Clear Liquor Scrubbing with Anhydrite Production

O. W. Hargrove, Jr. (buddy_hargrove@radian.com; 512 419-5075)
T. R. Carey (todd_carey@radian.com; 512 419-5653)
P. S. Lowell (phil_lowell@radian.com; 512 419-5635)
Radian International, LLC
P.O. Box 201088
Austin, TX 78720-1088

F. B. Meserole
Meserole Consulting (BYNG37A@prodigy.com;512 345-9232)
8719 Ridgehill Dr.
Austin, TX 78759

R. G. Rhudy (rrhudy@epri.com; 415 855 2421)
Electric Power Research Institute
P. O. Box 10412
Palo Alto, CA 94303-0813

Thomas J. Feeley, III (feeley@petc.doe.gov; 412 892 6134)
Federal Energy Technology Center
Department of Energy
P.O. Box 10940
Pittsburgh, PA 15236

Introduction

This project is funded by the U.S. Department of Energy's Federal Energy Technology Center (DOE/FETC) under a cost-sharing PRDA with Radian International. The Electric Power Research Institute (EPRI) is providing co-funding and technical oversight. The project is part of FETC's Advanced Power Systems Program, whose mission is to accelerate the commercialization of affordable, high-efficiency, low emission, coal-fueled electric generating technologies.

A process concept—Clear Liquor Scrubbing with Anhydrite Production—was proposed and accepted by FETC as a Phase I project in its Mega-PRDA program. The project integrates three process operations—chloride control upstream of the flue gas desulfurization (FGD) system, a clear liquor process for enhanced SO_2 removal performance, and production of anhydrite (anhydrous calcium sulfate) rather than calcium sulfite or gypsum (calcium sulfate dihydrate). The CLS/Anhydrite process is an advanced FGD process designed to remove greater than 99% of the SO_2 from flue gas, to avoid reliability problems associated with many existing FGD systems, and to produce a reusable byproduct—anhydrous calcium sulfate (anhydrite). Substantial cost reduction and greater byproduct marketing flexibility are projected for this process.

Phase I of the PRDA focused on anhydrite production since it was the least developed process and because byproduct disposal options are of more near-term interest to utilities. Anhydrite was produced successfully in the laboratory and during proof-of-concept testing at the 4 MW pilot scale.

Objective

The objective of this project to develop an advanced flue gas desulfurization (FGD) process that has decreased capital and operating costs, higher SO_2 removal efficiency, and better byproduct solids quality than existing, commercially available technology. A clear liquor process (which uses

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a scrubbing liquid with no solids) will be used to accomplish this objective rather than a slurry liquor process (which contains solids). This clear liquor scrubbing (CLS) project is focused on three research areas:
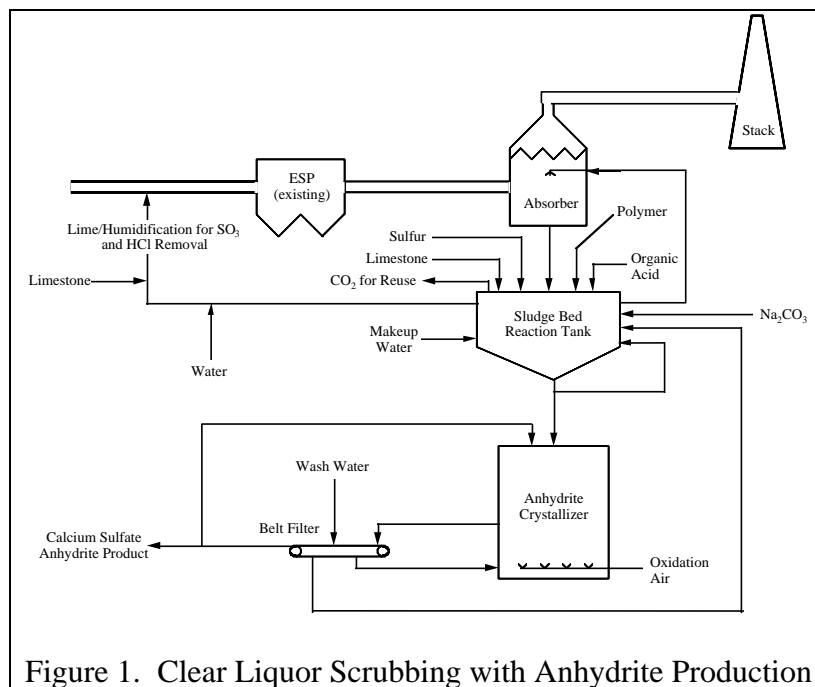
- Development of a clear liquor scrubbing process that uses a clear solution to remove SO_2 from flue gas and can be operated under inhibited-oxidation conditions;
- Development of an anhydrite process that converts precipitated calcium sulfite to anhydrous calcium sulfate (anhydrite); and
- Development of an alkali/humidification process to remove HCl from flue gas upstream of the FGD system.

The anhydrite process also can be retrofit into existing FGD systems to produce a valuable byproduct as an alternative to gypsum. This fits well into another of FETC's PRDA objectives of developing an advanced byproduct recovery subsystem capable of transforming SO_2 into a useable byproduct or high-volume valuable commodities of interest.

