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Effect of Surfactants on Phosphogypsum Crystallization and Filtration during Wet-Process Phosphoric Acid Production

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ABSTRACT

A nonionic surfactant (CMR-100) containing a mixture of C₆–C₂₂ sorbitan esters was tested to improve the filtration rate of phosphogypsum produced from a South Florida phosphate concentrate at different sulfate levels. Tests were carried out on a lab-scale under operating conditions that simulate, to a large extent, the industrial parameters. It was found that the mean diameter of the formed crystals was increased by addition of the surfactant. Equally important, a narrower size distribution was obtained as compared to that in the absence of the surfactant. A significant increase (23 to 31%) in filtration rate, expressed as tons P₂O₅/m²·day, was achieved due to the surfactant's addition. A correlation between crystal size distribution of phosphogypsum and filtration rate is presented in this paper.

Key Words. Solid liquid separation; Filtration; Dihydrate process; Phosphogypsum; Size distribution; Surfactant

INTRODUCTION

Phosphoric acid is an important intermediate product for the production of fertilizers. It is mainly produced by the dihydrate process in which a phosphate concentrate is leached with sulfuric and weak phosphoric acids to produce

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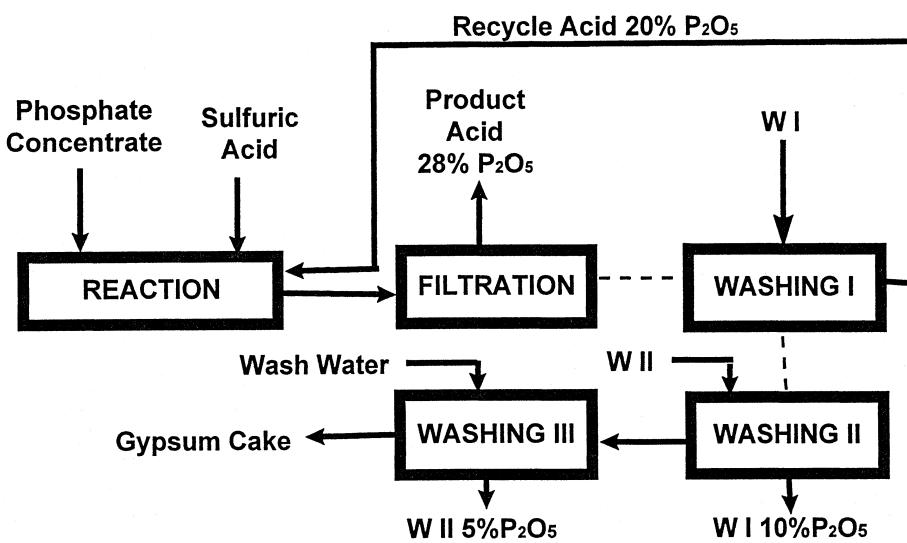
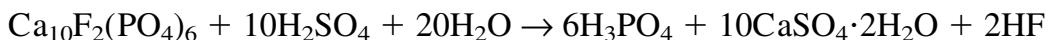
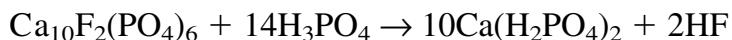


FIG. 1 Simple dihydrate process flow sheet.

phosphoric acid. A simple dihydrate process flow sheet is given in Fig. 1. Crystallization of calcium sulfate dihydrate (phosphogypsum) occurs as leaching is taking place. The primary reaction for the dihydrate process is (1):



After leaching, the slurry is filtered and countercurrent washed to separate the acid from the phosphogypsum cake (Fig. 1). The filtration operation represents a bottle neck in the wet-process phosphoric acid industry. Using the same filter area, the same production capacity with lower operation (running) costs can be achieved if the filtration rate is increased. It is known that the filtration rate depends on such filter cake characteristics as crystal size, size distribution, and morphology of the crystals. In other words, large, spherical, and narrow size distribution of crystals give a better filtration rate.

