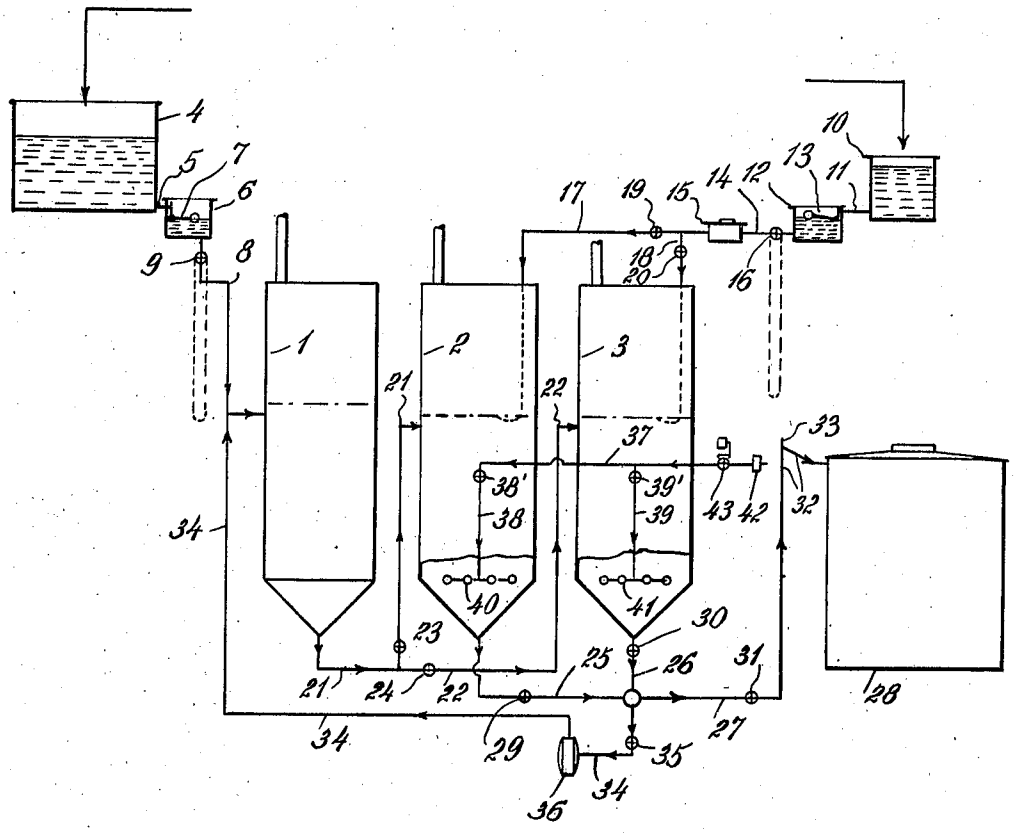
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MANUFACTURE OF SUGAR

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MANUFACTURE OF SUGAR

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This invention relates to the manufacture of sugar and has for its object certain improvements in the method of manufacturing sugar. The invention relates more especially to the treatment of juices in the manufacture of sugar, and particularly to improvements in the method of purifying and clarifying beet juices.

In the heretofore customary methods of manufacturing sugar from beet juices, the beets are washed and then sliced into cossettes. The sliced beets are next subjected to a counter current flow of warm water under pressure, in a so-called diffusion battery. The diffusion juice is dark in color and contains in suspension or solution the colloidal and soluble contents of the beet, i. e., sucrose, potassium and sodium salts of phosphoric, sulfuric, hydrochloric, oxalic, and tartaric acids, proteins, pectins, albumen, etc., and a small amount of invert sugar. The sugar content of the juice varies from about 9 to 16% by weight. In reaction it is slightly acid. The suspended fibre, cellular tissue and coagulated albumen are separated from the juice. The juice is then heated to about 85° C., which has the effect of coagulating a portion of the albumen, besides preparing it for clarification. Lime, usually in the form of milk of lime, or calcium saccharate, is added to the hot juice in amount varying from 2 to 3% of the weight of the beets employed. This throws the juice from its acid to an alkaline state. The limed juice is frequently subjected to prolonged agitation, varying from 15 to 20 minutes, at an elevated temperature.

