RESERVE COPY

PATENT SPECIFICATION 1,177,615

DRAWINGS ATTACHED.

1,177,615

Date of Application (No. 1372/67) and filing Complete Specification: 10 Jan., 1967.

Application made in Japan (No. 1434) on 10 Jan., 1966.
Application made in Japan (No. 2077) on 14 Jan., 1966.

Complete Specification Published: 14 Jan., 1970.

Index at acceptance:—B1 J(3A1, 3A2, 4A2, 6A2A, 6B4, 6C).

International Classification:—B 01 j 1/08.

COMPLETE SPECIFICATION.

Ion Exchange Apparatus.

We, KURITA WATER INDUSTRIES LIMITED, formerly Kurita Industrial Company Limited, of No. 1, 3-chome, Koraibashi, Higashi-ku, Osaka-shi, Japan, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to an improvement in ion exchange apparatuses which use ion exchange resin.

Various ion exchange apparatuses of the so-called moving bed type which are designed to effect ion exchange by the use of ion exchange resin and to automatically withdraw the resin and to regenerate it are known. Of the known apparatuses of the type described, one which comprises three operation sections, namely an ion exchange section, a regeneration section for regeneration of the spent resin and a washing section, which are arranged in series in a single column, is suited for conducting ion exchange on raw food liquids having a relatively high concentration. In view of the fact, however, that such an ion exchange apparatus utilizes highly pressurized water for the transfer of resin, it has the disadvantages that the resin tends to be destroyed by the pressurized water during the operation and also that the column requires a considerable overall height in order to enhance both the resin regeneration efficiency and the resin washing efficiency. On the other hand, an ion exchange apparatus comprising three separate columns, namley, an ion exchange column, a regeneration column and a washing column, is suited for the treatment of liquids of high or low concentration. However, because of the separate operation columns, an apparatus of this type has the disadvantage that it occupies a large area, it has a complicated structure and requires complicated operation mechanism.

Furthermore, moving bed type ion exchange apparatuses tend to cause disorder in the resin transfer in both the ion exchange column and the regeneration column, resulting in various disturbances. More specifically, in an apparatus whose column is not provided with means such as a perforated plate which serves to support resin in the column, the entire layer of resin tends to drop in a mass during the transfer operation and causes a great disorder in the operation, while an apparatus of the moving bed type provided with supporting means such as a perforated plate in the column is inconvenient in that when the resin is withdrawn from the bottom of the column, the layer of resin located in the central portion of the column is susceptible to motion, resulting in a decrease in the ion exchange efficiency, the regeneration efficiency and the purity of the treated liquid.

The present invention contemplates providing an improved ion exchange apparatus which is free of the foregoing defects inherent in the apparatus of the prior art.

The invention provides an ion exchange apparatus comprising in combination,

(a) an ion exchange column, (b) an upper strainer disposed in the upper portion within the ion exchange column, (c) an outlet for the liquid connected to the upper strainer, (d) a lower strainer within the ion exchange column and spaced from the lower end thereof, (e) an inlet for the liquid connected to the lower strainer, (f) a measurement tank connected to the lower end of the ion exchange column through a first valve, (g) a regeneration column, (h) an outlet for the regenerant disposed in the upper portion of the regeneration column, (i) a regenerant

[*Price 5s. 0d.*]

Price 25p

strainer disposed in an intermediate portion within the regeneration column, (j) an inlet for the regenerant connected to the regenerant strainer, (k) an inlet for washing water disposed in the lower portion of the regeneration column, (l) an outlet for washing water disposed between the regenerant strainer and the inlet for washing water, (m) a measurement tank connected to the lower end of the regeneration column through a second valve.

The present invention will now be described with reference to the accompanying drawings, wherein:

Fig. 1 is a diagrammatic representation, illustrating the structure of the apparatus of the present invention;

Fig. 2 is a sectional view taken on the line A—A in Fig. 1; and

Fig. 3 is a vertical sectional view taken on the line B—B¹ in Fig. 2.