There are no published reports about the effect of surfactants on the filtration rate of phosphogypsum on an industrial scale. There are, however, available data about nucleation and crystal growth of gypsum from pure chemicals (2–5). Also, the influence of different inhibitors on the growth rate of calcium sulfate dihydrate crystals has been studied (6–9). In addition, many papers have been published explaining the effects of additives and impurities on crystallization of calcium sulfate (10–15). There are numerous reports about improving the filtration rate using polymers (16–26) as filter aids. On



TABLE 1
Chemical Analysis of High Dolomitic Phosphate Concentrate
Sample

Constituent	%	Constituent	%
P_2O_5	27.22	Al_2O_3	0.95
CaO	42.79	Na_2O	0.73
MgO	1.58	K_2O	0.12
Fe_2O_3	1.56	Insolubles	8.05

the other hand, limited literature about improvement of the filtration rate of phosphogypsum using surfactants has been published (27–29). Information about the crystallization of calcium sulfate dihydrate from pure chemicals is given in the most of the published papers, but no data are available about the surfactant addition technique and its effect on crystal size distribution.

The main objective of the present work is to understand the effect of an applied surfactant on filtration rate and crystal size distribution of the phosphogypsum formed.

EXPERIMENTAL

Materials Characterization

The phosphate concentrate sample (high dolomite) was provided by IMC-Agrico Company (FL, USA). The chemical constituents and sieve analyses of this sample are given in Tables 1 and 2. As seen in Table 1, the phosphate con-

TABLE 2
Sieve Analysis of High Dolomitic Phosphate Concentrate Sample

Mesh ^a	Particle size		Cumulative weight % passing
	Inch	mm	
–10	0.0650	1.651	100
–48	0.0116	0.295	87.5
–65	0.0082	0.208	68.1
–100	0.0058	0.147	51.8
–150	0.0041	0.104	40.1
–200	0.0029	0.074	29.7
–270	0.0021	0.053	18.6
–325	0.0017	0.044	7.0

^a Tyler standard.



centrate contains a high MgO content (1.58%). A high MgO content increases the acid viscosity and consequently decreases the filtration rate. The accepted industrially limit in commercial phosphate concentrates is less than 1.0% MgO. The sieve analysis of the sample is given in Table 2. It contains 51.8% < 100 mesh and 29.7% < 200 mesh particle sizes, which are suitable for the dihydrate process. Most of the sample weight (80.5%) falls in the 44–295 μm size range.

Pure (95.5%) sulfuric acid of 1.835 g/mL specific gravity is used for the digestion. The recycle (return) acid (18–20% P_2O_5) is accumulated while carrying out the tests. It is adjusted to the required P_2O_5 content before recycling. The surfactant used is Crystal Modifier Reagent (CMR-100) supplied by Polimeros Sinteticos Co. (Tlalnepantla, Mexico).

Apparatus

The reaction is carried out in a cylindrical 1 L reactor of 10 cm diameter. It is fitted with a Teflon-coated stirrer and placed in water bath adjusted to 80°C. The impeller tip speed is adjusted at 1.44 m/s (550 rpm). The phosphate concentrate is fed using a vibrating rock feeder. The sulfuric acid is pumped using a peristaltic pump with Viton tubing. The surfactant/water suspension is added using a small graduated separating funnel. All the above feeding is done on a continuous bases. Filtration is performed using a Buchner-type filter of 4.6 inch diameter. Polypropylene filter cloth of 80 mesh aperture size is used. A vacuum pump is used for filtration. A Coulter Laser Diffraction Analyzer model LS 230 is used for determination of size distribution of the phosphogypsum crystals produced. A JEOL Scanning Electron Microscope model JSM-6400 was used for investigation of crystal morphology.

Procedure

Reaction

The required amount of the return recycle acid (R.A.) is added to the reactor. After the R.A. temperature increases up to 80°C, calculated amounts of phosphate concentrate, sulfuric acid, and the surfactant/water suspension are added continuously for 30 minutes and the reaction is continued for another 2.5 hours. The amounts of reacted materials and washwater are calculated using a material balance computer program (30).

Filtration

After the required retention time, the slurry is weighed and poured into the filter. Vacuum is applied and the time recorded from the moment of vacuum application to just before the surface appears dry to avoid effect of channeling



and cracking (wall effect). The required amounts of wash I (~10% P₂O₅), wash II (~5% P₂O₅), and washwater or simulated pond water are applied separately and the time is recorded. The cake is allowed to filter for 5 seconds for drying (removing as much as possible of the residual liquor from the cake). Pond water in phosphoric acid plant is pumped from an industrial pond that contains the produced phosphogypsum (gypsum pond). It contains about 1.5% P₂O₅.