This paper describes the proposed processes, outlines the test approach, and preliminary Phase I test results.

Process Description

Figure 1 illustrates the basic flow diagram for the CLS process. This figure includes incorporation of the alkali/humidification process for HCl reduction and the anhydrite production process. However, each of the three processes can be applied independently.

Figure 1. Clear Liquor Scrubbing with Anhydrite Production

Clear Liquor Scrubbing Process. The basic CLS concept is to recirculate clear liquor that contains a sufficient liquid-phase alkalinity to achieve the desired SO_2 removal efficiency without the need for solid-phase alkalinity. The liquor then flows to a limestone reactor and solid-liquid separator that precipitates a calcium-sulfur solid and returns clear liquor to the scrubber. The CLS process can be operated as an inhibited-oxidation system (calcium sulfite production) or as a forced-oxidation system (gypsum production). Either of these two products can then be converted to anhydrite; however, the economics of the anhydrite process are more favorable if calcium sulfite is produced in the CLS system.

To be successful, the CLS process must generate solids that are easily dewatered at high rates so that the high volume of liquor passing through the regeneration system does not require large or expensive tanks and equipment. Other key aspects for the success of the process include low consumption rates of the buffer, low L/G in the scrubber, and control of the chemistry to achieve good utilization and low scaling potential. The CLS process had been developed during prior EPRI

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testing using the 0.4-MW mini-pilot system at EPRI's Environmental Control Technology Center (ECTC).

Based on the previous studies of the CLS process, several issues require further investigation. The first need is to verify the findings observed on the mini-pilot system on the pilot system that is 10 times larger. Full-scale design criteria need to be developed and refined at the 4-MW pilot-scale before a full-scale demonstration is attempted. Variables to be tested at the larger scale include residence time, pH, suspended solids concentration, recycle ratio, and degree of agitation. In addition, the potential of combining the CLS process with the anhydrite process needs to be investigated. The high TDS concentration necessary for the anhydrite process may have a negative effect on the calcium sulfite solids properties in the CLS process.

Anhydrite Process. As shown in Figure 1, a process that produces anhydrous calcium sulfate (anhydrite) can be added to the CLS process. The anhydrite process could potentially be added to any conventional FGD system. Potential advantages of crystallizing anhydrite rather than gypsum from solution include the following: energy savings over thermally dehydrating gypsum, producing a material of lesser specific volume than gypsum, potentially displacing gypsum in some commercial applications, and producing a material for different commercial applications than gypsum. These potential advantages are somewhat speculative because the anhydrite market is currently small relative to that of gypsum in the U.S. However, if a relatively cheap source of anhydrite is developed, the use of anhydrite could increase in several different applications that are discussed later.

In a solution saturated with calcium and sulfate, anhydrite, rather than gypsum, is the thermodynamically stable form above about 100°F. However, typical forced-oxidation FGD systems operating at 120_F-140_F produce only gypsum. The gypsum product results because the kinetics of gypsum precipitation are very fast relative to the anhydrite precipitation kinetics. Figure 2 shows the solubility of gypsum and anhydrite as a function of temperature and ionic strength. Both solids become more soluble at lower temperatures, but only gypsum solubility is affected by ionic strength. This property is key to the anhydrite process.

In the proposed process, calcium sulfite produced in the CLS process is converted to anhydrite in a secondary reaction tank by oxidation of sulfite to sulfate and subsequent precipitation of anhydrite. As discussed above, the kinetics of anhydrite precipitation are relatively slow at typical FGD operating conditions. To increase the precipitation rate of anhydrite, the process must be operated at an elevated temperature and at a high ionic strength. As shown in Figure 2, gypsum solubility increases with ionic strength while anhydrite solubility does not. Based on information in Figure 2, conditions can be chosen that provide a driving force for anhydrite precipitation while the solution remains subsaturated with respect to gypsum (i.e., gypsum will not

Figure 2. Calcium Sulfate Solubility Data

be formed). Points A and B in Figure 2 represent such points at lower and higher ionic strengths, respectively. The driving force for anhydrite precipitation is represented by the distance between either Point A or B and the solid line. By increasing the solution ionic strength, the driving force for anhydrite precipitation can be increased without forming gypsum solids.
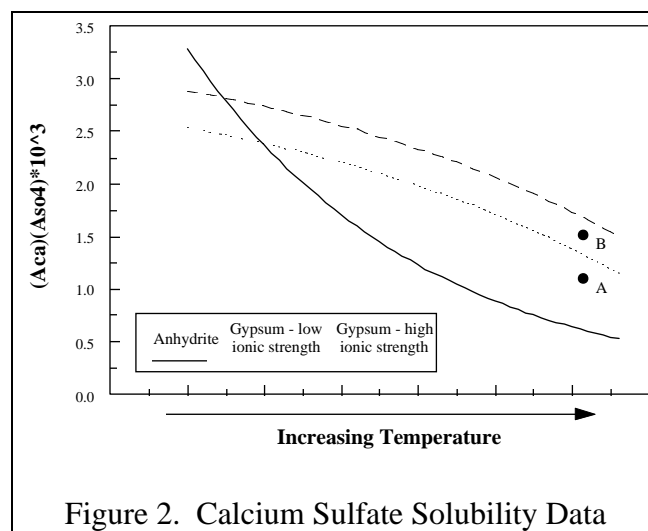
Previously conducted batch tests determined the precipitation rate of anhydrite at 140°F from a high ionic strength solution. Based on these data, an anhydrite precipitation rate was calculated from the

slope of a fitted exponential function. This rate was sufficiently high to warrant further investigation to determine how the rate might be further increased. The success of the anhydrite process depends on finding a set of operating conditions under which the anhydrite precipitation rate is sufficiently fast to yield an economically sized reaction tank and which minimizes gypsum production.

