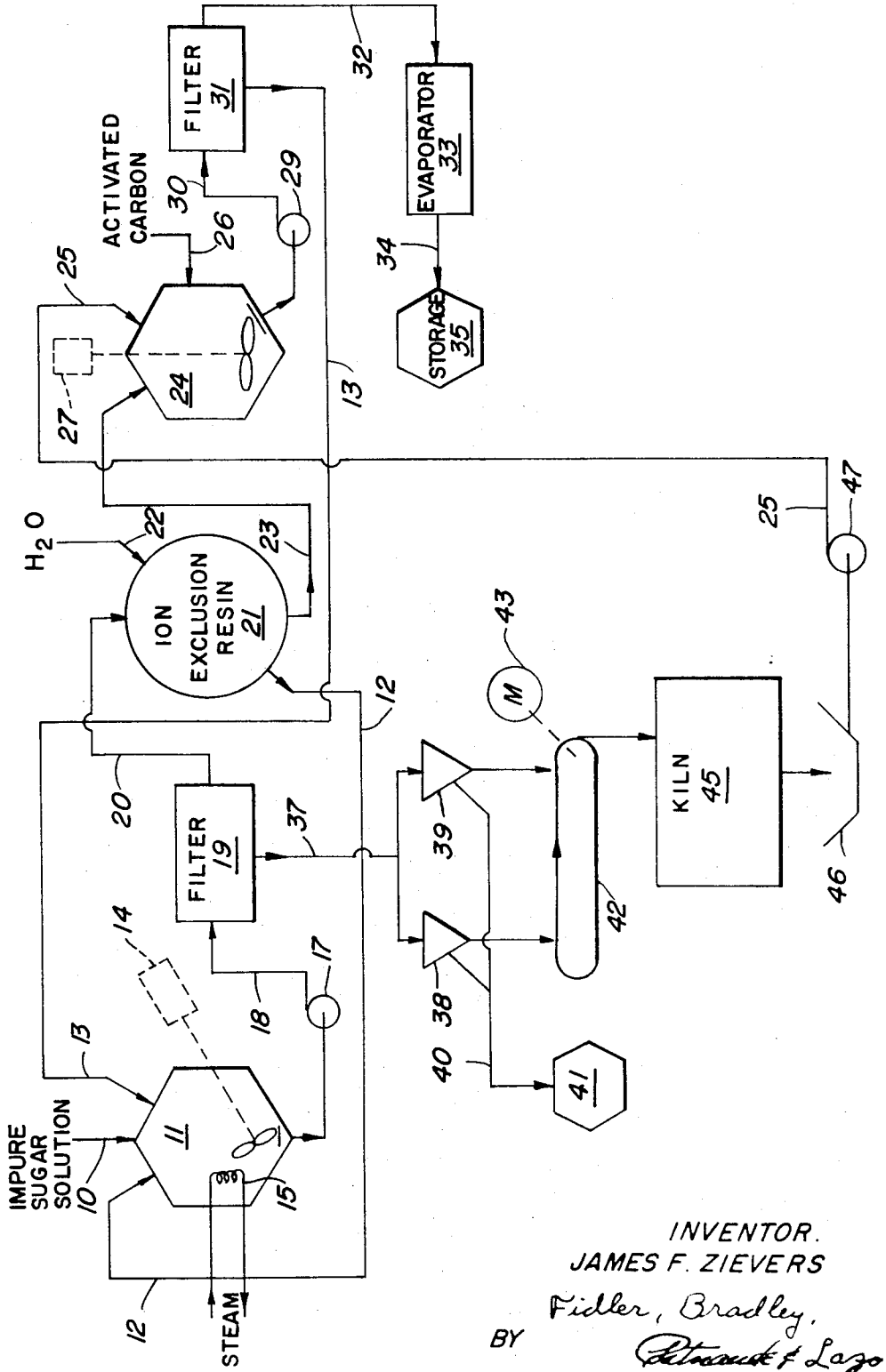
Jan. 16, 1973 J. F. ZIEVERS 3,711,329

SUGAR RECOVERY METHOD AND APPARATUS

Filed Feb. 22. 1971 2 Sheets-Sheet 1

FIG. 1



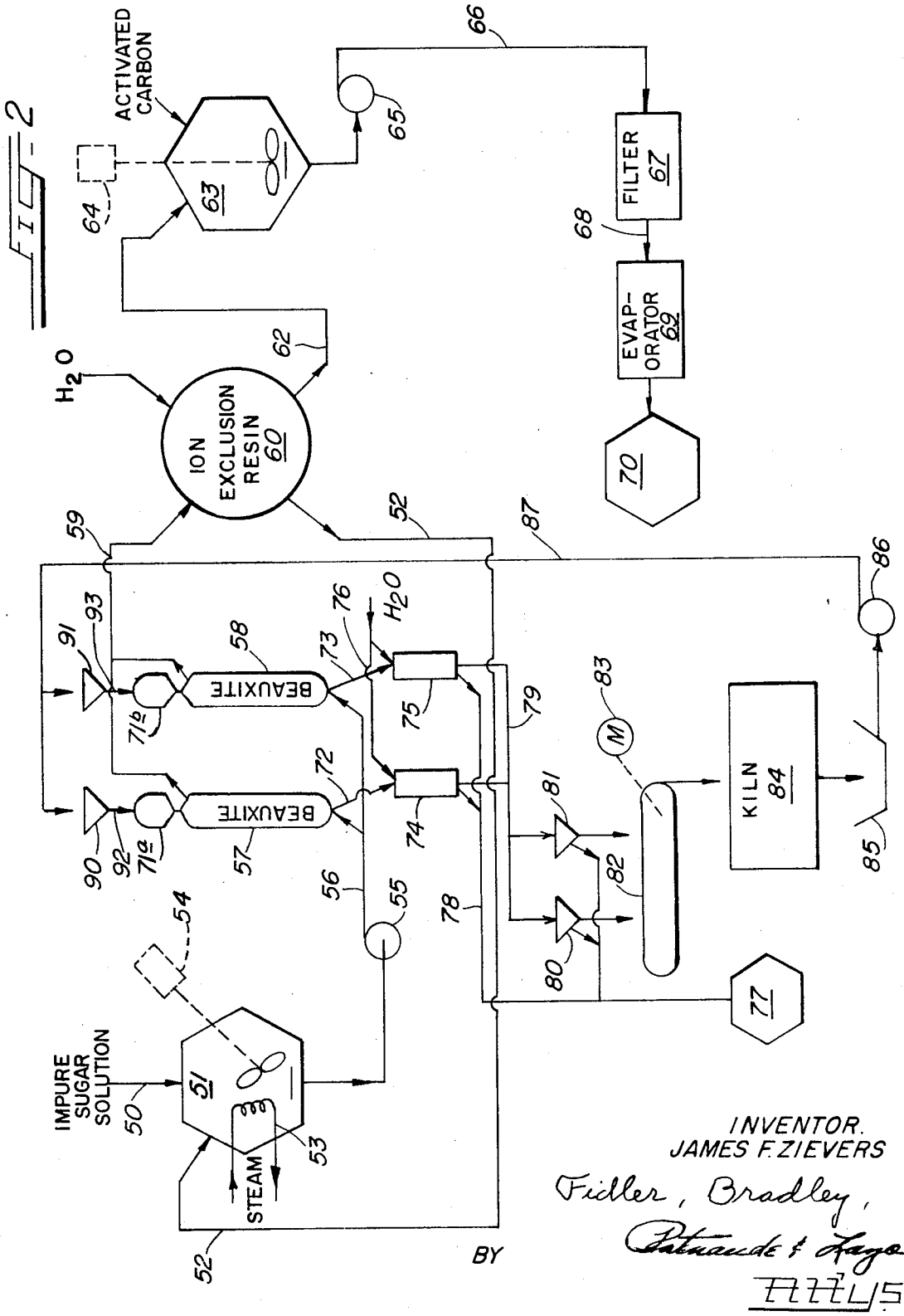
INVENTOR.
JAMES F. ZIEVERS
BY Fidler, Bradley, Strawn & Lazo
ATTYS.

Jan. 16, 1973 J. F. ZIEVERS 3,711,329

SUGAR RECOVERY METHOD AND APPARATUS

Filed Feb. 22. 1971 2 Sheets-Sheet 2

FIG-2



INVENTOR.
JAMES F. ZIEVERS

Fidler, Bradley,
Strauch & Hayes

BY

ATTYS.

