OST TECHNICAL PROGRESS REPORT
TEAM WORK PLAN--FY 1998 RESULTS

TITLE: Solid Fuels Formation Team

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DESCRIPTION: This report covers two projects in the areas of solid fuels dewatering and reconstitution. These projects are:
(1) GranuFlow Process Improvement
(2) Fundamentals of Dewatering Theory

RESEARCH OBJECTIVES: The overall research objectives for the first project were to improve the efficiency of the GranuFlow Process through several fundamental issues and the formulation of domestic emulsions, and to conduct a commercial plant test at the Terry Eagle Coal Preparation Plant in West Virginia. The overall research objectives for the second project were to explore a fundamental concept of dewatering of solid fuels through the thermodynamics of water (pore water and interstitial water) located between closely-spaced solid surfaces, and to explore the potential of dewatering of solid fuels at temperatures considerably below the normal boiling point of bulk water (100°C).

LONG TERM GOALS/RELATIONSHIP TO FETC'S PRODUCT LINE(S): The long-term goals of this team are (1) to fulfill the Solid Fuels and Feedstocks product line's objective of directly improving carbon recovery, dewatering, reconstitution, and handling of fine-sized coal, and (2) to help solve national energy and environmental problems.

(1) GranuFlow Process Improvement: When the GranuFlow Process becomes commercialized, it will accomplish the above long term goals in the following ways: (i) increased amounts of fine coal can be added to utility plant feedstocks without creating handling problems, (ii) the top size of coal fed to a preparation plant can be reduced to take advantage of increased liberation to improve the quality of the clean-coal product, (iii) coal fines (valuable fuel) can be reclaimed from waste ponds, thus extending the life of the pond, and (iv) handleability during transportation of coal can be improved by alleviating dust and freezing problems.

Coal preparation is used to upgrade as-mined coal before it is utilized by the end user, primarily electric utility power plants. It reduces the amount of noncombustible mineral impurities, sulfur-bearing minerals, and hazardous trace elements, and generally produces more uniform, higher energy content fuels. Most conventional and advanced coal cleaning processes, however, involve the use of water. Dewatering of the fine clean coal to a low moisture level is often a problem. Utility companies are concerned not only with the lower Btu content of the resulting wet, cleaned coal but more importantly with its handleability problems (stickiness, dustiness, freezing). Both moisture and handleability problems are exacerbated in the portion of the coal with the smallest particles, the fines. As a result, most utility coal contracts limit the amount of moisture and the quantity of fines in the final coal shipments. These contractual constraints pose difficulties for coal preparation plants in processing the fines and meeting the specifications to include them in the final product, and thus in many cases the fines are simply thrown away into slurry waste ponds. Today, it is estimated that 2-3 billion tons of fine coal exist in about 5000 ponds in the U.S. To alleviate the problems of fine coal processing and enhance the utilization of fines by utilities, DOE's Federal Energy Technology Center (FETC) developed and patented the GranuFlow Process.

This project featured a concept that combines fine-coal dewatering and reconstitution into one step. The process aimed at improving fine-coal handleability and reducing product moisture content. It minimizes coal losses and dust emissions during transportation, handling, and storage, and produces an economically reconstituted fine-clean-coal product that is in a free-flowing granular form and is easy to handle. The process requires the addition of a small amount of a specially selected binding material (Orimulsion) to the fine-clean-coal slurry before filtration or centrifugation. The process was scaled up and tests were conducted at FETC in both bench-scale screen-bowl and high-g force solid-bowl centrifuges. The pilot-scale and plant-scale tests were also successfully completed in FY1996 and FY1997, respectively. DOE granted an exclusive license agreement for commercial use of the process to CQ Inc. in FY1998.

(2) Fundamentals of Dewatering Theory: The fundamental dewatering theory contributes to the above long term goal by: (i) reducing the amount of energy necessary for dewatering solid fuels (particularly, low-rank coal) thereby reducing the costs of dewatering; (ii) reducing the cost of transporting the moisture that would otherwise accompany solid fuels; (iii) increasing the BTU value of solid fuels; and (iv) minimizing the problems associated with the freezing of solid fuels in rail cars during cold weather.

