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POST OFFICE BOX P
OAK RIDGE, TENNESSEEORNL
CENTRAL FILES NUMBER

CF-52-10-26

DATE: October 3, 1952

SUBJECT: $\text{Ca}(\text{OH})_2$ Contactors for Thorex Solvent Recovery System

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FROM: W. R. Winsbro

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SUBJECT: Ca(OH)₂ Contactors for the Thorex Solvent Recovery System

ABSTRACT

The operation and basic design of the Ca(OH)₂ Contactors for the Thorex Solvent Recovery System are discussed.

SUMMARY

A scale model of a Ca(OH)₂ Contactor is being constructed and will be tested in the Unit Operations Section prior to the design of full-scale units for the Pilot Plant.

This report presents design information obtained from the Laboratory Section during the course of detailing the design of the scale model and summarizes pertinent design variables which will require consideration during the Unit Operations Test Program.

INTRODUCTION

A Solvent Recovery System has been included in the Thorex Process to permit recycling of the process solvent stream. The requirements of this system are two-fold:

- (1) To remove certain "crud-forming" impurities from used solvent which accumulates at, and interfere with, the control of extraction column interface levels, and
- (2) To remove radioactive contamination from this solvent.

Earlier Solvent Recovery Systems have involved the countercurrent extraction of the stripped solvent with caustic and soda ash solutions. Investigations carried out in the Laboratory Section have shown that such systems are incapable of eliminating all of the "crud-forming" impurities from the Thorex solvent. An intensive search for a more effective scrubbing agent has resulted in the selection of powdered hydrated lime, Ca(OH)₂.

DESCRIPTION OF CONTACTOR OPERATION

This new system will be operated in a semi-continuous batch-wise manner.

Slurry Preparation:

A Ca(OH)₂ suspension containing 60 grams of Ca(OH)₂ per liter is prepared from fresh water-saturated solvent and charged to a vertical contactor vessel. Stripped solvent requiring refinement is then fed up through this contactor at a velocity below the interface settling

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velocity for the suspension. At first hand, it would appear that this velocity would be quite low since the ASTM specifications for hydrated lime call for a particle size such that 85% (by weight) will pass through a 200-mesh screen (mesh opening of 74 microns). Fortunately, however, these small particles agglomerate when contacted with solvent which is saturated with water, so that an interface settling velocity in the order of 3 cm/min is obtained with a newly-prepared suspension. Therefore, a solvent velocity in the order of 2.5 cm/min is expected to be safe for design purposes. Although the solvent fed to the contactor will be saturated with water, present information indicates that the Ca(OH)₂ particles will pick up very little additional water during operation. However, any additional water picked up, plus the added agglomerating effect produced by "crud" pick-up, should insure progressively safer performance with velocity of 2.5 cm/min.

Adsorption Mechanism:

As the solvent moves upward through the contactor, a number of different adsorption processes take place:

- (1) Removal of "crud-forming" impurities,
- (2) The removal of any residual radioactive contamination leaking past an upstream silica gel column in the system,
- (3) The neutralization of the nitric acid content of the solvent, and
- (4) Perhaps some dehydration of the solvent.

Laboratory information available to date indicates that the process of neutralization will limit the duration of the operating cycle for the contactor (the solvent will run about 0.002 N in HNO₃). Tentative plans call for arbitrarily terminating the contactor operating cycle when 50% of the Ca(OH)₂ charged has been neutralized.

Contact Time:

Preliminary Laboratory data also indicate that a solvent "residence time" or "contact time" of one hour in the contactor is ample for the above mentioned processes to take place. To obtain an "average" residence time of one hour in a single contactor, a contactor suspension volume equal to the hourly volumetric processing rate of the solvent is required. However, such a design only insures that the "average" residence time will be one hour and a good probability exists that some solvent will pass through the contactor with a residence time considerably below average. This probability is combatted

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more effectively by employing two contactors (each with a contact time of one hour) in series than by employing a single contactor with a two-hour contact time. Accordingly, the Thorex Solvent Recovery Process will employ two "one-hour residence time" contactors in series.

