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REGENERATION OF MAGNESIUM BISULPHITE PULPING LIQUOR AND
ABSORPTION OF SULPHUR DIOXIDE DURING REGENERATION
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FIG. 3

FIG. 4

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The cyclic recovery system shown in FIG. 1 is of the general character shown in U.S. Patents 2,238,456 and 2,285,676 of George H. Tomlinson. In such a cyclic system, a cooking liquor consisting of a relatively pure acid sulphite compound of magnesium, i.e., magnesium bisulphite with or without an excess of sulphur dioxide, is supplied to a digester from a cooking acid storage tank. As shown, the digester 10 is illustrated as being equipped for indirect heating so that the strength of the cooking acid is not reduced during the cooking or digestion process, as would happen in a direct heating system of digestion. By using an indirect heating digester, a more economical concentration of the residual liquor preparatory to combustion is possible.

When the cooking operation is completed, the contents of the digester 10 are discharged into a blow tank 11 from which gases are vented and the pulp and liquor mixture is pumped to suitable pulp washers 12 and 13 for the separation of the liquor from the pulp. Thereafter, the pulp is transferred to the usual pulp treating equipment for the production of the desired product.

Wash water from a hot water tank 14 is delivered to the pulp washer 13 and the filtrate discharged into a storage tank 15 from which the liquid is delivered to the other pulp washer 12 where the filtrate is used as the washing medium. The filtrate obtained from the washer 12 is delivered to an acid waste liquor tank 16 from which a portion may be returned to the tank 11 to increase the fluidity of the pulp and to facilitate its transportation to the washers, while the remaining filtrate, or all of the filtrate, depending upon the need for the liquid in the blow tank, is discharged to multiple effect evaporators 17 for concentration. In the embodiment of the invention the liquor is concentrated to the range of 5 to 70% solids prior to its delivery to a concentrated liquor storage tank 18 for controlled introduction into the combustion zone 20 of a heat recovery apparatus or boiler 21.

As described in the referred to U.S. patents, the concentrated residual liquor delivered to the combustion zone 20 is burned in suspension and under conditions of temperature and flow velocity such as to insure the production of a highly reactive magnesium oxide solid. The magnesium oxide passes through the combustion zone and the associated heat absorbing surfaces of the vapor generator 21 in a gas foamed condition. The gases entraining the magnesium oxide particles will contain, as major constituents, nitrogen, carbon-dioxide and sulphur dioxide. The mixture of gases and entrained solids is discharged from the heat recovery surfaces of the boiler 21 at a temperature of the order of 500°F, for example. From the boiler, the mixture of gases and solids is passed through a dust separating zone which in the present embodiment consists of a plurality of multi-clones 22. The multi-clones remove substantially all of the magnesium oxide from the gases of combustion, with the gases thereafter passed through an induced draft fan 23 which discharges the gases into the SO₂ recovery apparatus hereafter described.

As disclosed in said patents, the magnesium oxide particles discharge from the collecting hoppers of the multi-clones are passed through suitable washing and treating equipment 24 for the removal of solids inert to the process, and are delivered to magnesium oxide slaking tanks 25 wherein the magnesium oxide is substantially converted to magnesium hydroxide. The magnesium hydroxide slurry is delivered to a storage tank 26. Make-up chemicals should be added at the slaking tanks so, if in the oxide form, it can be converted to hydroxide. For example, the magnesium oxide make-up may be of the order of 20 to 40 lbs. of MgO added to the system for each ton of pulp produced in the process. The magnesium addition

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5 may be added as magnesium hydroxide or may be added directly in the oxide form. In accordance with the present invention, the flue gases leaving the induced draft fan 23 are subsequently contacted with magnesium bisulphite-monosulphite solution to which magnesium hydroxide slurry has been added, for the absorption of the SO₂ in the gases and the formation of magnesium bisulphite liquor (Mg(HSO₄)₂) of sufficient strength for cooking purposes in the pulp digesters. The cyclic loss of sulphur in the system is a relatively minor amount, but some make-up sulphur must be added. This sulphur may be conveniently added in the form of gaseous SO₂ obtained from burning pyrites or elemental sulphur.

