

# The Activation Effect of K<sub>2</sub>SO<sub>4</sub> on the Hydration of Gypsum Anhydrite, CaSO<sub>4</sub>(II)

N. B. Singh<sup>†</sup>

Department of Chemistry, DDU Gorakhpur University, Gorakhpur-273 009, India

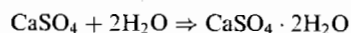
The effect of K<sub>2</sub>SO<sub>4</sub> activator on the hydration of chemical anhydrite obtained from burned FGD-gypsum has been studied by different experimental techniques. Results obtained show that the degree of hydration increases when the K<sub>2</sub>SO<sub>4</sub> concentrations increase from 0.5 to 3.3 wt%. Their heat evolution rate and maximum value also increase with the increase of K<sub>2</sub>SO<sub>4</sub> concentration. The highest values were obtained when the hydration degree was about 50%. Also a correlation between the hydration degree and the total heat evolved was obtained. The X-ray and SEM/EDX studies have shown that K<sub>2</sub>SO<sub>4</sub> is adsorbed at the surface of CaSO<sub>4</sub> even within 5 min of hydration and a syngenite—a double salt K<sub>2</sub>SO<sub>4</sub> · CaSO<sub>4</sub> · H<sub>2</sub>O is formed (even in the presence of 1.0 wt% K<sub>2</sub>SO<sub>4</sub>). Also, important changes in the morphology of the dihydrate crystal are detected. Finally, in the presence of 1.0 wt% K<sub>2</sub>SO<sub>4</sub> (water/anhydrite (*W/A*) ratio = 0.33), it was found that the resonance frequency, modulus of elasticity, compressive strength, and tensile strength increase with the degree of hydration whereas the total porosity decreases.

## I. Introduction

CONTROL of SO<sub>2</sub> emissions from power plants is often achieved by lime/lime stone scrubbers and produce a large amount of gypsum. This gypsum presents either a major disposal problem or constitutes a valuable resource depending on the technologies used. The importance of gypsum as an industrially usable product depends on its ability to transfer itself into various modifications when being heated. These modifications depend on the temperature, which causes partial or complete dehydration. This process is reversed to form the original gypsum (dihydrate) when water is added. These procedures, which are characterized by dehydration or calcination and by rehydration or bonding, are the basis of gypsum technologies.

In recent years, anhydrite of gypsum (CaSO<sub>4</sub>(II)) has gained considerable importance. It can be used for immediate and early bearing mining mortars, normal and self-levelling floor screeds, and double floor slabs.<sup>1</sup>

When water is added to anhydrite, the following reaction takes place:



This reaction occurs very slowly with a very low strength development. As such it is not of any practical utility. However, when certain activators are added to the hydrating anhydrite, the hydration and strength developments are enhanced considerably. A large number of chemical activators, mainly metal sulfates, have been used to accelerate the setting and increase the strength. The mechanism of action of activators on the hydra-

tion of anhydrite has been discussed by different researchers in different ways. Ottemann<sup>2</sup> and others<sup>3,4</sup> have proposed that the activators enhance the solubility of CaSO<sub>4</sub> by forming double salts, which in turn accelerate the formation of dihydrate crystals. Murat *et al.*<sup>5</sup> and El Hajjoui and Murat<sup>6</sup> postulated that hydration of anhydrite involves a dissolution–nucleation–growth process and the chemical activators increase the hydration rate by modifying essentially the nucleation step.

Ludwig *et al.*<sup>1</sup> systematically investigated the hydration of anhydrites and proposed that it is possible to have higher strengths by optimizing the anhydrite/activator ratio. According to them, the most effective activator is a combination of 1.8 wt% Ca(OH)<sub>2</sub> and 1.1 wt% K<sub>2</sub>SO<sub>4</sub>.

From the survey of the literature, it is quite clear that the mechanism of action of activators during the hydration of anhydrite is not fully understood and requires detailed investigation. The aim of the present investigation is to study the effect of different concentrations of K<sub>2</sub>SO<sub>4</sub> on the hydration of anhydrite of gypsum and to understand the mechanism.

## II. Experimental Procedure

### (1) Materials

Anhydrite CaSO<sub>4</sub>(II) obtained from burned FGD-gypsum was used for the study of hydration. The chemical composition determined by an X-ray fluorescence analyzer and atomic absorption spectroscopy is given in Table I.