Length of Operating Cycle:

The duration of the operating cycle for this dual contactor unit will be limited to that required to neutralize half the Ca(OH)₂ in the first contactor and is readily calculated to be 17 days as shown below:

Cycle Operating Time =

$$\frac{(V \text{ liters})(60 \text{ gms Ca(OH)}_2/\text{liter})(0.50)}{(37 \text{ gms Ca(OH)}_2/\text{equiv})(F \text{ liters/hour})(0.002 \text{ equiv/liter})(24 \text{ hr/day})=}$$

$$\frac{60 \times 0.5V}{37 \times 0.002 \times 24F} = \frac{30 \times 500V}{37 \times 24F} = 17 \frac{V}{F}$$

$$17 \frac{V}{V/\theta} = 17 \text{ days}$$

Where,

V = Contactor volume, liters

F = Solvent feed rate, liters per hour, which for a contact time of θ hours equals V/θ .

Waste disposal:

After 17 days of cumulative operation, both contactor units will be shut down and charged with fresh Ca(OH)₂ suspension. The spent suspension will be drained from the contactors to filtration units where the solvent will be recovered from the solids. The Ca(OH)₂ filter cake is disposed of by dissolving with 8M HNO₃ and then flushing to a hot drain. The Laboratory has indicated that the hot waste volume discharged from the Ca(OH)₂ System will be 1600 times smaller than that from the caustic-soda ash extraction system, an obvious advantage of the Ca(OH)₂ Contactor System.

Other Solvent Recovery Equipment:

Three other elements of equipment will be included in the main flow stream of the Thorex Solvent Recovery System:

- (1) Upstream from the Contactor will be located a silica gel column to eliminate the major portion of the radioactive contamination from the solvent, thus reducing the operating activity level of the contactors.

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- (2) Downstream from the Contactors will be located a micro-metallic stainless steel filter (porosity H; 5 micron) to remove suspended solids from the refined solvent.
- (3) Beyond the filter there will be located a simple two-phase contactor column where the solvent will be contacted with water. This water saturator will furnish a visible check on the "crud-eliminating" performance of the Recovery System and will restore the water content of the solvent, essential to the performance of the electro-magnetic flowmeters which will be used to run and control process flow.

Further details concerning the design of the Thorex Solvent System can be obtained from the T-5 Thorex Flowsheet (Dwg. No. D-9177).

DETAIL DESIGN FEATURES OF Ca(OH)₂ CONTACTORS

Previous discussion has described the contactor in general terms as a vertical sedimentation cell, cross-sectioned so that the maximum solvent velocity up through the cell will not exceed 2.5 cm/min. At some point near the top of the cell, a solid-liquid interface exists where the finer particles of Ca(OH)₂ drop out of the rising solvent. While this simple picture represents a convenient way of visualizing the essential function of the contactor, it is not a realistic picture in that such a cell would be subject to solvent "channeling" or "short-circuiting" through the cell.

Features to Improve Contact:

Channeling effects can readily be overcome by positioning a low-speed shaft mounted with several impellers in the suspension zone of the cell. Turbine type impellers having a diameter from 35 to 40% of the contactor diameter will be used for this purpose. For a low solvent processing rate of 20 to 50 gph, the height-to-diameter ratio of the contactor will be large and it will be necessary to locate the agitator shaft co-axially with the contactor. For such a geometry, several longitudinal baffles must be spaced about the inner wall of the contactor to obtain agitation free of swirl. Four such baffles, having a width of 7 to 8% of the tank diameter, will be employed.

Features to Improve Separation:

By including agitator equipment in the contactor to solve the problem of solvent channeling, a problem of establishing solid-liquid separation at the top of the contactor is created. For this reason, it is

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desirable to avoid the use of excessive agitation in the contacting zone. The operating speed of the agitator thus becomes a variable having an optimum value. It is expected that a speed between 200 and 400 rpm will prove adequate.

