Coarse sodium bicarbonate crystals of controlled size are produced by spraying an aqueous mixture of soda ash and sodium bicarbonate into an atmosphere containing CO₂ in a carbonating zone; simultaneously evaporating water from the aqueous mixture; collecting the resulting slurry of bicarbonate crystals in mother liquor in a crystallization zone; removing therefrom a slurry stream of coarse sodium bicarbonate crystals; separating coarse crystals of sodium bicarbonate from said slurry and recycling the resultant mother liquor. The separated crystals are suitably washed, dried and sized to obtain the sodium bicarbonate product.

15 Claims, 4 Drawing Figures
3,870,784

SODIUM BICARBONATE PRODUCTION
RELATED APPLICATIONS

This is a continuation-in-part of application Ser. No. 213,562 filed Dec. 29, 1971, entitled “Sodium Bicarbonate Production now abandoned.”

BACKGROUND OF THE INVENTION

1. Field of Invention

This invention relates to a process for producing sodium bicarbonate from soda ash or caustic by carbonation and particularly to a process for so producing sodium bicarbonate meeting wide ranges of size specifications under minimum scaling and heat load conditions.

2. Description of the Prior Art

Sodium bicarbonate has long been produced commercially by the ammonia-soda process and by carbonation of soda ash. In prior art processes, however, purity and particle size control are problems still susceptible of considerable improvement. It is difficult to devise improvements and to put them into commercial use economically. Satisfactory methods for producing coarse granular sodium bicarbonate, in particular, currently in demand for detergent compositions and for other purposes, have not been available. Furthermore where processes producing a satisfactory product are available, scaling of machinery utilized to manufacture the product is a constant problem. Frequently the problem has been solved by utilizing two carbonation towers and scaling one while operating the other. This represents a very inefficient use of processing equipment and increases the cost of producing the resulting product.

The principal objects of the present invention are to provide a carbonation process for producing granular sodium carbonate in which crystal size can be controlled and which can be utilized, for example, to economically produce a crystal size up to 20-mesh to provide such a process which circumvents the scaling problems discussed above and the economic consequences of the prior art solutions.

The present invention minimizes costs of starting materials by efficient use of low cost sources of CO₂ by utilizing any common form of soda ash including trona ash or “light” soda ash from the ammonia-soda process, or by utilizing caustic as a raw material in lieu of or in addition to soda ash. It further minimizes costs of the process by minimizing the heat load on the system and by promoting more efficient use of machinery utilized in the process.

SUMMARY OF THE INVENTION

According to the present invention, a process has been devised for producing sodium bicarbonate economically in controllable particle size and with controlled purity by the steps of:

1. Maintaining in a crystallization zone an aqueous mixture of sodium bicarbonate crystals in a solution of sodium carbonate saturated with sodium bicarbonate;
2. Maintaining in a contiguous carbonating zone above said crystallization zone an atmosphere having at least 4 percent carbon dioxide;
3. Spraying an aqueous mixture of soda ash and sodium bicarbonate into said carbonating zone, removing evaporated water from said absorbing zone and collecting the resulting carbonated mixture in said crystallization zone;
4. Removing from carbonated mixture an aqueous stream containing suspended coarse crystals of sodium bicarbonate;
5. Separating said coarse crystals from said aqueous stream and recycling the resultant mother liquor.

In the preferred embodiment of the process, a second aqueous stream is removed from the upper portion of the aqueous mixture in the classification zone; said second stream is heated and fresh soda ash is dissolved therein and the resulting fortified solution is sprayed into the carbonating zone. This modification is advantageous when said second aqueous stream contains suspended fine crystals of sodium bicarbonate.

In a first advantageous modification of the process the second aqueous stream is heated in a regenerative heat exchanger; soda ash is dissolved in the heated solution and the enriched solution is cooled by regenerative heat exchange with the entering stream and the fortified solution is returned to the carbonation zone. This modification is advantageous when said second aqueous stream contains suspended fine crystals of sodium bicarbonate.

In a second modification, fine, dry soda ash is fed directly into the sodium bicarbonate slurry in the crystallization zone where the added ash dissolves rapidly and does not substantially affect the purity of the bicarbonate crystals present in the crystallization zone.

In a third modification of the process aqueous caustic is used in lieu of soda ash in the process and the above-identified modifications.

