



Preparation of calcium sulfate whiskers from FGD gypsum via hydrothermal crystallization in the H_2SO_4 – NaCl – H_2O system



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ABSTRACT

Little attention has thus far been paid to the potential effect of solution composition on the hydrothermal crystallization of calcium sulfate whiskers prepared from flue-gas desulfurization (FGD) gypsum. When purified FGD gypsum was used as raw material, the morphology and phase structure of the hydrothermal products grown in pure water, H_2SO_4 – H_2O , NaCl – H_2O , and H_2SO_4 – NaCl – H_2O solutions as well as the solubility of purified FGD gypsum in these solutions were investigated. The results indicate that calcium sulfate whiskers grow favorably in the H_2SO_4 – NaCl – H_2O system. When prepared using 10–70 g NaCl /kg gypsum –0.01 M H_2SO_4 – H_2O at 130 °C for 60 min, the obtained calcium sulfate whiskers had diameters ranging from 3 to 5 μm and lengths from 200 to 600 μm , and their phase structure was calcium sulfate hemihydrate (HH). Opposing effects of sulfuric acid and sodium chloride on the solubility of the purified FGD gypsum were observed. With the co-presence of sulfuric acid and sodium chloride in the reaction solution, the concentrations of Ca^{2+} and SO_4^{2-} can be kept relatively stable, which implies that the crystallization of the hydrothermal products can be controlled by changing the concentrations of sulfuric acid and sodium chloride.

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1. Introduction

Calcium sulfate dihydrate (DH), calcium sulfate hemihydrate (HH), and calcium sulfate anhydrite (AH) are three phases that are most commonly observed in several industrial and environmental precipitation processes. A calcium sulfate whisker (CSW) is an HH. Recently, much attention has been given to the preparation of CSWs because of their wide range of application in plastics, rubber, and friction materials (Liu et al., 2011; Luo, Li, Xiang, Li, & Ning, 2010; Wang, Pan, Xue, & Cang, 2011; Xu, Li, Luo, & Xiang, 2011; Yuan, Wang, Li, & Han, 2010; Zhu, Xu, & Chen, 2011). Luo et al. (2010) have reported that calcium sulfate whiskers with diameters of 5–20 μm and lengths of 80–500 μm were prepared from $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ plates that were formed via the co-precipitation of CaCl_2 and Na_2CO_3 at room temperature. Yuan et al. (2010) have also prepared such whiskers using hydrothermal methods, mainly using natural gypsum of high purity as raw material.

However, the low crystallization speed for the preparation of CSWs in organic or dilute aqueous media remains an unsolved

problem. Moreover, the morphology of hydrothermal products has been found to be sensitive to parameters such as pH, reactant concentration, the molar ratio of $[\text{Ca}^{2+}]$ to $[\text{SO}_4^{2-}]$, and the presence of certain metal ions or organics (Deng, Yuan, Sun, Shi, & Jiang, 2009; Hamdona & Al Hadad, 2007; Luo et al., 2010; Sargut, Sayan, & Kiran, 2010). Factors that affect the crystal morphology of sparingly soluble salts from hydrothermal reactions are of interest. Many researchers have studied these factors, including temperature, supersaturation, seeding, and organic or inorganic additives (Abdel-Aal, Rashad, & El-Shall, 2004; Deng et al., 2009; Hamdona & Al Hadad, 2007; Sargut et al., 2010). However, there remain many uncertainties regarding some significant effects on the morphology of CSWs, especially when flue-gas desulfurization (FGD) gypsum is used as raw material. Because of the sparing solubility of gypsum, it is not effective to control its crystallization by changing the temperature and supersaturation of the solution. Moreover, compared with other chemical agents, purified FGD gypsum may still contain some impurities, which could have a significant effect on the crystallization of the hydrothermal products. Therefore, it is a challenge to control the crystal morphology of the hydrothermal products when preparing CSWs using FGD gypsum as raw material.

In our previous studies, we have investigated the influences of K_2SO_4 dosage, reaction temperature, and pH on the phase,

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Table 1

Chemical compositions of the purified FGD gypsum.

