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PRODUCTION OF HYDROQUINONE
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PRODUCTION OF HYDROQUINONE

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The present invention relates to a process and apparatus for the production of hydroquinone by means of reduction and quinone. More particularly, the invention is concerned with an improvement in the process and apparatus as disclosed in Von Bramer and Zabristke U. S. Patents No. 1,880,534 and No. 1,998,177.

The process as disclosed in those patents includes steam distillation of a quinone-containing mixture to obtain quinone vapor, which is conducted along with water vapor produced in the distillation to an eductor where it meets a circulating current of reducing liquid comprising an aqueous slurry of iron dust. The quinone and water vapor, due to a comparatively low temperature of the circulating liquid reducing slurry and a comparatively high vacuum maintained on the reducing system are instantly converted or condensed to the liquid phase and the quinone is reduced to hydroquinone. The mixture of water and dissolved hydroquinone passes to a vacuum tank wherein the greater proportion of the condensed steam which has entered the system from the steam distillation step flashes into steam vapor. The vapor is removed by a pump and the hydroquinone, being non-volatile, remains behind in the iron-water slurry. The process is continued until all of the quinone has been reduced, the reducing slurry becoming continuously more concentrated in hydroquinone and iron oxide and additional iron dust being added from time to time.

While the process as described above has proved quite efficient, there are certain operational difficulties which cause considerable inconvenience from time to time. One major problem arises in connection with feed of fresh iron dust to the system. The conventional procedure calls for preparation of a water slurry of fresh iron dust and addition of increments thereof, as required, to the reducing liquid recirculating in the system, the fresh slurry being introduced by passage thereof through a pipe line into the partially evacuated reduction tank. Not only does the pipe line frequently become plugged under the conditions of operation but it has proven quite difficult to obtain a uniform addition of iron to the system in this manner. Furthermore, as a requirement for the iron to be added to the system as a slurry necessitates subsequent evaporation procedures for removal of the additional slurry water. Another difficulty results because of the custom of adding iron in large amounts at intervals throughout the reduction process. This custom, while adapted for present commercial installations, frequently causes the circulating pump to choke. Additionally, the circulating pump in present practice operates with a vacuum at its intake and therefore has a tendency to vapor lock.

In the present systems a large amount of space must be devoted to the reduction tank since it must have sufficient capacity not only to hold the entire batch, but also to provide sufficient head-room for disengaging entrained liquid from the flashed vapor. The size of the reduction tank is not only inconvenient because of its excessive use of space, but also because of the replacement difficulties resulting from attrition due to abrasive action of the iron powder on the inside of the tank walls. Reduction tank maintenance sometimes amounts to a major item in the presently used systems.

It is an object of the present invention to reduce or entirely eliminate the above mentioned objectionable features which result in practical inconvenience. Other objects will be apparent from the accompanying specification and claims.

The system of the invention includes means for separating, under vacuum, the condensed water vapor from the circulating slurry at a point between the eductor and the reduction tank. The invention also comprises a reduction tank maintained at atmospheric pressure and means whereby the separated hydroquinone and iron dust may be fed by gravity from the evacuated separating means to the reduction tank. Accordingly the reduction tank may be open to the atmosphere whereby delivered iron dust may be fed to it with conventional conveyors, whereby no vapor disengaging action need be performed therein, whereby little abrasive action upon the reduction tank walls results from the iron dust, whereby the circulating pump need not pull against a vacuum, whereby no additional water need be introduced into the system in order to add additional iron dust, and whereby the reduction tank may be quite small in size relative to the tanks now in use.

The invention will be understood by reference to the accompanying drawing which is a more or less schematic representation of a commercial quinone reduction system.

Referring to the drawing, a quinone steam slurry generated in still pot 11 passes upwardly through stripping column 12 and into eductor 13. The quinone steam vapor mixture is drawn into a recirculating aqueous slurry of iron dust in the eductor and immediately condensed, the quinone thereupon being reduced to hydroquinone. The aqueous recirculating slurry of iron dust is prepared in reduction tank 14 by the addition thereto as required of measured amounts of iron dust from a feeder 15. The slurry is withdrawn from reduction tank 14 by means of recirculating pump 16 and continuously passes through recirculation line 17 and the eductor 13.