The limed or defecated juice is subsequently subjected to a so-called carbonation step, which consists in subjecting the juice to gas containing about 30% carbon dioxide. As a result of this treatment, calcium carbonate is precipitated. As many as five tanks are employed in the carbonation step. The operation is usually carried on in batches. One tank of the limed juice is subjected to the carbonation step, while the other tanks are being prepared for the same operation. When the gas is first introduced into the limed juice, a gelatinous and practically non-settling material is produced. As gassing is continued, the nature of the precipitate changes, and when the alkalinity has been reduced to about 0.10–0.15 grams of lime (calcium oxide) per 100 cc. of filtrate, the precipitate becomes flocculent and it can be readily settled or filtered. If the gassing is continued, and the alkalinity of the juice is still further reduced, the precipitate becomes more and more flocculent, but a point is soon reached where some of the impurities that have been removed from the solution begin to go back into solution.

This heretofore customary practice of manufacturing sugar from beet juice is highly complex, involves many operations, and requires the attention of very skilled operators. For example, if the first carbonation step is carried too far or is not carried far enough, it is substantially impossible to obtain a clear filtrate. In other words, unless the gassing operation is conducted to a precise end point, it is substantially impossible to obtain a clear separation of solids from liquids. Due to the fact that the operations are conducted in batches and the controls are all exerted manually, by turning valves, it has been extremely difficult to obtain substantially uniform purified and clarified sugar juices at the end of the process.

As a result of my investigations, I have determined that the treatment of beet juice in the manufacture of sugar may be greatly simplified and made far more efficient than is the case in the present practice. The purifying and clarifying steps may be accurately controlled, a better separation of solids and liquor may be effected, and the beet juice obtained at the end of the process is of a uniformly high grade.

In accordance with the practice of the present invention, beet juice is treated for the manufacture of sugar in such manner that the dissolved lime in the juice does not reach the saturation point. This may be accomplished either by introducing into the juice an amount of lime substantially less than that which will produce saturation when completely dissolved, or by subjecting the juice to the action of carbon dioxide or the like so promptly after the introduction into the juice of the lime that the lime does not have sufficient time to

form a saturated solution, and thereafter gassing the juice continuously at a rate that will prevent saturation. To this end, the invention involves the step of adding lime only to juice that is being substantially simultaneously gassed. My invention also contemplates mixing raw diffusion juice with previously carbonated juice before subjecting the raw juice to a gassing operation. The present invention also contemplates the step of repeatedly circulating such juice, at least in part, through a gassing zone. In the present preferred practice of the invention the above steps are advantageously combined, though they may also be used singly.

In referring to carbonation herein the expression is used in the sense customary in the art to indicate the usual treatment with carbon dioxide or its equivalent; and in accordance with customary usage references to carbonated juice are intended to designate juice that has been subjected to such customary carbonation, and which according to the usual practice contains a residual amount of dissolved lime.

In the copending application of Messrs. Ramsey and Bull, Serial No. 107,579, filed May 8, 1926, of which I am a co-applicant, there is disclosed a method and apparatus for automatically and quickly controlling the carbonation of sugar juices to predetermined degrees of alkalinity. Moreover, the method and apparatus therein described may be employed in such manner as to effect the continuous carbonation of sugar juices by such automatic control. The practice of the invention therein disclosed may advantageously be followed in the practice of the present invention.

I have found that the addition of a liming agent soluble in the juice, ordinarily comprising a suitable compound of calcium, such as fresh lime or calcium saccharate, to beet juice that is being substantially simultaneously gassed offers very important advantages. This step (liming) results in a precipitation of calcium carbonate from a solution much lower in concentration with respect to dissolved lime, for example, than is the case when lime is added outside the gassing zone. When the lime is added to the juice some distance away from the gassing zone, and particularly when the limed juice is agitated as in the general practice heretofore resorted to, a sufficient amount of lime will go into true solution to reach the saturation point. Under such operating conditions the initial precipitating of calcium carbonate from the gassing zone is from a solution completely or practically completely saturated with dissolved lime. In the practice of the present invention, on the other hand, the precipitation of calcium carbonate is effected from a solution of limed juice that is only partially saturated with lime in true solution.

