July 7, 1936. G. A. RICHTER 2,047,032

PROCESS OF CORRELATING CHEMICAL RECOVERY IN PULP MILLS OPERATING WITH DIFFERENT KINDS OF LIQUORS

Filed Sept. 28, 1934 2 Sheets-Sheet 1

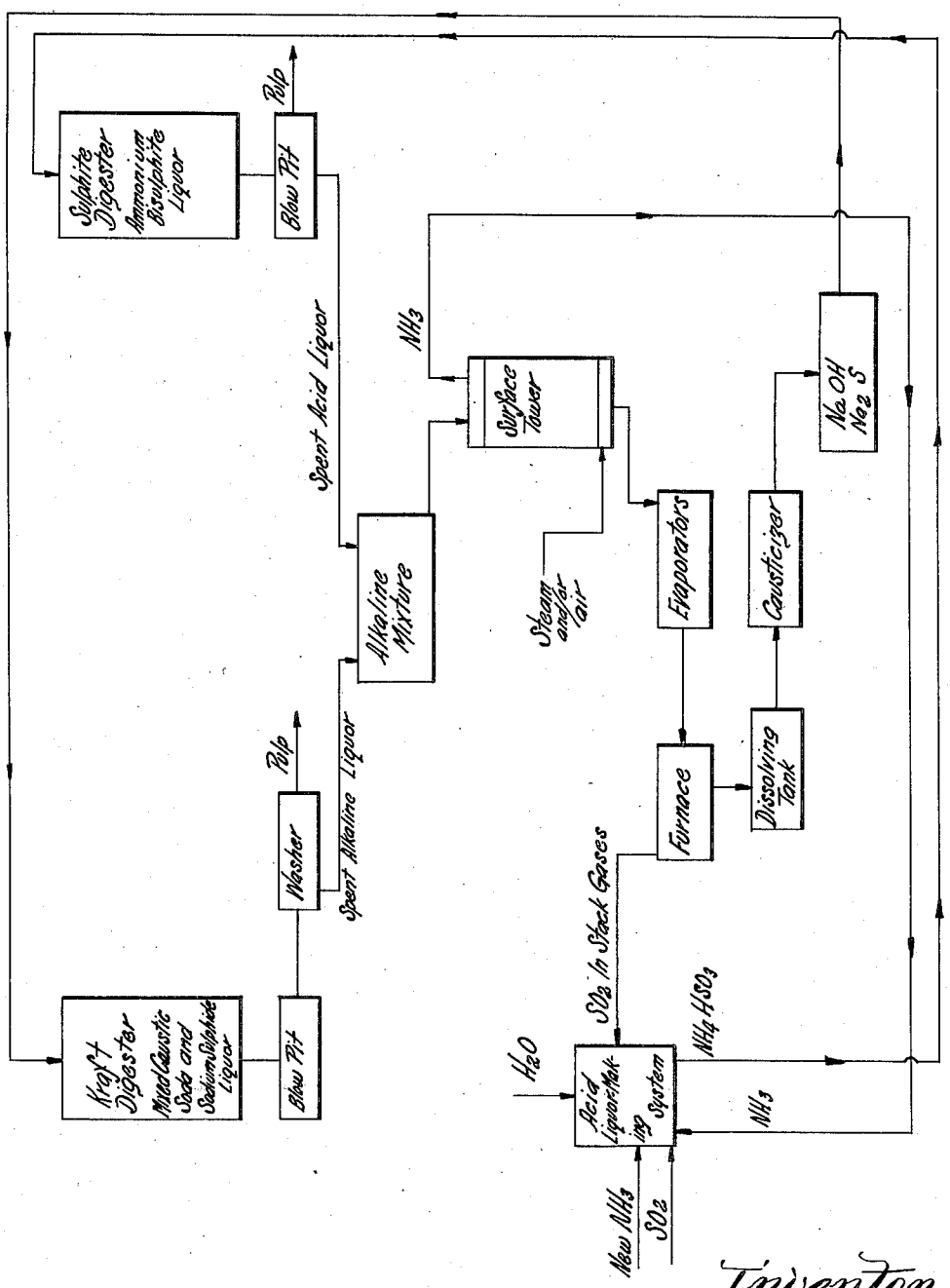


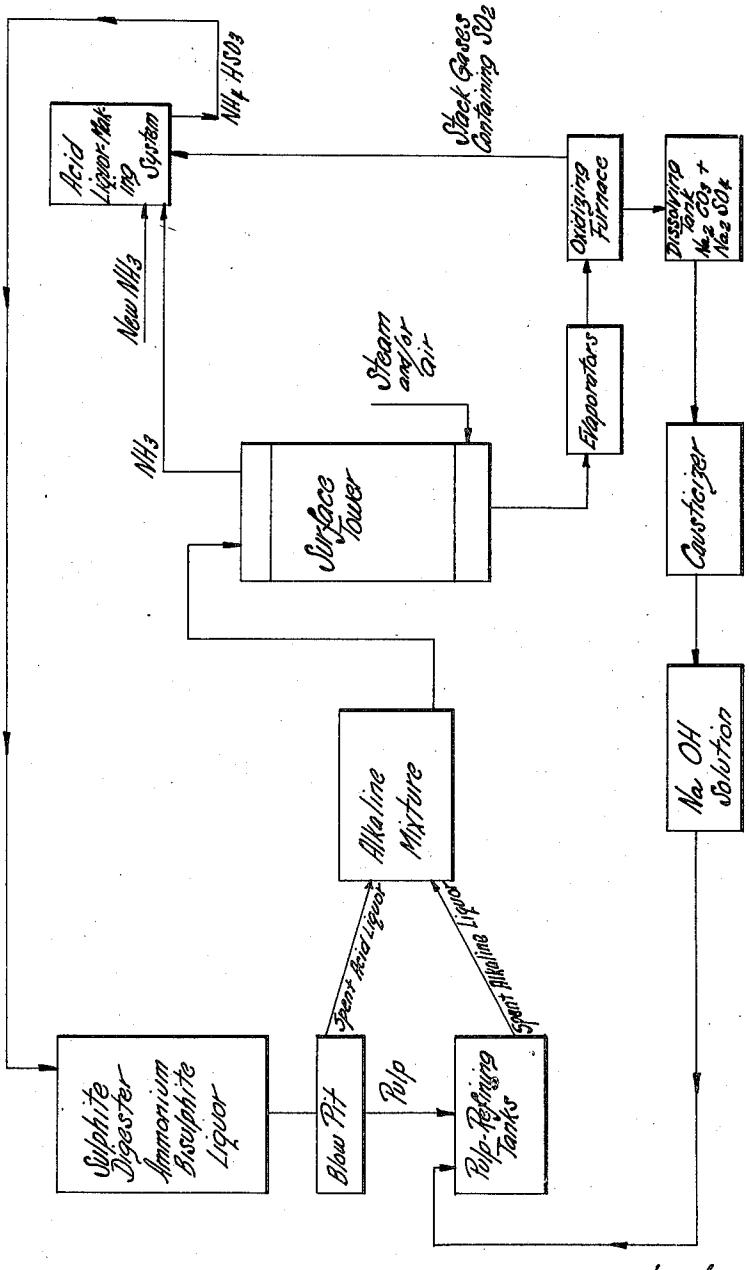
Fig. 1.

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PROCESS OF CORRELATING CHEMICAL RECOVERY IN PULP MILLS OPERATING WITH DIFFERENT KINDS OF LIQUORS

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6 Claims. (Cl. 23—48)

This invention relates to a process of correlating chemical recovery in pulp mills operating with different kinds of liquors but in the same locality and under the same auspices. It relates more particularly to the correlation of chemical recovery in a pulp mill wherein a cooking liquor containing an ammonium-sulphur salt is employed with chemical recovery in a pulp mill wherein an alkaline sodium base liquor is employed for cooking or fiber-liberating purposes and/or for the refinement or other treatment of preliberated pulp.

In accordance with the present invention, the spent cooking liquor containing the ammonium-sulphur salt is mixed with the spent alkaline sodium base liquor so as to cause a liberation of the ammonia content of the first-named liquor and a substitution of sodium therefor. The ammonia thus liberated is recovered and the sodium salts present in the mixed spent liquors are also recovered. More specifically, the ammonia may be recovered by causing it to react with water and an acid radical containing sulphur to form a fresh cooking