Alkali/Humidification Process. Maintaining a low chloride concentration in a FGD system has several potential benefits. Corrosion should be minimized at low concentrations that may allow materials of construction to be cheaper. In limestone systems, the limestone utilization will generally be higher. Finally, the scaling potential is reduced in the CLS process.

EPRI has examined a number of chloride control options including prescrubbing, tail-end separation or concentration, and injection of scrubber blowdown into the flue gas. The first two options are relatively expensive and result in some waste water streams. The last option has been tested with limited success, but chloride removal is limited by off-gassing of HCl in the process of drying the liquid.

The proposed chloride removal process involves injection of an alkaline slurry upstream of an ESP or other particulate removal device. HCl in the flue gas reacts with the alkali to form a solid which is dried by the flue gas and collected in the particulate removal device. EPRI has developed a similar process to react flue gas SO_3 with alkali reagent to mitigate opacity problems and has completed considerable work on the effect of injection on SO_2 removal.

Approach

Each of the three processes proposed in this program can benefit the power industry individually and are at different stages of development. Program plans were to develop each of the processes at the appropriate scale in Phase I. Since the anhydrite process was the least developed, the initial plans were to perform bench-scale tests in the laboratory followed by further development at the 4-MW pilot scale. The chloride removal and clear liquor scrubbing processes had been tested previously by EPRI on the 0.4-MW mini-pilot scale, and tests were planned the 4-MW scale for further development. Thus, all three processes were to be tested on the pilot unit at the ECTC during Phase I but will not be operated in an integrated, steady-state manner. This was to be included in Phase II.

However, the majority of the pilot work in Phase I was devoted to the anhydrite production process because of the small scale of development of the anhydrite process before this project, progress in producing reactive anhydrite from solution, and potential interest in an alternative byproduct by the utility industry.

Laboratory Testing. Development of the anhydrite process began with a series of batch and flow-through reactor tests in the laboratory. The objective of these tests was to provide kinetic data over a range of operating conditions. Batch test variables included solution ionic strength, temperature, and initial liquid-phase calcium-to-sulfate ratio. These tests were conducted by generating the desired initial solution composition with reagent grade chemicals and seeding the batch reactor with anhydrite. The anhydrite seed material was generated in the lab by thermally converting gypsum in a high ionic strength solution at 80-90°C. Complete conversion of the gypsum to anhydrite was confirmed using thermal gravimetric analysis (TGA) and Infrared (IR) spectroscopy. During each batch test, the liquid calcium and sulfate concentrations were measured with time by analyzing liquid samples with either atomic absorption spectroscopy (AA) or ion chromatography (IC). Changes in the calcium and/or sulfate concentration with time yield the anhydrite precipitation rate. Solids collected at the end of each test were analyzed using TGA and IR to confirm that no gypsum was generated.

Based on the batch test results, laboratory flow-through reactor tests were conducted at the most promising conditions. Variables included temperature, ionic strength, residence time, solids loading, and seed crystal size. The objective of these tests was to provide determine the best conditions for initial pilot-testing. Liquid flow-through tests generally provide more realistic kinetic data than batch tests since the data is generated at a constant liquid composition (i.e., constant anhydrite relative saturation).

Figure 3 illustrates the configuration used during the flow-through tests. The desired solution composition was established, and a known charge of anhydrite seed material was added to the reactor at the beginning of each test. Throughout each test, liquid was filtered from the reaction vessel while "fresh" solution, saturated with respect to gypsum, was added to the reaction vessel. In this way, these experiments approached steady-state with respect to the solution chemistry. Since the charge of seed material was large relative to the mass of anhydrite precipitated during most tests, the change in solid surface area was small, and changes in the soluble species concentration was also small. The rate of reaction was determined by a liquid-phase material balance between the inlet and outlet streams. The weight percent solids was measured at the beginning and end of each test to confirm the liquid-phase measurements, although the error in the solid-phase balance is much larger given the relatively small change in suspended solids concentration.