In industry the filtration rate is expressed as tons P₂O₅ produced per square meter per day, so the same expression was used. The filtration rate was calculated by applying the following equation:

$$F.R. = \frac{SW \cdot SC \cdot F}{T}$$

where F.R. = filtration rate tons P₂O₅/m²·day)

SW = slurry weight (g)

SC = solid content (%)

F = filtration factor

T = total time of filtration, washing, and drying (seconds)

The filtration factor is related to filter area, conversion of time, weight, and area units, as well as P₂O₅ recovery and phosphogypsum purity.

Crystal Size Distribution Determination

For crystal size distribution analysis, a sample (3 mL) is taken from the slurry during reaction and after 0.5, 1.0, 2.0, and 3.0 hours from the start-up of the test. Then the sample is dispersed in 100 mL methanol, sieved using a 106-μm screen, and the crystal size distribution is determined.

RESULTS AND DISCUSSION

During industrial crystallization of phosphogypsum, the presence of free (excess) sulfuric acid in the reaction medium is the most effective factor governing crystal quality. Its effect is not only on the crystal shape and size, but also on cocrystallized P₂O₅ losses and unreacted P₂O₅ losses (1). Free sulfate concentration affects supersaturation, which is the driving force of both nucleation and crystal growth. At low sulfate content the supersaturation is low and the rate of both nucleation and crystal growth are low, and vice versa. The range of free sulfate industrially applied is 2.0–3.0% (1). However, sulfate as low as 1.5% and as high as 3.5% can be encountered. Consequently, the South Florida phosphate concentrate (high dolomitic) was tested for phosphoric acid production with and without surfactant at different sulfate contents (1.5–3.5%). The amount of applied surfactant corresponds to 1.5 kg/ton P₂O₅ produced.



TABLE 3
 Filtration and Reaction Data at Low Sulfate Content (1.5–1.8%)

Item	Without surfactant	With surfactant
Filtration rate, tons $P_2O_5/m^2 \cdot day$	4.24 ± 0.2	5.54 ± 0.3
Moisture %	28.4	27.5
<i>Increase in filtration rate, %</i>	—	31
Reaction efficiency, %	93.2	97.1
P_2O_5 recovery, %	92.5	94.1
Washing efficiency, %	99.3	97.0

Testing at Low Sulfate Content (1.5–1.8%)

The filtration and reaction data obtained are given in Table 3. The filtration rate of 4.24 ton $P_2O_5/m^2 \cdot day$ without surfactant was improved to 5.54 ton $P_2O_5/m^2 \cdot day$ with the addition of the surfactant. The percentage of filtration rate improvement corresponds to about 31%. This is attributed to modification and growth of the crystals produced upon surfactant addition. The size distribution of gypsum crystals, which is given in Tables 4 and 5 and Fig. 2, show that the volume percentage of fine crystals ($<10 \mu m$) is decreased to about 31% upon addition of the surfactant as compared to its high value of 64% without surfactant addition. It is clear that after 3 hours of retention time d_{90} (diameter of crystals passing 90% by volume) are 25.5 μm without surfactant

 TABLE 4
 Comparative Size Analyses of Gypsum Crystals (at low sulfate content and after 3 hours)

μm	Mesh ^a	Cumulative volume % passing	
		Without surfactant	With surfactant
125	120	100.00	100.00
106	140	100.00	99.91
90	170	100.00	99.05
75	200	99.99	95.68
63	230	99.82	89.70
53	270	99.15	82.11
45	325	97.93	74.91
38	400	96.21	68.12
10	—	64.28	30.75

^aASTM standards.


TABLE 5
Statistics of Phosphogypsum Crystal Size Distribution (at low sulfate and after 3 hours)

Item d_x^a	Phosphogypsum crystal size distribution (μm)					Mean diameter of crystals (μm)	Average specific surface area (cm^2/g)
	d_{10}	d_{25}	d_{50}	d_{75}	d_{90}		
Without surfactant	1.0	2.3	6.3	14.3	25.5	10.3	10,453
With CMR-100 surfactant	1.9	7.2	22.2	45.1	63.5	28.1	6,599

^a d_x = diameter of crystals passing $x\%$ by volume.

and 63.5 μm with surfactant. Also, the mean crystal size increases with addition of the surfactant while the specific surface area decreases.