The invention of course is especially concerned with the portion of the system between the eductor and the reduction tank. As mentioned above, conventional procedure calls for passage of the slurry containing the condensed water vapor and the condensed and reduced quinon directly from the eductor to a large reduction tank maintained under vacuum. In accordance with the improvement of the present invention, and as illustrated in the drawing, the slurry containing the condensed water vapor and the condensed and reduced quinone passes directly from the eductor 13 to a centrifugal separator (cyclone) 18 which is maintained under vacuum. The separator advantageously may be fabricated of cast chrome-iron, which is highly resistant to abrasion by the iron slurry. Separation takes place within the centrifuge, water being flashed into vapor therein, withdrawn and passed to a condenser (not shown). From the bottom of the separator 18 extends a barometric leg 19 of sufficient proportions to maintain a pressure seal. The leg 19 of course projects beneath the level of the slurry maintained within reduction tank 14. Sulfuric acid may be added to the system as required through the line 20.

In operation, the circulating pump is started and sufficient water added to the reduction tank to maintain good circulation. The iron dust feeder then is started and its operation controlled either manually or automatically whereby a uniform feed of iron dust and correct con-
centration thereof in the slurry may be continuously maintained. The sulfuric acid feed likewise is commenced, and steam is admitted to the still pot after which feed containing quinone is admitted to the top of the stripping column. The centrifugal separator is put into operation, maintained under a suitable vacuum, e.g., 15" of mercury and the reduction process is allowed to operate until all of the quinone-bearing feed has been processed to produce a resultant mixture containing hydroquinone.

The invention is not limited to single specific structural designs for the centrifugal separator, barometric leg, open-top reduction tank, and dry iron dust feed conveyor, and it will be clear to those skilled in the art that various embodiments of each of these items may be employed as the occasion demands.

I claim:

1. In a process for the reduction of quinone to produce hydroquinone wherein a vapor mixture of quinone and steam is continuously introduced into a recirculating reducing slurry of iron dust in water, the slurry being subjected to flashing during each cycle whereby to remove excess condensed steam, the improvement comprising the steps of passing the slurry containing condensed steam and condensed and reduced quinone into a centrifugal separator maintained under vacuum, flashing said slurry within said separator whereby to remove a portion of said condensed steam as water vapor, passing the unflashed portion of the slurry from said separator through a barometric leg into a reduction tank, maintaining said reduction tank at atmospheric pressure, and introducing iron dust as required into said reduction tank as a dry powder under atmospheric pressure.

2. In a process for the reduction of quinone to produce hydroquinone wherein a vapor mixture of quinone and steam is continuously introduced into a recirculating reducing slurry of iron dust in water, the slurry being subjected to flashing during each cycle whereby to remove excess condensed steam and increments of additional iron dust being added to the recirculating slurry from time to time, the improved method of introducing said additional increments comprising passing the recirculating slurry through a zone maintained at atmospheric pressure and feeding said increments into said slurry through an exposed surface thereof within said zone.

3. Process as defined in claim 2 wherein said increments are added as a dry powder.

4. Process as defined in claim 2 wherein prior to its passage into said zone said slurry is first treated in a separating zone and excess condensed water vapor is removed from said slurry therein.

5. Process as defined in claim 4 wherein said separating zone is maintained under reduced pressure.

6. Apparatus for the reduction of quinone to hydroquinone comprising means for continuously recirculating an aqueous reducing slurry of iron dust, means for continuously introducing a vapor mixture of steam and quinone into said recirculating slurry, a centrifugal separator, means for maintaining said separator under vacuum, means for introducing slurry containing newly added condensed steam into said separator, an open topped reduction tank, a barometric leg connecting the separator with said reduction tank and extending to below the level of liquid in said reduction tank, and means for feeding iron dust into the open top of said reduction tank.

7. Apparatus for the reduction of quinone to hydroquinone comprising a line for the recirculation of an aqueous reducing slurry of iron dust, an eductor for the introduction of a vapor mixture of steam and quinone into said line, a centrifugal separator, means for maintaining said separator under vacuum, means for introducing eductor effluent into said separator, an open topped reduction tank below said separator, a barometric leg connecting the separator with said reduction tank and extending to below the level of liquid in said reduction tank, pump means for withdrawing slurry from said reduction tank and forcing it into said circulating line and means for feeding dry iron dust into the open top of said reduction tank.

8. An apparatus for reduction of quinone to hydroquinone comprising a recirculating slurry line, means for promoting recirculation of slurry therein, means for introducing into said line a vapor mixture of quinone and steam and means for removing excess condensed steam, an improved excess steam removal means comprising a vacuum centrifugal separator, an open-topped reduction tank disposed beneath said separator and a barometric leg providing communication between said separator and said tank to a point beneath the normal slurry level of the latter.

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