In a further modification of the process aqueous caustic is subjected to two stage carbonation utilizing recycled mother liquor and carbon dioxide vapor exhausted from a bicarbonate crystallizer-absorber.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a flow chart showing the preferred embodiment of the invention.
FIG. 2 is a flow chart showing the first modification of the preferred embodiment utilizing a regenerative heat exchanger.
FIG. 3 is a flow chart showing the second modification of the preferred embodiment wherein dry soda ash is introduced directly into the crystallization zone without dissolution or filtration of feed solution.
FIG. 4 is a flow chart showing the further modification utilizing two stage carbonation of caustic.

DETAILED DESCRIPTION

The method of the present invention is illustrated in the accompanying flow sheets. In FIG. 1, fortified but unsaturated aqueous solution of soda ash is introduced via line 35 through sprays 12 into an upper region of the gas filled crystallization zone 13 of the classifying crystallizer shown generally at 14. Sprays 12 are arranged and are controlled to promote impingement of the droplets on the roof and walls of crystallizer 14 to provide a flow of unsaturated feed solution over the roof and down the walls of carbonation zone 13 and into crystallization zone 11 in order to minimize scale formation.

Crystallization zone 11 and classification zone 15 contain a mixture of sodium bicarbonate crystals in an aqueous solution of sodium carbonate saturated and in equilibrium with sodium bicarbonate. As used herein,
crystallization zone is generally defined at the top by the liquid level below carbonization zone 13, and at the bottom by the termination of conical baffle 77. Appended to the lower edge of the baffle is cylindrical screen 78 which blocks passage of scale fragments too large to pass through orifices in nozzles 24. Conical baffle 77 and screen 78 thus physically separate crystallization zone 11 from classification zone 15.

The upper portion of liquor in classification zone 15 comprises crystal free mother liquor and/or mother liquor containing suspended relatively fine crystals of sodium bicarbonate; the lower portion thereof contains relatively coarser crystals of sodium bicarbonate.

A stream containing relatively larger volumes are drawn from crystallization zone 11, through classification zone 15, through line 21, cooled as desired in cooler 22, returned via line 23 to crystallizer 14 and dispersed through nozzles 24 in the lower region of carbonation zone 13. Nozzles 24 are positioned to direct the spray upward and inwardly from the perimeter of the tower to maximize mid-air collision of droplets moving in opposite directions and to minimize direct wall impingement of the droplets.

Carbon dioxide is provided in carbonation zone 13 in the form of washed stack gas via line 16. Exit gas is vented via line 17. Additional cooling is suitably provided if desired by directing a portion of vent gas via line 18 through cooler 19 and recycling it via line 20 with the incoming stack gas.

An aqueous stream 25 containing suspended fines of sodium bicarbonate is removed from classification zone 15 of crystallizer 14, heated in transit as desired in steam heater 26 and returned to dissolver 29. Fresh soda ash is introduced to dissolver 29 via line 30. Line 31 is provided for the introduction of fine, solid soda ash as desired directly into the liquor in crystallizer 14. Fortified feed solution leaves dissolver 29 via line 32, passes through filter 33 and is transferred via line 35 to sprays 12.

A slurry of coarse crystals is removed from the lower portion of classification zone 15 of crystallizer 14 via line 40 to centrifuge 41. Mother liquor is recycled via line 42 preferably to the classification zone. Purge line is provided at 43. Suitably, at other times, or using a duplicate of centrifuge 41, a slurry of fines is removed from classification zone 15 via line 44 to centrifuge 41. Alternatively, the fines are returned via line 25 to dissolver 29. The centrifuged product is washed with water from line 45 and then transferred via line 46 to rotary drier 47 heated by burning fuel and air in burner 48 and transferring the burner gas via line 49 to drier 47. Effluent gas containing fines and CO₂ pass via line 50 to fines collector 51 from which fine product is removed via line 52. Effluent gas from fines collector 51 is dehumidified by cool water introduced by line 53 into dehumidifier 54 and the dehumidified gas is vented via line 55 or recycled to drier 47 via line 56. Water from dehumidifier 54 is returned via line 57 and heater 26 to dissolver 29. Crystal sodium bicarbonate product is removed from drier 47 via line 58.

The method of the present invention is further illustrated in the accompanying flowsheet of FIG. 2. As in FIG. 1, fortified but unsaturated aqueous solution of soda ash is introduced via line 35 through sprays 12 in the gas filled carbonating zone 13 of crystallizer 14. Recirculation of the suspension is as in FIG. 1. Carbon dioxide is provided and exit gas is vented and/or recirculated with cooling as in FIG. 1. Aqueous stream 62 is removed from classification zone 15 and introduced into regenerative heat exchanger 66. The liquor, heated by vapor condensation, leaves the heat exchanger 66 via line 80 and is transferred to dissolver 29, additional optional heating as desired is provided by heater 26. Fortified liquor leaves dissolver 29 via line 32, passes through filter 33 and leaves via line 64 to the first stage of the lower section of regenerative heat exchanger 66. A portion of the uncooled, fortified liquor from line 64, heated by dissolution of soda ash therein, is split off for washing the roof of the absorber via line 55. The effluent liquor, cooled by evaporation in heat exchanger 66, is returned via line 65 to crystal suspension zone 11 of crystallizer 14.