The realization, from literature data, that the physical properties of water located between closely-spaced solid surfaces ("vicinal water") exhibit unique temperature dependence, when compared with those of bulk water, led to the current work. Specifically, the physical properties of vicinal water (e.g., pore water or interstitial water) have been reported to undergo "thermal anomalies," whereby they change abruptly *within specific temperature ranges* (14-16, 29-32, 44-46, and 59-62 degrees Celsius), almost irrespective of the type of physical property being measured or of the kind of solid material . An examination of the literature data on "thermal anomalies" led to the development of a theory, which suggested that solid fuels could be effectively dewatered at temperatures considerably below 100°C. This suggestion gave birth to the present project, which involved applications of the theory, and refinements thereof.

Dew point hygrometric data (moisture levels) and flow calorimetric data (heats of water sorption and desorption) were simultaneously collected, as functions of time and temperature, during the hydration and subsequent dewatering of a single pulverized coal sample. As a necessary step in quantifying the dewatering of the coal, similar experiments were also performed on the empty sample cell of the calorimeter, which was situated upstream from the dew point hygrometer. These data were collected both at various temperatures between 30 and 100°C (isothermal conditions) and under temperature-programmed conditions, whereby the coal sample was heated from 30 to 100°C at various rates. The amount of water removed from the coal during dewatering was calculated from the difference between the area bounded by the hygrometer signal for the coal sample and the area bounded by a theoretical curve for the empty sample cell.

SUMMARY ACCOMPLISHMENTS:

(1) GranuFlow Process Improvement:

- ***Collaborated with CQ Inc. toward the commercialization of GranuFlow Process***: A license agreement for two of the GranuFlow-related U.S. Patents was signed between FETC and CQ Inc. on October 16, 1997. The Solids Processing and Separations Division is preparing a CRADA with CQ Inc. for their continuous consultation and assistance during the commercialization efforts.
- ***Demonstrated the applicability of the GranuFlow Process to coal waste pond fines for the Greenfields Energy:*** The GranuFlow Process was tested on flotation concentrate prepared from the coal waste pond materials. Outstanding product moisture reduction was obtained from 29.8 to 14.6 wt% with an Orimulsion addition at 4.6 wt%.
- ***Completed plant-scale demonstration tests at the Terry Eagle Coal Preparation Plant in West Virginia***: Plant scale demonstration tests were successfully completed in FY1997. There were two presentations and 3 publications in 1998 from this work.
- ***Consultation with outside parties on the GranuFlow related commercialization projects***: Meetings with Consolidation Coal Company (CONSOL), American Electric Power Company (AEP), and CQ Inc. were held to further commercialize the GranuFlow process.
- ***CRADA with Russell Standard Corporation***: A CRADA with Russell Standard was signed in March 1998 to help develop modified domestic asphalt emulsions for use in the GranuFlow Process.
- ***CRADA with Mineral Technologies International Inc. (MTI)***: A CRADA was signed in May 1998 in support of a Phase II SBIR Grant to demonstrate a novel pilot-scale process system which integrates the high performance of the MTI packed column flotation cell with the dewatering ability of the GranuFlow Process.
- ***Collaborated with Monsey-Bakor***: Domestic emulsions to make the GranuFlow Process more economic have been identified by Monsey-Bakor and tested at FETC.

(2) Fundamentals of Dewatering Theory:

- A theoretical model explaining the "thermal anomalies" of water located between closely-spaced solid surfaces was developed on the basis of unique "temperature-domain" calculations. One application of the above-mentioned model resulted in the development of a theoretical empty-cell curve with which the sorption and desorption of water to and from porous solids can be quantified by means of dew point hygrometry under either isothermal or temperature-programmed conditions. This accomplishment could potentially elevate the status of the dew point hygrometer.
- Another application of the theoretical model established the first known linkage between water data from two entirely different kinds of experiments and two widely different energy realms. Specifically, linkage was found between dew point hygrometer data collected during the present investigation, showing how water behaves when removed from a metallic frit, and literature data describing the thermal behavior of the disjoining pressure of water located between closely-spaced silica plates. Thermodynamic calculations indicate the hygrometer data belong to the "*macrodynamic*" energy realm, while the disjoining pressure data belong to the "*mesodynamic*" energy realm. Because of the above-mentioned linkage, it is envisioned that the theoretical

model of "thermal anomalies" developed during the present project could be applied to ANY case, in which water is located between closely-spaced solid surfaces.