United States Patent Office 3,711,329
Patented Jan. 16, 1973

3,711,329

SUGAR RECOVERY METHOD AND APPARATUS

James F. Zievers, La Grange, Ill., assignor to Industrial Filter & Pump Mfg. Co., Cicero, Ill.

Filed Feb. 22, 1971, Ser. No. 117,296
Int. Cl. C13d 3/14
U.S. Cl. 127—9 12 Claims

ABSTRACT OF THE DISCLOSURE

A method and apparatus for purifying a sugar bearing solution such as molasses treats the impure solution with bauxite prior to the passage thereof to an ion exclusion resin.

The present invention generally relates to a method and apparatus for removing ionic and large molecular impurities from an impure sugar bearing solution, and it relates more particularly to a new and improved method and apparatus which may be used to extract a substantially pure liquid sugar solution from molasses.

The use of an ion exclusion resin to separate sugar molecules from ionic and other impurities in a sugar bearing solution is known and has met with some success. For example, Pat. 3,214,293 to Mountfort discloses such a system particularly adapted to the treatment of affination syrup. Similarly, Pat. 2,937,959 discloses an ion exclusion system for use in recovering sugar from molasses. Pat. 2,890,972 to Wheaton discloses another such system. In all of these systems it is necessary to first treat the impure sugar solution with an ion exchange resin before the ion exclusion step to prevent premature fouling of the ion exclusion resin.

An object of the present invention is to provide a new and improved method and apparatus for eliminating the use of an ion exchange resin and associated regeneration equipment ahead of the ion exclusion step.

Another object of the present invention is to provide a less expensive method and apparatus for recovery sugar from impure sugar solutions.

A further object of the present invention is to provide a new and improved apparatus utilizing bauxite in an ion-exclusion sugar recovery process.

Briefly, the above and further objects may be realized in accordance with the present invention by treating the impure sugar bearing solution with activated bauxite prior to the passage thereof to an ion exclusion resin. In one embodiment of this invention, granular activated bauxite is mixed with the impure solution and then filtered out before the ion exclusion step. In another embodiment the impure solution is passed through a bed of activated bauxite prior to the ion exclusion step.

Further objects and advantages and a better understanding of the present invention may be had by reference to the following detailed description taken in connection with the accompanying drawings, wherein:

FIG. 1 is a schematic flow diagram of a system using the process of the present invention to purify a sugar solution such as molasses; and

FIG. 2 is a schematic flow diagram of another system embodying the present invention.

Referring now to the drawings and particularly to FIG. 1 thereof, an impure sugar solution such as molasses is supplied from a line **10** to a mixing and heating tank **11**. A purified, dilute aqueous solution of sugar is supplied to the tank **11** through a line **12** and particulate activated bauxite at mesh size of about **100** is supplied to the tank **11** by a line **13**. A motor driven mixer **14** is disposed in the tank to thoroughly mix the bauxite particles with the solution and a heating coil **15** immersed in the solution is fed with steam to heat the solution in the tank to a

temperature within the range of about 170 to 180-degrees F. The impure sugar solution will generally have a relatively high Brix value as, for example, a value of about 80 in the case of molasses. It is desired that the effluent from the tank have a Brix value of about 60, and therefore, additional water may be added to the tank **11** if the sweet water supplied through the line **12** is insufficient to reduce the Brix value to that desired.

The effluent from the tank **11** is pumped by a means of a pump **17** through a line **18** to a filter **19** wherein the solids entrained in the impure sugar solution are removed. The filter **19** is a mechanical type filter and may be of the type described in United States application (Henry Schmidt, Jr.), Ser. No. 65,393, filed Aug. 20, 1970. The effluent from the filter **19** flows through a line **20** into a column **21** containing a bed of an ion exclusion resin. This resin may be of either the anion or cation type. At the present time, however, because of the relative costs of cation and anion exchange resins, it is preferable to use a cation resin for ion exclusion purposes. Any suitable resin, such as, for example, a strongly acid cation exchange resin comprising a sulfonated styrene-divinylbenzene polymerizate having a cross-linkage or percent of divinylbenzene in the hydrogen form no greater than four and a mesh range of 50 to 100, i.e., a particle size of .149 to .297 millimeter. One such resin is sold by Dow Chemical Company under the trade name Dowex 50X4. The ion exclusion process is carried out in the tank **21** in the usual manner as described, for example, in Pat. 3,214,293, Mountfort. In this process the tank **21** is substantially filled with the resin particles and the sugar molecules are separated from the larger particles such, for example, as the color bodies by virtue of the sugar molecules being absorbed into the gel within the resin beads while the other solutes occupy the interstitial space between the resin particles. After a sufficient time has elapsed to permit the resin beads to become saturated with the sugar molecules, water from a line **22** is flowed through the tank. Initially, the effluent from the tank **21** is an aqueous solution highly concentrated with the impurities contained in the interstitial liquid. As the flow of water through the tank **21** continues, the concentration of the impurities diminishes as the water begins to displace the sugar molecules in the resin beads. At this time the effluent is a sugar bearing solution having a considerably greater purity than the impure sugar solution initially supplied to the system and is usually retained for further purification.