Vapor transfer lines of heat exchanger 66 are shown at 67, 68 and 69. Liquid transfer lines of heat exchanger 66 are shown at 71, 72, 73, and 74. A similar heat exchanger is described in Journal of Metals, July 1966, pages 811-818. Product recovery and drying is as in FIG. 1. A simpler form of the invention is illustrated in FIG. 3. Fine soda ash is fed directly into the saturated crystallizer via line 31. An aqueous stream 25 containing suspended fines of sodium bicarbonate is removed from classification zone 15 of crystallizer 14, heated in heater 26 and transferred via line 35 to sprays 12. Recirculation of the crystallizer liquor and recovery of product is as described in FIG. 1. This modification of the invention eliminates the cost of dissolving the feed and filtering the feed solution. The cost of the steam for dissolving the feed is also eliminated. The product does not meet food grade specifications but capital investment and utility costs are reduced to a minimum.

To meet food grade specification with minimum utility costs, ash must be dissolved and filtered before this stream is admitted to the crystallizer. The liquor introduced into the feed dissolving circuit from the crystallizer must be heated to establish sufficient dilution to serve as a dissolving medium for the ash. Steam to supply this heat is a major cost item in the production of the crystal bicarbonate. The heat of solution of soda ash in the liquor is moderately exothermic. Therefore, regenerative heating and cooling by efficient counter-current contacting of the liquor entering the ash dissolver with the liquor leaving the ash dissolver is utilized. Scaling may occur in the regenerative heater due to the occurrence of supersaturation as the enriched mother liquor is cooled. Heat transfer by vapor exchange is not impaired by scaling. Scale which forms in a vapor exchanger can also be quickly dissolved by intermittent steaming. Absorber wall scale formation is minimized by distribution of hot, enriched, but slightly dilute mother liquor from the feed dissolving circuit directly onto the upper walls of the absorber.

To meet food grade specifications with minimum occurrence of scale formation, all of the hot enriched liquor from the feed dissolving circuit is directed to the roof of the absorber. The regenerative heat exchanger in the feed dissolving circuit is therefore omitted. This also eliminates scaling at this point in the process. Larger amounts of steam are required for heating the liquor in the feed dissolving circuit. An additional cooling load is also imposed on the crystallizer since the added steam load must be removed along with the exo-
thermic heat of reaction between soda ash and CO₂. However, scale formation in the feed dissolving circuit and on the walls of the absorber is minimized.

If desired caustic may be substituted for soda ash in the modifications shown by FIGS. 1-3 without substantial modification of the systems shown in the flow charts. As used herein the term "caustic" means caustic soda or aqueous sodium hydroxide. Thus, in FIG. 1 caustic rather than soda ash would be introduced into the crystallizer via line 31. Mixing of the caustic with sodium bicarbonate containing liquor converts the caustic to sodium carbonate according to the following reactions:

\[(A) \ NaOH + NaHCO₃ \rightarrow Na₂CO₃ + H₂O \]

\[(B) \ Na₂CO₃ + CO₂ + H₂O \rightarrow 2NaHCO₃ \]

The exothermic heat of reaction is about 505 Btu/LB. NaHCO₃ formed as opposed to about 400 Btu/LB. NaHCO₃ utilizing soda ash, thus creating an additional heat load on the system which must be counteracted by increasing cooling effected through cooler 19. The expense of increased cooling is substantially offset by economies gained in recycling mother liquor to sprays 12. As best seen in FIG. 4, the use of caustic eliminated the need for steam heater 26. Thus an aqueous stream 25 containing suspended fines of sodium bicarbonate may be transferred directly to dissolver 29 where fines are dissolved upon addition of aqueous caustic introduced into dissolver 29 via line 30a. Further, the lack of impurities in caustic eliminates the need for filter 33 direct transfer of fortified feed solution from dissolver 29 to sprays 12.

