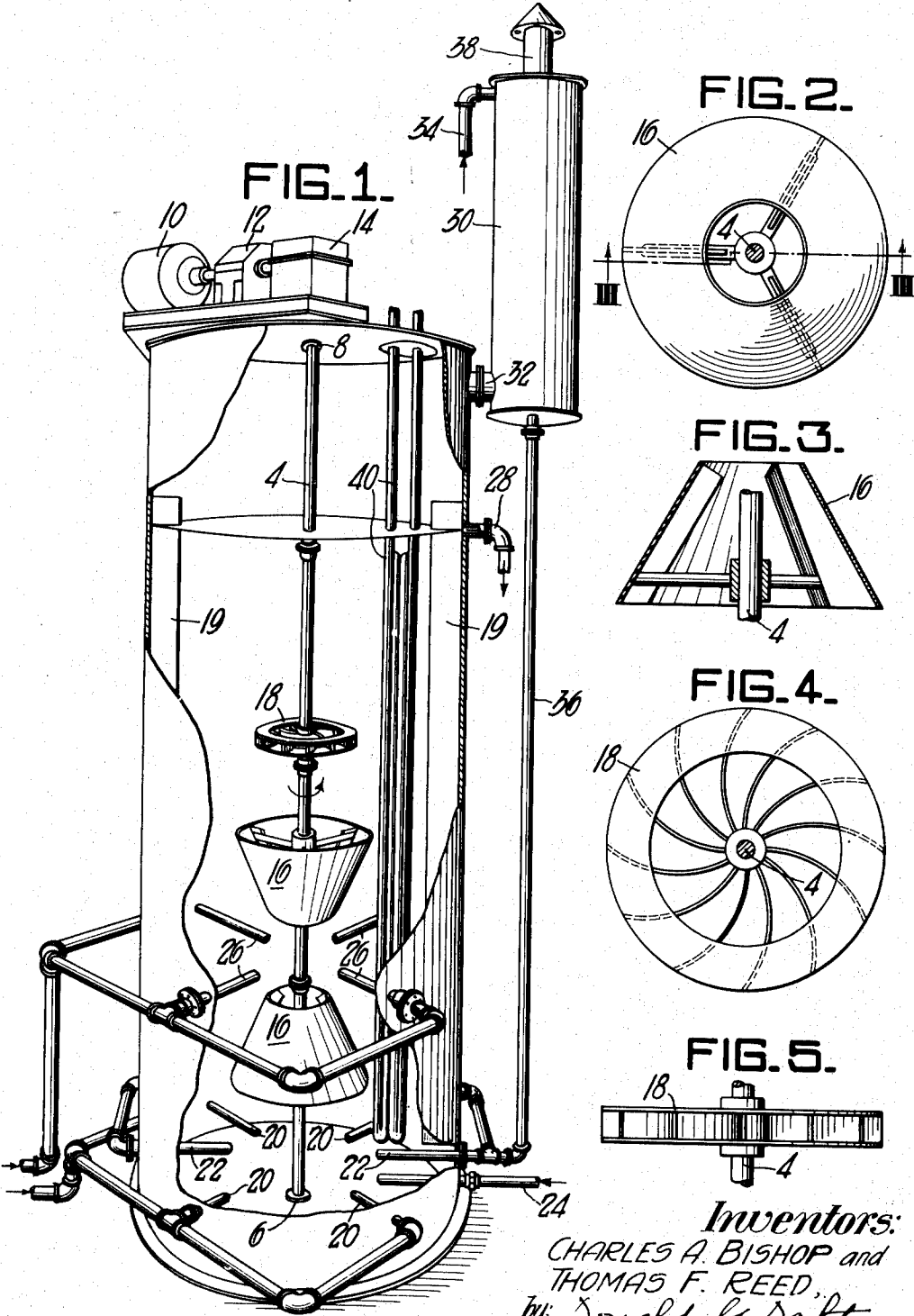


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TANK REACTOR

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TANK REACTOR

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This invention relates to apparatus for treating iron salt solutions and more particularly to apparatus for treating waste pickle liquor to obtain a black granular iron oxide and an ammonium salt substantially free from iron. The problem of disposing of waste pickle liquor has existed for many years and many proposed solutions have been advanced. However, none of these former processes have proved to be completely successful. In some instances, the cost of equipment is excessive or the operation is so slow that the space requirements for the equipment is excessive. In other instances the end product of the process is such that it cannot be readily disposed of. The ideal process would be one in which the cost of the equipment is not excessive and in which the value of the end products is such that they can be disposed of at a profit. Such a process is described in the pending application to Robert G. Thompson, Serial No. 83,227, filed March 24, 1949. The apparatus of this invention is suitable for carrying out the Thompson process and is described in the Thompson application. Such apparatus can also be used in carrying out the process described in Wilson Patent No. 2,419,240, dated April 22, 1947.

