.14	1.22	1.21	1.20
15	1.18	1.20	1.11	1.23	1.21	1.13	1.22	1.21	1.18
30	1.18	1.17	1.11	1.22	1.18	1.12	1.22	1.18	1.17
60	1.13	1.14	1.08	1.18	1.15	1.07	1.19	1.16	1.16
120	1.12	1.12	1.05	1.15	1.14	1.05	1.18	1.15	1.12
360	1.07	1.04	1.03	1.08	1.07	1.02	1.11	1.10	1.03
	1.0 g, 25 µm			1.0 g, 48 µm			1.0 g, 75 µm		
0	1.30	1.26	1.23	1.30	1.26	1.23	1.30	1.27	1.23
2	1.23	1.17	1.17	1.24	1.18	1.14	1.25	1.21	1.20
4	1.20	1.15	1.15	1.20	1.15	1.13	1.24	1.21	1.17
6	1.19	1.15	1.13	1.18	1.14	1.11	1.22	1.18	1.17
8	1.19	1.15	1.12	1.17	1.14	1.12	1.21	1.18	1.17
11	1.18	1.12	1.12	1.16	1.13	1.11	1.22	1.19	1.15
15	1.16	1.11	1.10	1.14	1.10	1.10	1.20	1.18	1.14
30	1.13	1.08	1.10	1.12	1.08	1.06	1.18	1.14	1.11
60	1.09	1.05	1.05	1.10	1.08	1.05	1.15	1.12	1.08
120	1.06	1.04	1.02	1.07	1.03	1.03	1.11	1.09	1.05
360	1.03	1.02	1.01	1.04	1.03	1.01	1.05	1.04	1.02
	1.5 g, 25 µm			1.5 g, 48 µm			1.5 g, 75 µm		
0	1.30	1.26	1.25	1.30	1.26	1.23	1.30	1.27	1.23
2	1.20	1.15	1.16	1.23	1.16	1.12	1.25	1.19	1.16
4	1.17	1.11	1.11	1.16	1.13	1.11	1.23	1.17	1.16
6	1.15	1.11	1.12	1.16	1.11	1.09	1.20	1.16	1.16
8	1.15	1.10	1.10	1.15	1.11	1.08	1.19	1.14	1.14
11	1.13	1.08	1.09	1.15	1.09	1.06	1.20	1.14	1.10
15	1.11	1.06	1.07	1.12	1.07	1.05	1.17	1.11	1.08
30	1.08	1.04	1.04	1.09	1.05	1.03	1.16	1.08	1.06
60	1.06	1.04	1.03	1.06	1.03	1.02	1.15	1.05	1.04
120	1.04	1.03	1.02	1.04	1.01	1.01	1.09	1.04	1.02
360	1.00	1.00	1.01	1.01	1.00	1.00	1.02	1.00	1.01

a systematic one-factor experiment design as listed in Table 2 was employed. Twenty-seven experiments were performed at various conditions. The results were presented in Table 4.

Generally, within the investigated temperature range from 5 °C to 25 °C, the introduction of gypsum seed crystals into the brine could efficiently decrease the calcium sulfate concentration as shown in Fig. 4. It is indicated that the rate of elimination increases when temperature increases. With 0.5 g/l seed crystals (75 µm), nearly a total elimination of supersaturation was obtained at 25 °C in 360 min while 63% elimination of supersaturation was obtained at 5 °C as presented in Table 4. Further, most of the elimination of supersaturation was obtained in 15 min even at low seed crystals concentration. For 0.5 g/l seed crystals (75 µm) at 5 °C, the supersaturation

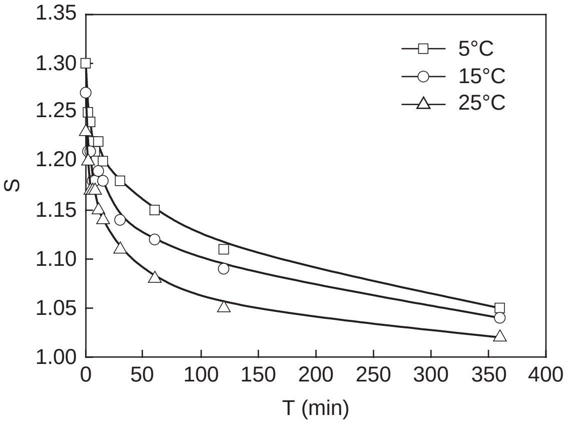

Fig. 4. The supersaturation elimination kinetics of $CaSO_4$ for 1.5 g, 75 µm crystals.

decreased from 1.30 to 1.11 in 360 min while 1.22 was reached in 15 min.