Referring now to Fig. 1 the apparatus comprises an ion exchange column 1 and a regeneration column 2. Interiorly of the column 1 there are provided an upper strainer 3, a lower strainer 3¹ and a perforated plate 4 for supporting resin in the column said perforated plate 4 being located a little above said lower strainer 3¹. A hopper 5 is provided above said ion exchange column and a resin measurement tank 6 is provided below said column. The hopper 5 and the measurement tank 6 are connected to the ion exchange column through conduits having valves 7 and 8, respectively.

The regeneration column 2 has a strainer 9 at an intermediary portion thereof, and a water collecting means 10 just below the strainer 9. Outside the regeneration column there are provided, in a manner similar to those of the ion exchange column, a hopper 11 and a measurement tank 12 which is of a type similar to that of the ion exchange column.

The strainers 3 and 3¹ of the exchange column 1 and the strainer 9 of the regeneration column 2 are of a similar structure. Figs. 2 and 3 respectively represent a plan view and a cross sectional view of the strainer 9 in the regeneration column 2.

The strainer 9 comprises a hollow plate having a plurality of resin passage holes 13 and an internal compartment 14. On the upper face of the strainer is evenly distributed a plurality of liquid spray means 16 each communicating with the compartment 14 and having a number of slits 15 formed circumferentially thereof. With respect to the strainers in the ion exchange column, the parts are indicated by the following reference numerals: 17 representing a resin passage hole, 18 representing the hollow compartment, and 19 representing a liquid spray means. In the ion exchange column the upper strainer collects the treated feed liquid, the liquid spray means of the upper strainer being directed downwardly to face the upwardly directed liquid spray means of the lower strainer.

During use of this apparatus, the ion exchange column 1 is kept packed with regenerated ion exchange resin, namely that which has been regenerated in the regeneration column 2, and the valves 7 and 8 are closed, while an inlet valve 25 for supplying the raw feed liquid to be treated and an outlet valve 26 for discharging the treated liquid are open. The ion exchange treatment is then conducted by passing the raw feed liquid in through said inlet valve 25 and the treated liquid is withdrawn from the column through the upper strainer and the valve 26, the slits in the spray means permitting withdrawal of the liquid but not the resin. In order to transfer spent resin from the column 1 for regeneration and to transfer regenerated resin to the column 1 to replace the spent resin valves 8 and 27 are opened. With the opening of these valves, the spent resin located in the column 1 below the strainer 3¹ effects a sudden downward movement to enter into the measurement tank 6, and a layer of feed liquid forms in the portion of the column below the strainer 3¹ where resin was previously located. Then, valves 25 and 26 are closed while valve 7 is oepned. This causes the resin remaining in the ion exchange column 1 to move downwards, with the result that regenerated resin contained in the hopper 5 moves into the upper part of the ion exchange column 1. The perforated plate 4 controls the downward movement of the resin located in the portion of the ion exchange column above said plate so that the resin moves downwardly in a uniform piston-like manner. After fresh resin has been supplied to the ion exchange column from the hopper 5, the valves 7, 8 and 27 are closed and the valves 25 and 26 are opened to resume the passage of the raw feed liquid. The valve 35 is kept open while the passage of raw feed continues and water is forced through a pipe 24 into the measurement tank 6 so that the resin contained in said tank is transferred through the valve 35 and a conduit indicated by a dotted line to the hopper 11 of the regeneration column.