The density of the anhydrite determined by helium pycnometer is 2.95 g/cm<sup>3</sup> whereas the surface area determined by the air permeability method (Blaine) is 4.66 m<sup>2</sup>/g. The particle size was determined by sieving/laser granulometry and the particle size distributions are given in Table II.

### (2) Activator

The activator used is K<sub>2</sub>SO<sub>4</sub>. The concentration of the activator has always been expressed with reference to anhydrite. The concentrations of K<sub>2</sub>SO<sub>4</sub> used are 0.5, 1.0, 2.0, and 3.3 wt%.

### (3) Methods

The conductivities in the presence of K<sub>2</sub>SO<sub>4</sub> were determined at 20°C at different water/anhydrite (*W/A*) ratios (*W/A* = 1, 10, and 100) up to 24 h (in some cases upto 72 h) with the help of a microprocessor conductivity meter LF 537. Conductivities of the paste and the corresponding filtrates were also determined after 30 min of hydration.

Hydrations were carried out in polythene bags in the presence of activator at a *W/A* ratio equal to 1.0 at room temperature. The hydrations from 1 h to 15 days were stopped with isopropyl alcohol, dried at 40°C, and stored in polythene bags. The degrees of hydrations were determined by measuring the weight loss in the hydrated samples at 340°–350°C with the help of Ultra-X. The degrees of hydrations were also determined by measuring the density of the hydrated samples with a helium pycnometer.

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<sup>†</sup>Author to whom correspondence should be addressed. e-mail: dr\_n\_b\_singh@rediffmail.com

Table I. Chemical Composition of the Anhydrite

Component	CaO	SO <sub>3</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	Mn <sub>2</sub> O <sub>3</sub>	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	Cl	Total
Composition (wt%)	41.2	56.8	0.81	0.45	0.11	0.01	0.02	0.034	0.01	0.02	0.003	99.773%

CO<sub>2</sub> = 0.93%, weight loss at 500°C = 0.23%.

Table II. Particle Size Distribution in the Anhydrite

Particle size (μm)	> 90	90-63	63-40	40-32	32-25	25-10	10-5	< 5
Fraction (wt%)	0.1	1.42	24.34	14.76	15.92	30.05	6.80	6.71

During hydration, the liquid phase was filtered;  $K^+$  and  $Ca^{2+}$  ions were estimated with the help of an atomic absorption spectrometer.

The heat of hydration of the anhydrite was measured with the help of a conduction calorimeter.<sup>7</sup> Two grams of anhydrite was weighed in a cylindrical cell. The same amount of finely ground quartz was weighed in another cylindrical cell. The two cells were then separately fitted with pippets attached to a syringe containing 2 g of activator solutions in order to obtain a  $W/A$  ratio of 1.0. The two cells were then placed in the calorimeter set at 20°C. On thermal equilibration, the activator solutions were injected into the cells and the rate of heat evolution and total heat evolved were recorded with a computer.

X-ray diffraction patterns of anhydrite hydrated for 5 min and scanning electron microscopic pictures of anhydrite hydrated for 10 days were recorded. Microanalysis of the ions at the surface of anhydrite hydrated for 5 min in the presence of activator was also carried out by the EDX technique.

In order to perform the physical testing, the anhydrite was mixed with water ( $W/A = 0.33$ ) in presence of 1.0 wt%  $K_2SO_4$ . Mixing and compaction were carried out with a mechanical working standard apparatus for the testing of cements. The flow of the paste was measured with a flow table and was found to be 20 cm.

Prisms of dimension 4 cm × 4 cm × 16 cm were made with stainless-steel gauge points inserted on the sides. The prisms were taken out from the mold after 1 day and then kept at relative humidities (RHs) of 95% and 100% (under water). Weight, length, resonance frequency, tensile, and compressive strengths of undried prisms were measured at 1, 2, 5, and 10 days of hydration. Modulus of elasticity was calculated from resonance frequencies. Each value is the average of two measurements, whereas the compressive strength values are the average of four measurements. The densities and the weight losses at 340°–350°C were also determined for the broken pieces. Total porosities of the specimens were calculated from their bulk density and the density of the hydrated materials, both in the dried state.