10 The absorption of the SO₂ which may be of the order of 1% by volume in the flue gases by the sprayed liquid of the present invention occurs by reason of the chemical affinity of the sprayed liquid for the SO₂ gas under relatively low temperature and controlled pH conditions. The sprayed liquid has, as a constituent, magnesium bisulphite which contains both free and combined SO₂. The terms “free” SO₂ and “combined” SO₂ are used herein as defined by the Technical Section, Canadian Pulp and Paper Association (Data Book, 4th Ed., p. 12, OCo, June 1955). When the bisulphite is mixed with magnesium hydroxide, monosulphite is formed with the free SO₂ converted to combined SO₂. Thereafter, when the mono-sulphite contacts the gaseous SO₂ the gas is absorbed by the monosulphite to form magnesium bisulphite.

15 The apparatus illustrated in FIG. 2 includes a SO₂ absorption zone wherein liquid is atomized into cooled combustion gases containing the SO₂ with the mixture thereafter passed through a venturi-like accelerating zone for intimate mixing of the droplets of liquid and the entraining gas. The SO₂ absorption is efficiently accomplished with a series arrangement of the venturi-like zones where each zone is followed by a pressure regain section and a liquid-gas separating chamber wherein the liquid containing the absorbed SO₂ is collected and utilized in part to provide the liquid for the absorption of the SO₂. A portion of the collected liquid from each of the venturi-like SO₂ absorption zones is passed from zone to zone in a direction counter-current to the gas flow direction to the zones to increase the SO₂ content in the acid discharged from the first gas contact zone and is delivered to the acid tanks for use in the pulp digestion process.

20 The process of gaseous SO₂ absorption in a liquid solution containing magnesium bisulphite and monosulphite is known. In such a process the solution is mixed with magnesium hydroxide to convert bisulphite to monosulphite generally according to the formula:

\[ \text{Mg(OH)}_2 + \text{Mg(HSO}_4\text{)}_2 \rightarrow 2\text{MgSO}_4 + 2\text{H}_2\text{O} \]

With the solution thereafter introduced into the SO₂ containing gas stream, the magnesium monosulphite absorbs the SO₂ to form magnesium bisulphite generally according to the formula:

\[ \text{MgSO}_4 + \text{SO}_2 + \text{H}_2\text{O} \rightarrow \text{Mg(HSO}_4\text{)}_2 \]

Herefore the amount of magnesium monosulphite present in a liquid solution has been limited by its solubility under the gas-liquid contact conditions in the known apparatus used for SO₂ absorption, for example, packed towers. When the solubility limit has been exceeded, with the liquid under relatively quiescent conditions, such as occurs in the multiplicity of relatively small areas in a packed tower, the excess magnesium monosulphite normally precipitates in a relatively non-reactive form incapable of efficiently absorbing gaseous SO₂. It has been found that the use of the venturi type gas and liquid contact apparatus of the present invention permits the use of higher percentages of magnesium monosulphite in the liquid than herefore without adverse precipitation, and thus makes possible correspondingly increased chemical absorption of the SO₂ by the liquid.

25 The increased chemical absorption of the SO₂ is due at least in part to the maintenance of continuous positive movement of the liquid in the liquid flow path from the formation of the SO₂ to the point at which the magnesium hydroxide to the magnesium bisulphite at, for example, the circulating pump) to its intimate turbulent spray contact with the SO₂-containing gases.

30 It has been observed that a monosulphite concentration greater than the normal saturation solubility limit is obtained in these operating conditions because of the formation of a suspension of microscopic and submicroscopic magnesium monosulphite crystals which are reactive with gaseous SO₂ in the venturi type contact apparatus within well defined limits of monosulphite concentration in the liquid.

The effect of solubility on absorption is illustrated in FIG. 5 where the curve A represents the heretofore known solubility limit of magnesium monosulphite content over a temperature range of 100°F. to 120°F. in a comparatively quiescent magnesium bisulphite liquid having 4% MgO (Data Book, p. 12, OCo, June 1955). The increased chemical absorption of SO₂ by the liquid through the temperature range of 100°F. to 120°F. is due at least in part to the maintenance of continuous positive movement of the liquid in the liquid flow path from the formation of the SO₂ to the intimate turbulent spray contact with the SO₂-containing gases.