The obtained reaction efficiencies and P_2O_5 recoveries in the presence and absence of the surfactant are given in Table 3. It is most interesting that both P_2O_5 recovery and reaction efficiency are increased with the addition of the surfactant. This may be attributed to a decrease of both lattice (cocrystallized) P_2O_5 and unreacted P_2O_5 losses. The lattice (cocrystallized) P_2O_5 losses generally represent about 60% of the total P_2O_5 losses (1). In this regard it has been reported that the presence of a surfactant has resulted in reduction of

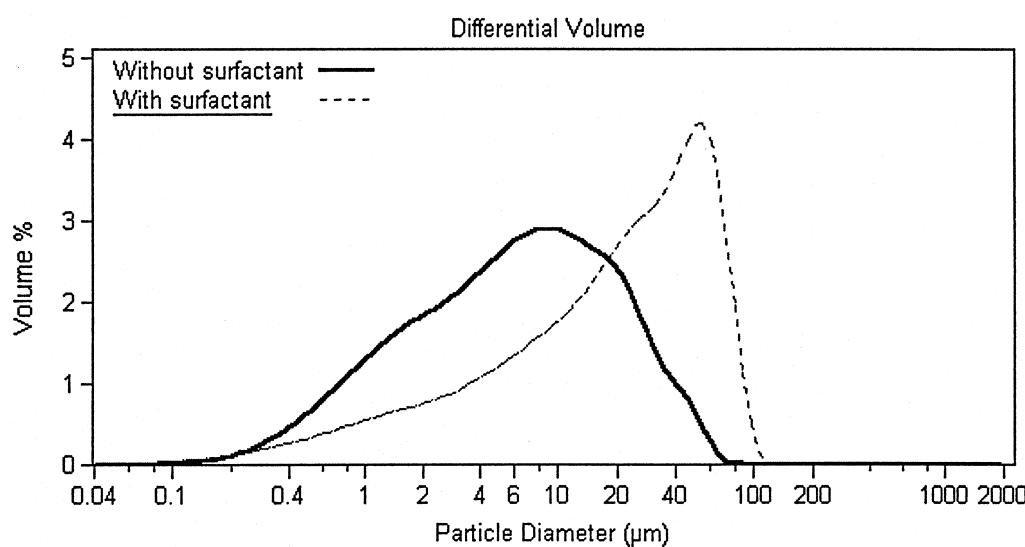


FIG. 2 Comparative size distribution of phosphogypsum crystals (at low sulfate content and after 3 hours).



TABLE 6
Filtration and Reaction Data at Medium Sulfate Content (2.2–2.4%)

Item	Without surfactant	With surfactant
Filtration rate, tons $P_2O_5/m^2 \cdot day$	5.13 ± 0.3	6.42 ± 0.4
Moisture, %	28.3	27.8
<i>Increase in filtration rate, %</i>	—	25
Reaction efficiency, %	95.8	98.6
P_2O_5 recovery, %	95.0	96.4
Washing efficiency, %	99.2	97.9

P_2O_5 in the precipitated calcium sulfate, and this is attributed to the surfactant's effect in reducing the capture of phosphate ions from solution by crystallizing gypsum crystals (31). It was found that the addition of surfactant increases the phosphate concentrate solubility and consequently increases reaction efficiency thus decreasing the amount of unreacted phosphate. In another study it was found that the presence of surfactant increases the solubility of calcium phosphate in thermal and wet-process phosphoric acid of 10 to 40% P_2O_5 concentration in a temperature range of 40 to 75°C (32). The increase in reaction efficiency is significant as it approaches 3.9% and the increase in P_2O_5 recovery is 1.6%.

TABLE 7
Comparative Size Analyses of Gypsum Crystals (at medium sulfate content and after 3 hours)

μm	Mesh ^a	Cumulative volume % passing	
		Without surfactant	With surfactant
125	120	100.00	99.85
106	140	99.88	99.18
90	170	98.95	98.06
75	200	95.63	95.65
63	230	89.86	90.55
53	270	82.47	80.54
45	325	74.98	66.31
38	400	67.09	49.95
10	—	22.06	10.38

^aASTM standards.