### III. Results and Discussion

#### (1) Electrical Conductivities

In order to understand the role of  $K_2SO_4$  during the hydration of anhydrite, the electrical conductivities of the paste and suspensions were measured. When the  $W/A$  ratio was 1.0, the conductivity of the paste in the absence of any activator (Fig. 1) increased with time up to a certain time interval and then became constant. These changes in conductivity are due to dissolution of  $CaSO_4$  in water and saturation of the solution with respect to  $Ca^{2+}$  and  $SO_4^{2-}$  ions. The conductivities of the paste in the presence of activator decreased immediately (Fig. 1) as soon as the anhydrite was added to the activator solution and after that became practically constant till the time of measurements. This showed that probably the activator is adsorbed on the surface of anhydrite as soon as they come in contact with each other.

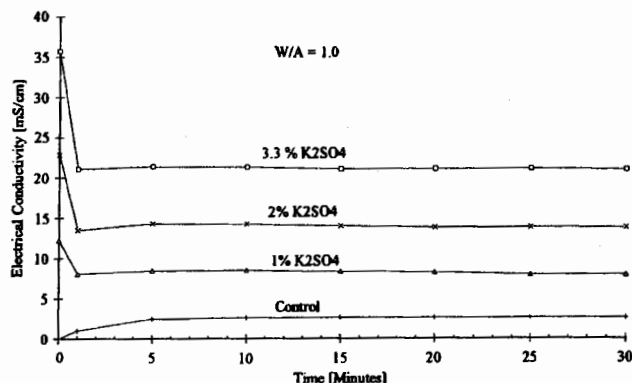


Fig. 1. Effect of  $K_2SO_4$  on the electrical conductivities of hydrating anhydrite at different intervals of time.

The conductivities of the paste ( $W/A = 1.0$ ) in the presence of different concentrations of  $K_2SO_4$  after 30 min of hydration and those of the filtrates of the corresponding pastes (after 30 min) were also measured (Fig. 2). The conductivities increase with  $K_2SO_4$  but the values are lower in the paste as compared with those of the corresponding filtrates. The lower conductivities of the paste indicate that the anhydrite paste is so thick that it hinders the easy contact of the liquid phase with the electrode. The conductivity of the filtrate in the presence of 1.0 wt%  $K_2SO_4$  is more than that of the corresponding  $K_2SO_4$  solution, but at 2.0 and 3.3 wt%  $K_2SO_4$ , the conductivities of the solutions are almost same as that of the corresponding filtrates; in all the cases, the values are lower than the sum of the conductivities of  $K_2SO_4$  solutions and that of the filtrate from the control. However, this difference is higher at higher concentrations. These results show that in the presence of  $K_2SO_4$ , the effective number of conducting ions is decreased and this decrease is

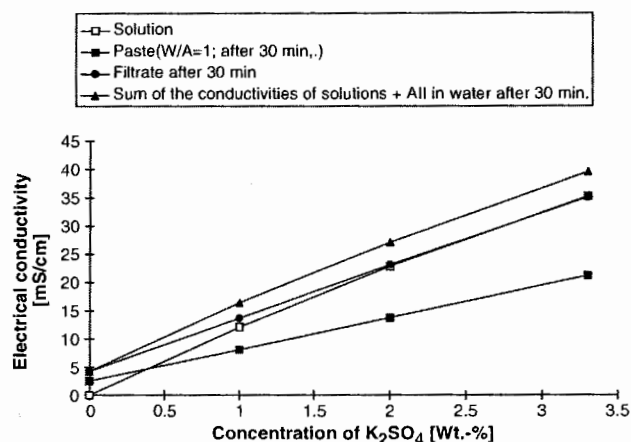


Fig. 2. Effect of different concentration of  $K_2SO_4$  on the conductivities of the paste and the filtrates after 30 min of hydration of anhydrite.

**Table III.** Concentration of  $K^+$  and  $Ca^{++}$  Ions in the Liquid Phase and Amount of Double Salt Formed in the Solid Phase  $W/A = 1.0$ 

Time of hydration (min)	Concentrations in presence of $K_2SO_4$						
	Control $Ca^{++}$ (g/L)	1.0 wt% $K_2SO_4$			3.3 wt% $K_2SO_4$		
		$Ca^{++}$ (g/L)	$K^+$ (g/L)	Doublesalt (wt%)	$Ca^{++}$ (g/L)	$K^+$ (g/L)	Doublesalt (wt%)
—	—	—	4.46	—	—	14.45	—
2	0.71	0.40	3.97	0.21	0.46	8.93	2.32
5	0.73	0.43	3.86	0.25	0.46	11.10	1.41
20	0.88	0.42	3.61	0.36	0.47	10.35	1.72
60	0.75	0.47	3.65	0.34	0.44	11.88	1.06

more when the concentration of  $K_2SO_4$  is higher. This may be due to adsorption of  $K_2SO_4$  on the surface of anhydrite and consequently the formation and precipitation of double salt— $K_2Ca(SO_4)_2 \cdot H_2O$  also known as syngenite.