The transfer cycle of the ion exchange resin in the whole system also includes the following steps. First, regenerated and washed resin contained in the measurement tank 12 of the regeneration column 2 is transferred to the hopper 5 of the ion exchange column 1, as is indicated by the dotted line, by forcing water into the measurement tank 12 through a pipe 20. After a predetermined amount of refreshed resin has thus

been transferred, charging of regenerant for the resin to the column 2 through a pipe 21 and the supply of washing water through the pipe 22 are suspended, and the valve 23 is opened so that the resin contained in the regeneration column 2 is caused to move downwards to be received in the measurement tank 12. On the other hand, predetermined amounts of spent resin are transferred from the measurement tank of the ion exchange column 1 to the hopper 11 of the regeneration column 2 by means of the pressurized water which is delivered through the pipe 24. The capacity ratio between the measurement tank 6 and the hopper 11 is predetermined so as to ensure that the resin collected in the measurement tank 6 is transferred to the hopper 11 after the movement cycle of the resin located in the regeneration column 2 has been repeated a predetermined number of times. The transfer of the layer of resin in the lower portion of the ion-exchange column is carried out swiftly and this is immediately followed by the closure of valves 25 and 26 to suspend the supply of raw feed liquid. Thus the liquid supply to the ion exchange column is suspended almost at the time the liquid supply to the regeneration column is suspended. The resin located in the bottom of the ion exchange column 1 is transferred into the measurement tank 6 and the resin contained in the hopper 5 is transferred into the ion exchange column 1, in a manner as has been described. After the measurement tank 6 has received the predetermined volume of spent resin, the regeneration column 2 commences a regeneration and washing operation, while the valves 7 and 8 are closed so that the supply of raw feed to the ion exchange column 1 can be resumed. After repeating this cycle in the regeneration column 2 a predetermined number of times in a manner as has been described, one displacement of resin is effected in the ion exchange column 1. Reference numerals 41 and 42 in Fig. 1 represent overflow pipes of the hoppers 5 and 11, respectively, for the pressurized water used for the transfer of the resin. Because of the apparatus of the type hereinabove described is provided with measurement tanks for both the ion exchange column and the regeneration column, the resin is measured with precision. In addition, because of the fact that the measurement tanks are connected to the columns through valves, it is possible to freely control the resin transfer speed. While it has been quite difficult, in general, to effect uniform flow of resin with the conventional apparatus of multiple-column type, such a shortcoming is eliminated in the apparatus of the present invention due to the provision of the measurement tanks. Furthermore, the controlled downward movement of the resin located in the upper portion of the ion exchange column, along with the downward displacement of the resin located in the bottom portion of the column by the layer of liquid formed therein, ensures that the entire movement of the resin contained in the ion exchange column is orderly and piston-like. Also, because of the fact that the amount of resin transferred in the ion exchange column 1 is determined by the amount of the resin transferred in the regeneration column 2 to the measurement tank of the regeneration column, the flow of resin in the entire apparatus can be markedly stabiilzed.

Description will next be directed to the structure of the regeneration column 2 and the regeneration and washing operations which are carried out on the resin contained in the regeneration column 2.

A washing water supply pipe 22 is connected adjacent to the lower, or resin outflow, end of the regeneration column 2, and a liquid drain pipe 30 is connected adjacent to the upper, or resin inflow, end of the regeneration column 2. The strainer 9, equipped with a liquid spray means, and constructed as described previously, is disposed in an intermediary portion of the regeneration column 2. A water collecting means 10 is provided adjacent to the lower side of the strainer 9 from which the resin flows. The water collecting means 10 communicates with said strainer 9 by means of a bypass pipe 34. A regenerant supply pipe 21 is connected to said bypass pipe 34. Fig. 1 shows a regeneration column which is arranged in such manner that ion exchange resin is permitted to move downwards from the upper portion to the lower portion of the column while washing water and regenerant are passed in an upflowing stream. Reference numerals 36 and 37 in Fig. 1 represent screens, which are disposed respectively at lower portion and an upper portion of the circumference of the regeneration column 2 so as to communicate respectively with the washing water supply pipe 22 and the liquid drain pipe 30. The strainer 9 comprises a hollow plate and is so disposed in the regeneration column 2 as to divide the interior of the column into an upper section and a lower section. The strainer 9 is provided, as is clearly seen in Fig. 2, with a plurality of resin passage holes 13 and a plurality of liquid spray means 16 in such manner that they are uniformly distributed across the column. Such structures of the strainer and the liquid spray means apply to those for the ion exchange column 1. The water collecting means 10 is disposed below the strainer 9 and comprises a cross-shape perforated pipe, and is entirely covered with a net which is not shown. The bypass pipe 34 extends

