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PRELIMINARY TESTS USING A LASER
PARTICLE-SIZE ANALYZER ON GEOTHERMAL BRINE

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November 18, 1977

MASTER

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Prepared for U.S. Energy Research & Development Administration under contract No. W-7405-Eng-48.

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PRELIMINARY TESTS USING A LASER PARTICLE-SIZE ANALYZER ON GEOTHERMAL BRINE

ABSTRACT

A laser light-scattering particle-size analyzer was used for a two-day period under limited field conditions to observe the occurrence and growth of particles in geothermal brines. Tests were conducted under conditions where both pH and rate of cooling to room temperature strongly influence the size and nature of the particulate population, with a low (4.5) pH and rapid quenching favoring slow growth rates. A method for estimating particulate mass in time periods shorter than 6 h from quenching is presented. Some implications for sampling and solids analysis are discussed.

INTRODUCTION

Suspended solids in low-temperature (100°C) liquid effluents from geothermal facilities may seriously interfere with injection of the effluent into rock formations for disposal (see Fig. 1). Current gravimetric methods for determining the amount of suspended solids are extremely time-consuming (6 h or more), especially if the particles are difficult to filter. An instrument that could rapidly provide even relative value for suspended solids would greatly facilitate injection and waste treatment studies.

The instrument I selected for exploring this possibility is a Phototron particle counter manufactured by SPECTREX, Inc., which employs a scanning laser beam to measure the concentration of particles larger than a selected threshold.

INSTRUMENT DESCRIPTION

The theoretical relationship between the intensity of light scattered by colloidal particles and the size of the particles was developed by Mie¹ and Jobst.² For particles that are large with respect to the wavelength of the incident light, the intensity of the scattered light is proportional to the square of the particle radius.

Fig. 1. Test facility schematic. Brine is flashed in four successive stages from wellhead conditions to atmospheric pressure. Acid is added twice: after the first stage and after the second. [This configuration closely simulates the conditions at the Geothermal Loop Experimental Facility (GLEF) operated by San Diego Gas & Electric Co. near Niland, California.]

The incorporation of this principle into the design of a laboratory instrument is described in detail by Porter.³ Basically, the focus of the beam from a low-power laser is mechanically scanned through a 10-ml volume. The transmitted portion of the beam is absorbed, while that portion scattered through a near-forward angle (approximately 6.5 to 19°) falls on a photodetector. Associated electronics convert the photodetector output into pulses directly related to particle size. The pulses are then fed to a conventional threshold detector/counter or to a pulse-height analyzer. A novel feature of the instrument is that the laser beam is defocused when it passes through the sample container wall, thus being unaffected by reasonable quantities of dirt and other foreign matter on the container.

The containers I used for the brine sample analysis described in this paper were of a type commonly used for dispensing pharmaceuticals: clear glass, approximately 25 mm diam, 70 mm high. The containers were prerinsed with distilled water that had been filtered to be essentially free of particles >5 μ m and wiped externally.

TEST CONDITIONS

The particle counter was available for only two days at the Lawrence Livermore Laboratory test site near Niland, California. My intent was not to conduct a set of highly controlled tests but rather to determine whether the instrument had potential for us under field conditions and whether it should be considered for further investigation. Accordingly, I did not attempt to vary effluent samples in any systematic way; rather, I selected a range of samples that represent typical conditions encountered by experimenters and facility operators. The range of samples was further reduced by variations in test facility operating conditions over which I had no control.

FIRST-STAGE BRINE (UNTREATED)

Brine leaves the first-stage separator at nearly wellhead conditions of temperature and pressure (200°C at 180 psig) that hamper analysis. Quenching a sample to a more manageable temperature range (28 to 37°C) and placing it in the counter took 5 min. By then, the population of particles in the >2 μ m

range* was too high to be measured without dilution (in fact, the number of particles in the range 2 to 5 μm remained too high throughout the experiment to measure, even with dilution). Accordingly, I limited the measurements to particles $>5 \mu\text{m}$ and plotted the growth of four size ranges vs time for 1-1/2 h after sampling (see Fig. 2). After 2 h, turbidity had appeared, and I halted the test.

FIRST-STAGE BRINE (ACIDIFIED)

In the second experiment, I decided to test acidification of a sample to a very low pH (<1.0) as a technique for halting particle growth and allowing analysis of the brine in a "frozen" state.[†] My procedure was to withdraw a quenched sample of first-stage brine, conduct an immediate (5 min) particle-size analysis, and add 0.5 ml of 12 N HCL for a final 0.7 pH. I then reanalyzed the sample three times: immediately, after 1 h, and after 2 h. Results are shown in Fig. 3. The particle population changed very little, and the suspension remained stable for more than an hour.

I also attempted to analyze brine samples that were not quenched but only flashed to 105°C and held at 85°C. However, particle growth was so rapid that I was unable to obtain useful data after 20 min; by 30 min, the samples became visibly turbid. Following that, I considered combining dilution with acidification of brine samples held at higher temperatures but did not pursue the plan because of the press of time and because acidified fourth-stage brine became available for analysis.