The effect of different quantities of seed on supersaturation removal is shown in Fig. 5. In all investigated levels, it was observed that when the concentration of seed crystals increases, a faster elimination of supersaturation was obtained. For instance, the supersaturation decreased from 1.23 to 1.13 at 25 °C in 15 min when 0.5 g/l seed (48 µm) was used. As the crystal concentration increases to 1.5 g/l, a higher elimination of supersaturation of 1.03 was achieved.

From Table 4 it can also be derived that smaller the seed crystals the faster elimination of supersaturation was obtained, which also can be observed directly in Fig. 6. The results are in agreement with the early studies [3,15]. It is to be noted that the size of seed crystals plays a more effective role when less seed crystals were added.

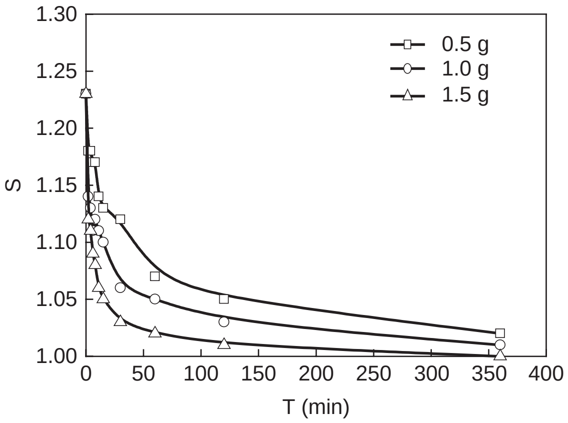

Fig. 5. The supersaturation elimination kinetics of $CaSO_4$ for 48 µm crystals at 25 °C.

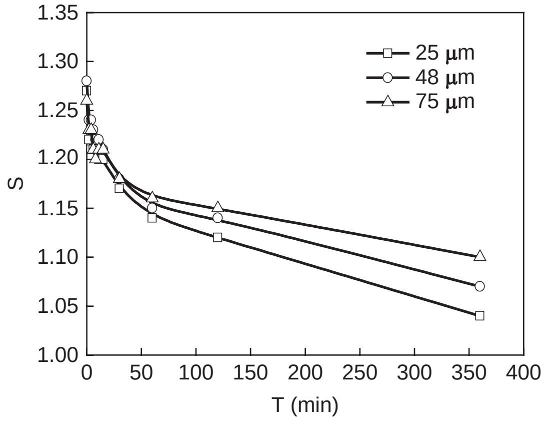

Fig. 6. The supersaturation elimination kinetics of $CaSO_4$ for 0.5 g seed crystals at 15 °C.

3.3. Crystal growth model: Seeded with calcium sulfate dihydrate in high salinity brine

Smith and Sweet [16] studied the crystallization of calcium sulfate dihydrate from diluted NaCl solutions at 30 °C. They have confirmed that the crystal growth of gypsum was a surface reaction control. After an initial surge, the rate of growth was found to be proportional to $(m/m_o)^{2/3}(C–C_{sat})^{2.0\pm0.2}$. Liu and Nancollas [14] had shown that the rate constant was proportional to the quantity of seeding crystals added initially, and the result is consistent with the idea that no new growth sites are formed during crystallization. Such would be the case if the growth is confined to the screw dislocations presented on the seed crystals. Their studies on precipitation of calcium sulfate dihydrate have focused on diluted salt solution (<0.1 M NaCl). In this work, the crystal growth of gypsum seed in concentrated brine (NaCl: 300 g/l, Na_2SO_4: 20 g/l) was performed. Under the conditions in which no induction period is observed, the rate of crystallization in supersaturated brine would be expected to follow an equation of the form [15]

