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The lead lined steel boiling tank in the bead catalyst plant at the Socomey Vacuum Oil Co. plant at Paulsboro, N.J., is now giving good service after alterations which involved doubling the number of straps (now 24 in. apart), increasing the thickness of the lead from 1/4 in. to 1/2 in. and changing from chemical to tellurium lead. This flat bottomed tank has eliminated sagging encountered in hemispherical and "V" bottom installations. The time of service has not been sufficiently long to judge fully its merits but to date performance has been highly satisfactory.

In addition, 3/16 in. lead lining is hung from a single horizontal steel strap near the top of the bead forming tower. This lead lining has served with no signs of failure since November, 1944, at room temperature and in the presence of dilute acid alum solution as shown in Drawing No. 3.

Steam ejected through a bronze ejector jet for heating the solution has eliminated the use of lead steam coils, agitates the solution and does away with the lead paddle wheel formerly used for agitation.

Flexible hose connections into diversion boxes have eliminated the use of many large lead lined valves. Application of other materials in the alum handling is also discussed.

a. INTRODUCTORY HISTORY

A total of 125 tons of lead was installed to handle the corrosive acid alum solution in the bead catalyst plant of the Socomey Vacuum Oil Co., Inc., at Paulsboro, N.J. Nearly two billion beads or 50 tons of finished catalyst per day are produced in the lead lined forming towers from gel forming solutions consisting of sodium silicate and acid-alum (aluminum sulfate).

The synthetic bead catalyst is used in the Thermoform catalytic cracking process which has recently come into extensive use in the production of high octane gasoline. The alum used is produced in a lead lined boiling tank from aluminum trihydrate reacted with sulfuric acid. Aluminum sulfate (alum) is produced from the basic raw materials rather than from commercial alum because of high purity requirements.

November 16, 1944

The use of aluminum sulfate and alum are similar and are largely identical. Alum has been very largely replaced by alum sulfate. The use of alum sulfate is a more economical method (or method, even) for the production of aluminum sulfate.

For a complete description of the process and flow sheet of this process, see the report of the Chemical & Metallurgical Division, April and May 1944, 1944, for comparison with alum tank used in water treatment plant, and Chemical Division, Bulletin No. 1.

The alum sulfate is a mixture of the solutions in a lead lined tank. The solutions of alum sulfate and alum are stirred through lead pipes of the same material as the tank. This is used for each of the wet processes of the heavy metals, other materials used throughout the plant are described in Division 10.

b. Process Description

Demineralized water, freed of catalyst poisons such as iron, copper, chromium, and sodium by use of the cation-anion exchange process, is first introduced to the alum reactor. Concentrated sulfuric acid is added until the solution is 50% acid which fills the tank to a depth of 18 in. Aluminum trihydrate is added which starts the exothermic reaction and additional alumina is added to maintain temperature at 240-260° F. The reaction is continued for 1-1/2 to 2 hr. at this temperature and is continuously agitated with steam and kept above solidification temperature of 210° F. Then the batch is diluted to 2% aluminum sulfate and kept at 130-140° F. for four hours. Further dilution is done in this tank until it is 90% full of 2% alum solution which remains liquid at room temperature. For use this is further diluted to 10% in a solution preparation tank. Several cycles are run daily when the plant is in full operation.

c. EQUIPMENT DESCRIPTION AND PERFORMANCE

The reaction tank is 17 ft. 7 in. inside diameter and 7 ft. 3 in. deep. In November, 1944, a 1/4-in. chemical lead lining was first installed with vertical straps every 4 ft. This proved unsatisfactory after a bare five months and was ripped out at that time. The most severe stage of the reaction occurs when the tank is filled to a depth of 18 in. and the lead penetration by the alum solution after five months was so extensive as to require replacement of the steel bottom and sides to a depth of 18 in.

The new tank lining was installed in March, 1945, and has served to date with only slight buckling of the lead and an occasional small leak. The bottom and sides are now 3/8-in. tellurium lead and the slightly convex top is 3/16-in. chemical lead. Additional straps were installed so that the new lining is strapped every 24 in. on the side walls. The bottom is secured by parallel straps, 18-20 in. apart. The low liquid level at height of reaction followed by dilution and lower temperatures place little strain on the upper tank wall area.

The original agitator was a lead covered paddle blade type but the lead covering came off exposing the steel below to the corrosive acid alum solution. The steel bolts holding the paddles corroded away and the paddles fell off. This was replaced by the eductor system described below.

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When first strand with lead, lining by hand, usually bonding regularly spaced small squares of lead was tried. This actually failed outright.

Each container is inspected by means of deep holes at the bottom edge of the reaction tank. This allows considerable corrosion to take place before the lead is discovered and it is possible to determine readily the actual point of leakage.

The 100-gal. lead lining of the bead forming tower is supported by a horizontal lead collar. In Drawing No. 3 the entire tower is shown with the lead lining. All lead lining is the old crucible material which contains the lead. A sluice water inlet and an oil inlet allow the liquid proportions to be maintained as shown.