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reason of the absorption of the SO2 from the combustion gases. Moreover, the difference in temperature between the entering liquid and the flue gases will result in cooling the gases below saturation or dew point temperature with the condensation of water from the gases so that upon leaving the upper end of the cooling tower through the outlet duct 33 the flue gases may be substantially saturated at a temperature of, for example, 104° F.

To maintain the cooling effect of the liquid sprayed into the tower 30, the liquid collected in the bottom of the tower is passed through the heat exchanger 27. The apparatus disclosed consists of a series of radiant tubular heat exchange elements arranged for counter-flow of the liquid in indirect cooling relationship with cooling water. For example, with the circulation of 2270 gallons per minute of the cooling liquid utilized in the cooling tower, the liquid will enter the heat exchanger at a temperature of approximately 149° F. and leave at a temperature of 88° F.

The cooling water entering the heat exchanger may be at a temperature of approximately 60° F. and leave at a temperature of approximately 110° F. The quantities of cooling water utilized under these conditions will be of the order of 2750 gallons per minute. This heated cooling water from the heat exchanger is passed through the connecting pipe to the MgO washing and treating portion of the system, with the excess water available for use in other portions of the plant.

The flue gases leaving the cooling tower 30 through a connecting duct 33 to an inlet in the upper portion of a gas absorption zone 34. The gas absorption zone consists of an upright closed body having a restricted venturi-like throat 35 in the upper portion thereof through which the gases are passed downwardly at an accelerated velocity. A plurality of spray nozzles 36 are positioned in the upper portion of the zone, upstream of the venturi-throat, with each of the nozzles supplied with SO2 absorbing liquid in predetermined quantity from a connecting pipe 37 leading from a pump 38. The nozzles atomize the liquid which contacts the entering gases so that the liquid spray is well distributed throughout the cross-sectional flow path of the flue gases. The acceleration of the gases in passing through the venturi throat intimately mixes the atomized liquid and the gases for absorption of SO2 from the gases into the liquid droplets. The lower portion of the absorption zone is provided with a gas outlet 40 upwardly adjacent a hopper bottom 41. The hopper bottom is provided with a liquid outlet 42 which is connected through a pipe 43 to the suction side of a pump 45 which in turn discharges through a connecting pipe 46 leading to an acid storage tank 47 (see FIG. 2).

The gases leaving the absorption zone 34 tangentially enter the lower portion of an upwardly elongated separator 48 of cylindrical shape. The separator 48 separates the liquid droplets from the entraining gases by centrifugal force with the separated liquid collecting in the bottom of the tower which is provided with an outlet 50 and is connected with the outlet 42 which discharges the collected liquid into the pipe 43.

The gases leaving the upper portion of the separator 48 pass into the upper end portion of a second absorption zone 51 which is a substantial duplicate, insofar as structure is concerned, of the absorption zone 34. The absorption zone is provided with a plurality of spray nozzles 52 in the upper portion thereof adjacent the flue gas inlet duct 53. The lower end portion of the absorption zone is provided with a gas outlet 54 connected with a separator 55 which separates the liquid droplets from the entraining flue gases. The separator 55 is provided with a liquid outlet 56 which is connected to the suction inlet of the circulating pump 57 to recirculate liquid from the separator through a pipe 60, and the pump is arranged to discharge liquid into a connected pipe 61 leading to the spray nozzles 52 positioned in the upper portions of the absorption zone 51. The absorption zone 51 is provided with a hopper bottom having a liquid outlet. The bottoms of each of the absorption zones and each of the separators are connected by a pipe 62 which maintains a constant level in all bodies to maintain a head of liquid and equilibrium conditions in flow to the storage tank 47. The gases discharge from the separator 55 through an outlet duct 63 to the atmosphere.

Since the absorption system provides recovered chemicals for forming the cooling acid used in the digestion process, during normal operation, it is necessary to provide treated make-up water to the system to maintain the system rate of acid flow. Moreover, it is necessary to provide a source of SO2 gas for the system during starting up periods.