Since at  $W/A = 1.0$  the paste becomes very thick and hinders the easy contact of the liquid phase with the electrode, the conductivities were measured at a higher  $W/A$  ratios ( $W/A = 10$  and  $100$ ). In all the cases, first of all the conductivities increased slightly and then became constant. The slight increase in conductivity at a higher  $W/A$  ratio may be due to partial dissolution of double salt. The conductivities of the suspension in the presence of 3.3 wt%  $K_2SO_4$  at  $W/A = 10$  were higher than those at  $W/A = 100$  because the concentration of  $K_2SO_4$  in the suspension with  $W/A = 10$  was 10 times higher than that of the suspension with  $W/A = 100$ . In both the cases, the conductivities of the suspension were lower than the sum of the conductivities of  $K_2SO_4$  solution alone and that of the corresponding control suspension. This shows that when anhydrite is added to  $K_2SO_4$  solution, the number of conducting ions decreases. This may be due to precipitation of double salt.

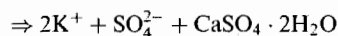
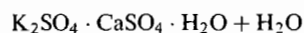
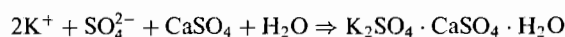
In order to have some idea about the formation of double salt,  $K^+$  and  $Ca^{2+}$  ions were estimated in the liquid phase in the presence of 1.0 and 3.3 wt%  $K_2SO_4$  (Table III). From the table, it is seen that in the presence of  $K_2SO_4$ , the concentration of  $Ca^{2+}$  ions is decreased and the concentration of  $K^+$  ions is also lower than the  $K^+$  ions in the corresponding  $K_2SO_4$  solutions. The decrease in concentrations of  $K^+$  and  $Ca^{2+}$  ions may be due to precipitation of double salt and gypsum. From the decrease in  $K^+$  ion concentrations, the amounts of double salt have been calculated and are given in Table III. The amounts of double salt formed are lower in the presence of 1.0 wt%  $K_2SO_4$  as compared with 3.3 wt%  $K_2SO_4$ .

## (2) Hydration Degree

The degrees of hydrations in the presence of activator at different times of hydration were determined by weight loss and density methods. The values determined by both the methods were found to be almost identical. The degree of hydrations determined by weight loss method in the presence of  $K_2SO_4$  is plotted against time of hydration (Fig. 3). The figure shows that in the absence of any activator, the degree of hydration is very low and

increases continuously with time. With the increase of  $K_2SO_4$  concentration (0.5 to 3.3 wt%), the degree of hydration increases considerably and reaches a maximum value within 5 days, irrespective of the concentrations used. After this, it became practically constant up to 15 days (the time of investigation). It appears that when the degree of hydration reaches a certain maximum value, the dihydrate structure becomes very dense. This hinders the hydration of the remaining anhydrite and further hydration becomes negligible.

From Fig. 3, it is quite clear that in the presence of 2.0 wt%  $K_2SO_4$ , nearly 50% hydration takes place within 16 h whereas in the presence of 3.3 wt%  $K_2SO_4$ , 50% hydration occurs within 8 h. Thus, one can infer that a higher concentration of  $K_2SO_4$  ( $>1.0$  wt%) is more effective within one day of hydration. When the  $W/A$  ratio is changed from 1.0 to 0.33, the effectiveness of 3.3 wt%  $K_2SO_4$  ( $W/A = 1.0$ ) becomes almost equal to that of 1.0 wt%  $K_2SO_4$  ( $W/A = 0.33$ ) within 2 days of hydration. In both cases, the concentration of  $K_2SO_4$  in the liquid phase is nearly the same. It appears that higher concentrations of  $K_2SO_4$  in the liquid phase interact with  $CaSO_4$  rapidly with the formation of double salt, which precipitates and then decomposes to dihydrate. The formation and decomposition of double salt can be represented as



The compound  $K_2SO_4 \cdot CaSO_4 \cdot H_2O$  in lower concentrations will be soluble and in contact with a saturated solution of  $CaSO_4$  may be thermodynamically unstable and as a result decompose to dihydrate—a stable phase. The formation and decomposition of double salt continue till a major portion of the anhydrite is converted to dihydrate. However, when the amount of double salt formed is more than a certain optimum value, it will precipitate and when not in contact with the water solution of  $CaSO_4$ , it may be stable.

