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CONTROL TECHNOLOGY FOR IN-SITO OIL SHALE RETORTS, JUNE MONTHLY REPORT

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July 9, 1980

TO: Charles Grua, Brian Harney, and Art Hartstein
FROM: Peter Persoff, Joe Ratigan, Bill Hall, Mohsen Mehran, and Phyllis Fox
RE: June Monthly Report
Control Technology for In-Situ Oil Shale Retorts
LBID-250

TASK 3. BARRIER OPTIONS

Development of cementitious properties in spent shale

The experimental program of heating spent shale without any added material under controlled atmospheres is progressing. A system to deliver 100 percent steam to the tube furnace has been assembled. Lurgi spent shale or dolomite (as a control) has been heated in flowing steam, flowing N₂ gas, or evolved CO₂. X-ray diffraction patterns have been taken on the products. A qualitative test of cementing, suitable for use with small quantities of material, will be devised and used to determine whether these treatments produce or enhance cementing properties.

Testing of grouted core samples

Unconfined compressive strength has been measured on duplicate specimens of all candidate grouted cores. Results of these tests are summarized in Table 1. The specimens were 2-inch diameter by 4-inch long cylinders consisting of simulated in-situ spent shale and grout. The grout solids were 2/3 Lurgi spent shale and 1/3 sand, plus small amounts of added cement, as shown in Table 1. Details of the method of sample preparation are contained in the monthly report for February (LBID-177). Samples were cured under moist conditions at 73 ± 2°F for the length of time indicated and tested with a slow loading rate. Good reproducibility was observed for all duplicates except Q-5, for which another sample will be tested.
### Table I. Summary of Unconfined Compressive Strength Tests on Grouted Cores

<table>
<thead>
<tr>
<th></th>
<th>Q-0</th>
<th>Q-1</th>
<th>Q-3</th>
<th>Q-4</th>
<th>Q-5</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Grout formula</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water-Solids Ratio</td>
<td>0.587</td>
<td>0.620</td>
<td>0.607</td>
<td>0.678</td>
<td>0.818</td>
</tr>
<tr>
<td>Added portland cement, percent</td>
<td>0</td>
<td>2.5</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Added Lurgi spent shale cement, percent(^a)</td>
<td>0</td>
<td>0</td>
<td>2.5</td>
<td>5</td>
<td>10</td>
</tr>
<tr>
<td>Curing time, days (duplicate specimens)</td>
<td>68,69</td>
<td>69,82</td>
<td>84,101</td>
<td>103,103</td>
<td>102,102</td>
</tr>
<tr>
<td>Compressive strength, psi (duplicate specimens)</td>
<td>29,30</td>
<td>77,84</td>
<td>32,36</td>
<td>50,54</td>
<td>55,80</td>
</tr>
</tbody>
</table>

\(^a\) It should be noted that the Lurgi spent shale cement used in grouts Q-3, Q-4, and Q-5 was inferior to that reported in previous monthly reports. The limestone-spent shale ratio was 0.9 and the 28-day strength (ASTM C 109) was only 528 psi. This is lower than expected and may indicate poor process control during calcining. The best Lurgi spent shale cement produced in earlier experiments is much stronger and would contribute to a stronger grout.
Triaxial compressive strength tests were also run on all candidate grouted cores, with confining pressures of one and two times the unconfined compressive strengths measured in Table 1. A novel specimen capping technique was devised to produce specimens with flat parallel ends. Specimens were placed within a mold with flat, parallel end cavities into which hydrostone, a proprietary plaster, was injected. This obviated the necessity of trimming the caps, which could have weakened or damaged the specimens. Results and interpretations of these tests will be available next month.

Use of electrical conductivity measurements to monitor progress of saturation. It is important for accurate permeability measurements that specimens be fully saturated. Ordinarily, specimens are saturated by being submerged in de-aired water with a vacuum over the water for an arbitrary length of time. It is known that as saturation of a porous medium increases, the electrical resistivity decreases. It is proposed to use this fact to ensure that grout specimens for permeability measurements are indeed fully saturated. A device for measuring electrical resistance of specimens has been fabricated. The specimen is held between spring loaded contacts and a 10 hz AC signal is imposed upon the specimen and a reference resistance in series. The voltage drop across the reference resistor is compared to the oscillator signal voltage to calculate the resistance of the sample.