It is therefore an object of our invention to provide apparatus particularly adapted for efficiently treating iron salt solutions.

This and other objects will be more apparent after referring to the following description and attached drawings, in which:

Figure 1 is a perspective view of apparatus for carrying out our invention with parts thereof broken away;

Figure 2 is a plan view of a centricone impeller;

Figure 3 is a sectional view taken on the line III—III of Figure 2;

Figure 4 is a plan view of a turbine impeller; and

Figure 5 is a side elevation of Figure 4.

Referring more particularly to the drawings, the reference numeral 2 indicates a vertical tank having a shaft 4 having its bottom end resting in a bearing 6 in the bottom of the tank and its top end extending through an anti-friction thrust bearing 8 in the top of the tank. The shaft 4 is rotated from a motor 10 through a gear reducer 12 and a set of bevelled gears 14. Two opposed centricone impellers 16 are fastened to the lower part of the shaft 4. A turbine impeller 18 mounted on the shaft 4 a short distance above the top impeller 16 prevents the formation of a vortex at the top of the tank.

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A plurality of vertical baffles 19 are provided on the tank walls to aid agitation. A plurality of ammonia inlets 20 are provided near the bottom of the tank and are distributed around the periphery of the tank so as to avoid high local concentrations of the ammonia. A plurality of pickle liquor inlets 22 and a steam inlet 24 are also provided at the bottom of the tank adjacent the ammonia inlets 20. Located between the two centricones 16 are a plurality of air inlets 26 arranged around the periphery of the tank. An outlet 28 for the treated liquid is provided a short distance below the top of the tank. Located adjacent the top of the tank 2 is an ammonia absorber tower 30 which is packed with ceramic Raschig rings. The bottom of the tower 30 is connected to the top of tank 2 by means of a pipe 32. Waste pickle liquor is fed to the tower 30 through a pipe 34. A pipe 36 leads from the bottom of tower 30 to the acid inlets 22. An air exhaust 38 is provided at the top of the tower 30. Cooling coils 40 are provided to aid in maintaining the proper reaction temperature.

In carrying out the process described in the above identified Thompson application, the tank 2 is first filled with water slightly above the top of upper impeller 16, after which the motor 10 is started so as to rotate shaft 4 and the impellers thereon. Steam is introduced through pipe 24 until the temperature of the water is between 150° and 160° F. and the maximum amount of air is introduced through the inlets 26. Ammonia is fed into the tank until the water therein contains at least one gram of ammonia per liter, after which the flow of pickle liquor at the operating rate is started. The flow of ammonia is continued at a rate in excess of the stoichiometric amount corresponding to the analyses and feed rate of the pickle liquor. The above described steps are continued until the tank 2 is filled to the outlet pipe 28. At this time the temperature of the liquid will be between 150° and 160° F., the ratio between ferric and ferrous iron will be at least 2.2, and the pH value will be at least 8.4. Continuous operation of the process is then started.

While it is preferred to operate the process at a temperature between 150° and 160° F. experience has shown that the process can operate satisfactorily in a temperature range between 140° and 170° F. Heat is developed by the reaction in the tank 2 and therefore it may be necessary to use the cooling coils 40 to prevent the temperature from rising too high. The air

delivered into the tank aids the impellers 16 and 18 in agitating the bath, thus providing for more efficient reaction; and also oxidizes part of the iron originally present in the ferrous state to the ferric state. This is necessary in order to have at least 50% of the total volume of oxide produced settle within 2½ minutes. Any slower settling rate will not be commercially acceptable. In order to have a commercially salable product, the total iron content in the filtrate should not exceed .05 gram per liter. To determine the iron content, specimens of the filtrate are sampled and a few drops of hydrogen peroxide are added thereto. If an orange precipitate forms, the product will be unsatisfactory. The effect of the ferric to ferrous iron ratio on the efficiency of the method can be seen in the following table:

TABLE NO. I

Ferric:ferrous ratio	Time for 50% settling, min.	Fe content in filtrate
1.7-----		Present.
1.9-----	3	Do.
2.0-----	2.9	Do.
2.2-----	1.7	None.
2.3-----	2.2	Do.
2.4-----	2.4	Do.

This ratio should be maintained between 2.2 to 1 and 6 to 1.

The rate of ammonia admission which can be expressed as a function of ammonia content of the liquor at the moment of precipitation largely controls the pH of the system and represents one of the major factors involved in an efficient operation of the process. The following table gives the ammonia content of the solution expressed in grams per liter, the corresponding pH value and the iron content of the filtrate which defines the efficiency of the method.

TABLE NO. II

NH ₃ , g/l.	pH	Fe content in filtrate
0.00-----	6.9	Present.
0.3-----	7.2	Do.
1.5-----	8.4	None.
2.6-----	8.8	Do.
3.2-----	8.4	Do.

It has been found that an ammonia content of at least 1.5 grams per liter and a pH value of at least 8.4, other conditions being constant, lowers the iron content of the filtrate below the permissible maximum.