## (3) Calorimetric Studies

The rate of heat evolution and the total heat evolved during the hydration in the presence of different concentrations of  $K_2SO_4$  are shown in Fig. 4. In the absence of  $K_2SO_4$ , as soon as anhydrite comes in contact with water, rapid heat evolution takes place and reaches its maximum value within 2 min (Fig. 4a), then decreases, and ultimately after 1 h it becomes practically zero. This increase in heat evolution is due to exothermic dissolution of  $CaSO_4$  in water. Since the rate and degree of hydration of anhydrite are very low, the rate of heat evolution and total heat evolved during hydration are also very low. In the presence of different concentrations of  $K_2SO_4$ , again there is an early high rate of heat evolution with a peak at around 2 min but the values are lower as compared with that of the control and lowest for 1.0 wt%  $K_2SO_4$ . After this first peak, again there is a slight increase with a broad peak and then the rate of heat evolution decreases (Fig. 4a). It appears that in the presence of

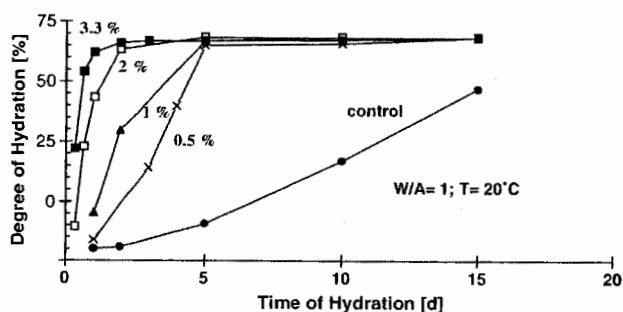


Fig. 3. Effect of different concentrations of  $K_2SO_4$  on the degree of hydration of anhydrite at different intervals of time (weight loss method).

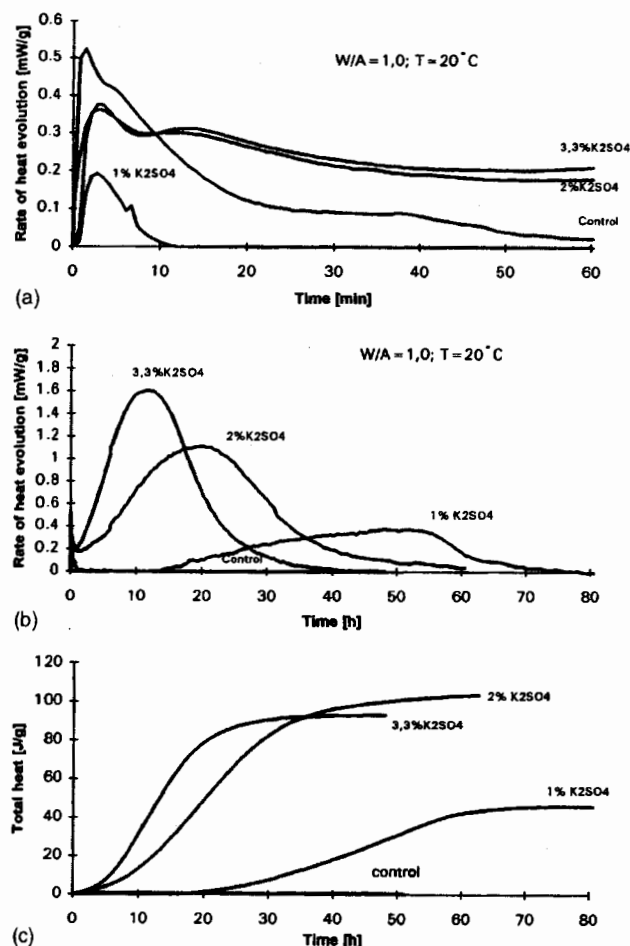


Fig. 4. Effect of different concentrations of  $K_2SO_4$  on the: (a) and (b) rate of heat evolution, and (c) total heat evolved during the hydration of anhydrite at different intervals of time.