A further modification of the system utilizing caustic in lieu of soda ash is illustrated in FIG. 4. Aqueous caustic is introduced via line 75 into a first stage carbonation unit 76 in which it is carbonated to form a finely divided monohydrated crystalline Na₂CO₃ precipitate. The first stage carbonation is carried out at elevated temperature increasing the efficiency of CO₂ absorption over that which can be obtained by preliminary carbonation in the crystallizer-absorber. Carbonation may be effected in this first-stage unit either by passing an aqueous caustic solution through an atmosphere of CO₂ or by passing CO₂ through the aqueous solution, both such methods being well known in the art.

Following carbonation the aqueous solution of sodium carbonate and unreacted sodium hydroxide, if any, is introduced into the absorber-crystallizer via line 31a where sodium bicarbonate is formed in accordance with steps discussed in connection with FIGS. 1, 2, and 3.

The suspension in the first stage carbonator should contain a ratio of 25 pounds of monohydrated Na₂CO₃ to 60 pounds saturated ash solution. Due to the lack of water entering the system in the aqueous caustic (about 50 percent H₂O) and the high evaporation resulting from high-operating temperatures dilute mother liquor is recycled via line 43a to maintain the ratio.

Operating the first stage absorber at a temperature of up to 90°C, as much as 40° above the temperature at which the second stage absorber-crystallizer operates, increasing carbon dioxide absorption about 9 fold. Thus it is desirable to utilize gas vented from the second stage absorber crystallizer and to introduce this into the absorber 76 as a source of CO₂ for the first stage absorber through line 17a. The absorber 76 thus acts as a scrubber for gases which would normally be vented through a special scrubber from the bicarbonate absorber-crystallizer. If additional carbon dioxide is required, it may be supplied via line 16a.

By utilizing the two stage carbonation process described above for processing caustic about 60 percent of the heat load is dissipated through the first stage carbonation unit 76, thus avoiding the necessity of increasing cooling capacity of cooler 19. When coupled with the elimination of heater 26 and filter 33, (shown in FIGS. 1 and 2) the utilization of caustic becomes a practical and economical way to use an alternate starting material without sacrificing product quality or operating efficiency in the process of producing course granular sodium bicarbonate.

In the process of the present invention, the crystal suspension in the crystallizer is re-circulated and sprayed into an atmosphere containing at least 4 percent CO₂ in an open absorption column immediately above an exposed crystal suspension. Supersaturation is induced by the absorption of CO₂ in the carbonate-containing mother liquor. The sprayed liquor then drops into the contiguous crystallization zone below.

Fine crystals are segregated and removed from the suspension in the connecting classification zone as required to balance the amount of seed in the suspension with the crystal production rate.

Published solubility data for solutions of sodium carbonate and sodium bicarbonate in equilibrium with sodium bicarbonate crystals show that at 45°C, for example, the liquor composition may range from a maximum of about 18 percent by weight of Na₂CO₃ and 6 percent of NaHCO₃ to a minimum of 0 percent Na₂CO₃ and 12 percent NaHCO₃. As the temperature increases, the concentrations of NaHCO₃ required to saturate the solution also increase. At intermediate Na₂CO₃ concentrations within this solubility range the absorption of CO₂ results in a decrease in the concentration of the Na₂CO₃ and an increase in the concentration of NaHCO₃ according to the equation:

\[Na₂CO₃ + CO₂ + H₂O \rightarrow 2NaHCO₃\]

Addition to the mother liquor of Na₂CO₃ either in solution or in solid form compensates for carbonate depletions by bicarbonate formation and sustains the Na₂CO₃ concentration of the mother liquor in the range indicated above. Na₂CO₃ in solid form dissolves in the mother liquor and induces crystallization of NaHCO₃ by salting it out.

Commercial grades of sodium bicarbonate crystal are recovered from the suspension. Supplemental heat is not required to dissolve the ash and costs are minimized by elimination of the ash dissolving liquor circuit.

To meet U.S.P. purity requirements, the soda ash feed solution is filtered to avoid accidental entry of insoluble foreign matter into the suspension. Heat requirements are minimized if the ash is dissolved in heated mother liquor. Further minimization of heat requirement results form regenerative heating and cooling in a countercurrent exchanger of solution entering and leaving the feed ash dissolver. Crystal growth is induced by cooling the enriched feed solution in contact with the suspension.