Composition	CaO	SO ₃	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	MgO	TiO ₂	K ₂ O	Na ₂ O
Content (wt%)	31.46	44.95	0.95	2.16	0.26	<0.01	<0.01	<0.01	<0.01

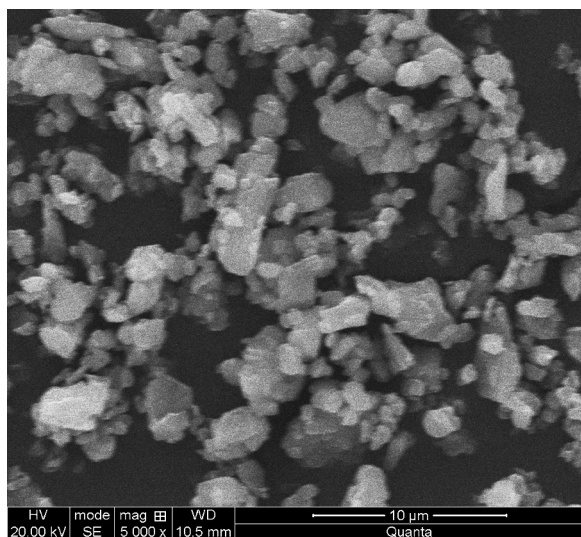
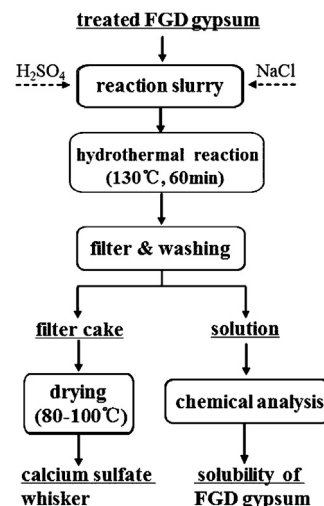
morphology, and quality of CSWs prepared via hydrothermal crystallization using treated FGD gypsum as the raw material (Wang, Yang, Wang, Liu, & Zhu, 2012; Yang, Wang, Zhu, & Du, 2013). However, the effect of K₂SO₄ dosage on the composition of the reaction solution was not addressed, and no investigations of the relation between the composition of the reaction solution and the crystallization of the hydrothermal products were conducted. Furthermore, the length of the prepared CSWs was less than 200 μ m, and the distribution of their diameters was not uniform. To overcome these shortcomings, NaCl was used as a crystal modifier to promote the one-dimensional growth of the products in this study. In addition, sulfuric acid was used to adjust the composition of the reaction slurry. The morphology, structure, and composition of the products formed under various hydrothermal conditions were characterized to identify the phase-conversion processes and the crystal-morphology diversification. In particular, the effect of the reaction-slurry composition on the solubility of CaSO₄·2H₂O was investigated to explore the detailed crystallization processes of the calcium sulfate whiskers.

2. Experimental

2.1. Experimental materials and procedures

Commercial sodium chloride (NaCl, >99% purity) of analytical grade and sulfuric acid (H₂SO₄, density = 1.84 g/cm³) were provided by Luoyang Chemical Reagent Co. Ltd., China. The FGD gypsum used in this study was supplied by a power plant in Henan Province, China. The FGD gypsum was purified following the procedure used in our previous studies, and the CaSO₄·2H₂O content of the purified FGD gypsum was approximately 96% (Wang et al., 2012; Yang et al., 2013). The chemical composition of the purified FGD gypsum is listed in Table 1, and its morphology is shown in Fig. 1.

The purified FGD gypsum, approximately 29.5 g, was first wet-milled at a rate of 300 rpm for 240 min (polyethylene bottle, ZrO₂ medium) in a ball mill (QM-3SP2, Nanjing University Instrument

**Fig. 1.** SEM image of the purified FGD gypsum.**Fig. 2.** Experimental procedure.

Co., China), while maintaining the mass ratio of purified FGD gypsum to distilled water at 1:1. After ball milling, the slurry was transferred into a double-walled jacket autoclave (CJK-1, Weihai Newera Chemical Co. Ltd., China), and the solid–liquid ratio was adjusted to between 1:20 and 1:30 using boiling water. In the meantime, 0.01 M sulfuric acid and specific amounts of sodium chloride (with respect to the amount of purified FGD gypsum, g/kg) were added into the slurry. The slurry was then heated at a rate of 2–3 °C/min and maintained at a constant temperature of 130 °C for 40–90 min. The slurry was stirred at a constant rate of 260 rpm. After reaction, the autoclave was cooled to 100 °C, and the suspension was rapidly filtered. The hydrothermal products were washed with boiling water and then dried to constant weight at 80–100 °C. The filtrate was subjected to chemical analysis. The experimental procedure is illustrated in Fig. 2.

2.2. Characterization of samples

The chemical compositions of the samples were investigated using a wavelength-dispersive X-ray fluorescence spectrometer (XRF, Magix PW2403, PANalytical B.V., Netherlands). Morphological studies of the samples were performed via scanning electron microscopy (SEM, Quanta 450, FEI, Holland) at an operating voltage of 20 kV after the samples were coated with gold, and EDS analysis was performed using an X-ray energy-dispersive spectroscope (X-Max EDS, Oxford, England) equipped with a Quanta 450 SEM. The structures of the samples were examined using an X-ray diffractometer (XRD, X'Pert PRO MPD, PANalytical B.V., Holland) using Cu K α radiation ($\lambda = 1.54178$ Å) at a scanning rate of 0.02°/s for a 2θ range of 5–80°. Thermal analyses of the samples were performed in a corundum crucible using a simultaneous thermal TG/DSC analyzer (NETZSCH STA 409, Selb/Bavaria, Germany) at a heating rate of 10 °C/min and under an N₂ atmosphere with a gas flow rate of 20 mL/min. The soluble Ca²⁺ was analyzed via EDTA titration.