from the water collecting means 10 and communicates with the hollow portion 14 of the strainer 9. A regenerant supply pipe 21 is connected to an intermediary portion of the bypass pipe 34. The hopper 11 is connected to the top of the regeneration column 2 through a resin introduction pipe 38. The measurement tank 12 is connected to the bottom of the regeneration column 2 through a resin drain pipe 43.

As has been discussed, the regeneration column 2 is packed initially with the spent ion exchange resin which has been supplied from the measurement tank 6 of the ion exchange column 1 via the hopper 11. Regeneration of spent resin is carried out in the regeneration column 2 while spent resin is supplied thereto from the hopper 11. In operation, the valve 28 of the regenerant supply pipe 21, the valve 29 of the washing water supply pipe 22 and the valve 31 of the liquid drain pipe 30 are opened, while the valves 23 and 32 for transfer of resin are closed. Under such state of the valves, washing water is passed into the regeneration column 2 through the washing water supply pipe 22, and regenerant is supplied through the regenerant supply pipe 21, while the liquid comprising mainly the used regenerant with some washing water is drained from the liquid drain pipe 30. The washing water fed through the washing water supply pipe 22 advances upwardly through the layer of resin in the lower part of the column, washing away regenerant adhering to the regenerated ion exchange resin. The majority of the washing water after completing the washing operation is drained from the pipe 33 and substantially all the remainder is collected by the water collecting means 10 and enters into the bypass pipe 34. In the bypass pipe 34, strong regenerant which is fed through the pipe 21 is mixed with the washing water containing washed regenerant, and the regenerant which is thus uniformly diluted enters into the strainer 9, and after pasisng through the liquid spray means 16, advances to the top of the regeneration column 2. In the area above the strainer 9, the regenerant discharged from the liquid spray means 16 is mixed with the washing water which is moving upwards after passing through the resin passage holes 13 and is thus further diluted. The mixture of water and regenerant advances further upwards while regenerating the spent resin, and flows out of the column from the liquid drain pipe 30.

The resin contained in the regeneration column 2, on the other hand, is caused to move downwards when the supply of the regenerant and washing water which is effected at predetermined intervals is stopped and the valves 23, 32 and 39 are opened. The resin which is regenerated when in contact with the regenerant drops downwardly through the resin passage holes 13 in the strainer 9, and is washed by the washing water supplied through the pipe 22, and flows into the measurement tank 12 through the resin drain pipe 43.

The washing water which enters into the regeneration column 2 through the pipe 22 advances upwardly in the column while washing the resin stacked in the lower portion of the column. The majority of the washing water which has washed the resin is drained from the pipe 33, and the remainder is drained through the water collecting means 9 into the bypass pipe 34. The washing water which drains into the bypass pipe 34 is uniformly mixed with strong regenerant supplied from the pipe 21 and the resulting mixture enters into the column 2. As a consequence, the regenerant which passes through the layer of ion exchange resin located in the upper portion of the column 2 has a substantially uniform concentration, resulting in an enhanced regeneration efficiency. In Fig. 1, the respective operation zones in the regeneration column 2 are indicated by the letters X, Y and Z. X represents the regeneration section, Y represents the extrusion section ,and Z represents the washing section.

It will be appreciated that regeneration and washing of the spent ion exchange resin are carried out simultaneously in the regeneration column as has been described. In addition, the orderly flow of resin effected through the measurement tank, along with the actions of the strainer, the liquid spray means and the water collection means, contribute to a remarkable enhancement of the regeneration efficiency.