$$-dC/dT = KA(C-C_{sat})^2 \tag{3}$$

The linearity plots of the integrated form of Eq. (3) at 25 °C with different seed sizes is illustrated in Fig. 7. As it can be observed, for the experiments carried out with different seed masses, $(C–C_{sat})^{-1}–(C_i–C_{sat})^{-1}$ is proportional to the stirring time. The results indicate that the growth rate is proportional to the square of supersaturation. Fig. 8 shows the *KA* of different masses for the seed crystals with size of 25 µm at 25 °C, and it is seen that the *KA* gives a linear relationship with different masses. The result is consistent with the idea that the rate constant is proportional to the crystal surface area.

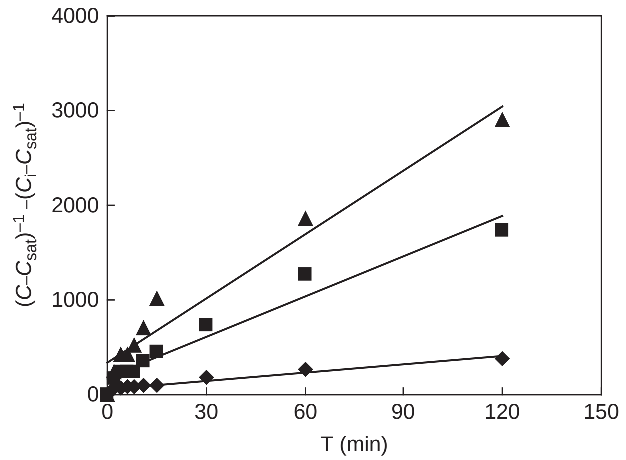

Fig. 7. The crystal growth kinetics of gypsum seed at 15°C with seeds of 25 μm: -♦- 0.5 g; -■- 1.0 g; -▲- 1.5 g.

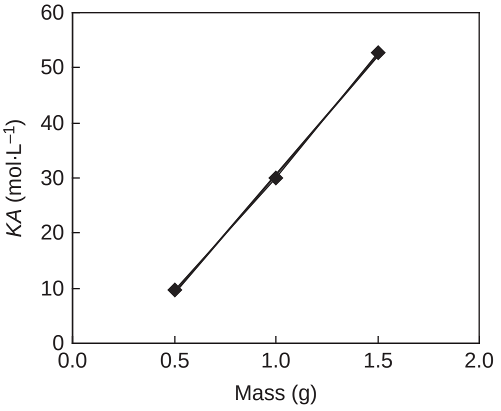

Fig. 8. Plots of *KA* as a function of the seed mass at 25 °C.

Table 5
Solubility and supersaturation of calcium sulfate in the diluted brines at 25°C

V (mL)	ρ (g/L)	ρ_{sat} (g/L)	*S*
0	1.99	1.74	1.14
1	1.97	1.77	1.11
2	1.95	1.82	1.07
3	1.93	1.85	1.04
4	1.91	1.89	1.01
5	1.90	1.92	0.99
10	1.81	2.12	0.85
15	1.73	2.31	0.75
20	1.66	2.50	0.66

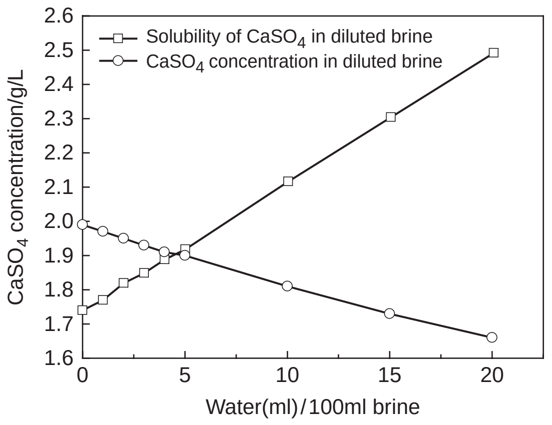

Fig. 9. Solubility and concentration of calcium sulfate in diluted brine.