To promote good solid-liquid separation at the top of the contactor, despite turbulence in the contacting section proper:

- (1) A conical annular baffle has been located at the top of the contacting zone just beyond the upper extremity of the longitudinal baffles. The periphery of this baffle fits tightly against the contactor wall and will eliminate the upward velocity which the top impeller imparts to the suspension in the region above the impeller and near the contactor wall. The width of this ring is another variable of considerable design importance; a large width reduces solvent through-put rate materially (due to the limiting design velocity of 2.5 cm/min), while a small width will not completely eliminate excessive vertical velocity components above the plane of the baffle.
- (2) Radial baffles have been provided to eliminate all residual swirl carrying over above the annular baffle. These baffles will be welded to the external circumference of a short section of pipe which will be located about the agitator shaft and will extend all the way to the contactor wall. Solvent leaving this section should be essentially free of all excessive vertical and horizontal velocity components relative to the main flow velocity.
- (3) A conical section of enlarged cross-section has been provided. A slope of 60° from the horizontal is the minimum that can be employed in this section and yet obtain good solid drainage back into the contactor. A cross-section enlargement ratio of three was arbitrarily selected for this section.

A number of circular 60° conical partitions located within the enlarging section further improve the efficiency of clarification. These extend throughout the entire height of the section and are so spaced that the total cross-section for flow is broken down into a number of annular passages of approximately one-inch width. Solvent passing through the section is thus forced to pass through a number of narrow

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annular channels which diverge at a 60° angle. Within such a passage, clarified solvent quickly moves to the upper surface and the solids to the lower surface with the result that the two phases are thus able to flow countercurrent to each other at maximum velocity. The Laboratory, who suggested this refinement, has observed that the settling rate for the suspension in a sloping channel is about twice that in a vertical channel. Combining this factor with the cross-sectional area factor, the over-all separation factor for the annular partitioned enlargement section becomes six.

SCALE MODEL Ca(OH)₂ CONTACTOR

The above discussion of the design and operation of the proposed contactor for the Pilot Plant Thorex Process has emphasized a number of factors which are of importance for contactor design. For this reason, and also because the contactor is basically a new type of contactor for the CTD, a scale model has been designed for Unit Operations testing. Detail drawings for the test model are completed and fabrication is underway. This unit contains all the features previously described, along with provisions for the independent insertion and removal of all elements so that the relative effectiveness of each can be studied. The contactor section proper, six inches in diameter and forty-eight inches long, is of glass so that the contacting performance of the unit can be observed. The solvent processing capacity of the model will be in order of 4 gpm (one-fifth scale of the Pilot Plant Thorex Process).

Detail drawings of this unit are available (drawings D-9185 and D-9186).

SUMMARY OF DESIGN VARIABLES REQUIRING INVESTIGATION

Design variables which will require consideration during the Unit Operations Test Program for the Scale Model are summarized below:

1. The maximum safe linear solvent velocity that can be employed up through the minimum traverse cross-section of the contactor should be checked. In other words, the figure of 2.5 cm/min requires confirmation. Since it is probable that the degree of agglomeration will increase somewhat with operating time, this test should be made with fresh slurry and perhaps checked for used slurry.
2. A satisfactory shaft speed range for the agitator should be defined. The matter of impeller spacing, or number of impellers, should probably receive consideration in this connection. The variables, impeller

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type and impeller-to-tank diameter ratio, conform to usual practice and probably do not require investigation.

3. An optimum width for the annular baffle should be established. The greater the width of this element, the less free cross-section there is available for solvent flow and the smaller the contactor through-put capacity.
4. A suitable length of operating cycle should be established. While information available to date points to an operating cycle of 17 days, this fact should be confirmed by actual test.
5. In connection with item 4, the effect of temporary shut-downs before the conclusion of the operating cycle should be considered. Is it difficult to start up again after such shut-downs? Is the performance of the contactor adversely affected in any way?
6. The cross-section enlargement ratio for the contactor clarification header should be considered. Would it be desirable to use a ratio larger or smaller than three? This can be determined from observations of the relative effectiveness of the solid-liquid separation.
7. The adequacy of a porosity-H micro-metallic stainless steel filter for eliminating solids from the refined solvent should be confirmed.
8. The desirability of using 8M HNO₃ to dissolve the Ca(OH)₂ filter cake should be analyzed. Is it accompanied by excessive steaming?

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