As the flow of water continues and all of the impurities have been washed from the tank **21**, the effluent becomes a highly purified sugar solution. This fraction is fed through a line **23** to a second mixing tank **24** where it is mixed with bauxite fed to the tank **24** through a line **25** and with activated carbon fed through a line **26**. A suitable motor operated mixer **27** is disposed in the tank to thoroughly mix the bauxite and carbon with the purified sugar solution. As the flow of water through the tank **21** continues, the concentration of sugar effluent decreases and when it falls below about 40 degrees Brix the effluent is connected by suitable valve means (not shown) to the line **12** so that it is returned to the original mixing tank **11** at the head of the system wherein it is mixed with the initial impure sugar solution. The effluent from the tank **24** is pumped by a pump **29** through a line **30** to a filter **31** wherein the activated carbon particles and the bauxite particles are removed. The effluent from the filter **31** flows through a line **32** to an evaporator **33** wherein water is evaporated from the sugar solution so that the effluent from the evaporator **33** which flows through line **34** to a storage tank **35** has a Brix value of between sixty and seventy. The solids, i.e., bauxite and carbon particles filtered from the sugar solution by the filter **31** are fed to the line **13** from which they flow into the mixing tank **10**

at the head of the system. The solids which are removed by the filter **19** are fed through a line **37** to a pair of settling tanks **38** and **39**. The tanks **38** and **39** are alternately filled and retain the solids for a sufficient time to permit the liquid carried thereby to settle to the bottom of the tank from which it is fed through a line **40** to a suitable retention tank **41**. The solids from the tanks **38** and **39** are dropped onto a conveyor belt **42** suitably driven by a motor **43** and fed to a kiln **45** wherein the bauxite particles are purified by burning off the impurities which were captured thereby in the mixing tank **11**. Also, the carbon particles are disintegrated in the kiln **45**.

The thus purified particulate bauxite from the kiln **45** is fed by gravity to a hopper **46** from which it is pumped by a pump **47** through the line **25** to the second mixing tank **24**.

Referring now to FIG. 2, the impure sugar solution is supplied from the line **50** to a heating and mixing tank **51** to which a dilute aqueous sugar solution, such for example, as sweet water, is supplied through a line **52**. The contents of the tank **51** is heated by a steam coil **53** immersed therein to a temperature of about 170 to 180 degrees F., and a motor driven mixer **54** may be provided for thoroughly mixing the contents of the tank **51** to maintain it in a fluid state. When the impure sugar solution is molasses it may have a Brix value of about 80 and if the amount of sweet water from the line **52** is insufficient to reduce this value to about 60, additional water should be added. The effluent from the tank **51** is pumped by a pump **55** through a line **56** to a plurality of columns **57** and **58** which are substantially filled with a granular activated bauxite having a mesh size of about 12 by 20. In addition to removing monovalent ions from the sugar solution, the bauxite bed also functions as a mechanical filter to remove entrained solids from the solution as it passes therethrough.

The columns **57** and **58** are alternately used so that the effluent from the tank **51** flows through one of the columns while the bauxite from the other column is being renewed. Suitable valving, not shown, is used for this purpose. The effluent from one of the other columns **57** and **58** passes out of the top of that column into a line **59** which feeds into a column **60** containing a bed of ion exclusion resin of either the cation or anion type as is used in the column **21** in the embodiment of FIG. 1 described hereinabove. The column **60** is operated in the same manner as is column **21** to provide a plurality of fractions beginning with an effluent which is substantially free of sugar and containing a high concentration of the impurities from the initial sugar solution, a second fraction containing some sugar but also including a high concentration of impurities, a third fraction which is a substantially pure sugar solution having a concentration of about 40 degrees Brix and finally a fourth fraction which is a pure but an extremely dilute aqueous sugar solution having a Brix value less than 40.