3. Results and discussion

3.1. Effects of solution composition on the morphology of calcium sulfate whiskers

Fig. 3 shows the effect of NaCl concentration on the morphology of CSWs prepared from purified FGD gypsum in the H₂SO₄ (0.01 M)–NaCl–H₂O system. CSWs can be obtained without the

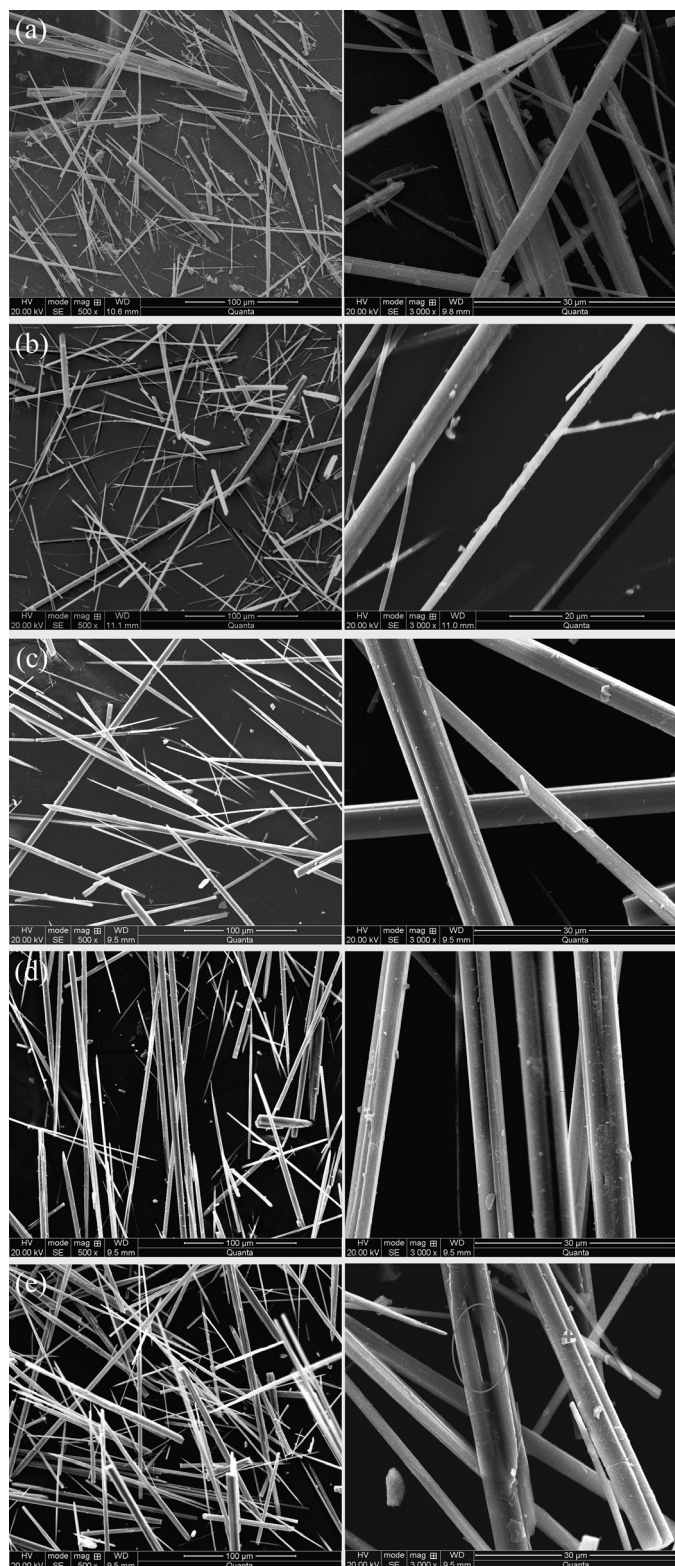


Fig. 3. SEM images of the calcium sulfate whiskers prepared at 0.01 M H_2SO_4 and different concentrations of sodium chloride. (a) 0 g/kg, (b) 10 g/kg, (c) 30 g/kg, (d) 50 g/kg, and (e) 70 g/kg.

Table 2

Results of the EDS analysis on the spot and whisker marked in Fig. 3(e).