TABLE 8
Statistics of Phosphogypsum Crystal Size Distribution (at medium sulfate and after 3 hours)

Item d_x^a	Phosphogypsum crystal size distribution (μm)					Mean diameter of crystals (μm)	Average specific surface area (cm^2/g)
	d_{10}	d_{25}	d_{50}	d_{75}	d_{90}		
Without surfactant	2.4	11.9	26.5	45.1	63.2	30.3	8069
With CMR-100 surfactant	9.4	26.2	38.1	49.6	62.3	38.4	3995

^a d_x = diameter of crystals passing $x\%$ by volume.

Testing at Medium Sulfate Content (2.2–2.4%)

To cover the range of sulfate contents applied in industry, tests at medium sulfate content (2.2–2.4%) were carried out with and without addition of the surfactant. The filtration and reaction data obtained are given in Table 6. The filtration rate increased by 25% with the addition of surfactant. This is related to the formation of a narrow size distribution of phosphogypsum crystals with a low volume percentage of fines as confirmed by the size distribution of phosphogypsum crystals given in Tables 7 and 8 and Fig. 3. It is clear that the

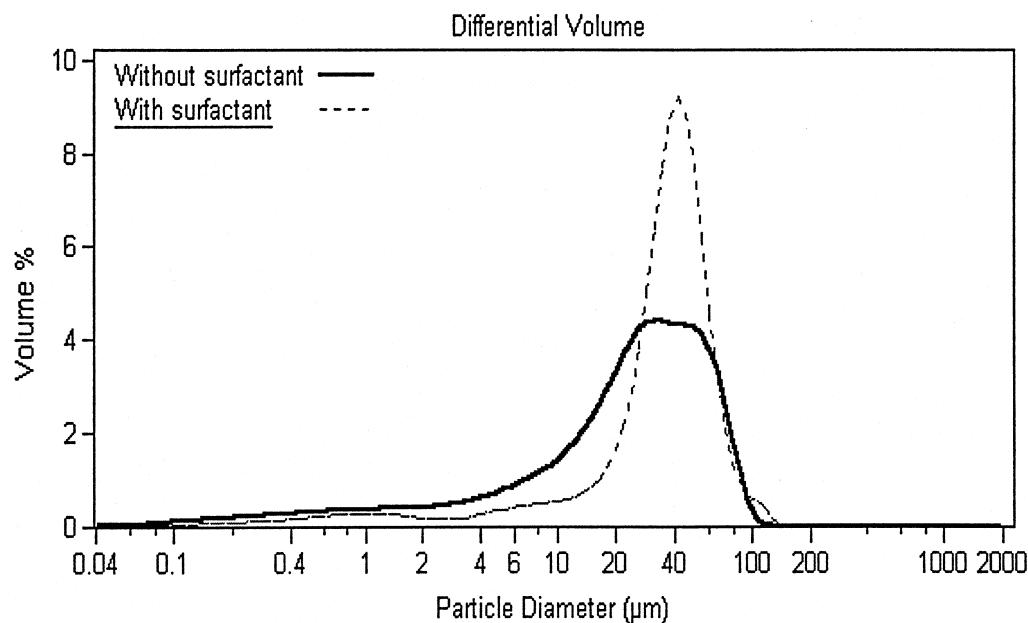


FIG. 3 Comparative size distribution of phosphogypsum crystals (at medium sulfate content and after 3 hours).



TABLE 9
Filtration and Reaction Data at High Sulfate Content (3.2–3.5%)

Item	Without surfactant	With surfactant
Filtration rate, tons $P_2O_5/m^2\cdot day$	6.77 ± 0.4	8.33 ± 0.5
Moisture, %	27.9	28.4
<i>Increase in filtration rate, %</i>	—	23
Reaction efficiency, %	96.9	99.0
P_2O_5 recovery, %	96.4	96.9
Washing efficiency, %	99.5	97.8

volume percentage of fine phosphogypsum crystals ($<10 \mu m$) decreases with the addition of surfactant. Also, the mean crystal size increases with the addition of surfactant. The phosphogypsum crystal size distribution data show that the mean diameter of crystals without surfactant is $30.3 \mu m$ and increases to $38.4 \mu m$ with surfactant. Also, the average specific surface area of the formed phosphogypsum without surfactant is $8069 \text{ cm}^2/g$ and decreases to $3995 \text{ cm}^2/g$ with surfactant.