- Finally, it was discovered while heating samples from 30 to 100°C that there is a threshold heating rate (1.5±0.3°C/minute), beyond which there is a significant increase in the amount of water removed from solid fuels (in the present case, *coal*) during dewatering. This threshold heating rate, which manifests itself through abrupt and significant changes in both calorimeter signals and dew point hygrometer curves, can be explained entirely by means of the above-mentioned theoretical model for "thermal anomalies."

RESULTS:

(1) GranuFlow Process Improvement

GranuFlow Process feasibility/handleability work (CONSOL/AEP) During early FY1998, FETC began cooperative work with CONSOL in an attempt to determine if FETC's patented GranuFlow Process might improve the handleability characteristics of CONSOL's Pocahontas seam coal sufficiently so that AEP could make use of this coal as a compliance coal. CONSOL R&D provided FETC with Pocahontas coal slurry from their Buchanan, Virginia, preparation plant, along with detailed information about their current processing and marketing of this coal. Upon receiving the Pocahontas coal slurry, a baseline characterization was performed, including ash content, particle size distribution, and solids content, and then the slurry was used in a variety of tests directed toward two primary goals: 1) to characterize the performance of the GranuFlow Process as applied to this type of coal, and 2) to evaluate the effect of the GranuFlow Process on the flowability of this coal. Past studies of the handling and combustion characteristics of GranuFlow-treated coal were reviewed; CQ Inc. was consulted regarding their experience with coal handleability evaluation; and Bob James of FETC's Combustion Engineering Research Facility (CERF) was consulted to ensure that our investigation of coal handleability would proceed along practical lines.

The characterization of GranuFlow performance with the Pocahontas coal began with an extensive battery of laboratory-scale vacuum filtration tests. Both a computer-interfaced laboratory filtration apparatus and a larger-scale bench top Denver press filter at the FETC GranuFlow Process Development Laboratory were used in these tests. In addition to determining the effect of varying bitumen dosages on filter cake moisture content and dust index, the interaction between a bitumen dosage and a number of other process variables was examined. The results of these tests indicated that the GranuFlow Process could lower filter cake moisture content from about 20% to as low as 12% (during static filtration to an equilibrium moisture content), and could lower filter cake dustiness by more than 90% using Orimulsion and Monsey-Bakor emulsions. The high solids content of the Buchanan Plant slurry was found to have no effect on GranuFlow performance, whereas both the intensity/duration of treated slurry mixing and the aging of treated slurry were found to have significant effects. Filtration tests with this slurry revealed the presence of an unidentified variable that can make the difference between either attaining a substantial moisture content reduction or attaining no moisture reduction at all. Two series of tests seemed to link this variable to the slurry flocculent content, and therefore to the surface chemistry of the slurry coal particles, but subsequent tests with different slurry samples failed to repeat those results.

GranuFlow performance was also characterized in tests using a 14-inch high-g force solid-bowl centrifuge at FETC's Solids Processing Research Facility (SPRF) and a 6-inch screen-bowl centrifuge in the GranuFlow Process Development Laboratory. The solid-bowl centrifuge test, which was the largest-scale performance test conducted with the Pocahontas slurry, showed very substantial reductions in dewatered product moisture and dust index. With addition of bitumen at a dosage of 9 wt%, product moisture decreased from 16.7% to 9.8%, and product dust index decreased by over 70%. In the screen-bowl centrifuge test no significant reduction in product moisture was attained with bitumen addition, although the product dust index did decrease by 92% with bitumen addition at a dosage of 5 wt%. The reason for the absence of a moisture effect with the screen-bowl was not apparent, but it might possibly be related to an inadequacy in mixing intensity during the addition of the bitumen.