Figure 3. Bench-Scale Flow-Through Crystallizer
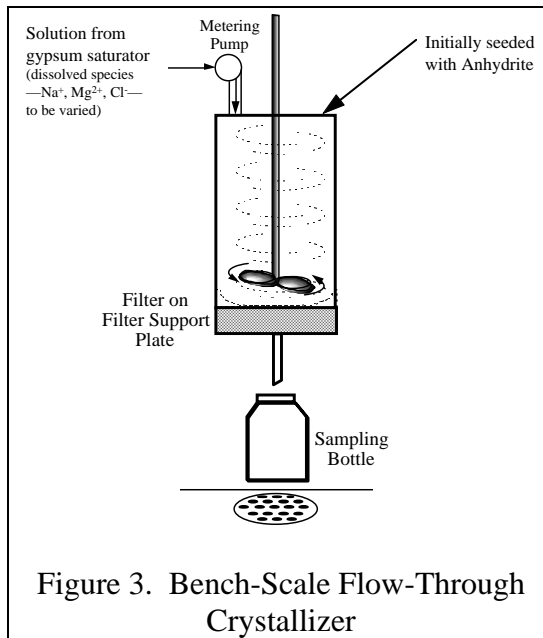
Pilot Testing. Figure 4 shows the equipment configuration used to test the anhydrite process at the ECTC. The major objective of the pilot testing was to demonstrate that anhydrite could be produced from FGD-generated calcium sulfite slurry. Once it had been demonstrated that anhydrite production was feasible, important variable interactions were to be explored. Several modifications were made to the original test plan because the pilot anhydrite reactor was an existing pilot reaction vessel, not a newly constructed one. Because of some limitations, test conditions had to be modified to achieve the desired reaction temperature. However, a good understanding of several important variable interactions was developed during the Phase I testing.

A number of techniques were employed to determine the phase of calcium sulfate produced in the pilot unit including:

- % Bound Water (measure solid weight loss at about 180°C) - provides estimate of anhydrite wt.% assuming solids are mixture of only anhydrite and gypsum;

- X-Ray Diffraction (XRD) - indicates the presence of gypsum, hemihydrate, and/or anhydrite; and

- Visual Microscope and Scanning Electron Microscope (SEM) - indicate changes in particle size and shape.
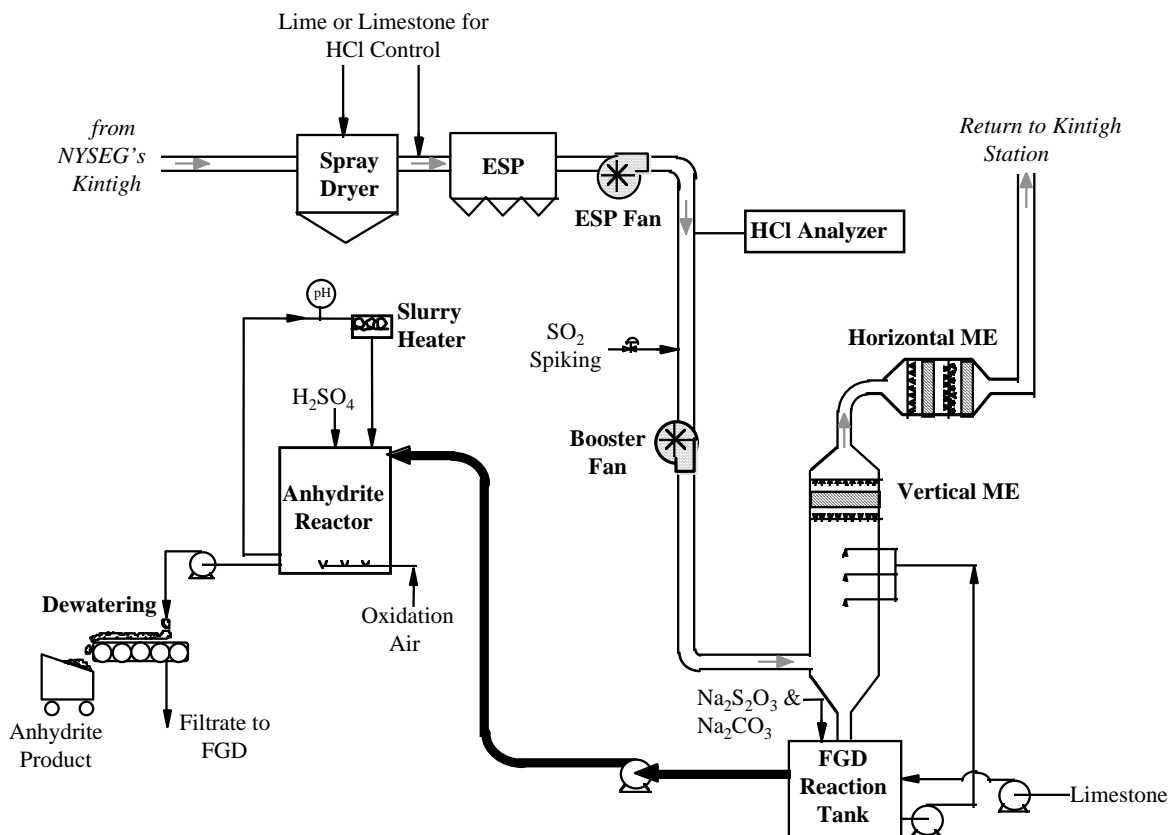
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Figure 4. ECTC Flow Configuration for Anhydrite Test Block.

The percentage of bound water and the visual microscope were available on site and were the primary means of characterizing the solids to provide operating feedback. In using the bound water as an indication, it was assumed that the solids were either anhydrite or gypsum. For 100% gypsum solids, the bound water should be about 21% by weight. Anhydrite should have no bound water. Visual observations of the centrifuge product were also valuable once experience had been gained. The XRD and SEM were used in Radian's Austin laboratories to confirm on-site results.