The reaction efficiencies and P_2O_5 recoveries obtained in the presence and absence of surfactant are given in Table 6. Both P_2O_5 recovery and reaction efficiency are higher with the addition of surfactant. As previously mentioned, this is attributed to a decrease of lattice P_2O_5 and unreacted P_2O_5 losses. The increase in reaction efficiency and P_2O_5 recovery are 2.8 and 1.4%, respectively.

TABLE 10
Comparative Size Distribution of Gypsum Crystals (at high sulfate content and after 3 hours)

μm	Mesh ^a	Cumulative volume % passing	
		Without surfactant	With surfactant
125	120	99.91	99.70
106	140	99.01	97.75
90	170	96.15	92.97
75	200	89.76	84.77
63	230	81.11	76.14
53	270	71.31	68.25
45	325	62.10	61.57
38	400	53.26	55.06
10	—	17.96	20.07

^a ASTM standards.



TABLE 11
Statistics of Phosphogypsum Crystal Size Distribution (at high sulfate and after 3 hours)

Item d_x^a	Phosphogypsum crystal size distribution (μm)					Mean diameter of crystals (μm)	Average specific surface area (cm^2/g)
	d_{10}	d_{25}	d_{50}	d_{75}	d_{90}		
Without surfactant	3.9	15.8	35.5	56.6	75.4	38.1	6988
With CMR-100 surfactant	3.7	13.2	33.1	61.6	83.7	39.3	7389

^a d_x = diameter of crystals passing $x\%$ by volume.

Testing at High Sulfate Content (3.2–3.5%)

The results obtained are given in Tables 9–11 and Fig. 4. It is interesting to note that the filtration rate (6.77 ton $\text{P}_2\text{O}_5/\text{m}^2\cdot\text{day}$ without additive) improved to 8.33 ton $\text{P}_2\text{O}_5/\text{m}^2\cdot\text{day}$ with the addition of surfactant. The increase in filtration rate is related to the growth of the crystals produced upon surfactant addition and the formation of a high volume percentage of coarse crystals ($>75\ \mu\text{m}$). The results show that, the volume percentage of fine fraction ($<10\ \mu\text{m}$) and the average specific surface area are slightly increased. However, the

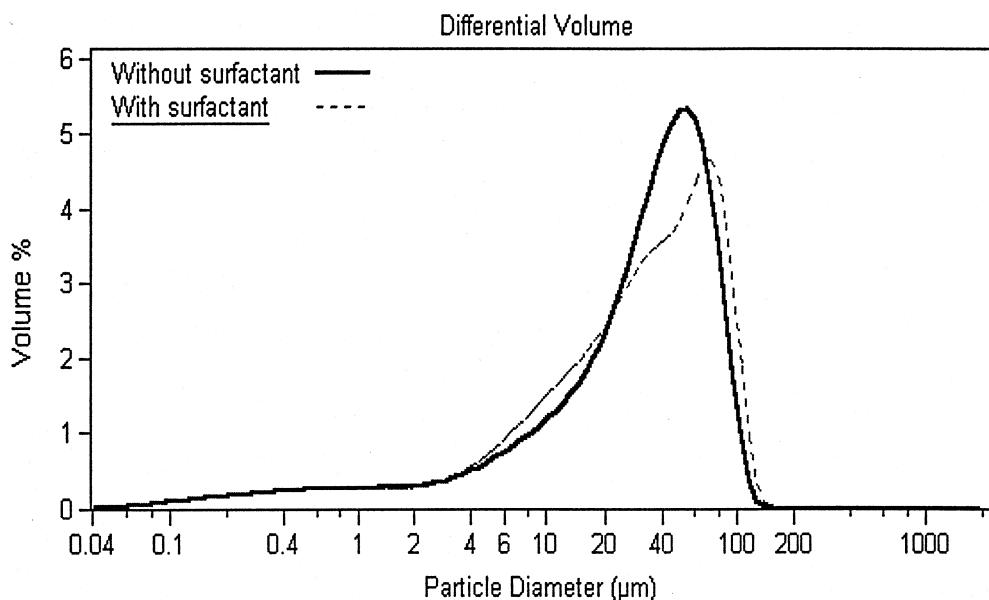


FIG. 4 Comparative size distribution of phosphogypsum crystals (at high sulfate content and after 3 hours).



coarse crystals ($>75 \mu\text{m}$) are increased to about 15% upon addition of surfactant as compared to a low value of 10% without surfactant addition. The phosphogypsum crystal size distribution statistics show that d_{90} (diameter of crystals passing 90% by volume) of the formed crystals is $75.4 \mu\text{m}$ without surfactant and increases to $83.7 \mu\text{m}$ with surfactant.