$K_2SO_4$ , various processes such as dissolution of  $CaSO_4$ , adsorption of  $K^+$  and  $SO_4^{2-}$  ions on the surface of  $CaSO_4$ , formation, precipitation, dissolution, and decomposition of double salt may take place. The rate of heat evolution may correspond to the average value of heat evolved during these processes. In the presence of higher concentration of  $K_2SO_4$ , the processes might be taking place with a faster rate and as such more rate of heat

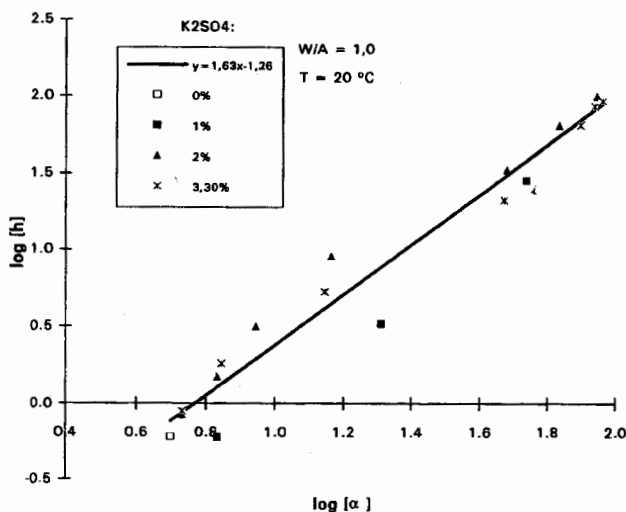


Fig. 5. Correlation between the degree of hydration and total heat evolved during the hydration of anhydrite in the presence of  $K_2SO_4$ .

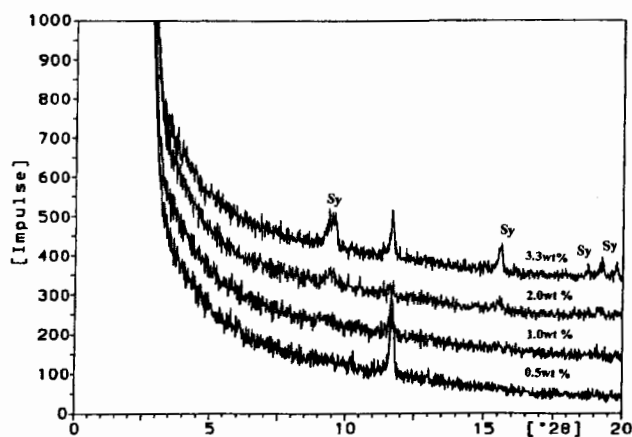


Fig. 6. X-ray diffraction pattern of  $CaSO_4(II)$  hydrated for 5 min in the presence of  $K_2SO_4$ .

evolution. After the first peak, the rate of heat evolution became very low and was almost constant for a certain period of hydration called "pre-induction time." When the  $K_2SO_4$  concentration increases, the pre-induction time decreases and the value of heat evolution rate increases (Fig. 4b). After this second peak the rate of heat evolution decreased continuously and ultimately to a negligible value. The time for the maximum rate of heat evolution (peaks) in the presence of different concentrations of  $K_2SO_4$  followed the sequence:

$$3.3 \text{ wt\%} < 2.0 \text{ wt\%} < 1.0 \text{ wt\%}$$

It appears that the higher the rate of hydration and crystallization of dihydrate, the higher the rate of heat evolution. If we compare the rate of heat evolution (Fig. 4) with the degree of hydration, it is observed that the maximum rate of heat evolution (peak) is achieved when the degree of hydration is about 50%. After this value, the rate of hydration becomes slow, so the rate of heat evolution also decreases. When  $\log(h)$  is plotted against  $\log(\alpha)$ , a straight line (Fig. 5) showing the validity of Eq. (1) is obtained.

$$h = a\alpha^n \quad (1)$$

where  $h$  is the total heat evolved in J/g and  $\alpha$  is the degree of hydration in percent, and  $a$  and  $n$  are constants. From the graph, the values of  $a$  and  $n$  were found to be  $5.49 \times 10^{-2}$  J/g and 1.63, respectively. Thus, if one knows the degree of hydration, one can find out the heat evolved in the presence of  $K_2SO_4$ , irrespective of its concentration.

#### (4) Mineralogical and Microstructural Characterization

In order to confirm the adsorption of  $K_2SO_4$  on the surface of  $CaSO_4$  and the formation of double salt, the hydrations in the presence of 0.5, 1.0, 2.0, and 3.3 wt%  $K_2SO_4$  with  $W/A = 1.0$  were stopped after 5 min of hydration and X-ray diffraction patterns were recorded (Fig. 6). It is observed that syngenite ( $K_2SO_4 \cdot CaSO_4 \cdot H_2O$ ) is formed even within 5 min of hydration except in the presence of 0.5 wt%  $K_2SO_4$ . The amount of syngenite formed increased with the increase in  $K_2SO_4$  concentration. It is possible that in the presence of 0.5 wt%  $K_2SO_4$  very little syngenite is formed, which dissolved in solution or was below the detectable limit.