Results

Laboratory. This section presents the results of batch and flow-through laboratory experiments. In both batch and flow-through tests, anhydrite precipitation rates were measured that are equivalent to gypsum precipitation rates at typical FGD conditions (0.06 g/hr-g seed at 125°F). This indicates that tanks of an economically attractive size may be used in the production of anhydrite at the commercial scale, so long as another process not simulated in the laboratory (such as calcium sulfite dissolution) does not become rate limiting.

A total of 13 valid batch reactor tests were completed. The precipitation rate continuously changes throughout a batch experiment and can be calculated by measuring the change in solution composition over time. By comparing the anhydrite precipitation rate 10 minutes into each run, the effects of several important variables were determined. Although the amount of solids precipitated was relatively small compared to the initial seed inventory, IR and TGA analyses indicated that gypsum was not formed during the batch experiments.
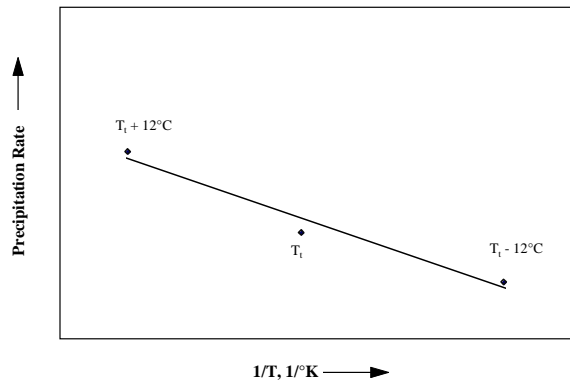
The effect of temperature on the anhydrite precipitation rate in an NaCl solution is shown in Figure 5. These results show the expected increase in reaction rate as the temperature increases over a 25°C range. If the rate constant were to exhibit an Arrhenius-type relationship with temperature ($k = Ae^{-E^*/RT}$), a plot of the log of the precipitation rate versus 1/T (in °K) should yield a straight line. Figure 4-3 shows this relationship. The precipitation rate increases by slightly more than a factor of two for each 10°C temperature rise over the temperature range.

Figure 5. Effect of Temperature on Batch
Precipitation Rate

According to theoretical calculations (see Figure 2), the anhydrite precipitation rate should be greater at higher ionic strength (higher concentration of dissolved solids). This effect was observed in the batch tests as shown in Figure 6. There was more than a 20-fold increase in the precipitation rate as the NaCl concentration increased from by a factor of 6. Clearly, for anhydrite production to be successful, higher ionic strength solutions have to be used.
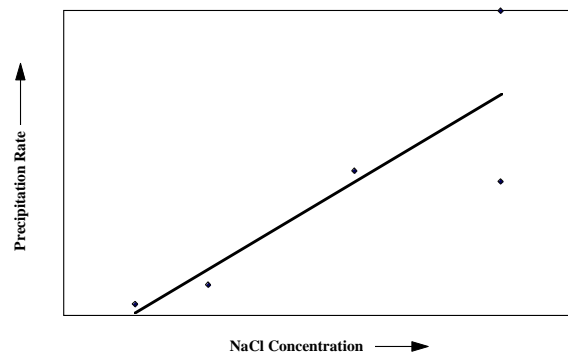
Figure 6. Effect Solution Strength on Anhydrite Precipitation Rate (NaCl Solutions)

Several liquid flow-through, batch solids runs were conducted to confirm rates measured in the batch experiments. The batch rates are determined by differentiating concentration data obtained from a solution that is continually changing in composition. The flow-through tests approach a steady-state solution composition more similar to a continuous full-scale process, and, for this reason, the flow-through results are considered to be more representative. A total of 12 valid flow-through experiments were completed.

The initial runs were conducted such that the composition of the inlet stream (calculated after assumed mixing) was slightly subsaturated with respect to gypsum. Different anhydrite seed loading and different flow rates were tested at different temperatures to develop an understanding of the steady-state precipitation rates. The pH was also decreased substantially in one run and iron added to the reactor in another run to determine if either had a positive effect on the precipitation rate.

The anhydrite precipitation rates measured in the first several tests were much lower than the initial rates measured in the batch experiments (based on comparison of batch rates at similar solution composition and temperature). Changes in the feed rate to the reactor, calcium concentration in the feed solution, and amount of anhydrite in the initial charge to the reactor were made. After these changes were made, anhydrite precipitation rates of 0.03 to 0.04 g/g_{seed}-hr were achieved. These anhydrite precipitation rates in the flow-through reactor compare favorably with gypsum precipitation rates of about 0.06 g/g_{seed}-hr at typical FGD forced oxidation conditions.

The effect of pH and iron (a known modifier of crystal surface reactions) addition were examined in an attempt to increase the anhydrite formation rate. The effect of pH was negligible. The iron addition decreased the anhydrite precipitation rate compared to runs without iron that were

conducted under otherwise similar conditions. Apparently iron affects the anhydrite surface reaction but in a deleterious manner.

The final two runs were conducted at two different temperatures with substantially higher feed stream calcium concentration to determine if the precipitation rate could be further increased. The experiment conducted at the higher temperature showed that a rate of 0.11 g/g_{seed}-hr was achieved. Analysis by IR indicated that no gypsum was formed. At a lower temperature, a rate of 0.8 g/g_{seed}-hr was measured, but the IR analysis indicated presence of substantial amounts of gypsum at the end of the run.

