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TECHNOLOGY — FEED MATERIALS

UNITED STATES ATOMIC ENERGY COMMISSION

EXTRACTION TEST IN A PULSE COLUMN USING
A SLURRY FEED CONTAINING 20% SOLIDS

By
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Price \$ 0.25

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Intra-Laboratory Correspondence

OAK RIDGE NATIONAL LABORATORY

Date: 24 July 1951

From: A. C. Jealous

Subject: Extraction Test in a Pulse Column Using a Slurry
Feed Containing 20% Solids

1.0 Introduction

The selection of a contactor for the extraction column in the Slurrex uranium ore refining process will depend on the ability of the contactor to handle a feed solution containing insoluble solids. The Catalytic Construction Company, architect engineer for the Feed Materials Processing Center, is evaluating several types of contactors for use in the Slurrex process, including the pulse column. Previously in the Unit Operations section a six-hour trial made in a 0.5 in. I. D. pulse column with plates incorporating 0.04 in. holes and using a slurry feed had favorably indicated the feasibility of using unclarified or slurry type feeds in this contactor. No other contactor under consideration has the unique feature of intermittently reversing the flow direction which is desirable for its effect on preventing solids collection or agglomeration anywhere in the contactor. Hence a test in the Unit Operations 2 in. I. D. x 24 ft. pulse column using a sample of acidified uranium ore slurry provided from the St. Louis pilot plant was arranged with Catalytic Construction Company and the AEC. The test was conducted on July 2 and 3, 1951, in Bldg. 3503, ORNL.

2.0 Summary

Use of the pulse column contactor for solvent extraction of uranium from acid slurry containing ~25% by volume of insoluble solids was satisfactorily tested in a 24 hr. long scouting run in the Unit Operations test column.

Operation with aqueous phase continuous was far superior to that with the aqueous phase dispersed. Less than 0.01% of the feed uranium was lost in the slurry raffinate flowing from the bottom of the 2 in. I. D. x 24 ft. tall glass pulse column. The column was operated at ~800 gal/sq. ft.-hr. [The results indicate that a shorter column (i. e. 16 ft. vs. the 24 ft. test column) would satisfactorily effect extraction. At no time during the 24-hour period was there any indication of solids build-up anywhere in the contactor; nor was plugging of plate holes noted. No scale formation was noted on either the column walls or the plates. Emulsification tendencies prevented flowrates above 800 gal/sq. ft.-hr; however, this was probably the result of (1) not washing the solvent before use and (2) plate geometry, requiring extraordinary pulsing.] Clean-up was readily accomplished with a routine flush of dilute nitric acid. Such encouraging results from one exploratory run obviously demand further studies looking towards plant-scale application of this contactor.

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A second run is proposed using the remaining 50 gallons of feed sample. In this run the column may be shortened to 16 ft. the plate hole diameter may be increased 50% from 0.125" to 0.188" but without changing the 23% free area and improved slurry flow controls may be used to permit steadier operation.

Purpose of the run would be to test higher throughputs, check operability under steadier flow conditions, and determine uranium recovery in the shortened column.

3.0 Equipment

The trial run was carried out in the same "true-bore" glass column previously used for scrub and strip tests. The only alterations of the system made in anticipation of the run were connection of the sample drums directly to the inlet of the Milton Roy Constametric feed pump and installation of propeller mixers in the sample drums for continuous agitation during the run. Use of the Constametric pumps for handling the slurry was considered as a calculated risk. It was predicted that they would pump the slurry although probably with an erratic flow.

Flow measurement of the slurry feed was accomplished solely through use of the Kellex magnetic flow meter. This unit apparently responded well to the flow of slurry and also served to indicate the nature and degree of the flow variation.

4.0 Feed Material

The aqueous feed was a sample of uranium slurry (~ 25% solids by volume) provided from the St. Louis pilot plant. Supernatant obtained by clarifying a small sample of the feed material analyzed 235 μ g/l and 2.6 N in nitric acid.

The solvent feed stream analyzed 25% TBP in Amsco diluent. The solvent feed was made from fresh stock and was not washed prior to the run.

5.0 Slurrex Extraction Run

5.1 Preparatory Tests

Initial tests were made to determine operable pulse and flow conditions to be used in a run of 24 hours duration. Based on previous experience reported at the Mallinckrodt pilot plant, the pulse column was first operated with the organic phase continuous, 0.7 in. pulse amplitude, 50 cycles/min pulse frequency and about 800 gals./sq. ft.-hr. total throughput at an organic aqueous flow ratio of 2.8:1. It was quickly noted that the aqueous (slurry) phase was not dispersing through the plates and hence the pulse conditions were readjusted principally towards higher amplitudes. Operation improved somewhat, however, even at 1.3 in. amplitude (maximum available), continued operation would probably have led to flooding. The surface tension of the aqueous phase apparently was sufficiently great as to prevent the downward

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pulse flow and hence prevented dispersal of the aqueous phase through the 0.125 in. plate holes. Hence operation of the column was revised to aqueous phase continuous.

5.2 Description of the Run

Operating with the aqueous phase continuous also required the maximum pulse amplitude available (1.3 in) for best results. For the throughput rate used (800 gal/sq. ft.-hr) a pulse frequency of 50-60 cycles/min. was required. Higher frequencies tended to cause flooding by emulsification, while lower frequencies tended to lead to flooding by failing to surge the dispersed organic phase completely through the plates.

The fact that the solvent feed solution was not washed with sodium carbonate prior to use as uranium extractant may have enhanced the tendencies towards emulsification during the run. Also the solvent was not acidified before feeding to the column.