FOURTH-STAGE BRINE (ACIDIFIED)

I obtained fourth-stage samples at 105°C and 0 psig at a time when acid was being injected between the first and second stage to maintain a pH of 4.5 in the fourth-stage effluent. Several samples were quenched to a temperature

* Figures given for particle sizes and size distribution throughout this report are highly qualitative; they are suitable only for relative comparisons — they are by no means absolute measurements.

[†] There has been some concern that such a technique may redissolve some silica; certainly carbonates and sulfides are dissolved by acidification.

Fig. 2. Unmodified separated brine quenched to 37°C and held at 27°C.

Fig. 3. Unmodified separated brine quenched to 37°C and held at 27°C.

of 36 to 37°C and held at about 30°C. Particle-size analyses were conducted immediately and after 1 h and 2 h. The results are shown in Fig. 4. (I cannot account for the odd behavior in the 20- to 30-μm range.) A second test under similar conditions was made over a 6-h timespan; the results are shown in Fig. 5. Suspensions remain clear to the eye throughout the measurements and, in fact, only a few ppm of solids are present.

A FLIGHT OF FANCY

I repeat the caution that the results of these tests — on the size distributions indicated by the instrument — are only qualitative and not suitable for rigorous comparison. Nevertheless, the data can be used to give estimates of mass distribution with size and total particulate mass per unit volume. As a speculative exercise, therefore, let us assume the particles are spheres with a uniform density of 2.1 g/cm³ (silica). Then the mass of solids per unit volume of suspension is given by

$$M = \frac{\pi}{6} D^3 N \rho. \quad (1)$$

For N in particles per cm³, D in μm, and ρ in appropriate units,

$$M = (1.1 \times 10^{-12}) N D^3 \text{ g/cm}^3. \quad (2)$$

Now assuredly the particles are not spheres and do not have a uniform density of 2.1 g/cm³. However, two interesting results are obtained by applying Eq. (2) to the data from the fourth-stage brine tests. As Fig. 6 shows, a pronounced shift occurs in the size range: most of the particulate mass resides toward >20 μm as early as 1 h after sampling. Further, as Fig. 7 shows, there is a remarkably pure exponential dependence of total suspended mass on time. Calculations repeated with suspensions held 17 h, however, do not conform to the curve of Fig. 7. This is probably because the calculation does not consider the large particles that constitute the major fraction of the mass. The technique seems worth pursuing, nonetheless.

Fig. 4. Fourth-stage brine at pH 4.5 quenched to 30°C and held at that level.

Fig. 5. Fourth-stage brine at pH 4.5 quenched to 30°C and held at that level.

Fig. 6. Fourth-stage brine at pH 4.5 quenched to 30°C and held at that level.

Fig. 7. Fourth-stage separated brine at pH 4.5 quenched to 30°C.

SAND COLUMNS AND CORES

While I was testing the particle counter, other experimenters were passing samples of fourth-stage brine at pH 4.5 through a sand filter and through a core sample from a typical injection formation. Since both the sand bed and the core contained about 10% calcite (CaCO_3), effluent pH had increased to about 5.8 by the time I obtained samples for testing. Results are shown in Figs. 8 and 9. The trend toward the production of small (2 to 10 μm) particles is apparent in both cases; however, the source of these particles is not known. The sand-column samples were immediately acidified to pH 1.0, which should rule out calcite as a source of small particles. Silica, decomposed micas, or clays are potential contributors, but further investigation is clearly needed.

IMPLICATIONS FOR SAMPLING AND SOLIDS MEASUREMENT

These few experiments have led me to conclude that gravimetric measurement of suspended solids in geothermal brine is extremely sensitive to the method of sampling, the time before analysis, and the temperature at which the sample is held. The best solution to the problem of particle growth seems to be filtering the sample at the time of sampling -- at the pressure and temperature of the process brine. Brine samples have been filtered under these conditions, but measurement accuracy is poor when solids concentrations are low. Moreover, it is difficult to take measurements when fluids are sampled at atmospheric pressure.

For consistent and meaningful results with conventional vacuum filtration techniques, I therefore suggest that samples be quenched at sampling time and filtered immediately. Acidifying samples to a pH near 1.0 can retard particle growth if analysis is conducted at some later time. In any event, the sampling technique, holding temperature, and time between sampling and filtration should be recorded to permit comparisons between samples.

SUMMARY

I found the light-scattering analyzer simple to use. In two days of work under field conditions, it produced the data presented in this report.

Fig. 8. Sand column (10% calcite) operating on brine at pH 4.5± at 0.5 l/min.

Fig. 9. Sandstone core (~10% calcite) flushed with brine at pH 4.7.

I believe the technique of estimating total suspended mass with an instrument of this type could be refined and calibrated for both routine analysis in effluent treatment experiments and quality control during injection.

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Printed in the United States of America
Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161
Price: Printed Copy \$; Microfiche \$3.00

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