In brief, the operation of the continuous process is as follows:

Into a solution of waste pickle liquor and ammonia having a pH value of at least 8.4 is continuously delivered ammonia and waste pickle liquor with the ammonia being admitted in an excess over the stoichiometric amount necessary to react with the waste pickle liquor being admitted. The solution is agitated by means of the impellers 16 and 18 as well as by the air being introduced into the system. The air also oxidizes the iron in order to obtain the desired ferric to ferrous iron ratio of between 2½ to 1 and 6 to 1 in the precipitate. The temperature of the solution is maintained between 140° and 170° F. The treated liquid is continuously removed from the tank 2 through the opening 28 and the iron oxide is allowed to settle out therefrom. The operation is controlled by testing

the resulting effluent to determine if the amount of excess ammonia is at least 1.5 grams per liter and preferably between 1.5 and 5 grams per liter and the precipitate is tested to determine whether the ratio of ferric to ferrous iron therein is at least 2.2 and is preferably between 2.2 to 1 and 6 to 1. Under these conditions the operation of the process will be such that the settling rate and the purity of the effluent is that desired. Even though the conditions are slightly outside of these ranges the process will still operate successfully. However, any test result outside these ranges indicates that the conditions in the tank 2 are progressively becoming worse and that soon an unsatisfactory product will be obtained. Therefore, if the tests show that the desired conditions are not present, the feed rate of the pickle liquor and/or the ammonia is changed so that the control conditions will be met. Thus, by this method of control, departure from the desired product is anticipated and the conditions controlled to prevent unsatisfactory products from being produced. The effluent and precipitate are then treated in a conventional manner to obtain crystalline ammonium sulphate and dry magnetic iron oxide. Air leaves the tank 2 through the tower 30 and is stripped of ammonia therein by the raw pickle liquor entering the tower 30 through pipe 34. The air escapes to the atmosphere through the outlet 38 and the pickle liquor partly neutralized by the ammonia is introduced through pipe 36 into the tank 2.

While one embodiment of our invention has been shown and described it will be apparent that other adaptations and modifications may be made without departing from the scope of the following claims.

We claim:

1. Apparatus for treating iron salt solutions comprising a tank, a shaft extending vertically through said tank, means for rotating said shaft, a pair of opposed centricone impellers fastened to the lower part of said shaft, the small diameter ends of said centricone impellers being adjacent each other with a space therebetween, a turbine impeller fastened to said shaft above said centricone impellers, a plurality of ammonia inlets near the bottom of the tank around the periphery thereof, a plurality of iron salt inlets adjacent the ammonia inlets, a plurality of air inlets around the periphery of the tank at a level between the centricone impellers, and an outlet from said tank a short distance below the top thereof.

2. Apparatus for treating iron salt solutions comprising a tank, a shaft extending vertically through said tank, means for rotating said shaft, a pair of opposed centricone impellers fastened to the lower part of said shaft, the small diameter ends of said centricone impellers being adjacent each other with a space therebetween, a turbine impeller fastened to said shaft above said centricone impellers, a plurality of air inlets around the periphery of the tank at a level between the centricone impellers, an outlet from said tank a short distance below the top of the tank, an ammonia absorber tower adjacent the tank, a connection between the top of the tank and the tower, and means for separately introducing iron salt solutions and ammonia into the bottom of said tank.

3. Apparatus for treating iron salt solutions comprising a tank, a shaft extending vertically through said tank, means for rotating said shaft, a pair of opposed centricone impellers fastened

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to the lower part of said shaft, the small diameter ends of said centricone impellers being adjacent each other with a space therebetween, a turbine impeller fastened to said shaft above said centricone impellers, a plurality of ammonia inlets near the bottom of the tank around the periphery thereof, a plurality of iron salt inlets adjacent the ammonia inlets, a plurality of air inlets around the periphery of the tank at a level between the centricone impellers, an outlet from said tank a short distance below the top of the tank, an ammonia absorber tower adjacent the tank, a connection between the top of the tank and the tower, and a conduit leading through said tower to said iron salt inlets.

4. Apparatus for treating iron salt solutions comprising a tank, a shaft extending vertically through said tank, means for rotating said shaft, a pair of opposed centricone impellers fastened to the lower part of said shaft, the small diameter ends of said centricone impellers being adjacent each other with a space therebetween, a turbine impeller fastened to said shaft above said centricone impellers, a plurality of vertical baffles fastened to the walls of said tank, a plurality of ammonia inlets near the bottom of the tank around the periphery thereof, a plurality of

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iron salt inlets adjacent the ammonia inlets, a plurality of air inlets around the periphery of the tank at a level between the centricone impellers, an outlet from said tank a short distance below the top thereof, an ammonia absorber tower adjacent the tank, a connection between the top of the tank and the tower, and a conduit leading through said tower to said iron salt inlets.

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