It is a very definite advantage to prevent the alkalinity of the juice from ever reaching the high value corresponding to that obtaining when the juice is saturated with respect to dissolved lime. Under such circumstances the calcium carbonate particles settle very slowly and the mixture may only be filtered with considerable difficulty. The introduction of lime to the raw diffusion juice at some distance and time before the gassing operation, as well as the agitation of the limed juice, is harmful because the juice will become saturated with dissolved lime, and a high alkalinity is established. Albuminoids and the like present in the juice are attacked by the lime in solution. This reaction produces a relatively large proportion of very fine colloids. These finely divided colloids in turn are adsorbed on the calcium carbonate particles and inhibit crystal growth. On the other hand, when the juice is only partially saturated with lime in solution, and a relatively low alkalinity is established, a very much smaller amount of the fine colloids is produced. Hence the adsorption of colloids on the surfaces of the calcium carbonate particles as a result of carefully controlling liming is materially inhibited, and crystal growth is encouraged. The establishment of relatively low alkalinities in the juice by controlled liming aids in the crystallizing step in that fewer and larger calcium carbonate particles are precipitated, since the number and size of crystals increase and decrease, respectively, with increased concentration of lime in solution. The lime in suspension, that is, undissolved lime, has no effect as such on the alkalinity of the juice, and hence plays no important role in the reactions just discussed. High alkalinities are therefore to be avoided as much as possible, and I have accomplished this desirable result by introducing the lime into the juice substantially simultaneously with the introduction of the carbon dioxide gas.

My investigations have also shown that the mixing of previously carbonated juice with raw diffusion juice before introduction into the gassing zone results in certain marked advantages in the purification and clarifying operation. Since the raw diffusion juice is slightly acidic and the carbonated juice is alkaline (due to the lime in true solution), the mixture of the two in proper proportions gradually renders the raw juice alkaline. The purification of the juice is both mechanically and chemically effected. The mechanical purification is due to the fact that the suspended calcium carbonate particles become covered or coated with colloidal matter in suspension throughout the raw juice. A portion of these colloidal constituents contact with and are adsorbed on the calcium carbonate particles. The removal of such colloidal matter in this manner aids the further nor-

mal precipitation of calcium carbonate particles, since such adsorbed colloids are effectively removed from the reaction zone.

In addition to the mechanical removal of colloidal constituents, as just pointed out, certain chemical reactions take place to purify the juice. Dissolved lime in true solution reacts chemically with certain dissolved impurities in the raw juice, such as potassium and sodium salts of phosphoric, sulfuric, oxalic and tartaric acids, to form substantially water-insoluble compounds. For example, raw diffusion juice undoubtedly contains some sodium phosphate in solution. Sodium phosphate reacts with the lime in solution to form relatively water insoluble calcium phosphate. As the carbonated and raw juices are circulated, the solids in suspension contact with one another and tend to build into larger particles or agglomerates that are readily separable from their liquid vehicle either by settling or filtering. Such water insoluble particles also operate to purify the juice mechanically by adsorption on their surfaces of objectionable colloidal matter.

In this manner raw diffusion juice eventually reaches the gassing zone in at least a partially purified condition. As a result of this preliminary purification, the surfaces of the freshly precipitated calcium carbonate particles formed during the gassing period are less contaminated with colloidal matter than in the process heretofore practiced. The substantially clean surfaces of the freshly precipitated calcium carbonate particles lend themselves to particle growth. Such particle growth increases the average size of the calcium carbonate crystals, which is particularly desirable for efficient and rapid settling and filtering.

Marked advantages are to be obtained when carbonated juice is, at least in part, repeatly circulated through a gassing zone. Such recirculation of carbonated juice tends materially to aid in the building-up of calcium carbonate particles. Particle growth is particularly fostered on such individual calcium carbonate particles recirculated as have substantially clean surfaces. It is therefore preferred to combine this feature of recirculation with the steps outlined above.

The calcium carbonate precipitation step itself is materially aided by the presence of previously precipitated calcium carbonate particles. That is to say, the calcium carbonate particles tend to act as nuclei for the precipitation of further amounts of calcium carbonate out of solution.