Prior art methods for the absorption of gases in liquids are by contact in packed towers, by dispersing liquid in the gas as a spray or cascade, and by dispersing gas bubbles in the liquid. In the prior art methods, sca-
Heating of the mother liquor de-saturates the liquor and permits it to be utilized to dissolve additional sodium carbonate. Heating of the mother liquor also destroys excess crystal nuclei removed from the elutriation zone. A particularly advantageous method of heating and cooling the liquor in the feed dissolving circuit is by passing the solution countercurrently to itself through a regenerative heat exchanger. The heat of solution of dry soda ash is exothermic and the solution leaving the feed dissolving tank is hotter than the solution entering. This differential in temperature is utilized in a regenerative heat exchanger to cool the heated, enriched filtered solution returning to the crystallizer by countercurrent contact with the cooler mother liquor flowing from the crystallizer to the dissolving tank. The regenerative heat exchanger obviates the need of an additional source of external heat for de-saturating the mother liquor for dissolution of fresh soda ash. The fortified feed solution containing dissolved soda ash is filtered and returned to the crystallizer directly or via the heat exchanger. The heat requirements for dissolving soda ash in the heated mother liquor are much less than the heat that would be required for first dissolving the fresh soda ash in water and then providing additional heat for evaporation of the solvent water so added.

Acceleration of the nucleation rate by artifical means, such as a high speed attrition impeller operating in the suspension, also requires an associated increase in the rate of fines removal from the classifying zone to maintain the coarse crystal growth rate in balance with the seed rate. The fines streams so withdrawn from the crystallizer may then be charged to the centrifuge and drying system and then packaged as fine product. Drying is slower and dust losses are higher but this is an advantageous improvement compared to the production of powdered bicarbonate by milling of larger crystals to meet the demand for fine grades of sodium bicarbonate.

The slurry of larger crystals removed from the crystallizer is centrifuged to separate the mother liquor from the product crystals. The centrifuged crystals are suitably washed and transferred to a rotary dryer where residual moisture is volatilized. In the rotary dryer, the crystals are dried at relatively low temperature in a CO₂ atmosphere to prevent decomposition of the bicarbonate to carbonate by loss of CO₂. The dryer is suitably a variable speed, automatic-batch type which minimizes the breakage of crystals and increases the production of coarse granular grades. The dry bicarbonate from the dryer is transferred to screening and milling operations by screw conveyors, bucket elevators or by an integral air-veyor system whereby the product is entrained in the dryer exhaust air stream and transported to the screens by air ducts.

Finer grades of bicarbonate are suitably produced from the coarser grades grown in the crystallizer by milling and classification in a closed-loop system.

In the process of the present invention, substantially all of the heat is removed by evaporative cooling of the droplets of dispersed liquor in the CO₂ absorber, including the exothermic heat of solution of the soda ash, the exothermic heat of crystallization of the bicarbonate, the exothermic heat of absorption of CO₂ and any heat from external sources required to dissolve the dry soda ash in recycled mother liquor. This process avoids surface coolers such as shell-and-tube coolers in the suspension recycle circuit supplying the spray nozzles.
Problems of local supersaturation adjacent to the cooling surfaces resulting in scale formation and impairment of cooler performance are avoided. This permits the supply of unsupersaturated solution in high volume for deterring the formation of scale on the absorber walls.

A particular advantage of the process of the present invention resides in providing a large, e.g., 5 to 10 hours of production, dynamic reserve of sodium bicarbonate crystals in the crystallizer suspension. This reserve serves to slow the rate of crystal growth and to assure the production of coarse crystals of high strength. Substantial fluctuations in this reserve over periods of several hours do not adversely affect crystal quality. This reserve stabilizes the production rate over extended periods of time and effects economies in other sections of the plant. Intermediate surge silos between major sections of the plant are therefore unnecessary. The slurry transfer rate from the crystallizer to the centrifuges is suitably adjusted to accommodate normal batch-wise operation. A plurality of centrifuges including a spare unit is advantageous to minimize the magnitude of surges in crystal flow rate through the drier and to assure continuity of flow to the mills in the event of centrifuge breakdown. In contrast, the reserve of crystals in conventional bicarbonate towers covers only about 2.5 hours of tower product. This reserve must be maintained in order to maintain productivity and cannot be varied to stabilize subsequent plant production. Surge silos are required to stabilize flow through screens and mills whereas such silos are necessary in the process of the present invention.

CO₂ concentration in the tower suitably varies from 4 to 100 percent, preferably about 8 to 40 percent. Novel nozzle design permits operation with 4 to 5 percent CO₂.

CO₂ supplied is suitably boiler stack gas containing 9 to 15 percent CO₂ or, for example, a plant stream containing 95 percent CO₂ and from 0.5–1.5 percent H₂ such as is recovered from ammonia synthesis plants. Normally gas pressure in the carbonate prevents intake of air and no hazard is presented by the selective absorption of CO₂. However, suitable provision is made for abnormal conditions by injecting nitrogen into such hydrogen-containing CO₂ supply.