Further, the adsorption of  $K_2SO_4$  on the surface of anhydrite is confirmed by the EDX technique. At the surface of  $CaSO_4$  hydrated for 5 min in the presence of 1.0 wt%  $K_2SO_4$  (Fig. 7),  $K^+$ ,  $Ca^{2+}$ , and  $SO_4^{2-}$  ions were detected.

SEM photographs of anhydrite hydrated for 10 days ( $W/A = 0.33$ ) are shown in Fig. 8. In the absence of any activator (Fig. 8a), the etching of anhydrite crystal surfaces has occurred and small grains of dihydrate with no intergrowth between the

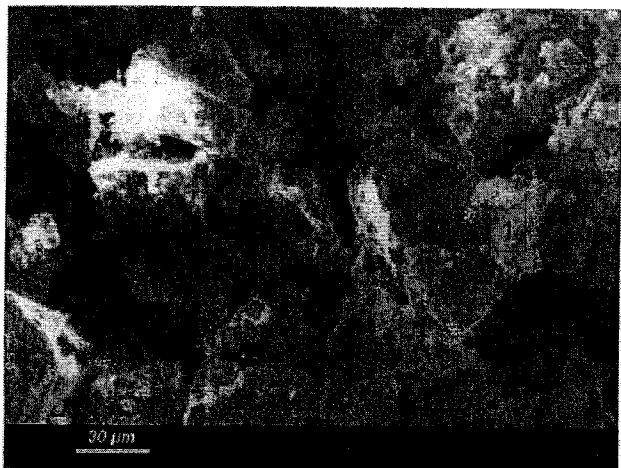


Fig. 7. EDX of  $\text{CaSO}_4(\text{II})$  hydrated for 5 min in the presence of 1.0 wt%  $\text{K}_2\text{SO}_4$ . At point 1, the Ca/S and K/S ratios are:

	Atom ratio	Wt ratio	Count ratio
Ca/S	0.897	1.12	0.713
K/S	0.178	0.217	0.163

individual grains can be seen. In the presence of 1.0 wt%  $\text{K}_2\text{SO}_4$ , well-shaped, dense plate-like compact dihydrate crystals with sharp edges are formed (Fig. 8b). There is an intergrowth between the individual crystals. It appears that the effectiveness of  $\text{K}_2\text{SO}_4$  as an activator is associated with its role in modifying the dihydrate crystal morphology during the hydration of anhydrite.

##### (5) Physical Test

The changes in resonance frequency, modulus of elasticity, length, compressive strength, and tensile strength with hydration time in the presence of 1.0 wt%  $\text{K}_2\text{SO}_4$  ( $W/A = 0.33$ ) at a RH of 95% and 100% (under water) are shown in Fig. 9. At 95% RH, all the values except length change increased up to 5 days and then became constant. However, at 100% RH all the values are lower than those corresponding to a RH of 95%. However, the length change increased with the hydration time at a RH of 100%. It appears that the decrease in the values with time (RH—100%) is due to an increase in length. Also, it is



(a)



(b)

Fig. 8. SEM photographs of anhydrite hydrated for 10 days ( $W/A = 0.33$ ) (a) control (b) in the presence of 1.0 wt%  $\text{K}_2\text{SO}_4$ .

possible that water enters the compact crystal layers of dihydrate crystals, causing a decrease in the above values.

The variations of resonance frequency, modulus of elasticity, compressive strength, tensile strength, and percent total porosity with degree of hydration are shown in Fig. 10. Total porosity decreased whereas other properties increased with an increase in