These and other advantages of the present invention will, it is believed, be better understood by reference to the accompanying drawing, taken in conjunction with the following description, in which is diagrammatically shown a form of apparatus adapted to practice the invention.

The set-up shown in the drawing consists of three main treatment tanks 1, 2 and 3, provided with outlet vents at the tops thereof for the escape of unabsorbed gas. A juice storage tank 4, preferably located at a level above the main treatment tanks, connects by means of pipe 5 with a constant level tank 6. This latter tank is provided with a float valve 7. The constant level tank connects with the first treatment tank 1 by means of a pipe line 8. This pipe line is provided with a chain operated valve 9.

A lime storage tank 10, preferably located at a level above the main treatment tanks, connected by means of a pipe 11 with a constant level tank 12. This constant level tank is also provided with a float valve 13. A pipe line 14 connects the constant level tanks and a "sight-box 15." This box contains a weir for the measurement of the amount of lime passing therethrough. The pipe line is equipped with a chain operated valve 16. Carbonation tanks 2 and 3 are connected to the "sight-box" by means of pipe lines 17 and 18, both pipes being provided with valves 19 and 20, respectively.

The main treatment tanks 1, 2 and 3 are appropriately interconnected by means of suitable pipe lines. Tank 1 connects with tank 2 by means of a pipe line 21 that extends from the bottom of tank 1 to near the middle of tank 2. Another pipe line 22 connects with pipe line 21 and tank 3 near its middle. Both of these pipe lines are provided with valves 23 and 24, respectively, so that tanks 2 and 3 may be alternatively put in operation with tank 1.

The bottoms of tanks 2 and 3 connect by means of interconnecting pipe lines 25 and and 26 and 27 with a settling or filtering device 28. Each of these lines is suitably provided with valves 29, 30 and 31, respectively. The pipe line 27 is provided with a loop 32 to maintain the level of carbonated juice in the tanks at a desired point. A vent 33 is provided in the loop to overcome any siphon action that may develop in the feed line to the thickener.

Tanks 2 and 3 also connect at their bottoms by means of pipes 25 and 26, respectively, with a recirculating pipe line 34, provided with a valve 35. The recirculating line is provided with a pump 36 to force the contents of tank 2 or tank 3 to tank 1.

Tanks 2 and 3 are suitably connected with gas distributing means. A main gas line 37 is provided with offtake pipes 38 and 39, having valves 38′ and 39′, at tanks 2 and 3, respectively. These offtake pipes are in turn connected with a plurality of distributors 40 and 41, leading into tanks 2 and 3 near the bottoms thereof. A constant pressure regulator 42 is provided in the main gas line 37. The main gas line is also provided with a valve 43, interposed between the pressure reg-

ulator and offtakes, to regulate the amount of gas fed to the carbonation tanks. This valve is preferably automatically operated, in the manner disclosed in our copending application above referred to.

The apparatus just described may be operated as follows: Raw diffusion juice is fed into storage tank 4. A sufficient amount of this juice is conducted by gravity into tank 6 to build up a constant pressure as indicated and determined by the float valve 7. The chain operated valve 9 is suitably opened to conduct the beet juice through the pipe line 8 into the first treatment tank 1.

Beet juice is passed from tank 1 by means of pipe line 21 into the carbonation tank 2, until tank 2 is filled to its working level. Valve 24 in the pipe line 22 leading to tank 3 is closed while tank 2 is in operation.

Milk of lime is fed into the storage tank 10, and finds its way by gravity through pipe 11 into the constant level tank 12. The chain operated valve 16 is suitably opened to conduct the milk of lime through the "sight-box" 15. Valve 20 in line 18 leading to tank 3 is closed so that the milk of lime may pass through pipe 17 into tank 2, where it mixes with the beet juice.

When the tanks are filled to their normal operating level, the juice and lime supplied are temporarily shut off, the recirculation pump 36 is started and gas is admitted for the carbonation of the mixture already in the tanks.

Carbon dioxide gas in fed through the main gas line 37. The pressure of the gas fed to the line is suitably regulated by the pressure valve 42. The valve 39′ in offtake pipe 39 leading to tank 3 is closed so that the gas may be fed into tank 2. Since the gas is made to enter the mixture of beet juice and milk of lime near the bottom of tank 2, the bubbles of gas percolate up through the body of liquid.