In one exemplary embodiment of the present invention, an ion exchange apparatus comprising an ion exchange column having a diameter of 500 mm and a height of 1000 mm with an annexed measurement tank having a capacity of 40 litres, and a regeneration column having a diameter of 250 mm and a height of 3500 mm (of which the regeneration section measuring 1500 mm, the extrusion section 1000 mm, and the washing section 1000 mm) with an annexed measurement tank having a capacity of 10 litres, arranged as shown in Fig. 1, was used. Ion exchange operation was conducted with a satisfactory result, with the regeneration cycle being set for once in 8 minutes and the ion exchange cycle once in 32 minutes, and under the following conditions:

Resin used: Dia Ion SKIB (a product of Mitsubishi Kasei Co.)
Concentration of raw feed: 790 ppm (as $CaCO_3$). Sodium: 35%
Liquid supply rate: 7.1 m^3/hr.

Resin circulation rate: 76.8 litres-resin/hr
Regeneration concentration: 85.5g (HCl)/litre-resin.
Amount of regenerant used: 53% (which was the effectively used quantity. In case of a fixed bed, this rate was 25 to 30%)
Resistivity of treated water: 80000 Ω cm.

WHAT WE CLAIM IS:—

1. An ion exchange apparatus comprising in combination,

(a) an ion exchange column,
(b) an upper strainer disposed in the upper portion within the ion exchange column,
(c) an outlet for the liquid connected to the upper strainer,
(d) a lower strainer within the ion exchange column and spaced from the lower end thereof,
(e) an inlet for the liquid connected to the lower strainer,
(f) a measurement tank connected to the lower end of the ion exchange column through a first valve,
(g) a regeneration column,
(h) an outlet for the regenerant disposed in the upper portion of the regeneration column,
(i) a regenerant strainer disposed in an intermediate portion within the regeneration column,
(j) an inlet for the regenerant connected to the regenerant strainer,
(k) an inlet for washing water disposed in the lower portion of the regeneration column,
(l) an outlet for washing water disposed between the regenerant strainer and the inlet for washing water,
(m) a measurement tank connected to the lower end of the regeneration column through a second valve.

2. Apparatus as claimed in claim 1, in which the lower strainer comprises a hollow plate having through passageways for the passage of resin, a liquid inlet and liquid spray means for spraying into the interior of the column liquid introduced into the hollow plate through the inlet.

3. Apparatus as claimed in claim 2 including a perforated plate located in the ion exchange column just above the lower strainer.

4. Apparatus as claimed in any preceding claim in which the upper strainer comprises a hollow plate having through passageways for the passage of resin, and spray apertures for the entry of liquid from the column into the hollow plate, the liquid outlet being connected so as to withdraw from the column liquid which enters the hollow plate of the upper strainer.

5. Apparatus as claimed in any preceding claim wherein the resin regeneration column contains a strainer and water collecting means disposed below the strainer, the strainer comprising a hollow plate dividing the regeneration column into upper and lower sections and having through passageways for the passage of resin and liquid spray means on the upper surface of the plate for spraying liquid into the interior of the column.

6. Apparatus as claimed in claim 5 wherein the resin regeneration column includes a peripheral water inlet adjacent to the base of the column, a peripheral water outlet located between the peripheral water inlet and the water collecting means, and a peripheral fluid outlet adjacent to the top of the column.

7. Ion exchange apparatus substantially as described herein with reference to the accompanying drawings.

8. A method of transferring resin in an ion exchange apparatus as claimed in claim 1, characterized in that the resin contained in the ion exchange column is caused to moved downwardly by the formation of a layer of liquid below the lower strainer in the bottom portion of said ion exchange column with the liquid forced into said column.

9. A method of regenerating resin in an ion exchange apparatus of the type as described in claim 1, characterized in that said regeneration of resin is effected by a solution comprising a mixture of a strong regenerant and washing water introduced into said apparatus.

10. A method for operating an ion exchange apparatus, including cyclically using and regenerating an ion exchange resin, substantially as described herein with reference to the accompanying drawings.