The data for reaction efficiencies, P_2O_5 recoveries, and washing efficiencies with and without surfactant are given in Table 9. It is important to note that even at high sulfate content the reaction efficiency and P_2O_5 recovery are increased with the addition of surfactant. The increase in reaction efficiency is significant as it approaches 2.1% and the increase in P_2O_5 recovery is 0.5% with addition of surfactant.

Correlation between Crystal Size Distribution of Phosphogypsum and the Filtration Rate

The correlations between the crystal size distribution of phosphogypsum and the filtration rates at different sulfate levels are given in Table 12. These results show that by increasing the mean of crystal size, d_{75} , median, and/or mode, the filtration rate increases at each sulfate level.

Phosphogypsum Morphology

One of the most important factors affecting filtration rate is gypsum morphology (size and shape of the crystals). For best results, crystals of uniform sizes are desirable. In this study the crystal shape was changed from individual tabular crystals to clusters by the addition of surfactant as shown in SEM photomicrographs (Figs. 5 and 6). Similar results were obtained at all sulfate

TABLE 12
Correlation between Crystal Size Distribution and Filtration Rate

Item	Phosphogypsum crystal size (μm)				Filtration rate (tons $\text{P}_2\text{O}_5/\text{m}^2\cdot\text{day}$)
	Median	d_{75}	Mode	Mean	
Low sulfate content:					
Without surfactant	6.3	14.3	9.4	10.3	4.24
With CMR-100 surfactant	22.2	45.1	55.1	28.1	5.54
Medium sulfate content:					
Without surfactant	26.5	45.1	31.5	30.3	5.13
With CMR-100 surfactant	38.1	49.6	41.7	38.4	6.42
High sulfate content:					
Without surfactant	35.5	56.6	55.1	38.1	6.77
With CMR-100 surfactant	33.1	61.6	73.0	39.3	8.33



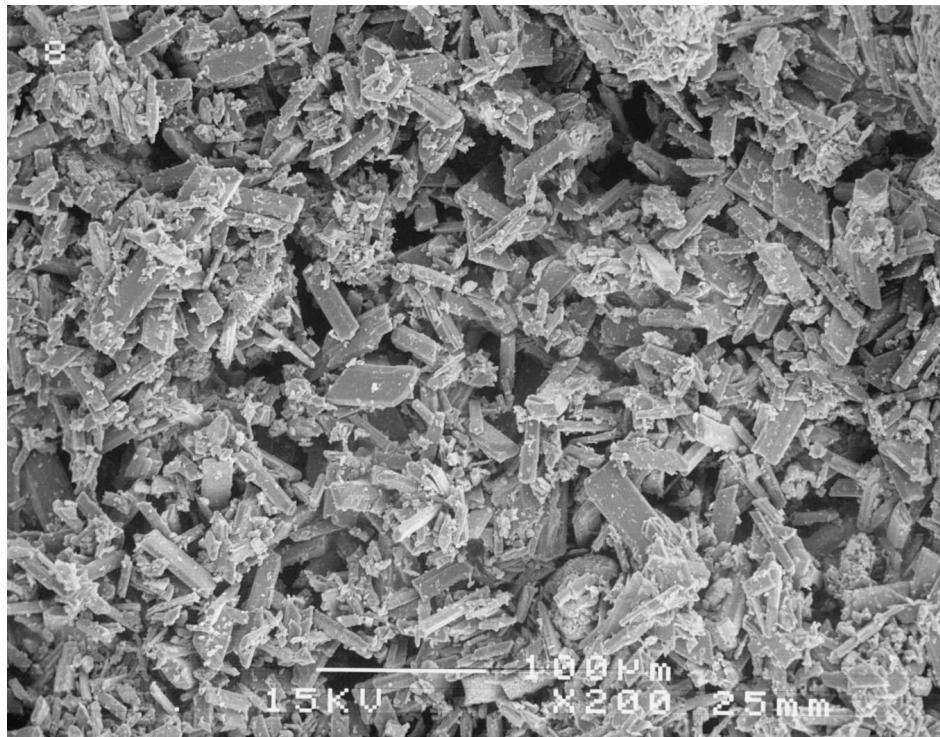


FIG. 5 SEM photomicrograph of phosphogypsum without surfactant (magnification 200).