A 5% solution of aluminum sulfate, 5% sulfuric acid, and 90% deionized water is heated from steam through lead pipes to a flow controller. The non-corrosive sodium silicate solution is safely handled in steel pipe. Saram tubing then carries both solutions to the cast silicon bronze distributing head, through a Lucite nozzle, over a plaster of paris fluted distributing cone into the lead lined tower where beads are formed as the droplets fall through a layer of oil. Sluice water at the bottom of the tower carries the beads through lead pipe to a weir box, then through rubber hose to a waxed steel flume and on to wet bead processing.

d. USE OF MATERIALS COMPETITIVE TO LEAD

With the exception of the perforated lead standpipe, all parts of the eductor system in the alum reactor are made of bronze (88% copper, 10% tin, 2% lead). Bronze pipe and fittings of this composition give good service but when first installed brass was used with resulting severe corrosion by dezincification. Steam for heating is jetted through the eductor nozzle eliminating the use of lead steam coils for heating.

Cast mixing heads (See Drawing No. 2) of silicon bronze are used in mixing the acid alum with the sodium silicate but proved to be very porous and the solution seeped through. The mixing heads were continued in use with diminished seepage after impregnating the castings with a phenolic resin. Connections to the mixing head are of Saram tubing (a vinyl plastic) which has given good service and eliminated a leakage problem around threaded bronze joints.

A nozzle of Lucite provides an exit from the mixing head and the mixture is discharged over the apex of a fluted cone of plaster of paris. A thin anti-wetting wax coating originally applied to the cone surface to prevent gel adherence wore off in spots necessitating daily redipping. An Americoat resin paint was applied and increased cone life nearly thirty times.

Lucite has also been found suitable for use in replacing parts in bronze flow control valves in acid alum lines. Worthite pumps in these lines performed well but bronze valve parts were severely corroded in less than a year. Lucite parts machined in the refinery shops replaced these and are now serving well as plugs, seats, and rods. Bronze has served adequately in acid alum lines not requiring close tolerances, but longer service is expected from the Lucite replacement.

Chemical Bulletin No. 1

The use of steel tanks with a lead lining is a common method of resisting a corrosive medium. The lead is applied in the form of a composition on a structural base. The composition has been used in the form of a mixture of four oxides and is a white solid with a melting point of 1100° F. (melting point is 165-175° F.) The lead is applied in the form of a mixture of four oxides and is a white solid with a melting point of 1100° F. (melting point is 165-175° F.) The lead is applied in the form of a mixture of four oxides and is a white solid with a melting point of 1100° F. (melting point is 165-175° F.)

Testling was found satisfactory for acid handling. Valves and fittings of the same design should be avoided through porosity. Acid also will seep right through the pores in the casting.

There are 16 wet processing tanks of reinforced precast concrete, lined with wax on the inside surface to prevent acid leakage. Wire braiding covers the rough spots and a micro-crystalline wax, known as C, is applied by spraying at 100° F. (melting point is 165-175° F.) Several coats are applied to build up a coat about 1/16 in. thick. One gallon of wax will cover about 100 sq. ft.

Also used in the wet processing system are stainless steel (18-8) 8-mesh wire screen strainers over the effluent liquor outlets. These are on the bottom of the lead pipe and prevent the beads from flowing off through the liquor outlets.

After wet processing the beads are sluiced through a waxed steel flume to the dryers. In large areas of the flume wax is worn off but the steel below has not corroded because the pH of the wash liquor is around 8 or 9 and a tenacious white surface coating forms on the steel.

CONCLUSIONS

A. ALUM REACTION TANKS.

1. Chemical lead, 1/4 in., strapped every 48 in. failed badly in less than five months.
2. Tellurium lead, 3/8 in., strapped every 24 in. has given good service since March 1945 (21 months). Length of service is as yet too short to judge the performance of this lining.
3. Regularly spaced squares of homogeneously bonded lead lining failed over night, proving inadequate to support the lead lining under conditions of fluctuating temperatures.
4. Flat bottom design has eliminated sagging and leakage troubles encountered in hemispherical and "V" bottom tanks. This is where all leakage occurred in the alum reactor of the Baltimore waterworks (see Chemical Bulletin No. 1).
5. The plant engineers would like to know of any methods other than weep holes by which leaks in lead lined steel tanks can be readily located.
6. Brass for interior fittings in the alum reactor failed because of dezincification. Eductor parts of bronze have served in the alum reactor with no trouble.

B. BEAD FORMING TOWERS.

1. The bead forming tower, lined with 3/16-in. chemical lead is unaffected by corrosion from the dilute acid and aluminum sulfate solutions.

C. OTHER MATERIALS.

1. Jarrah connections, worthite pumps, stainless steel and Ameripat resin paints are materials proven satisfactory for particular phases of acid alum handling.