The make-up water pipe 32 is connected directly with the spray nozzles 31, and is provided with a branch pipe 65 leading to the pipe 67 which in turn connects with both the pipe 60 and the pipe 66. With such an arrangement the cooling tower 30 may be supplied with make-up water through pipe 32 or with cooled liquid from the heat exchanger 27 through the pipe 66. In a like manner, the spray nozzles 52 of zone 51 may be supplied with make-up water through pipe 65 or with cooled liquid from the heat exchanger 27 through the pipe 67. The pipes 32, 65, 66 and 67 are provided with valves so that flow through the pipes may be controlled.

To obtain efficient absorption of SO2 in the liquid in the zones 34 and 51, the liquid spray is controlled as to its pH value by the addition of magnesium hydroxide slurry which is obtained from the tank 25 and pumped through a closed pipe system 68. The system 68 includes a pump 70 with the return line of the system discharging into the tank 26 (as shown in FIG. 1). Suitable valved pipes 71 and 72 connect the system 68 with the pumps 57 and 36, respectively, so as to separately regulate the pH value of the liquid delivered to the spray nozzles 52 and 36, respectively. Best results have been obtained by maintaining a pH value of about 4.5 in the spray liquid in zone 34 and about 5.6 in zone 51. These values are obtained by controlling the rate of admission of magnesium hydroxide slurry introduced through pipes 71 and 72. The control can be accomplished manually or automatically by regulation of valves, or by the use of proportioning pumps. It will be understood that pH values are generally indicative of the monosulphite concentration under specific conditions, such as temperatures and acid strengths.

In the modification of the invention shown in FIG. 3, the flue gases leaving the multiclones 22 and the induced draft fan 23 (shown in FIG. 1) pass through the duct 28 directly to the SO2 absorption apparatus of the invention. The gases are at a higher temperature entering the venturi-like scrubber 80 than entering the venturi-scrubber zone 35 as shown in FIG. 2, and to attain efficient absorption of the SO2 gases in the magnesium bisulphite-monosulphite solution, the liquid is cooled in an indirect heat exchanger 81 before it is introduced into the duct 28 through the pipe 83 and the spray nozzle 85. The gases are cooled by contact with the spray liquid and are at a temperature of, for example, 120° F. in passing through the pressure regain portion of the venturi 80 and discharging into the expansion and gas turning chamber 84. With the reduction in gas flow velocity and the abrupt turn in the direction of gas movement, liquid droplets are separated from the gases to collect in the lower inverted frusto-conical portion 85 of the chamber 84. The gases leave the chamber 84 through a duct 86 which opens to a second venturi scrubber 87 where the gases are again contacted by a spray of SO2 absorbing liquid introduced through nozzles 88 positioned in the duct 86 upstream of the throat of the venturi 87. The absorbing liquid should be cooled in heat exchanger 100 to maintain a gas temperature of approximately 104° F. Thereafter, the gases flow through a pressure regain section 89 of the venturi scrubber 87 tangentially into a
cylindrical separator 34 where the centrifugal action of the gases separates suspended liquids, with the gases discharging to the stack 91. The collected liquid is withdrawn from the inverted frusto-conical bottom 92 of the separator 90.

While the venturi scrubbers 89 and 87 are shown in a vertical arrangement, the second scrubber 87 may be arranged with its axis of flow in a horizontal plane without appreciably affecting the SO₂ absorption efficiency of the arrangement. In any arrangement wherein hot gases are delivered directly to the venturi, as in the FIG. 3 version of the invention, it is desirable to enclose the gas duct 28 with a water jacket, or the like, to avoid an accumulation of solids in the duct. Such a water jacket is illustrated at 89 in FIG. 4 where its function is to condense moisture from the hot gases to provide a wetted duct surface to wash the walls of the duct and to prevent deposition of solids in the duct 28 and in the entrance to the venturi. This is accomplished by encircling the duct 28 adjacent the nozzles 83 in the entrance to the venturi throat. With the duct 28 extending into the enlargement, the spray droplets will contact the wetted wall of the enlargement where the moisture is formed by condensation of moisture from the gases by reason of the cooling effect of the water jacket.