Pilot Results. The initial operating conditions specified did not produce anhydrite at the ECTC pilot unit. Many of the problems were associated with converting an existing reaction tank to the anhydrite reaction tank. To achieve the shorter residence times specified required the operating slurry level to be relatively low which increased the air rate required to achieve complete sulfite oxidation. Since one of the major heat losses associated with this process is the heat of vaporization of water vapor leaving with the spent oxidation air, the higher air rate prevented the target temperature from being reached. There were other heat losses as well, including inadequate insulation around the tank and lines to the pH measurement and sampling locations. Much of the first month was spent understanding and resolving the temperature limitations of the pilot anhydrite reactor.

Once the temperature issues were resolved satisfactorily, anhydrite was produced with varying degrees of success for the remainder of the pilot testing. Figure 8 shows how the percentage anhydrite changed as a function of time during the first 3 months of testing. For purposes of simplifying the presentation, Figure 8 considers the solids produced to be either gypsum or anhydrite. Actually, there were periods in which other hydrate phases was also observed, but the predominant phases were gypsum and anhydrite.
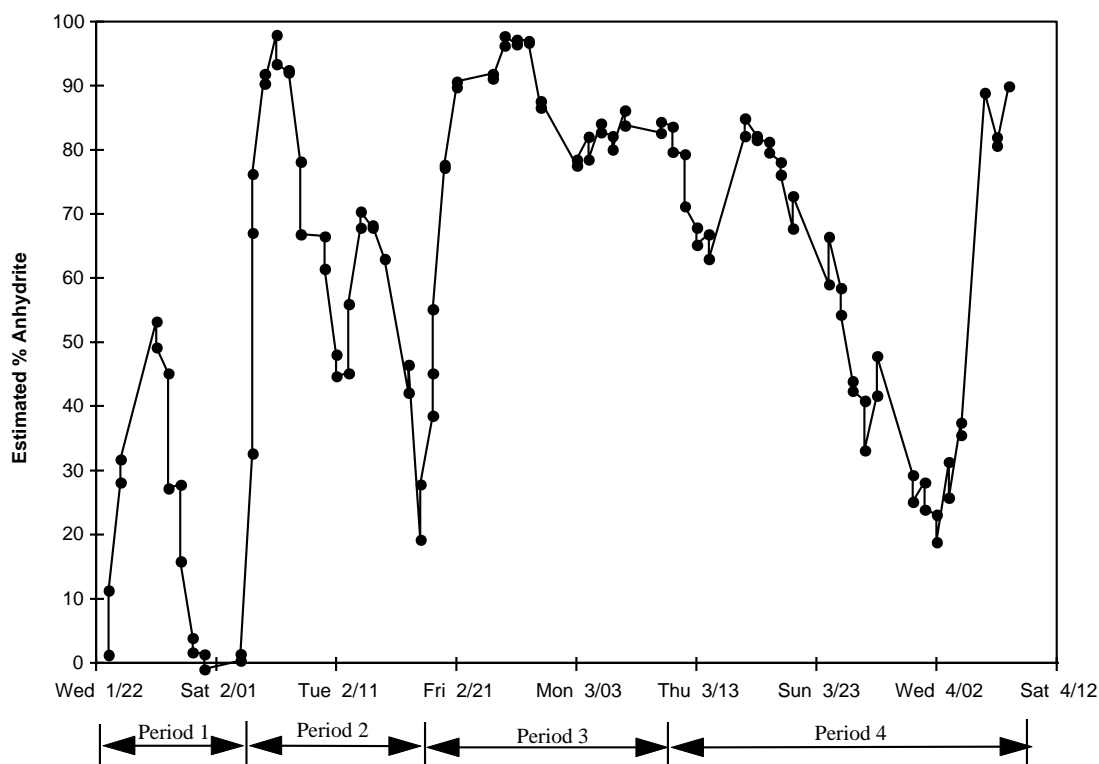

Figure 8. Anhydrite Content as a Function of Time

Period 1 was the first attempt to make anhydrite during which the temperature issues had to be resolved. The target temperature was not achieved until late January. During January, Figure 8 shows that some anhydrite was formed, but only a maximum of about 50%. Near the end of January the temperature dropped in the reactor and the anhydrite converted back to gypsum. Figure 9 shows the SEM results for the gypsum formed at the end of Period 1. An of the XRD scan showed the sample to be all gypsum.
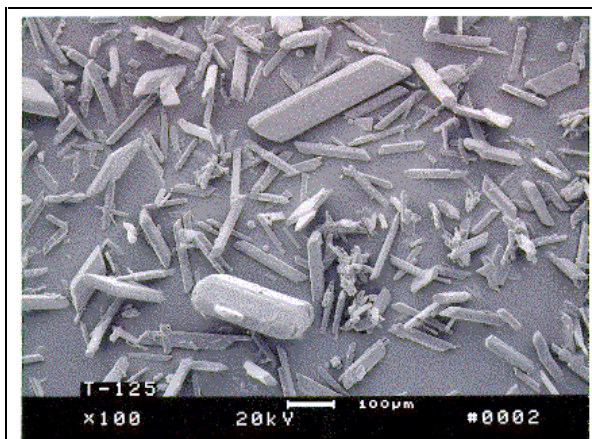
Following an excursion during which the oxidation sparger plugged in the anhydrite reactor, conditions conducive to the formation of anhydrite were achieved in early February. Figure 8 shows that significant amounts of anhydrite were produced in Period 2. XRD analyses showed the solids