Composition		O	S	Ca	Al	Si
Content (wt%)	Spot	60.46	14.84	21.12	1.56	2.02
	Whisker	49.53	21.91	28.56		

addition of NaCl, and some short columnar, fascicular, and gradual products that co-exist with the CSWs can be easily observed in this case. However, the crystal distributions in terms of diameter and length are not uniform, and the crystallization of the CSWs is very poor in the absence of NaCl. As the NaCl concentration was increased, the occurrence of these short columnar, fascicular, and gradual products was gradually reduced. At the NaCl concentration of 30–50 g/kg, the diameters of the CSWs were 3–5 μm , and their mean length markedly increased from 200 to 600 μm . However, with the further increase of the NaCl concentration, the mean length of the CSWs was reduced. This result indicates that the NaCl concentration has a significant effect on the crystal morphologies of the hydrothermal products.

In Fig. 3(a)–(e), a small number of spots on the surfaces of the CSWs are apparent. To determine the influence of these spots on the crystallization of the CSWs, EDS was performed on them and the whiskers, as indicated in Fig. 3(e) (see Fig. 4). In the case of the whiskers, their chemical composition was nearly equivalent to the theoretical composition of hemihydrate gypsum ($\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$), with 27.59 wt% calcium, 22.07 wt% sulfur and 49.66 wt% oxygen (see Table 2), implying that the whiskers are composed of hemihydrate gypsum. As for the spots, however, their sulfur and calcium contents were lower than those of the whiskers, while some aluminum and silicon were detected, which indicates that the spots were most likely composed of HH and a compound of calcium, aluminum, silicon, and oxygen. From Fig. 3(a)–(e), it can be seen that the presence of impurities, such as aluminum and silicon, in the purified FGD gypsum has an insignificant impact on the crystallization of the CSWs. However, they can adsorb on the surface of a CSW and decrease its quality.

As reported in the literature, some metal ions have a strong effect on the crystallization of gypsum (Hamdona & Al Hadad, 2007; Luo et al., 2010; Sargut et al., 2010). For example, the morphology of gypsum crystals has been found to change from a rose-like form to a leaf-like plate form in the presence of Cr^{3+} ions (Sargut et al., 2010). However, it is difficult to identify the role of NaCl in the process of CSW crystallization from Fig. 3 alone because of the presence of sulfuric acid in the reaction slurry. To gain more insight into the effect of NaCl on CSW crystallization, SEM images of hydrothermal products grown in pure water and in the presence of NaCl were acquired. Many short columnar, irregular block, plate, and gradual crystal morphologies can be observed from Fig. 5(a), while needle-like crystallization is very scarce. In the presence of NaCl media, most hydrothermal products are in the form of CSWs, as shown in Fig. 5(b). Compared Fig. 5(b) with the products grown in the presence of H_2SO_4 , shown in Fig. 3(a), the lengths of the CSWs were not markedly different, while the diameters were clearly smaller. However, the CSWs in both systems were shorter and smaller than those grown in the H_2SO_4 –NaCl– H_2O system. These results indicate that the crystallization of hydrothermal products grown in pure water, the NaCl– H_2O system, the H_2SO_4 – H_2O system, and the H_2SO_4 –NaCl– H_2O system are very different.

3.2. Effects of solution composition on the phase of calcium sulfate whiskers

According to Ostwald's rule of stages, the least thermodynamically stable phase usually crystallizes first, followed by conversion

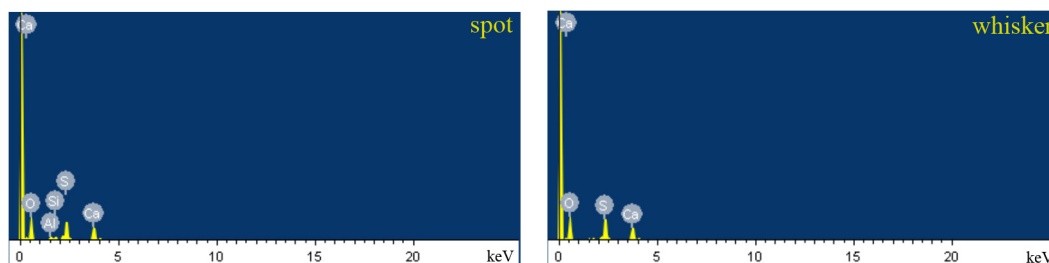


Fig. 4. EDS patterns of the spot and whisker marked in Fig. 3(e).