Agents for the Applicants:
STANLEY, POPPLEWELL, FRANCIS and ROSS,
Chartered Patent Agents,
Cursitor House,
9—11, Cursitor Street,
Chancery Lane,
London, E.C.4.

Printed for Her Majesty's Stationery Office by Burgess & Son (Abingdon), Ltd.—1970.
Published at The Patent Office, 25 Southampton Buildings, London, W.C.2,
from which copies may be obtained.

Resin circulation rate: 76.8 litres-resin/hr
Regeneration concentration: 85.5g (HCl)/litre-resin.
Amount of regenerant used: 53% (which was the effectively used quantity. In case of a fixed bed, this rate was 25 to 30%)
Resistivity of treated water: 80000 Ω cm.

WHAT WE CLAIM IS:—

1. An ion exchange apparatus comprising in combination,

(a) an ion exchange column,
(b) an upper strainer disposed in the upper portion within the ion exchange column,
(c) an outlet for the liquid connected to the upper strainer,
(d) a lower strainer within the ion exchange column and spaced from the lower end thereof,
(e) an inlet for the liquid connected to the lower strainer,
(f) a measurement tank connected to the lower end of the ion exchange column through a first valve,
(g) a regeneration column,
(h) an outlet for the regenerant disposed in the upper portion of the regeneration column,
(i) a regenerant strainer disposed in an intermediate portion within the regeneration column,
(j) an inlet for the regenerant connected to the regenerant strainer,
(k) an inlet for washing water disposed in the lower portion of the regeneration column,
(l) an outlet for washing water disposed between the regenerant strainer and the inlet for washing water,
(m) a measurement tank connected to the lower end of the regeneration column through a second valve.

2. Apparatus as claimed in claim 1, in which the lower strainer comprises a hollow plate having through passageways for the passage of resin, a liquid inlet and liquid spray means for spraying into the interior of the column liquid introduced into the hollow plate through the inlet.

3. Apparatus as claimed in claim 2 including a perforated plate located in the ion exchange column just above the lower strainer.

4. Apparatus as claimed in any preceding claim in which the upper strainer comprises a hollow plate having through passageways for the passage of resin, and spray apertures for the entry of liquid from the column into the hollow plate, the liquid outlet being connected so as to withdraw from the column liquid which enters the hollow plate of the upper strainer.

5. Apparatus as claimed in any preceding claim wherein the resin regeneration column contains a strainer and water collecting means disposed below the strainer, the strainer comprising a hollow plate dividing the regeneration column into upper and lower sections and having through passageways for the passage of resin and liquid spray means on the upper surface of the plate for spraying liquid into the interior of the column.

6. Apparatus as claimed in claim 5 wherein the resin regeneration column includes a peripheral water inlet adjacent to the base of the column, a peripheral water outlet located between the peripheral water inlet and the water collecting means, and a peripheral fluid outlet adjacent to the top of the column.

7. Ion exchange apparatus substantially as described herein with reference to the accompanying drawings.

8. A method of transferring resin in an ion exchange apparatus as claimed in claim 1, characterized in that the resin contained in the ion exchange column is caused to moved downwardly by the formation of a layer of liquid below the lower strainer in the bottom portion of said ion exchange column with the liquid forced into said column.

9. A method of regenerating resin in an ion exchange apparatus of the type as described in claim 1, characterized in that said regeneration of resin is effected by a solution comprising a mixture of a strong regenerant and washing water introduced into said apparatus.

10. A method for operating an ion exchange apparatus, including cyclically using and regenerating an ion exchange resin, substantially as described herein with reference to the accompanying drawings.

Agents for the Applicants:
STANLEY, POPPLEWELL, FRANCIS and ROSS,
Chartered Patent Agents,
Cursitor House,
9—11, Cursitor Street,
Chancery Lane,
London, E.C.4.

Printed for Her Majesty's Stationery Office by Burgess & Son (Abingdon), Ltd.—1970.
Published at The Patent Office, 25 Southampton Buildings, London, W.C.2,
from which copies may be obtained.