When the carbonation has proceeded to the usual end point, the supply of juice and lime is again started, valve 31 is opened and the process is then under continuous operation.

A portion of the carbonated juice is conducted from the bottom of tank 2 through lines 25 and 27 into the settling or thickening device 28, while the remainder is pumped into tank 1. The recirculation of carbonated juice is conducted, in the present preferred practice of the invention, at a ratio of eight to one. That is to say, about eight volumes of juice are recirculated for each volume of new juice added to the circuit. Completely carbonated juice is conducted to the settling or filtering device as rapidly as raw diffusion juice is fed into the system. In this manner beet juice may be continuously carbonated.

It will be observed that the addition of milk of lime to the juice in tank 2 is made while the mixture is being gassed. There is no agitation of juice with lime preceding the carbonation step. Rising bubbles of gas strike the juice substantially as soon as it is mixed with lime. The interval of time between liming and gassing is negligible under operating conditions. The object is to gas the limed juice before the juice is saturated with respect to lime in true solution. In other words, the juice is gassed when it is but partially saturated with lime, or when its alkalinity is relatively low compared with what it would be when completely saturated with lime in true solution.

Since carbonated juice from tank 2 is made to mix with raw diffusion juice in tank 1, it is apparent that the purifying step above discussed takes place. The carbonated juice contains calcium carbonate particles in suspension. These particles operate mechanically to purify the raw diffusion juice in tank 1 before it reaches the gassing zone in tank 2. Colloidal matter present in the raw juice contacts with and is adsorbed on these suspended calcium carbonate particles.

The lime in true solution removed from tank 2 to tank 1 operates chemically to purify the raw juice. This lime reacts with certain dissolved salts and the like in the raw juice to form compounds that are substantially insoluble in the mixture. Colloidal matter also contacts with and is adsorbed on these insoluble particles.

By the time the raw juice from tank 1 reaches the liming and gassing zone in tank 2, it is materially purified. This juice may then be gassed for the precipitation of fresh calcium carbonate particles without colloidal matter being immediately adsorbed on their surfaces. Clean calcium carbonate particles readily lend themselves to particle growth, and, moreover, act as nuclei in the precipitation of calcium as calcium carbonate.

Whenever it is desired to shut down tank 2, for example, to clean the gas distributor or tank, the valves in the pipe lines leading to this tank may be closed and those in the lines leading to tank 3 may be opened to throw it into operation.

It will be understood that the present invention is not to be confined to practice in apparatus above discussed. The invention may obviously be conducted in other and various ways. For example, instead of employing separate tanks in association with one another, such as tanks 1 and 2 or 1 and 3, one relatively high tank might very well be employed. The upper end of such a single tank, suitably baffled from the lower end, might serve as a place in which to mix raw diffusion juice with carbonated juice, while the liming and gassing operations are conducted in the lower end of the tank. This and other modifications of the practice of the invention will no doubt suggest themselves

to those skilled in this art, in view of the principles herein disclosed.

It will be noted that the process as set forth in the above description is essentially continuous. It is well known that with juice having a given composition and purity, a definite amount of the liming agent will be required for proper defecation, and a corresponding amount of carbon dioxide gas or the like will be necessary to reduce the alkalinity of the carbonated juice to the requisite point. Where juice is carbonated by the former batch method, the rate of introduction of the lime relative to the rate of gassing can be regulated as desired; but where the juice is flowing continuously through a treatment zone, it is necessary to add the lime at a fixed rate determined by the proportion of lime to juice required by the purity, etc. of the latter; and a reduction in the rate of lime addition will result in inadequate liming. A problem of the present invention involves the maintenance of the necessary continuous flow of lime without producing saturation of the juice with lime at any point; and the disclosed arrangement has solved this problem by providing a suitable method of introducing juice and lime at the top of the relatively narrow tanks directly in the active gassing zone, so that withdrawal of the carbonated or partially carbonated juice and calcium compound crystals beneath the carbonation zone will produce a uniform downward flow against a uniform upward gassing action, thereby preventing all parts of the juice from becoming saturated with the liming agent at any time.