EXAMPLE I

In an absorber-crystallizer 20 feet in diameter and 75 feet high a suitable plurality of spray nozzles are arranged in opposing groups at an elevation of about 30 feet above the bottom of the tower and provide spray interaction which increases the rate of CO₂ absorption. Residence time of the spray droplets is 2 to 3 seconds at a nozzle pressure of 30 psig. Suitable spray nozzles of the 30° to 45° solid cone or hollow cone type having rated capacities of 50 to 200 gpm (gallons per minute) operating at discharge pressures of about 15 to 30 psig provide satisfactory absorption rates of CO₂. The nozzles are directed to provide maximum interaction to enhance CO₂ absorption. Enhancement of the CO₂ absorption rate from gases low in CO₂ concentration is effected by design or selection of nozzles which disperse the recirculated suspension into smaller droplets. Gas dispersion nozzles are particularly effective in generating small drops but also require the dissipation of larger amount of pumping and compression energy.

A funnel-shaped conical baffle is sealed internally to the side walls with the top of the funnel at an elevation of 25 feet above the bottom of the tower and the bottom of the funnel at an elevation of 15 feet. The bottom of the funnel has a diameter of 12 feet. The concentric zone between this funnel and the tower wall provides a quiescent elutriation zone also intended to generate crystal free mother liquor. Ports fitted with throttle plates near the upper edge of this baffle provide for the regulation of the flow of clarified mother liquor upwardly in the peripheral zone of the baffle.

Suspension circulating pump intakes (2) are provided at an elevation of 1 foot above the bottom and these discharges in a battery of spray nozzles similar to those previously described at an elevation of 30 feet above the bottom. Fragments of scale too large to pass the smallest orifice in the spray nozzles are restrained by a large cylindrical screen affixed to the bottom of the conical elutriation zone baffle and extending to the bottom of the tower. Recirculated suspension must pass through the screen to reach the pump intakes. Suspension for supplying the crystal centrifuges is preferably drawn from a dynamic suspension zone in the bottom of the tower.

At an intermediate level 22 feet above the bottom, a stream of crystal free mother liquor is removed from the elutriation zone and heated in the regenerative heat exchanger to provide a solution unsaturated in soda ash in which fresh soda ash is dissolved. The regenerative heat exchanger is particularly advantageous in reducing auxiliary steam requirements for dissolving the ash but cooling is controlled to maintain the temperature of the returning fortified feed liquor above saturation temperature. Connections for drawing mother liquor with variable concentrations of fine crystal and nuclei are also provided at an intermediate level of about 17 feet. The concentration of crystals in this stream is dependent on the total rate of flow of crystal-free mother liquor through the elutriation zone. Crystals in this stream may be recovered as fine product or they may also be destroyed by reheating in the feed dissolving circuit.

A suitable rotary dryer is 6 feet in diameter and 25 feet long. It has a capacity of 15,000 pounds per hour of sodium bicarbonate. Hot air enriched with recycle CO₂ entering at 450°F. flows countercurrently through the dryer. This minimized air velocity, dust entrainment and bicarbonate decomposition. The maximum discharge temperature of the bicarbonate is 70°C. (about 158°F.). Internal dryer flighting improves heat transfer and contributes strength to the dryer shell.

EXAMPLE II

Absorber Operation

In an absorber-crystallizer tower having a diameter of 20 feet and an overall height of 76 feet, the cylindrical body has a height of 44 feet with an 18 foot high conical roof and a 14 foot deep conical bottom reducing the diameter to 6 feet. The steep roof angle minimizes scale formation on this surface and the conical bottom minimizes crystal sedimentation on the walls. The top 30 feet of the cylindrical body of the tower is the absorption zone and the bottom 28 feet of the tower is the crystallizer and classification zone. The remaining 18 feet constitutes the conical roof.

Mother liquor containing 13 percent Na₂CO₃ and 6.5 percent NaHCO₃ was sprayed at a temperature of
50°C. through 100 nozzles (Spraying Syst. Co. No. 2 CRC 30-45), each rated at 50 gpm at a pressure drop of 30 psig across the nozzle. The temperature of the liquid in the absorber was 53°C. The CO₂ concentration in the inlet gas varied from 80 to 85 percent and in the exit gas from 10 to 20 percent. Droplet residence time was 2 seconds. The CO₂ absorption rate was 4,000 lb./hr. and the sodium bicarbonate was crystallized at the rate of 15,300 lb./hr.