In order to evaluate the influence of the GranuFlow Process on fine coal handleability aside from its effect on moisture content, it was decided to have samples of GranuFlow-dewatered coal tested by H. Colijn & Associates, an outside lab that specializes in this type of testing. This decision was made following its original suggestion by CONSOL R&D, and subsequent discussions with CQ Inc., AEP, and the local H. Colijn & Associates testing engineer. The overall plan was to compare the flowability of the final, thermally-dried product of CONSOL's Buchanan coal preparation plant with analogous samples of GranuFlow-dewatered coal. Since only the minus 28 mesh fraction of the Buchanan plant coal is vacuum-filtered (this was the size fraction contained in the slurry obtained from CONSOL), this fraction was dewatered with GranuFlow and was blended with the proper portion of coarser material to produce the analogous GranuFlow samples. CONSOL R&D screened samples of the Buchanan thermally dried product and the Buchanan plant plus-28 mesh basket centrifuge material to minus 8 mesh, as was required for the flowability test method, and provided these samples to FETC. A Denver press was used to prepare GranuFlow-dewatered filter cakes at 0, 2, and 4 wt.% bitumen dosages using Orimulsion, and the hi-g force solid-bowl centrifuge in the SPRF was used to prepare GranuFlow-dewatered product at a 5.8 wt.% bitumen dosage. The filter cake and centrifuge product materials were then combined with plus-28 mesh material from the Buchanan plant in a dry weight ratio matching that prevailing at the Buchanan plant. The resulting mixed samples were submitted for flowability testing to H. Colijn & Associates at the close of FY1998.

Small-scale laboratory tests were performed on CONSOL Pocohontas coal to see the effect GranuFlow may have on the dewatering of this coal. Tests were run several times this year to support subsequent larger-scale testing of Pocohontas coal with GranuFlow. Results are listed in the following table.

Orimulsion added, %	Moisture content, %
0	19
2	14.4, 17.8
4	14.3, 15.6
6	13.6, 12.3
8	11.1, 10.6

Since a noticeable and repeated difference was noted between untreated and Orimulsion-treated coal samples, further testing was executed, including extensive handling tests.

Plant-scale demonstration tests at the Terry Eagle Coal Preparation Plant in West Virginia During FY1998, a plant-scale test of the GranuFlow process was conducted using two 36-inch (900 mm) Bird screen-bowl centrifuges for the dewatering and reconstitution of fine clean-coal slurry at AMVEST Coal Company's Terry Eagle Coal Preparation Plant in West Virginia. The plant capacity was about 425 tph of run-of-mine coal. The centrifuges were dewatering 28 mesh (600μm) x 0 fine clean-coal at a feed rate of about 40-50 t/h. This centrifuge feed consisted of the 100 mesh (150μm) x 0 froth flotation concentrate at about 12-15 t/h, and the 28 mesh (600μm) x 100 mesh (150μm) classifying cyclone underflow at about 28-35 t/h. In this test, Orimulsion was added into the froth flotation concentrate at varied dosages, and this stream was then mixed with the cyclone underflow before being fed into the centrifuges for dewatering and reconstitution. Test results indicated that the average moisture contents of the dewatered coal were 25.6, 25.0, 25.3, 22.2, 23.8 and 21.3 wt% with Orimulsion additions of 0, 0.7, 1.3, 2.3, 3.8 and 5.5 wt%, respectively. The handleability, dustiness, and recovery of the dewatered coal product were also improved.

***Application of GranuFlow Process to flotation-cleaned waste pond fines* (Greenfield Energy/Antaeus)** A study being conducted in the SPRF on the feasibility and logistics of using a Microcell froth flotation column to clean fines from the Greenfield Energy's Alpheus waste pond in West Virginia provided the opportunity to evaluate the application of the GranuFlow Process to the dewatering of the resulting flotation concentrate. The evaluation was conducted using the 14-inch high-g force solid-bowl centrifuge in the SPRF and also using a 6-inch screen-bowl centrifuge in the FETC GranuFlow Process Development Laboratory.