Figure 9. SEM of Gypsum in Period 1

to be a mixture of anhydrite, hemi-hydrate, and gypsum during this period. The operating conditions during Period 2 were the same as during Period 1 but with more consistent temperature control with the better insulated tank. After cleaning the plugged sparger, the reactor produced solids that were over 90% anhydrite and hemi-hydrate for 3 days. The decrease in anhydrite content occurred simultaneously with minor changes in oxidation air rate and reactor pH. However, after returning to the previous conditions, the anhydrite content of the solids did not return to 90%. Figure 10 shows the SEM results for Period 2. The SEM shows that the solids had changed with thinner rods with more rounded ends observed. Semi-quantitative results showed about 25% of the solids were hemi-hydrate. The dewatered solids exiting the centrifuge became relatively hard within 24 hours. There was no significant temperature rise upon mixing the solids with water. These setting properties are consistent with anhydrite so the hemi-hydrate was apparently not reactive, probably because of its large particle size.

Following the drop in anhydrite content at the end of Period 2, it was postulated that anhydrite would form initially small crystals which would subsequently grow in size until the surface area was insufficient to maintain the gypsum relative saturation below 1. Once the gypsum saturation exceeds 1 by any significant amount, gypsum will begin to form rapidly because of its faster precipitation kinetics. It was therefore decided to control the anhydrite surface area through external means. The initial method of surface area control began on February 19 and concluded on March 11.
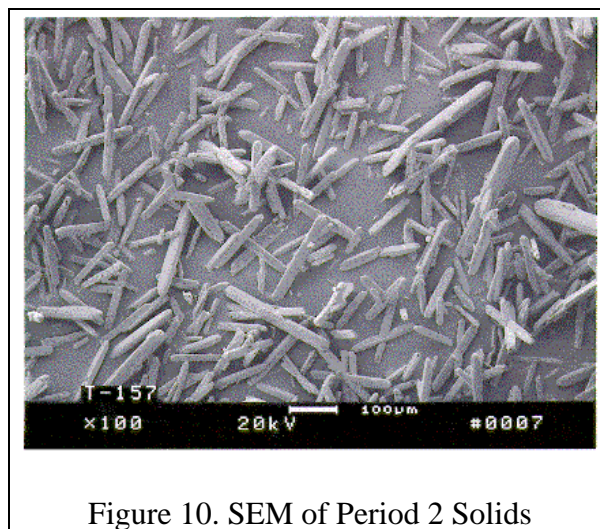
Figure 8 shows that the anhydrite content increased rapidly following initiation of anhydrite surface area control. The XRD of a sample taken

Figure 10. SEM of Period 2 Solids

on February 21 showed about equal amounts of hemi-hydrate and anhydrite with no gypsum. Over the next week, the solids gradually changed until they were mainly anhydrite with small amounts of hemi-hydrate and gypsum. The centrifuge product hardened upon cooling similar to the previous operation when anhydrite and hemi-hydrate were present.
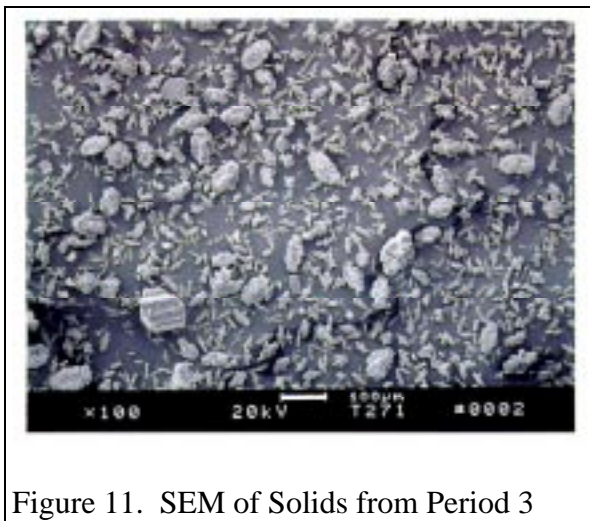
Figure 11. SEM of Solids from Period 3

Over a weekend, the characteristics of the centrifuge product changed dramatically. On March 3, these solids were hardening rapidly upon cooling and the solids were forming one large rock in the collection dumpsters used at the ECTC. When the dumpsters were emptied into the trucks for ultimate disposal, the solids were discharged in a single piece that did not break apart when colliding with the truck bottom as had been observed previously. The bound water measurement and the XRD analyses showed the solids to be 80 to 90% anhydrite with the remainder gypsum. These characteristics were observed until the end of the period when method of surface area control had to be changed. Figure 11 shows the SEM results from March 7 (end of Period 3). The XRD shows a majority of the solids to be anhydrite with small amount of gypsum. The SEM shows a change in crystal size and shape with smaller crystals that appear to be "stacked platelets" under the visual microscope.