EXAMPLE III

Crude light soda ash was charged to the stirred dissolver at the rate of 8,620 lb./hr. The soda ash had a chemical and mesh analysis as follows:

<table>
<thead>
<tr>
<th>Component</th>
<th>Wt. pct.</th>
<th>Mesh analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na₂O</td>
<td>58.0</td>
<td>+ 20</td>
</tr>
<tr>
<td>CO₂</td>
<td>41.1</td>
<td>- 20 + 50</td>
</tr>
<tr>
<td>NaCl</td>
<td>0.4</td>
<td>- 20 + 100</td>
</tr>
<tr>
<td>H₂O</td>
<td>0.3</td>
<td>- 100 + 150</td>
</tr>
<tr>
<td>Balance</td>
<td>0.2</td>
<td>- 150 + 200</td>
</tr>
</tbody>
</table>

Recycling heated liquor from the heat exchanger at a temperature of 68°C. (154.4°F.) was fed to the dissolver providing a solution containing 24,000 lb./hr. of Na₂CO₃, 16,000 lb./hr. of NaHCO₃, and 152,000 lb./hr. of water. An additional 4,665 lb./hr. of water was also added to the dissolver. The effluent solution from the dissolver, passed through the filter and heat exchanger contained 32,620 lb./hr. of Na₂CO₃, 16,000 lb./hr. of NaHCO₃ and 156,665 lb./hr. of water.

The slurry of fine crystals of NaHCO₃ was removed from the crystallizer at a temperature of 57°C. (134.5°F.) and heated in the heat exchanger to 66°C. (150.8°F.). The exothermic heat of solution of the ash raised the solution temperature to 68°C. (154.4°F.). Meanwhile, hot fortified feed liquor from the filter was charged to the heat exchanger at 68°C. (154.4°F.) and was cooled to 59°C. (138.2°F.) before returning to the crystallizer.

The CO₂ atmosphere in the absorber-crystallizer was provided by feeding washed stack gas containing 7,997 lb./hr. of CO₂, 5,009 lb./hr. of water, 1,683 lb./hr. of O₂ and 42,560 lb./hr. of N₂. The vent gas from the absorber contained 4,597 lb./hr. of CO₂, 6,457 lb./hr. of water, 1,683 lb./hr. of O₂ and 42,560 lb./hr. of N₂. Thus 3,400 lb./hr. of CO₂ was absorbed and 1,448 lb./hr. of water was evaporated.

A recycle stream of liquor and crystals from the bottom of the crystallizer was circulated at a rate of 10,000 gallons per minute to the lower bank of sprays in the absorber zone to induce absorption of 3,400 lb./hr. of CO₂.

Coarse crystal slurry was transferred from the bottom of the crystallization zone to the centrifuges and amounted to 12,600 lb./hr. of NaHCO₃ crystals in 37,600 lb./hr. of mother liquor. The wet bicarbonate removed from the centrifuges amounted to 13,000 lb./hr. holding 400 lb./hr. of water. This was dried and screened to provide 12,600 lb./hr. of coarse, crystalline sodium bicarbonate product having a bulk density of 55 lb./ft.³. It showed the following chemical and mesh analysis:

<table>
<thead>
<tr>
<th>Component</th>
<th>Wt. pct.</th>
<th>Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaHCO₃</td>
<td>99.8</td>
<td>- 10 + 20</td>
</tr>
<tr>
<td>Na₂CO₃</td>
<td>0.1</td>
<td>- 20 + 50</td>
</tr>
<tr>
<td>Balance</td>
<td>0.1</td>
<td>- 50 + 100</td>
</tr>
<tr>
<td></td>
<td>-100 + 150</td>
<td>30.0</td>
</tr>
<tr>
<td></td>
<td>-150 + 200</td>
<td>90.0</td>
</tr>
</tbody>
</table>