3.4. Dilution method

Various volume of water was added into the brine at 25°C, and then the solubility of calcium sulfate dihydrate was measured. The solubility and concentration of calcium sulfate in the diluted brines are listed in Table 5 and shown in Fig. 9. Obviously, the calcium sulfate concentration in brine decreases with increasing the dosage of water. The relationship between the concentration of calcium sulfate and the dosage of water is expressed as follows:

$$\rho = \rho_i/(1 + V/100) \tag{4}$$

In contrast, the solubility of calcium sulfate in the diluted brine increases as the dosage of water increased. The curve is linear, indicating a proportional relationship between the solubility and the dosage of water. An equation could be regressed from the graph using Microsoft excel.

$$\rho_{sat} = 1.737 + 0.0382 \times V \tag{5}$$

with the Pearson product moment correlation coefficient $R^2 = 0.9998$.

The supersaturation *S* in the diluted brine is also plotted against the dosage of water added (Fig. 10). It is obvious that the supersaturation of solution decreases sharply with increase in the dosage of water. The supersaturation decreased from 1.14 to 1.07, suggesting 50% elimination of supersaturation, when 2 ml water was added into 100 ml brine. The result indicated that the scale deposited in pipes would decrease more than a half when 2.0 wt% of water was added into the brine before transport. It also can be derived from the result that the

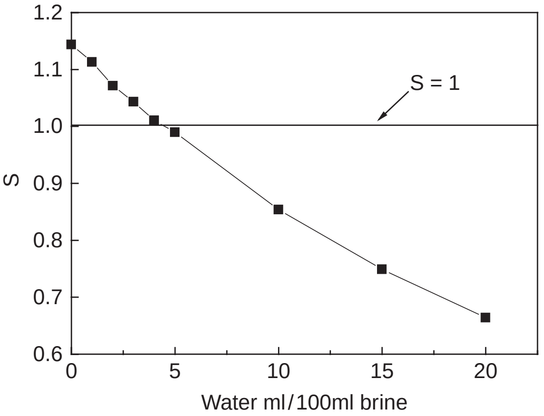

Fig. 10. The supersaturation of calcium sulfate in diluted brine.

depositing speed is proportional to the square of supersaturation.

Substituting Eqs. (4) and (5) into Eq. (6) yields the following relationship between supersaturation and the dosage of water:

$$S = 1.99/(1.737 + 0.05557V + 0.000382V^2) \qquad (6)$$

From Eq. (6), it can be concluded that a complete elimination of supersaturation was obtained when 4.3% of the volume of water was added into the brine.

4. Conclusions

Prevention of gypsum scaling in transportation pipes was studied. The results indicated that:

(1) The solubilities of calcium sulfate dihydrate in given brine (NaCl: 300 g/L, Na_2SO_4: 20 g/L) increases with increasing temperature, and solid phase transforms to calcium sulfate hemihydrate above 70°C.

(2) With the seeded method, it was found that a fast elimination of supersaturation was obtained when temperature increases. The more seed crystal was used, the faster elimination of supersaturation was achieved. After an initial period, the rate constant was proportional to the quantity of seed crystals. The crystal growth of calcium sulfate dihydrate in brine follows a second order reaction equation in supersaturation.

(3) With the dilution method, results showed that a complete elimination of supersaturation was obtained when 4.3% of the volume of water was added. The solubility of calcium sulfate dihydrate in brine was proportional to the percentage of water added in.

Symbols

A — surface area of seed crystals (cm^2)
C — calcium sulfate concentration (mol/L)
C_i — initial calcium sulfate concentration (mol/L)
C_{sat} — calcium sulfate concentration in saturated brine (mol/L)
K — growth rate constant (1/mole cm^2 min)
m — mass of seed crystals in brine (g)
m_0 — initial mass of seed crystals in brine (g)
S — supersaturation
T — time (min)
V — volume of water added into brine (ml)
ρ — calcium sulfate concentration in the diluted brine (g/L)
ρ_i — initial calcium sulfate concentration in brine (g/L)
ρ_{sat} — solubility of calcium sulfate in diluted brine (g/L)

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