In the absorption of SO₂ from the combustion gases, the absorption liquid should be maintained at optimum flow and pH values for most efficient chemical absorption of the SO₂ gases. As disclosed in FIG. 3, the liquid collected in the frusto-conical bottom 85 of the chamber 84 is passed through pipe 93 to the inlet pump 94 and through the indirect cooler 92 to the pipe 82. The pH of the liquid delivered to the spray nozzle 83 is regulated by the addition of controlled amounts of magnesium hydroxide slurry added thereto through the pipe 95, as much quantity is regulated by the operation of the proportioning pump 96 which is automatically regulated by a pH controller 97 positioned in pipe 82 downstream of the heat exchanger 81.

In a similar arrangement, the absorption liquid delivered to the nozzle 88 is withdrawn from the bottom of the cyclone separator 90 through a pipe 98 by a pump 99, passed through a heat exchanger 100 and a pipe 101 connected to the nozzle. The pH of the liquid is regulated by the addition of magnesium hydroxide slurry to the suction side of the pump 99 through a pipe 102, as determined by the operation of the proportioning pump 103 which is controlled by a pH controller 104. Each of the proportioning pumps 96 and 103 receive magnesium hydroxide slurry from a pipe 105 which is connected with a storage source of slurry.

The excess liquid collected in the separator 90 discharges by gravity through a pipe 106 to the liquid collected in the bottom of the chamber 84 with the liquid in excess of the withdrawal through pump 94 withdrawn by a pump 107 and delivered through pipe 108 to the acid plant for reuse in the digestion of pulp.

While in accordance with the provisions of the statutes we have illustrated and described herein the best form and mode of operation of the invention now known to us, those skilled in the art will understand the invention is also applicable to the recovery of SO₂ from the gaseous combustion products resulting from the incineration of any fuels by absorption in a solution prepared from a magnesium base material. Certain features of our invention may sometimes be used to advantage without a corresponding use of other features.

We claim:

1. The method of treating a magnesium base residual sulphite pulp liquor which comprises concentrating the residual liquor by evaporation, burning the combustible organic constituents of the concentrated liquor in suspension to obtain a dry ash consisting mainly of magnesium oxide and combustion gases containing SO₂, mixing said magnesium oxide with water to form a slurry of magnesium hydroxide, passing said combustion gases through a venturi-like SO₂ absorption zone in intimate direct co-current contact with a constantly moving atomized magnesium bisulphite-monosulphite liquid in an acid state containing magnesium monosulphite in an amount in excess of the normal solubility of the monosulphite in the liquid and at a temperature in the range between 100° F. and the dew point temperature of the said hot combustion gases to form magnesium bisulphite in the liquid, said excess monosulphite being in the form of a suspension of reactive microscopic crystals, separating the liquid containing magnesium bisulphite from said combustion gases, discharging a portion of the magnesium bisulphite containing liquid from said absorption zone to a storage zone, withdrawing the remainder of said magnesium bisulphite containing liquid from said absorption zone, and mixing said withdrawn liquid with a controlled amount of said magnesium hydroxide slurry to form magnesium monosulphite in excess of the normal solubility of the monosulphite in the liquid in the acid state while recirculating said mixed liquid to a successive absorption zone to absorb gaseous SO₂ in said liquid, said excess monosulphite being in the form of a suspension of reactive microscopic crystals.