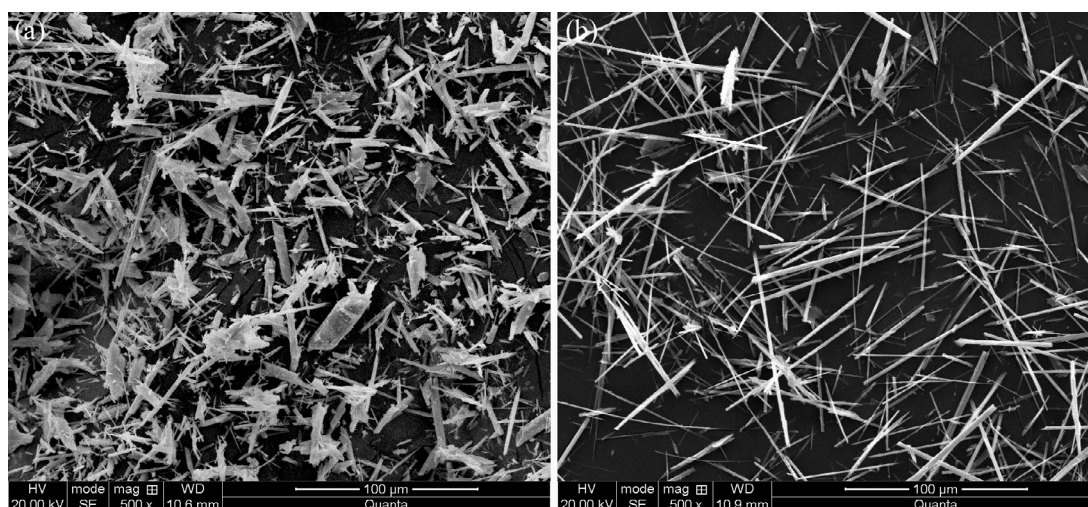


Fig. 5. SEM images of the calcium sulfate whiskers grown in (a) pure water and (b) the presence of 50 g/kg sodium chloride.

to a relatively stable phase (Fu, Guan, Jiang, Yates, & Wu, 2012). However, when multiple phases crystallize simultaneously, the kinetics of competitive nucleation plays a vital role in the formation of stable or metastable phases (Fu et al., 2012; Guan, Yang, & Wu, 2010). The crystallization of gypsum is a typical competitive nucleation process in water, especially in electrolyte solutions. Many studies have indicated that based on a dissolution–recrystallization mechanism, its phase transformation can take place among DH, HH, and AH, as shown in Table 3.

Therefore, it is necessary to investigate the phase structures of hydrothermal products grown under various solution conditions. The XRD patterns presented in Fig. 6 indicate that the phase of purified FGD gypsum is DH. Via hydrothermal reactions, DH

was completely converted into HH. However, in pure water, the characteristic diffraction peaks of HH were relatively weak, which indicates that the crystals had a weak structure and could be broken easily because of the hydrodynamic conditions in the autoclave, as shown in the SEM image in Fig. 5(a). The degrees of crystallinity of the hydrothermal products grown in both NaCl–H₂O and H₂SO₄–H₂O media were clearly heightened. Moreover, in the presence of NaCl and H₂SO₄, the crystals could fully grow into CSWs of excellent structure, as shown in the SEM images in Figs. 3 and 5 and the XRD patterns in Fig. 6.

Simultaneously, it was found that the NaCl concentration has a significant effect on the crystallization of CSWs, as shown in Fig. 6(c) and (e)–(h). When the NaCl concentration was increased, the

Table 3

Review of literatures on equilibrated phase of calcium sulfates under different experimental conditions.

Reference	Starting materials	Experimental conditions	Equilibrated phase
Marshall and Jones (1966)	DH, AH, HH	0–1.0 M H ₂ SO ₄ at 25–60 °C	DH
		0–1.0 M H ₂ SO ₄ at 125 °C	HH
		0–1.2 M H ₂ SO ₄ at 150–350 °C	AH
Dutrizac (2002)	DH	0–0.6 M H ₂ SO ₄ at 25–95 °C	DH
Ling and Demopoulos (2004)	DH	0–0.1 M H ₂ SO ₄ at 100 °C	DH
	DH		DH
Yang et al. (2009)	HH	1.0–18.0 M KCl at 85–100 °C	HH
	AH		AH
Li and Demopoulos (2005)	DH	3.0 M HCl at 80 °C	DH + minor AH
		3.0 M HCl + 2.0 M CaCl ₂ at 80 °C	AH + HH
Guan et al. (2010)	CaCl ₂ + Na ₂ SO ₄	0–0.2 M MgCl ₂ ·6H ₂ O at 90 °C	HH
		At 20–70 °C	DH
Luo et al. (2010)	CaCl ₂ + Na ₂ SO ₄	At 90–110 °C	DH + HH
		At 130–160 °C	HH
		At 170–200 °C	AH