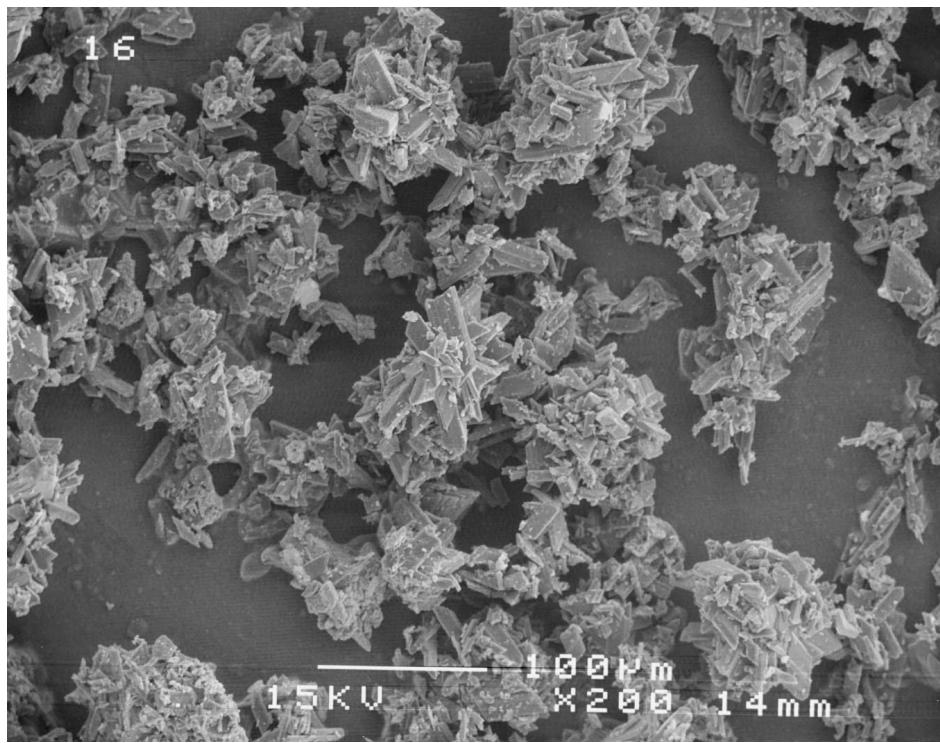


FIG. 6 SEM photomicrograph of phosphogypsum with CMR-100 surfactant (magnification 200).

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levels. This change in morphology could be the reason for the change in size distribution and, consequently, the enhancement in filtration rates. Fundamentally, these large clusters are composed of many small crystals. This may be due to adsorption of surfactants on crystal surfaces, causing hydrophobic (attraction) interactions. Similar findings have been reported in the literature (33). Further research is underway in an effort to illustrate the mechanism involved in the system. For example, by using pure chemicals and under simulated industrial conditions, it was found (34) that surfactant changes the aspect ratio of crystals formed and decreases the induction time. The results of these fundamental studies will be published later.

CONCLUSION

The effect of CMR-100 surfactant on phosphogypsum filtration rate and size distribution of phosphogypsum has been investigated at different sulfate contents. The results indicate that 31, 25, and 23% increases in filtration rate were achieved at low, medium, and high sulfate contents, respectively. The observed improvements are attributed to changes in crystal size distribution. With narrower size distributions with a higher mean crystal size, the filtration rate is highly increased. In addition, noticeable increases in reaction efficiencies and P_2O_5 recoveries are obtained. The increase in reaction efficiency is significant as it ranges between 2.1 and 3.9% and the increase in P_2O_5 recovery is 0.5 to 1.6% depending on the sulfate content with the addition of 1.5 kg CMR-100 surfactant per ton P_2O_5 produced.

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