The solid-bowl centrifuge tests showed substantial reductions in dewatered product moisture and dust index, and centrifuge effluent solids. With addition of bitumen at a dosage of 4.6 wt%, product moisture decreased from 29.8% to 14.6%, product dust index decreased by 86%, and effluent solids decreased from 440 to 220 ppm. In the screen-bowl centrifuge test, no significant reduction in product moisture was attained with bitumen addition, although the product dust index decreased by 92% and the screen effluent solids content dropped from 46 to 17 wt% with bitumen addition at a dosage of 6 wt%.

Exploratory/theoretical work and methods development An effort was made to improve and expand the equipment and methods used for the evaluation of the vacuum filtration of fine coal. Potential sources of error in tests conducted using the computer-interfaced laboratory filtration apparatuses were identified and their limits were assessed. Denver press filtration was adapted for controlled dewatering by incorporating a plastic film liner to prevent vacuum leakage upon cake contraction. A bench-scale drum vacuum filter was reconditioned and then operated to determine if it could be used to mimic the dewatering performance of the disc filters used at CONSOL's Buchanan coal preparation plant. Performance with untreated Buchanan plant slurry at different drum rotation speeds was characterized, and it was found that the slowest possible drum speed (14 rpm) duplicated the 20% moisture content of the disc filters. However, when bitumen-treated slurry was used with the filter, insurmountable clogging problems were encountered. In future work with the drum filter, a different feed pump will be used, and the slurry solids concentration will be limited so that bitumen-treated slurries can be used.

An ongoing effort to simplify the interpretation and characterization of vacuum dewatering rate curves through mathematical modeling continued. Instead of modeling the entire rate curve, the individual segments pertaining to the draining of free water and the removal of interstitial water were examined independently. This technique may allow the calculation of separate cake resistances for different filtration conditions and might provide better insight into filtrations in which moisture content changes do not correlate significantly with filtration rates. The increasing database of laboratory-scale filtration results provided an enhanced opportunity for improving the data analysis methods.

Additionally a thermistor grid was fabricated to permit the monitoring of filter cake temperature during filtrations run using the computer-interfaced laboratory filtration apparatus. Using this capability, a series of filtration tests was performed to characterize the temperature profile of filter cakes during the filtration of slurries heated to different temperatures. This preliminary experimentation precedes a proposed study to determine the effects of temperature on the vacuum dewatering of coal.

GranuFlow improvement – domestic emulsion Domestic emulsions were tested to evaluate their ability to dewater fine coal. A domestic emulsion is needed to replace the Orimulsion from Venezuela that is expensive to transport. A cheaper domestic source of emulsion would make the GranuFlow process much more marketable as an inexpensive way to process and recover fines, reduce dustiness, improve handleability, and keep the coal from freezing.

Five domestic emulsions manufactured by Monsey-Bakor were tested with Pocohontas coal in laboratory-scale tests. Two of the emulsions resulted in higher moisture contents of the coal cakes. The other three emulsions (Emulsions1-3) produced coal cakes that had moisture contents comparable to or lower than the Orimulsion treatments. Moisture contents for each treatment are listed in the table below.

Treatment	Moisture content, %
Untreated	15.1
5% Orimulsion	13.6
5% Emulsion 1	12.6
5% Emulsion 2	13.9
5% Emulsion 3	12.3

Larger scale dewatering tests were run with these experimental emulsions. Denver press test results with Pocohontas coal produced the following moisture content for each treatment:

Treatment	Moisture content, %
Untreated	21.8
4% Orimulsion	17.6
4% Emulsion 1	16.4
4% Emulsion 2	16.8
4% Emulsion 3	16.4

There is a definite reduction in moisture content in the coal cakes produced with the experimental emulsions. Dust index tests were run on the Denver press samples, and dust was reduced by each emulsion treatment as compared to untreated coal as follows: Orimulsion, 88%; Emulsion 1, 84%; Emulsion 2, 59%; Emulsion 3, 90%.