The run was continued for about 24 hours with the aqueous phase continuous. The column contents had the appearance of chocolate milk, hence, only the edges of the perforated plates could be observed. At no time during the run was there any indication of column plugging or collection of solids around the edges of the plates. The aqueous feed rate however, varied widely at times and particularly during the latter part of the run, apparently due to intermittent plugging of the constametric step valves.

During the early part of the run, total throughput was at the rate of 1000 gal/sq. ft.-hr. (flow ratio was 2.8 organic: 1 aqueous). After about 16 or 17 hours of operation severe emulsification began at the upper end of the column and proceeded down the column to about 4 ft. from the bottom. Flow rates were reduced to about 700 gal/sq. ft.-hr. and this situation was satisfactorily corrected in about an hour and did not re-occur during the remainder of the run. During the final six hours of the run the slurry feed rate was very erratic making it practically impossible to determine the flow rate.

5.3 Uranium Extraction

One other qualitative observation concerns the uranium extraction. For most of the run, the uranium as noted by presence or absence of yellow coloring of the column contents remained in the upper quarter of the 24 ft. column. During the latter part of the run when emulsification and near flooding conditions developed the uranium color dropped as low as 20 ft. from the top of the column. However, as the hourly grab-sample analyses show, the uranium content of the raffinate never rose above 0.02 g/l during the entire run. (See Table I). The uranium again remained in the upper part of the column when the near-flooding conditions were eliminated.

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The run was sampled hourly to check uranium extraction. Composite samples of the product and raffinate streams were collected for analysis by the New Brunswick Laboratory.

5.4 Interface Crud

A black, slick substance collected in fairly large quantity at the interface in the top section of the column - possibly half a liter all told for the entire run. A sample of this material was analyzed spectrographically and found to contain aluminum, silicon, and magnesium as major constituents. (See Table II). A sample of interface crud was also submitted to NBL for analysis.

5.5 Column Clean-Up and Inspection

After the run was terminated the system was flushed in routine manner with dilute nitric acid. None of the plates showed any evidence of being plugged; nor was there any evidence of scaling on the column walls.

The feed and aqueous raffinate Constametric pumps were disassembled and cleaned. The feed Constametric pump was found plugged at the inlet to one of the two parallel step valves. This was apparently the major cause of the wide fluctuations in feed flow rates.

Inspection of the step valve seats and the ball checks did not reveal any noticeable effects attributable to the pumping of the slurry.

6.0 Proposed Run

A second run will be made using the remaining 50 gallon sample of slurry feed. For this run the column would be modified as follows:

- (1) replace the existing plates containing 0.125 in. holes with new plates containing 0.188 in. holes intended to permit operation at higher flow rates and possibly lower pulse amplitudes
- (2) reduce the column length to 16 ft. overall.

During steady operation in the first run, only about eight or ten feet of column appeared necessary for satisfactory uranium extraction.

The Constametric pumps would be eliminated from the system for this run. The feed would be pneumatically fed through the Kellex magnetic flowmeter for satisfactory flow control. The slurry raffinate may be carried away by use of a Vanton or similar pump.

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TABLE I						
URANIUM AND ACID ANALYSES OF STREAM SAMPLES FROM PULSE COLUMN EXTRACTION RUN USING A SLURREX FEED						
Column: 24 ft. x 2 in. I. D. true-bore glass pipe. Plates: 18 ga. stainless steel, 2 in. spacing, 0.125 in. holes, 23% free area. Pulsator: reciprocating piston pump Pulse amplitude: 1.3 in. Pulse frequency: 50-60 cycles/min. Flow rate: 700-1000 gal/sq.ft.-hr. Flow ratio: 2.8 organic: 1 aqueous						
Feed Material: Aqueous: UNH solution from St. Louis Pilot Plant (200 gu/l; 25% solids by vol.) Organic: 25% TBP in Amesco (unwashed)						
Sample Hour	U S/l	H ⁺ N	U g/l	H ⁺ N	U g/l	H ⁺ N
1			84.7	0.07	0.07	3.25
2			67.3	0.26	0.046	2.19
3			88.7	0.07	0.0086	2.72
4			48.0	0.38	0.0092	1.81
5			64.3	0.28	0.0145	1.33
6			54.7	0.31	0.0066	2.50
7			70.3	0.09	0.008	1.79
8	2.36	2.79	13.4	0.73	0.0091	0.50
9			8.0	0.75	0.0011	0.33
10			16.3	0.44	0.0089	
11			10.2	0.14	0.0081	1.03
12	236	2.88	41.0	0.45	0.0086	1.06
13			77.5	0.13	0.0055	
14			84.0	0.11	0.0054	2.41
15			36.7	0.49	0.0054	2.61
16			67.3	0.63	0.0083	2.46
17			25.0	0.58	0.0048	3.33
18			27.7	0.59	0.0068	2.12
19			78.7	0.08	0.0092	3.11
20	232	2.67	90.0	0.05	0.0099	2.60
21			84.0	0.08	0.0105	1.53
22			68.0	0.23	0.0085	3.23
23			85.3	0.59	0.0120	1.53

*Analysis based on sample supernatant; not corrected for solid volume.

TBP content of solvent feed was 25%.

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TABLE II

SPECTROGRAPHIC APPROXIMATE ANALYSIS OF
INTERFACE CRUD FROM SLURREX EXTRACTION RUN

1. Major Elements

Al - very strong
Mg, Si - strong

2. Minor Elements

Ag, Mo, Pb, Pd -- moderate
Co, Ca, Cr, Cu, Fe, Mn, Ni, Ti, V - weak

3. Trace Elements

Au, B, Pt, Sr, Zr.

4. Elements sought but not found

As, Ba, Be, Bi, Li, Na, P, Sb, Ta.

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