I claim:

1. The method of treating beet juice in the manufacture of sugar which comprises liming and carbonating beet juice substantially simultaneously, mixing said limed and carbonated juice with raw beet juice, and subjecting said mixed juice to carbonation.

2. The method of treating beet juice in the manufacture of sugar which comprises incompletely saturating the juice with lime, carbonating said limed juice, mixing said carbonated juice with raw juice, and carbonating said mixed juice.

3. The method of treating beet juice in the manufacture of sugar which comprises partially saturating the juice with a liming agent soluble in the juice, subjecting said partially saturated juice to carbonation, and repeatedly circulating said carbonated juice at least in part through the carbonation zone.

4. The method of treating beet juice in the manufacture of sugar which comprises partially saturating the juice with a liming agent soluble in the juice, subjecting said partially saturated juice to carbonation, mixing said carbonated juice with raw juice, and repeatedly circulating said carbonated juice at least in part through the carbonation zone.

5. The method of treating beet juice in the manufacture of sugar which comprises incompletely saturating the juice with lime and then carbonating the partially saturated juice, said juice being in part at least repeatedly recirculated through the carbonation zone.

6. The method of treating beet juice in the manufacture of sugar which comprises mixing raw juice with carbonated juice.

7. The method of treating beet juice in the manufacture of sugar which comprises mixing raw juice with carbonated juice, and liming said mixture.

8. The method of treating beet juice in the manufacture of sugar which comprises mixing raw juice with carbonated juice, liming said mixture, and carbonating said limed mixture.

9. The method of treating beet juice in the manufacture of sugar, which comprises mixing raw juice with carbonated juice, liming said mixture, and carbonating said limed mixture, said carbonation taking place substantially simultaneously with the liming step.

10. The method of treating beet juice in the manufacture of sugar which comprises flowing a body of limed and carbonated juice into a body of raw juice, adding more lime to the mixture of juices, and passing said mixture through a carbonation zone.

11. The method of treating beet juice in the manufacture of sugar which comprises continuously mixing carbonated juice with raw juice, circulating said mixture through a carbonation zone, and withdrawing carbonated juice from the circuit substantially as rapidly as raw juice is introduced into the circuit.

12. The method of treating beet juice which comprises flowing a substantially constant stream of the juice to a liming zone, incompletely saturating the juice with lime, conducting the limed juice to a carbonation zone, subjecting said partially saturated juice to the action of carbon dioxide gas, flowing a substantial portion of said carbonated juice to the point of introduction of raw juice into the circuit, mixing said carbonated and raw juices, flowing said mixed juices to the liming zone, incompletely saturating the mixed juices with lime, conducting the limed juice to the carbonation zone, subjecting said partially saturated juices to the action of carbon dioxide gas, returning a substantial portion of said carbonated juices to the beginning of the circuit, and withdrawing carbonated juices from the circuit substantially as rapidly as raw juice is introduced into the circuit.

13. The method of treating beet juice in the manufacture of sugar which comprises passing the juice in continuous flow through a carbonation zone, subjecting the juice in

said zone to the continuous action of carbon dioxide gas, continuously adding a liming agent to the juice at the beginning of its flow through said zone, and maintaining throughout the juice as it passes through said zone uniform distribution of said liming agent and gas, adequate to maintain substantially all of said juice in a condition of incomplete saturation with said liming agent during its passage through the carbonation zone.

14. The method of treating beet juice in the manufacture of sugar which comprises passing the juice downwardly in continuous flow through a vertical elongated carbonation zone of substantially uniform width, subjecting juice in said zone to the continuous action of carbon dioxide gas passing upwardly through the flowing juices, and adding continuously a liming agent to the juice at the top of said zone in the field of gas action.

15. The method of treating beet juice in the manufacture of sugar which comprises initial treatment of the juice with a liming agent in an amount insufficient to saturate the juice, and thereafter passing the juice in continuous flow through a carbonation zone, subjecting the juice in said zone to the continuous action of carbon dioxide gas, continuously adding an additional amount of a liming agent to the juice at the beginning of its flow through said zone, and maintaining throughout the juice as it passes through said zone uniform distribution of said agent and gas adequate to prevent any substantial portion of the juice from becoming saturated with the liming agent.

In testimony whereof I affix my signature.

ARTHUR W. BULL.