A CRADA with Russell Standard Inc. signed during FY1998 will lead to further testing of domestic emulsions.

Microscopic studies of coal cakes Coal cakes formed by the laboratory dewatering device were examined microscopically. Under the stereo microscope, coal treated with Orimulsion and other emulsions appeared similar. Untreated and treated coal cake samples were epoxyed and polished for further examination. Something appeared to leach out of the treated coal samples when emersion oil was used in the analysis. No such effect was noted in the untreated samples. Photographs of the samples show small, round "snakes" oozing out of particular areas of coal. These "snakes" may be the emulsions being extracted from the coal by the oil. The patterns of these "snakes" can show where emulsions are present in the coal samples. More work in microscopic analysis may help identify the regions within the coal cake where the emulsion adheres.

Presentations/Publications

[1]. Yang, D.C., W.W. Wen, M. Zeng, "An Integrated Process for Recovery of Coal Fines from Waste Streams Using the Packed Flotation Column." Preprint 98-125 SME Annual Meeting, Orlando, Florida, March 1998.

[2]. Wen, W.W., R.P. Killmeyer, "The Application of the GranuFlow Process to Centrifuge Dewatering at the Terry Eagle Plant." Proceedings 15th International Coal Preparation Conference. Lexington, Kentucky, April 1998.

[3]. Wen, W.W., R.P. Killmeyer, "Field Testing and Commercialization of the U.S. Department of Energy's GranuFlow Process." Proceedings XIII International Coal Preparation Congress Conference, Brisbane, Australia, October 1998.

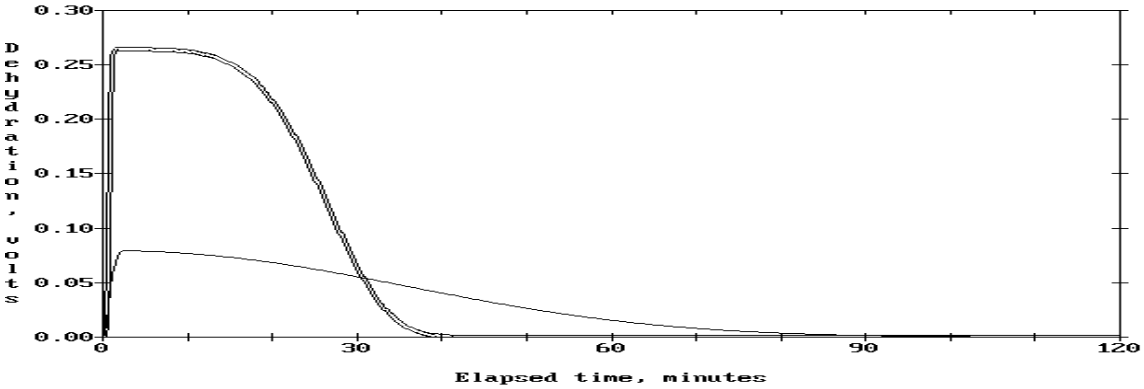
[4]. Parekh, B.K., J.G. Groppo, O. Tao, W.W. Wen, R.P. Killmeyer, "Pilot Scale Dewatering Studies of Ultra-fine clean coal." Proceedings XIII International Coal Preparation Congress Conference, Brisbane, Australia, October 1998.

[5]. Wen, W.W., "An Integrated Fine Coal Preparation Technology: The GranuFlow Process." Manuscript submitted for publication, International Journal of Mineral Processing, 1998.