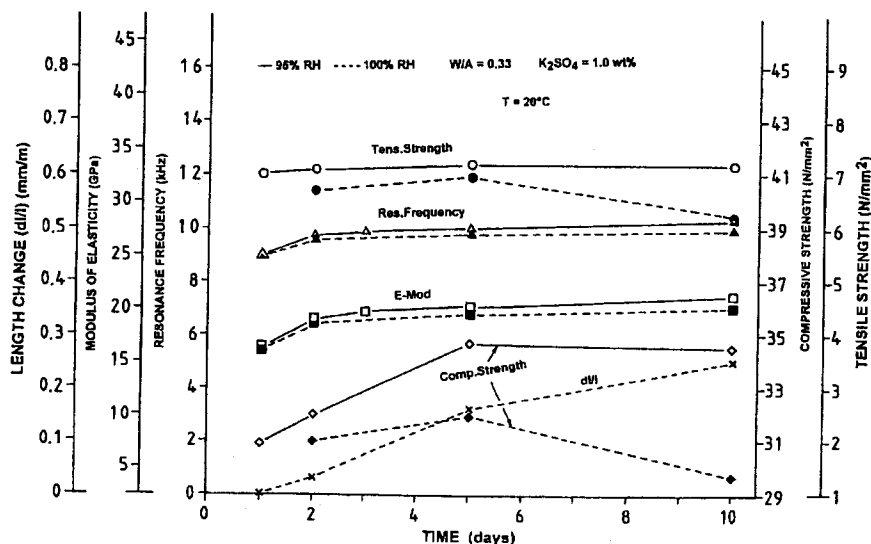


Fig. 9. Changes in resonance frequency, modulus of elasticity, length, compressive strength, and tensile strength of hydrating anhydrite with time in the presence of 1.0 wt%  $\text{K}_2\text{SO}_4$  (prisms—4 cm  $\times$  4 cm  $\times$  16 cm).

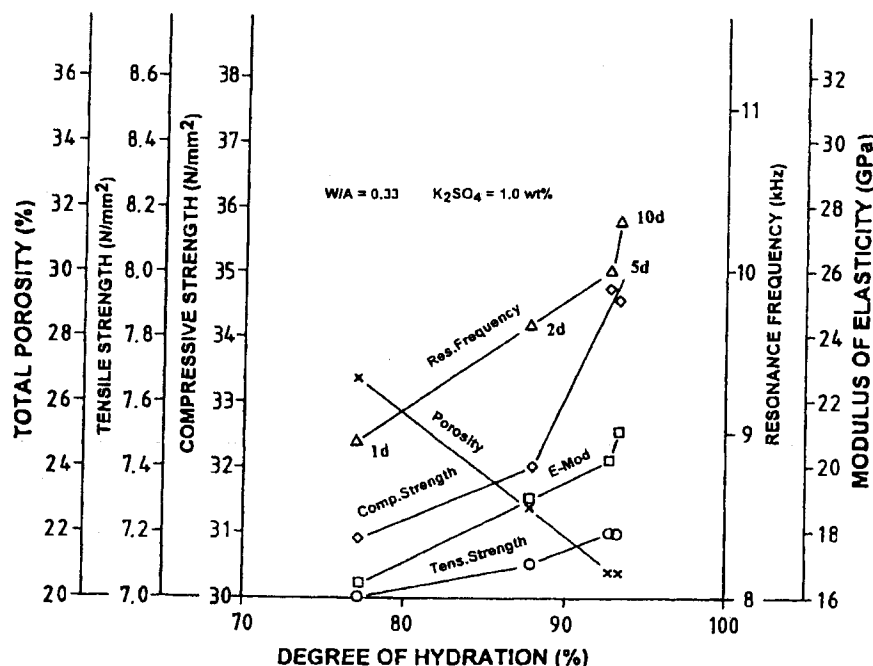


Fig. 10. Changes in resonance frequency, modulus of elasticity, compressive strength, tensile strength, and total porosity with the degree of hydration in hydrating anhydrite in the presence of 1.0 wt%  $K_2SO_4$  (prisms—4 cm × 4 cm × 16 cm).

the degree of hydration. As the degree of hydration increases, more and more compact crystals of dihydrate with low total porosity will be formed, which will increase the strength. According to Israel<sup>8</sup> and the result obtained by SEM, the changes in the physical properties of anhydrite activated with  $K_2SO_4$  can be associated with the changes observed in the morphology of crystals.

#### IV. Conclusions

From the results one can conclude that  $K_2SO_4$  is a very effective activator for the hydration of anhydrite. The effectiveness of  $K_2SO_4$  may be associated with the formation and stability of double salts. Heat of hydration studies have revealed that a correlation exists between total heat evolved and the degree of hydration, irrespective of  $K_2SO_4$  concentration. The higher strength in the presence of  $K_2SO_4$  is due to morphological changes.

#### Acknowledgment

The author is grateful to Prof. U. Ludwig for useful discussions.

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