Materials and equipment limitations necessitated a change in the method used for anhydrite surface area control on March 11. Over the next two days, the anhydrite content decreased from 85% to 65% on March 14. The centrifuge solids appeared to be drier and did not set as quickly or as hard. Experimentation with methods of surface area control continued throughout March. Figure 8 shows a gradual decrease in the anhydrite concentration through the latter half of March. The only exception was on March 17 when samples taken 17 showed 82 to 85% anhydrite, and the centrifuge product had returned to its more reactive properties. The surface area control method used at this time could not be maintained for extended periods of time with the equipment on site. The anhydrite content dropped to as low as 33% during this period. Figure 12 shows the SEM of solids produced toward the end of Period 4. The XRD results indicated a large amount of gypsum with some anhydrite. The SEM shows some very large gypsum crystals as well as some of the finer anhydrite crystals.
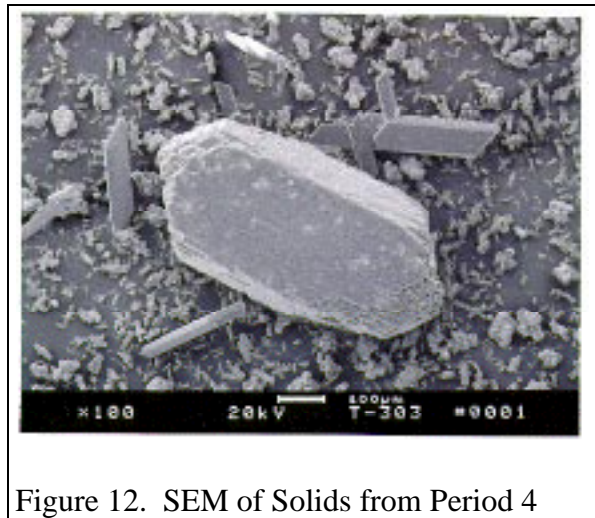
A successful method of control anhydrite surface area was re-established in late March, and the anhydrite concentration increased to 90% by mid-April. Properties of the anhydrite product returned to those observed during Period 3.

Figure 12. SEM of Solids from Period 4

Application

Prior to beginning the pilot anhydrite test program at the ECTC, a preliminary market survey to identify the most promising uses of anhydrite was conducted. Perhaps the most attractive alternatives include the self-leveling floor and aggregate markets.

Self-Leveling Floors. The use of anhydrite in Europe for self-leveling floor screeds is successful and growing. Some of the earliest use has been in Germany and the Netherlands[i]. There are some significant differences between the production of anhydrite by one of leading European processes

and our FGD solution precipitation method. The European method heats FGD byproduct gypsum to 800°C by a series of cyclones that contact hot air (produced through combustion of natural gas, oil, or coal) with the gypsum in a countercurrent manner. During the process, the gypsum is first dried, then converted to hemihydrate, and ultimately to anhydrite as the solids reach 800°C. The product crystals have a limited internal pore volume, probably as a result of fusion at high temperature. The product anhydrite is pulverized to obtain the desirable fluid properties.

The FGD anhydrite solution precipitation process should be much more thermally efficient than the Dutch process. This is major advantage since fuel would be expected to be a major operating cost in the Dutch process. On the other hand, the anhydrite produced in the Dutch process may be more reactive and therefore suitable for the self-leveling floor application. However, FGD precipitated anhydrite should have reactive characteristics similar to synthetic byproduct anhydrite from the hydrofluoric acid manufacture. This material has been successfully employed in the application of self-leveling floors. The Canadians have examined a process to produce self-leveling floors from natural anhydrite produced in Nova Scotia.[ii] Application of FGD anhydrite in self-leveling floors is one goal of Phase II of the PRDA project.

Synthetic Aggregate. The insoluble calcium sulfate anhydrite ($CaSO_4$) solids produced as a byproduct from wet lime or limestone FGD systems will, upon rehydration, "set up" to form a coherent, hard mass. This property, which is not characteristic of other byproduct options such as calcium sulfate dihydrate (gypsum), may yield potential market opportunities for the anhydrite byproduct option. In surveying the potential market for anhydrite as a synthetic aggregate, an existing commercial operation was identified in which more than 500,000 tons per year of anhydrite waste from hydrofluoric acid production is being converted to aggregate and sold as a substitute for crushed stone. This anhydrite product is being used by a number of municipal and county agencies as well as private companies for construction fill and road base material. Assuming that this material has similar properties to those of FGD anhydrite, there appears to be a large potential market for this material in or near urban areas where conventional crushed-stone aggregates are in short supply.

There does not appear to be any potential for using byproduct anhydrite to produce aggregate for use in concrete. In this use, sulfate is known to react with concrete, causing undesirable expansion and cracking.

Future Activities

Currently a final report on this project is being prepared and a plan to optimize the anhydrite process and test its use in potential markets is being developed.

Acknowledgments

[i] Kappe, J., "High Quality Anhydrite from Flue Gas Desulphuization Gypsum," Environment and Technology, November 1991.

[ii] "Anhydrite Self-Leveling Flooring Screed: Final Report," Microlog (233440-1-9044-01-SQ.)