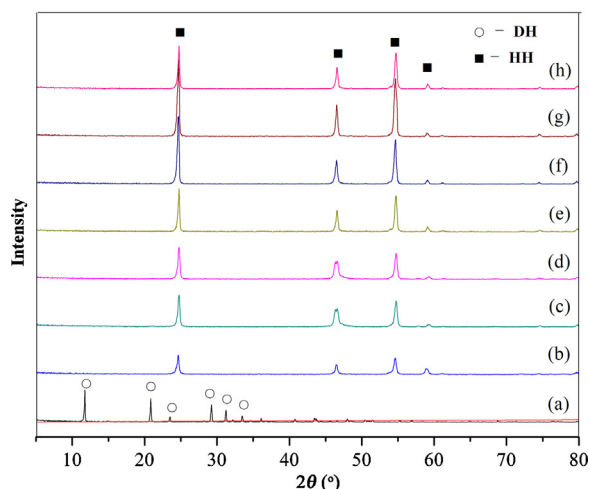


Fig. 6. XRD patterns of purified FGD gypsum (a) and calcium sulfate whiskers grown in (b) pure water, (c) H_2SO_4 (0.01 M)– H_2O , (d) NaCl (50 g/kg)– H_2O , (e) H_2SO_4 (0.01 M)– NaCl (10 g/kg)– H_2O , (f) H_2SO_4 (0.01 M)– NaCl (30 g/kg)– H_2O , (g) H_2SO_4 (0.01 M)– NaCl (50 g/kg)– H_2O , (h) H_2SO_4 (0.01 M)– NaCl (70 g/kg)– H_2O .

crystallization of the CSWs was improved. At the NaCl concentration of 30–50 g/kg, the intensities of the characteristic diffraction peaks of the hydrothermal products were comparatively stronger, which indicates that the crystallization achieved under these conditions was better than that achieved in the other cases. However, with the further increase of the NaCl concentration to 70 g/kg, the crystallization of the CSWs worsened, which was also apparent from the defects in the CSWs, such as the one marked with a red ellipse in Fig. 3(e). Therefore, the solution composition has a strong influence on the crystallization of hydrothermal products.

To gain further insight into the phase transformation and crystallization of the hydrothermal products, TG analysis was performed, and the results are shown in Fig. 7. The mass loss of the hydrothermal products grown in H_2SO_4 (0.01 M)– NaCl (50 g/kg)– H_2O was 6.33 wt% at 200 °C, closely corresponding to the theoretical content of crystal water in HH, which indicates that the hydrothermal products underwent a dehydration reaction, as shown in Eq. (1) (Dantas et al., 2007; Lou, Guan, & Wu, 2011). The result also implies that DH can be completely converted into HH and that the degree of crystallinity of hydrothermal products is good when purified FGD gypsum is used as raw material. In

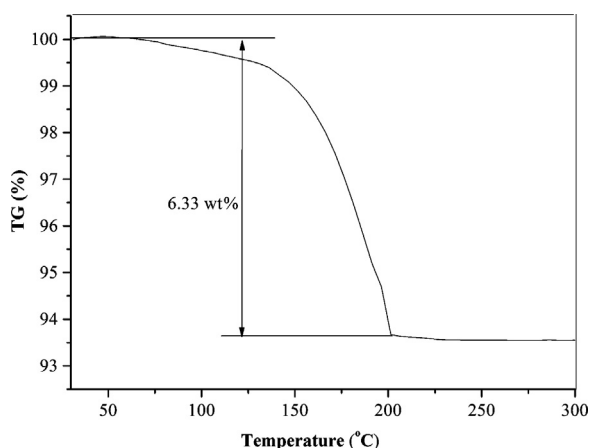
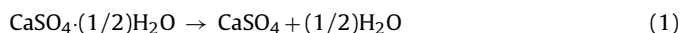


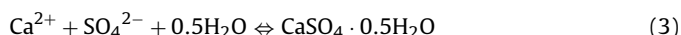
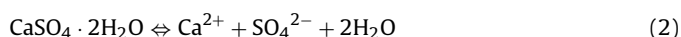
Fig. 7. TG curve of hydrothermal products grown in H_2SO_4 (0.01 M)– NaCl (50 g/kg)– H_2O .

addition, the crystallization of CSWs can be improved in the presence of NaCl and H_2SO_4 .



3.3. Effects of solution composition on the solubility of FGD gypsum

Regarding the crystallization of calcium sulfate whiskers, some reported studies have indicated that the gypsum underwent a process of dissolution–recrystallization, as shown in Eqs. (2) and (3), when CSWs were prepared via a hydrothermal method using natural gypsum or industrial-byproduct gypsum as raw material (Abdel-Aal et al., 2004; Deng et al., 2009; Xu et al., 2011; Yuan et al., 2010).