2. The method of treating magnesium base residual sulphite pulp liquor which comprises concentrating the residual liquor, burning the combustible organic constituents of the concentrated liquor in suspension to obtain a dry unsintered ash consisting mainly of reactive magnesium oxide and combustion gases containing SO₂, removing substantially all of the ash produced from the combustion zone by flotation in the gaseous products of combustion, separating the dry unsintered ash from said gaseous products of combustion and mixing said ash with water to form a slurry of magnesium hydroxide, passing said gases in series through a plurality of venturi-like SO₂ absorption zones in direct co-current contact with an atomized magnesium bisulphite-monosulphite liquid in an acid state and at a temperature in the range of 100° F. to 120° F., said liquid including magnesium monosulphite in an amount in excess of the normal solubility of the monosulphite in the liquid at the prevailing liquid temperature, said excess monosulphite being in the form of a suspension of reactive microscopic crystals, said monosulphite combining with the SO₂ to form magnesium bisulphite in the liquid, separating the liquid containing magnesium bisulphite from said combustion gases leaving each of said zones, withdrawing a portion of the magnesium bisulphite liquid in each of said absorption zones for discharge to a magnesium bisulphite liquor storage zone, withdrawing the remainder of said magnesium bisulphite liquid from each of said absorption zones, mixing each of said last named liquids with said magnesium hydroxide slurry, controlling the amount of magnesium hydroxide slurry mixed with said liquids for the formation of magnesium monosulphite therein in excess of the normal solubility of the monosulphite in the particular bisulphite containing liquid, said excess monosulphite being in the form of a suspension of reactive microscopic crystals, and recirculating each of said mixed liquids to a corresponding absorption zone.

3. The method of treating magnesium base residual sulphite pulp liquor which comprises concentrating the residual liquor, burning the combustible organic constituents of the concentrated liquor in suspension to obtain a dry unsintered ash consisting mainly of reactive magnesium oxide and combustion gases containing SO₂, removing substantially all of the ash produced from the combustion zone by flotation in the gaseous products of combustion, separating the dry unsintered ash from said gaseous products of combustion and mixing said ash with water to form a slurry of magnesium hydroxide cooling said gases of combustion substantially to dew
point temperature by direct contact with a cooling liquid which is heated thereby, passing said heated liquid through a cooling zone and recirculating said cooled liquid through said gas cooling zone, passing said cooled gases through a venturi-like SO₂ absorption zone in direct co-current contact with an atomized magnesium bisulphite-monosulphite liquid in an acid state and approximately at a temperature in the range of 100° F.-120° F., said liquid including magnesium monosulphite in an amount in excess of the normal solubility of the monosulphite in the liquid, said excess monosulphite being in the form of a suspension of reactive microscopic crystals, said monosulphite combining with SO₂ to form magnesium bisulphite in the liquid, separating the liquid containing magnesium bisulphite from said combustion gases, withdrawing a portion of the magnesium bisulphite liquid from said absorption zone for discharge to a magnesium bisulphite cooling liquor storage zone, withdrawing the remainder of said magnesium bisulphite liquid from said absorption zone, treating said liquid with said magnesium hydroxide slurry to form magnesium monosulphite in excess of the normal solubility of the monosulphite in the particular bisulphite containing liquid, said excess monosulphite being in the form of a suspension of reactive microscopic crystals, and recirculating said liquid to a successive absorption zone for absorption of SO₂ by the conversion of magnesium monosulphite to magnesium bisulphite in the liquid.

4. The process of absorbing sulphur dioxide contained in gases of combustion which comprises passing said gases through a venturi-like sulphur dioxide absorbing zone, spraying an acidic magnesium bisulphite-monosulphite liquid at a temperature in the range between 100° F. and 120° F. co-currently into the gases entering said absorbing zone to convert said magnesium monosulphite and gaseous sulphur dioxide to magnesium bisulphite, said liquid containing magnesium monosulphite in an amount in excess of the normal solubility of the monosulphite in the liquid at the prevailing temperature, said excess monosulphite being in the form of a suspension of reactive microscopic crystals, separating the liquid containing magnesium bisulphite from the gases in a separating zone, passing one portion of said separated liquid containing magnesium bisulphite to a storage zone, passing the remaining portion of said separated liquid containing magnesium bisulphite to the said venturi-like sulphur dioxide absorbing zone, mixing magnesium hydroxide slurry with said separated liquid enroute to said absorbing zone to convert magnesium bisulphite to magnesium monosulphite for regenerating said liquid for absorption of sulphur dioxide therein, controlling the quantity of said magnesium hydroxide added to said liquid for forming magnesium monosulphite therein in excess of the normal solubility limit of magnesium monosulphite in said bisulphite containing liquid, said excess monosulphite being in the form of a suspension of reactive microscopic crystals, and thereby increasing the sulphur dioxide absorbing capacity thereof.

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