The third fraction, namely, the purified concentrated sugar solution is fed from the column **60** through a line **62** to a second mixing tank **63** wherein powdered activated carbon is added thereto to inhibit the growth of thermofiles and to improve the color of the sugar solution. A motor operated mixer or agitator **64** is disposed in the tank **63** to thoroughly mix the carbon particles with the sugar solution.

The effluent from the tank **63** is pumped by a pump **65** through a line **66** to a filter **67** wherein the carbon particles are removed by the deposition thereof on a filter cake on the filter elements of the filter. The filter **67** may be of any suitable type for removing the fine carbon particles from the sugar solution. For example, it may be a pressure type leaf or tube filter. The effluent from the filter **67** is fed through a line **68** to an evaporator **69** wherein water is evaporated from the solution to increase the Brix value of the sugar solution to about 60 to 70 Brix. The effluent from the evaporator **69** is fed to a storage tank **70**.

As the bauxite in the columns **57** and **58** becomes spent, it must be replaced. In accordance with the present invention fresh bauxite is periodically added in small amounts to the tops of the columns **57** and **58** from a pair of pressurizable feed tanks **71***a* and **71***b* and an equal amount of spent bauxite is removed from the bottoms of the columns through a pair of lines **72** and **73** and fed to respective washing tanks **74** and **75**. The spent bauxite in tanks **74** and **75** is washed by flowing water therethrough from a line **76** and the liquid effluent from these tanks is supplied to a retention tank **77** through a line **78**.

The bauxite granules from the tanks **74** and **75** are fed through a line **79** connected to both tanks to a pair of hoppers **80** and **81** and from these hoppers to a conveyor belt **82** driven by a motor **83** which drops the semi-dry bauxite into a kiln **84**. The hoppers **80** and **81** are alternately fed by the tanks **74** and **75** and in turn alternately feed the conveyor belt to provide an additional retention time during which more liquid may flow off to the tank **77**. In the kiln **84**, the bauxite is heated to a temperature of about 600 degrees F. to burn off the impurities carried on the surface of the bauxite granules and in the pores thereof. The thus purified bauxite from the kiln **84** is fed to a hopper **85** from which it is pumped by a pump **86** through a line **87** to a pair of hoppers **90** and **91** mounted above the feed tanks **71***a* and **71***b* and respectively connected thereto by means of lines **92** and **93**.

As indicated hereinbefore, the columns **57** and **58** are alternately connected between lines **56** and **59**. When the column **57** is connected between the lines **56** and **59** it is sealed off from the line **72** and the feed tank while at the same time the column **58** is sealed off from the lines **56** and **59**. During this period of time the feed tank is connected to the hopper **91** and fresh bauxite falls by gravity therein. Thereafter, and very briefly, as, for example, twenty to thirty seconds, the feed tank **71***b* is pressurized by a gas and connections between the column **58** and the tank **71***b* and the line **73** are opened whereby a small pulse of fresh bauxite enters the top of the column **58** and an equal amount of spent carbon leaves the bottom. The column **58** thus remains filled with bauxite at all times. Similarly, while the column **58** is on line, i.e., connected between the lines **56** and **59**, the bauxite in the column **57** is renewed. It will be apparent that since small amounts of bauxite are added to the top of the column to replace an equal amount of bauxite removed from the bottom of the column, the sugar solution passing up through the columns **57** and **58** first passes the most used bauxite and as it travels up through the bauxite bed it encounters fresher and fresher bauxite. During the time that the columns **57** and **58** are being respectively pulsed, the other column is on line so that the flow of effluent through the line **59** may be continuous if desired. It will be apparent, however, that only one column, **57** or **58** can be used if desired. During the time that flow from the ion exclusion column **60** is interrupted, the flow through the filter **67** may also be interrupted to remove the filter cake from the filter elements.

If only a single ion exclusion tank **60** is employed, it will be apparent to those skilled in the art that the flow through the line **59** is not continuous. However, if a continuous flow is desired, two ion exclusion resin tanks can be provided and alternately connected between the line **59** and the line **62**. In such a case, when one of the ion exclusion columns **60** is connected to the line **59**, it will be disconnected from the line **62**. At that same time the other column is connected to the line **62** and disconnected from line **59**.