(2) Fundamentals of Dewatering Theory:

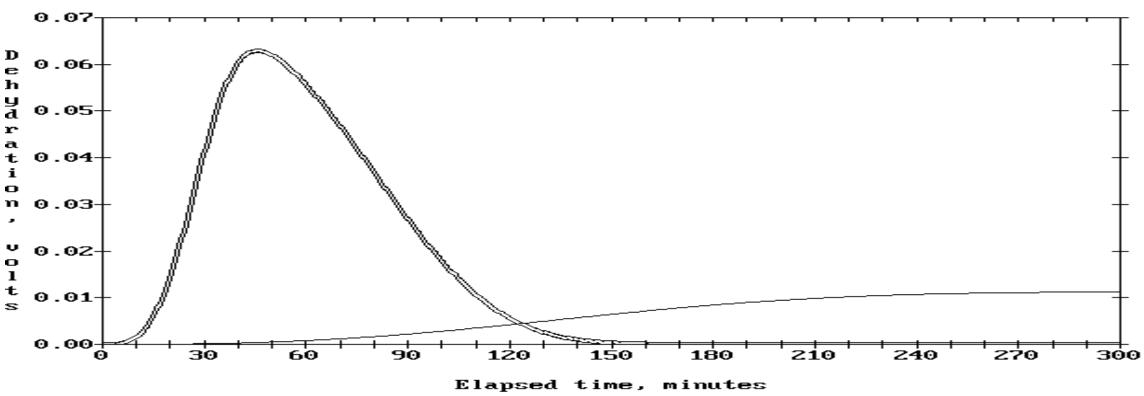
The theoretical model for "thermal anomalies" developed during the present investigation has enabled the hygrometric quantification of water removed from porous solids during a dewatering process. Moreover, this model has made it possible to analyze the water removed into two different categories: (1) **Site 1 water**-- a mixture of surface water and "intermediate water," consisting of all of the interstitial water and perhaps some pore water, and (2) **Site 2 water**--"deep" water, which consists of most, if not all, of the pore water.

The following figure shows coal dewatering curves, based on the above-mentioned theoretical model, *for Site 1 water.*



The double-lined curve represents dewatering of the coal sample from 30-100°C at a heating rate of 3.2°C/minute, which exceeds the threshold rate (1.5±0.3°C/minute), beyond which there is a significant increase in the dehydration level. The single-lined curve represents dehydration of the same sample at 30°C (isothermal conditions). Below the threshold heating rate, the amount of water removed from the coal is similar to that shown above for isothermal conditions. Hence, the above figure demonstrates that a significant advantage can be gained by exceeding the threshold heating rate. It is important to note, however, that without the calculated empty-cell curve based on the theoretical model developed during the present project, comparisons of the type shown in the above figure would be virtually impossible.

The following figure shows coal dewatering curves *for Site 2 water*.



Once again, the double-lined curve represents dewatering from 30-100°C at 3.2°C/minutes, while the single-lined curve is based on dehydration at 30°C. Similar to the Site 1 water results, the graph for Site 2 water shows that there was a significantly greater amount of water removed from the coal under the temperature-programmed conditions than under the isothermal conditions. In fact, the dewatering of Site 2 proceeded at an ex*tremely slow* rate when the temperature was fixed at 30 degrees.

Comparison of the areas bounded by the curves in the above two figures reveals that about twice as much water was removed from Site 1 under temperature-programmed conditions (30-100°C at 3.2°C/minute) than from Site 2 under the same conditions. This is consistent with the interpretation that Site 2 water is "deep" water and is therefore extremely difficult to remove. In fact, no significant amounts of Site 2 water were removed from the coal at heating rates below the threshold value.

In general, all of the questions that existed at the start of the project were answered. It was learned that knowledge of the thermodynamics of water located between closely-spaced solid surfaces can be applied toward more effectively removing water that, traditionally, has been extremely difficult or virtually impossible to remove from solids, such as coal. It was also determined that knowledge of such thermodynamics can be applied to dewatering solid fuels at temperatures considerably below 100°C. Theoretical calculations show that the enhanced removal of Site 1 water *began at 30°C, as long as the heating rate exceeded the threshold value*. Finally, it was learned that solid fuels can be effectively dewatered in the absence of chemical additives. *No* chemical additives were used during the present investigation. These results and the thermodynamic hypothesis pertaining to dehydration in solids are being summarized in a manuscript to be submitted for publication in the Journal of Chemical Physics.

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For additional information pertaining to the GranuFlow Process Improvement, please contact Wu-Wey Wen. For information on the Fundamentals of Dewatering Theory, please see Kenneth Jones.