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NEWPORT, DELAWARE

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Newport, Delaware
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PLANT TRIAL OF THE ACID FLUSHING PROCESS

This report is intended to summarize experience with subject process, which was evaluated in the plant April 10, 1951. The acid flushing process, which consists of dissolving a CPC synthesis slurry in a relatively large quantity of 98% sulfuric acid (7 parts per part of CPC) and drowning in water, offers a substantial improvement in quality and a means of obtaining an essentially kerosene-free crude presscake. The plant trial was scheduled solely for the latter advantage, since adequate facilities for drying crude CPC are no longer available in the plant.

Serious equipment limitatinns and several operating difficulties prevented a thorough evaluation of the "AF" process; however, it may be concluded that a number of major equipment changes would be required for continued operation. It is not believed that acid flushing could be adapted to present equipment at forecast production rates.

Since it appears that Newark will be successful in drying crude CPC for the Newport plant in tray dryers, and, since Newport is not properly equipped for acid flushing, the secondary goals, improved quality and crude CPC that may be useable <u>without</u> solvent milling in certain codes, actually become more important than a kerosene-free crude presscake. Accordingly, it is planned to determine the equipment and investment that would be required for acid flushing.

The acid flushing process is attached with comments showing the reasons for conclusions presented herein. The scheduled second trial of acid flushing was cancelled in view of the equipment limitations plus the fact that the synthesis intended for evaluation had reached 218°C (specification is 195-200°C) and pumping difficulties requiring an excessive volume of diluent kerosene were anticipated.

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ACID FLUSHING PROCESS

Flushing

1. Pump entire synthesis slurry from synthesis kettle to steam still. (Some difficulty was encountered pumping the charge. More kerosene was required for diluting and flushing than desired. The temperature in the still was 103°C, which is considerably lower than Semi-works experience (150°C).)

2. Add 6,300 lbs. H_2SO_4 (98%) to the sulfation tank.

3. Pump synthesis charge into sulfator over a 60 minute period. (Approximately 5 hours was required for pumping 85% of the charge into the acid due to line stoppages and the volume approaching the capacity of the tank. It was necessary to decant the kerosene from the sulfator prior to flushing the remaining 15% of the charge. The pumping troubles required steaming out pipe lines, which introduced water into the charge; consequently, a complete solution was not obtained. An additional 1,800 lbs. H_2SO_4 was required to give a slurry that could be handled.)

4. Discharge acid/pigment solution to the hydrolysis tank leaving the kerosene layer in the sulfator. (Due to the inaccessibility of the sulfator and inadequate equipment for measuring the levels, approximately 100 gallons of kerosene was inadvertently discharged into the hydrolysis tank.)

Drowning

1. Run 2,400 gallons of water into the extraction tank, which is to be used for drowning the acid solution.

2. Pump one-half the acid solution into the extraction tank. (Pumping difficulties were encountered which resulted in drowning two-thirds of the charge instead of half.)

Neutralization

1. Add 50% NaOH to the extraction tank to a pH of 9.0 - 10.0 using extreme caution. (The tank foamed over as a result of inadequate volume making it necessary to filter about one-third of the charge on the acid side before the required amount of NaOH could be added.)

2. Filter in press 33A or 33B.

3. Wash sulfate free. (After 24 hours washing, sulfate was still present. It was necessary to discharge filter press to avoid additional production losses.)