However, other studies (Jones & Ogden, 2010; Van Driessche et al., 2012) have reported that the crystallization of the gypsum underwent a three-stage process: (i) homogeneous precipitation of nanocrystalline hemihydrate bassanite below its predicted solubility, (ii) self-assembly of bassanite into elongated aggregates co-oriented along their c axes, and (iii) transformation into dihydrate gypsum. In other words, calcium sulfate whiskers could be obtained via the formation of a nanocrystalline hemihydrate phase and the self-assembly thereof. We deduce that for the preparation of CSWs via hydrothermal crystallization, both mechanisms play important roles. Regardless, the dissolution of the purified FGD gypsum in the reaction cannot be ignored. Hence, the influence of solution composition on the solubility of FGD gypsum should be investigated, thereby exploring the details of the crystallization processes of calcium sulfate whiskers.

When sulfuric acid is present, the sulfuric acid will dissociate, as in Eqs. (4) and (5), which will affect the reactions shown in Eqs. (2) and (3), and consequently the solubility of FGD gypsum. Moreover, a change in solution composition has a significant effect on the morphology of crystals produced in that solution (Guan et al., 2010; Hamdona & Al Hadad, 2007; Sargut et al., 2010). Therefore, the influence of solution composition on the solubility of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ should, in turn, affect the detailed crystallization processes of the hydrothermal products.



Fig. 8 shows the effect of H_2SO_4 concentration on the solubility of FGD gypsum at various temperatures. The solubility of FGD gypsum increases with increasing H_2SO_4 concentration in the range of 0–1.0 M. The data generated in this study are comparable to those available in the literature (Cameron & Breazeale, 1903; Farrah, Lawrance, & Wanless, 2007). However, in this study, the hydrothermal products were prepared at a low H_2SO_4 concentration of ≤ 0.01 M. Hence, for the purposes of this study, the investigation of the effect of a low H_2SO_4 concentration on the solubility of FGD gypsum is more important than that of a relatively high concentration.

Fig. 9 shows the solubility of FGD gypsum at lower H_2SO_4 concentrations, in the range of 0–0.1 M. The tendency is similar to that seen in Fig. 8 for 50–100 °C, which is consistent with the tendency presented in Farrah et al. (2007). For sulfuric acid solutions, the first dissociation (Eq. (4)) is considered to be complete, and the second dissociation (Eq. (5)) is partial (Guan et al., 2011; Prisciandaro, Lancia, & Musmarra, 2001). At lower temperatures and lower concentrations of H_2SO_4 , the second dissociation would

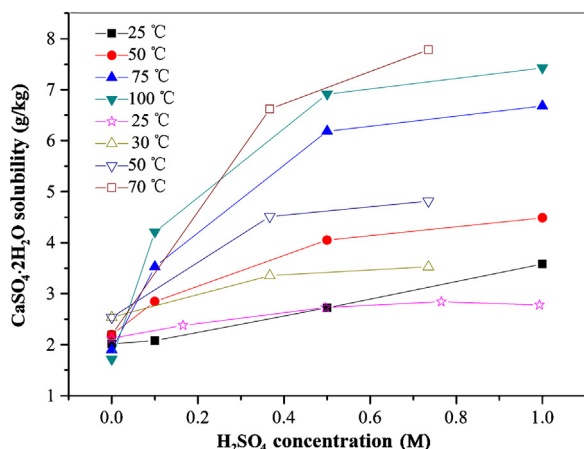


Fig. 8. Variations of the solubility of FGD gypsum with H_2SO_4 concentration at various temperatures: ■, ●, ▲, ▼, this work; ☆, Cameron and Breazeale (1903); △, ▽, □, Farrah et al. (2007).

be more complete, leading to a decrease in the solubility of FGD gypsum because of the increase in SO_4^{2-} in the solution. When the concentration of H_2SO_4 surpasses 0.07 M, the solubility begins to increase at 25 °C. With further increases in temperature and sulfuric-acid concentration, the solubility of FGD gypsum markedly increases. Previous studies have shown that the second dissociation of sulfuric acid sharply decreases with increasing temperature, reducing the SO_4^{2-} concentration and resulting in an increase in the solubility of FGD gypsum (Ling & Demopoulos, 2004; Luo et al., 2010; Marshall & Jones, 1966).