Three oven dried sandstone rods, 1.6 cm by 1.3 cm by 13.1 cm long, were placed in the saturating bath and periodically removed for weighing (the surface was patted dry for weighing) and electrical resistance measurement.

Figure 1 shows the increase in weight due to pore saturation of the sandstone rods as a function of time. This shows that about \(10^5\) seconds are needed for nearly complete saturation of sandstone. Figure 2 shows the change in electrical resistivity as a function of pore water. This shows that the decrease in resistivity can be used to monitor the saturation process.
A similar procedure will be used on grout specimens for permeability measurements. This should give the amount of time needed for saturation prior to permeability measurements. All permeability specimens will then be saturated for this amount of time or longer prior to measurement of permeability.

Availability of fly ash

Enquiry to the marketing agents for fly ash from the Craig (Moffat Co.) power plant indicates that 1000 to 1200 tons per day of fly ash could be delivered to tract C-a or C-b for $10 per ton. A sample of this fly ash has been obtained and will be tested as a grout ingredient. Fly ash samples from the Jim Bridger (Rock Springs) and Wyodak (Gillette) power plants in Wyoming have also been obtained and will be tested although these would be expected to cost more at the site.

Structural modeling of grouted retorts

A draft of the User Information Manual "SUBSID: A nonlinear, two-dimensional Finite Element Program for Static Evaluation of Mining Subsidence" has been completed. SUBSID is the program used for all of the subsidence calculations done to date in evaluating the effectiveness of grouted retorts of various strengths and stiffness. A draft report containing the results of previous subsidence calculations for various mine geometries and grouted retorts is presently in preparation.

TASK 5. LEACHING OPTIONS

Leaching of organics from spent shale

The batch study designed to measure the diffusion coefficient for total organic carbon (TOC) in spent shale continued. A slab of spent shale has been suspended in a stirred container of distilled water since May 5, 1980. The increase with time of TOC has been monitored. The diffusion coefficient of the TOC in the solid phase of the shale can be estimated
by following a method described by Crank (1975).

Results at this time are inconclusive. Measured TOC levels in the liquid do not show a gradual increase with time. Concentrations range from an initial 1.6 ppm to a maximum of 13 ppm. Recent measurements of TOC are generally lower than those taken earlier in the experiment. A rational analysis of the leaching mechanisms leads to the conclusion that the TOC concentration in the leachate should increase rapidly with time at first and then taper off as equilibrium is reached.

Possible reasons for the erratic results are the analytical methods used to determine TOC and a change with time of organics in the leachate due to biological activity or chemical reaction. Biological activity is unlikely since 3 ppm of silver sulfate was added to the leaching water as a bio-inhibitor. A new Beckman 915-B Total Carbon Analyzer, now being used for analysis of leachate samples, has a capability for reliable measurements of TOC in the 1 to 10 ppm range. This should improve the accuracy of TOC measurements in the lower ranges where many of the leachate samples lie.

Because of the importance of the TOC diffusion coefficient in the leaching and transport model previously described, the experiment is being repeated. At the same time, an attempt is also being made to develop the diffusion coefficient from the last two column leaching experiments.

Development of groundwater flow model

During the past months, parametric studies of dewatering by internal mine drainage were carried out for tract C-b. The results of these studies are being summarized in a report entitled "A Preliminary Investigation of Dewatering Scenarios for C-b Tract, Piceance Creek Basin, Colorado". The report will include a brief description of climatologic and hydrogeologic characteristics of the area, mine development and design, fundamentals of the program TRUST, sensitivity analysis of important parameters, results and discussion. The report is expected to be out by July 30, 1980.
Another series of parametric studies of dewatering by wells (as opposed to internal drainage) has been initiated. The program TRUST is being used to model constant-rate pumping from a multiaquifer system. The flow region is axisymmetric with the well located at the axis of symmetry. Special attention is paid to the seepage face where the well-bore potential drops below that of the surrounding medium. The approximate analytic solution of Kroszynski and Dagan (1975) is being used to check the validity of the numerical scheme.

REFERENCES


Figure 1. Increase in weight of sandstone rods with saturation time.
Figure 2. Decrease in electrical resistance of sandstone rods with increasing saturation.
Figure 1. Increase in weight of sandstone rods with saturation time.
Figure 2. Decrease in electrical resistance of sandstone rods with increasing saturation.
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