What is claimed is:
1. A method for producing sodium bicarbonate by carbonating sodium carbonate in an absorber-crystallizer to precipitate sodium bicarbonate crystals which comprises:
   1. maintaining in a crystallization zone a mixture comprising sodium bicarbonate crystals in an aqueous solution of sodium carbonate saturated and in equilibrium with sodium bicarbonate;
   2. maintaining in a contiguous carbonating zone above said crystallization zone an atmosphere having at least 4% carbon dioxide;
   3. spraying said mixture upwardly and inwardly into said atmosphere from the perimeter of said carbonating zone, through opposed nozzles adapted to maximize mid-air impingement of sprays emanating therefrom, removing evaporated water therefrom and collecting the resulting carbonated mixture in said crystallization zone;
   4. removing from said carbonated mixture an aqueous stream containing suspended crystals of sodium bicarbonate;
   5. separating said crystals as product from said aqueous stream and recycling the resultant mother liquor to said absorber-crystallizer.
2. The method of claim 1 wherein a second stream containing relatively fine crystals of sodium bicarbonate is removed from said carbonated mixture, said fine crystals are dissolved in said second stream and said second stream is introduced onto the roof and walls of said carbonating zone.
3. The method of claim 2 in which soda ash or caustic is introduced directly into said crystallization zone in amounts sufficient to substantially compensate for sodium carbonate converted to sodium bicarbonate and removed as product and maintain said equilibrium.
4. The process of claim 2 in which said second stream is fortified with soda ash or caustic.
5. The process of claim 4 in which supplemental soda ash or caustic is introduced directly into said crystallization zone in amounts which, when added to amounts added to said second stream, are sufficient to substantially compensate for sodium carbonate converted to sodium bicarbonate and removed as product and to maintain said equilibrium.
6. A method for producing sodium bicarbonate by carbonating sodium carbonate in an absorber-crystallizer to precipitate sodium bicarbonate crystals which comprises:
   1. maintaining in a crystallization zone a mixture comprising sodium bicarbonate crystals in an aqueous solution of sodium carbonate saturated and in equilibrium with sodium bicarbonate;
   2. maintaining in a contiguous carbonating zone above said crystallization zone an atmosphere having at least 4% carbon dioxide;
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3. collecting said mixture in a classification zone and classifying said mixture into an upper portion having relatively fine crystals of sodium bicarbonate suspended therein and a lower portion having relatively coarse crystals of sodium bicarbonate suspended therein;

4. removing a portion of said mixture from said classification zone and spraying the same upwardly and inwardly into said atmosphere from the perimeter of said carbonating zone through nozzles adapted to maximize mid-air impingement of sprays emanating therefrom, removing evaporated water therefrom and collecting the resulting carbonated mixture in said crystallization zone;

5. removing from said classification zone in an aqueous stream containing suspended crystals of sodium bicarbonate;

6. separating said crystals from said aqueous stream as product and recycling the resultant mother liquor to said absorber-crystallizer.

7. The process of claim 6 in which a second stream containing said relatively fine crystals of sodium bicarbonate is removed from said upper portion, said relatively fine crystals are dissolved in said second stream and said second stream is sprayed into an upper region of said carbonating zone to impinge on the roof and walls thereof to provide a flow of unsaturated feed solution thereover.

8. The process of claim 7 in which sodium carbonate or aqueous sodium hydroxide is introduced directly into said crystallization zone in amounts sufficient to substantially compensate for sodium carbonate converted to sodium bicarbonate and removed as product and to maintain said mixture in equilibrium.

9. The process of claim 7 in which said second stream is fortified with sodium carbonate or aqueous sodium hydroxide to provide a flow of fortified unsaturated feed solution over the roof and walls of the carbonation zone.

10. The process of claim 9 in which supplemental sodium carbonate or aqueous sodium hydroxide are introduced directly into said crystallization zone in amounts which, added to amounts added to said second stream are sufficient to substantially compensate for sodium carbonate converted to sodium bicarbonate and removed as product and to maintain said mixture in equilibrium.

11. The process of claim 6 wherein the mixture removed from said classification zone and sprayed into said carbonation zone is removed from a lower portion of said classification zone and contains relatively coarse crystals of sodium bicarbonate.

12. The process of claim 11 in which a second stream containing said relatively fine crystals of sodium bicarbonate is removed from said upper portion, said relatively fine crystals are dissolved in said second stream and said second stream is sprayed into said carbonating zone to contact the walls and roof thereof to form a flow of unsaturated feed solution thereover.

13. The process of claim 12 in which sodium carbonate or aqueous sodium hydroxide is introduced directly into said crystallization zone in amounts sufficient to substantially compensate for sodium carbonate converted to sodium bicarbonate and removed as product and to maintain said mixture in equilibrium.

14. The process of claim 12 in which said second stream is fortified with sodium carbonate or aqueous sodium hydroxide to provide a flow of fortified unsaturated feed solution over the roof and walls of the carbonation zone.

15. The process of claim 14 in which supplemental sodium carbonate or aqueous sodium hydroxide is introduced directly into said carbonation zone in amounts which, added to amounts added to said second stream, are sufficient to substantially compensate for sodium carbonate converted to sodium bicarbonate and removed as product and to maintain said mixture in equilibrium.

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