Table 4 presents the solubility of FGD gypsum under various solution conditions. It can be seen that the solubility decreases with increasing sodium-chloride concentration below 50 g/kg, and above 50 g/kg, the solubility increases with increasing sodium-chloride concentration. In the presence of strong electrolytes in the gypsum solution, the ionic strength increases with increasing electrolyte concentration; on the other hand, the presence of strong electrolytes promotes the desolvation of Ca^{2+} (Guan et al., 2010). Therefore, at a lower sodium-chloride concentration of 50 g/kg, Na^+ could compete for partial hydration water associated with Ca^{2+} due to the salt-out effect, which causes a reduction in the solubility of FGD gypsum. The opposite effect on the solubility of FGD gypsum can be seen from Table 4 for further increases in the sodium-chloride concentration, as the activity of water and the ionic activity

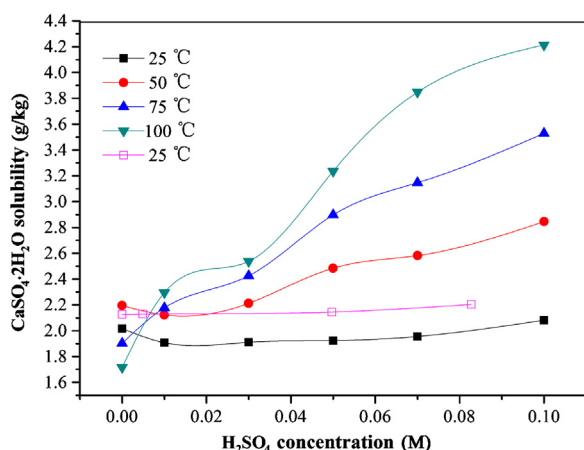


Fig. 9. Variations of the solubility of FGD gypsum with H_2SO_4 concentration at various temperatures: ■, ●, ▲, ▼, this work; □, Cameron and Breazeale (1903).

Table 4

Solubility of the purified FGD gypsum under different solution conditions.

Solution conditions		Temperature (°C)			
NaCl (g/kg)	H_2SO_4 (M)	25	50	75	100
0	0	2.017	2.195	1.902	1.716
10	0	2.006	2.038	1.853	1.701
30	0	1.982	1.996	1.774	1.639
50	0	1.965	1.981	1.667	1.592
70	0	1.991	2.016	1.781	1.744
0	0.01	1.907	2.122	2.178	2.395
10	0.01	1.886	2.068	2.103	2.184
30	0.01	1.757	1.977	2.002	2.118
50	0.01	1.642	1.716	1.824	1.956
70	0.01	1.796	1.985	2.113	2.201

coefficients decrease because of the higher ionic strength (Cameron & Breazeale, 1903; Guan et al., 2010; Prisciandaro et al., 2001). That is to say, at a sodium-chloride concentration in the range of 0–50 g/kg, the effect of salt on the solubility of FGD gypsum is much stronger than that of the ionic strength, while for concentrations exceeding 50 g/kg, the opposite effect can be observed.

As shown in Figs. 8 and 9 and Table 4, sulfuric acid and sodium chloride play opposing roles in their effects on the solubility of FGD gypsum. Because of the co-presence of sulfuric acid and sodium chloride in the hydrothermal reactions investigated in this study, it is necessary to further investigate the solubility under these conditions. Our experimental results indicate that the solubility of FGD gypsum in H_2SO_4 – NaCl – H_2O is between that in H_2SO_4 – H_2O and that in NaCl – H_2O , and its tendency is similar to that in NaCl – H_2O . Because of the opposing roles of sulfuric acid and sodium chloride, the levels of Ca^{2+} and SO_4^{2-} in the reaction solution can be kept relatively stable, which contributes to the improved crystallization of the hydrothermal products. Furthermore, the crystallization of gypsum is a competitive nucleation process in electrolyte solutions, and the presence of sodium chloride will contribute to the desolvation of Ca^{2+} and facilitate the formation of calcium sulfate whiskers (Guan et al., 2010). This has been demonstrated by the experimental results obtained in this study.

Based on the results of this study and those in the literature, it is clear that changes in solution composition will result in variations in the equilibrium concentration of Ca^{2+} and SO_4^{2-} . Therefore, the crystal morphology of CSWs can be manipulated by controlling the solution composition (Guan et al., 2010, 2011; Yang, Guan, Wu, & Wu, 2009).

4. Conclusion

The solution composition has a significant effect on the morphology and phase structure of calcium sulfate whiskers prepared from purified FGD gypsum via hydrothermal crystallization. When prepared using 10–70 g/kg NaCl –0.01 M H_2SO_4 – H_2O at 130 °C for 60 min, calcium sulfate whiskers with diameters of 3–5 μm and lengths of 200–600 μm were obtained, and their phase structure was calcium sulfate hemihydrate. Compared with pure water and H_2SO_4 – H_2O and NaCl – H_2O solutions, the H_2SO_4 – NaCl – H_2O solution is more favorable for the growth of calcium sulfate whiskers because the concentrations of Ca^{2+} and SO_4^{2-} can be kept relatively stable. Gypsum crystals with the desired morphology can be prepared by controlling the solution composition.

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