While the present invention has been described in connection with particular embodiments thereof, it will be understood that many changes and modifications of this invention may be made by those skilled in the art without departing from the true spirit and the scope thereof. Accordingly, the appended claims are intended to cover all

such changes and modifications as fall within the true spirit and scope of the present invention.

What is claimed is:

1. A method of purifying an impure sugar solution such as:

molasses containing monovalent and multivalent cations, comprising the steps of:

heating the impure sugar solution to about 170° F. to 180° F.,

contacting the heated impure sugar solution with bauxite to remove the monovalent ions therefrom,

passing the treated solution into a body of ion exchange resin particles capable of retaining in the pores of the resin particles a major portion of the sugar molecules while excluding a major portion of other solutes in said sugar solution,

thereafter contacting said resin particles with water to wash out said sugar molecules and separating the effluent into a plurality of fractions wherein the solids in one of said fractions are primarily sugar,

mixing activated carbon particles with said one of said fractions,

removing said carbon particles from said one of said fractions, and then

evaporating said one of said fractions to provide a purified sugar solution having a Brix value in the range of about 60 to 70.

2. A method according to claim 1 comprising the additional step of:

collecting from the effluent another fraction wherein the solids are primarily sugar and having a Brix value less than about 26, and

adding said another fraction to said sugar solution to be treated.

3. A method according to claim 1 in which said bauxite after being used to treat said sugar solution is oven heated to burn off the adsorbed solids and then reused to treat said sugar solution.

4. A method according to claim 1 in which:

said sugar solution is treated with bauxite by mixing particulate bauxite with said sugar solution, and comprising the additional step of

filtering said particulate bauxite from said sugar solution before passing said solution into said body of ion exchange resin.

5. A method according to claim 1 wherein said sugar solution is treated with bauxite by:

providing a bed of granular bauxite, and

passing said sugar solution through said bed,

whereby said bed of bauxite filters entrained solids from said sugar solution prior to the passage thereof into said body of ion exchange resin particles.

6. A method according to claim 1 wherein said sugar solution is molasses having a Brix value of about 80, and comprising the additional step of:

adding an aqueous solution to said molasses to reduce the Brix value to about 60 before passing said solution into said body of ion exchange resin.

7. A method according to claim 6 wherein said ion exchange resin is a strongly acid cation exchange resin com-

prising a sulphonated styrene-divinylbenzene polymer, said resin being a cross-linkage of divinylbenzene in the hydrogen form.

8. A method according to claim 1 comprising the additional step of treating said one of said fractions with bauxite prior to said evaporating step.

9. A method according to claim 8 wherein the step of treating said one of said fractions comprises the steps of:

mixing particulate bauxite with said one of said fractions and then

filtering the resulting mixture to remove the bauxite therefrom.

10. A method according to claim 9 comprising the additional step of reusing the bauxite filtered out of the sugar solution in said treating of the impure sugar solution with bauxite.

11. Apparatus for purifying molasses, comprising:

a first mixing tank for receiving said molasses and an aqueous solution,

heating means in said tank,

a receptacle containing an ion exclusion resin,

a pump and conduits connected between said tank and said receptacle for pumping liquid from said tank through said receptacle,

a column of bauxite connected between said first mixing tank and said receptacle containing an ion exchange resin,

a second mixing tank,

a conduit connected between said receptacle and said second mixing tank for carrying a purified sugar effluent from said receptacle to said second mixing tank,

an evaporator, and

a filter connected between said second mixing tank and said evaporator for removing entrained solids from the effluent from said mixing tank before said effluent is fed to said evaporator.

12. Apparatus according to claim 11 comprising:

filter means connected between said first mixing tank and said receptacle containing an ion exclusion resin for removing entrained solids from the effluent from said first mixing tank before passage thereof into said receptacle.

References Cited

UNITED STATES PATENTS

2,868,677	1/1959	Kopke	127—46 B
2,937,959	5/1960	Roents	127—9 X
3,563,799	2/1971	Zievers	127—46 A
3,098,045	7/1963	Allegrini	252—455 R

FOREIGN PATENTS

560,795	4/1957	Italy.

MORRIS O. WOLK, Primary Examiner

S. MARANTZ, Assistant Examiner

U.S. Cl. X.R.

127—46 B