liquor for the pulping mill whose cooking liquor is to contain an ammonium-sulphur salt, whereas the sodium salts present in the mixed spent liquors may be transformed into a fresh alkaline sodium base liquor by burning the combustible content and smelting the inorganic content of such mixed liquors to produce mixed sodium salts, including sodium-sulphur salt, dissolving the salts in water, and causticizing at least one salt in solution. Smelting may, if desired, take place in a reducing atmosphere, in which case the resulting smelt of mixed sodium salts includes essentially sodium carbonate and sodium sulphide. In such case, the sodium carbonate component of the smelt is preferably causticized. Smelting may, if desired, also take place in an oxidizing atmosphere, in which case the resulting smelt of mixed sodium salts includes essentially sodium carbonate and sodium sulphate. In such latter case, both the sodium carbonate and sodium sulphate components of the smelt are preferably causticized.

The principles of the present invention extend especially to a sulphite mill wherein raw cellulosic material, such as chipped wood, is subjected to the cooking or fiber-liberating action of a liquor containing ammonium sulphite and/or ammonium bisulphite in solution. In some instances, the sulphite cooking liquor may be one consisting of a sulphurous acid solution of ammonium sulphate or other ammonium salt, or of a mixture of such salt and ammonium sulphite. The chemical recovery in such a mill may, in accordance with the present invention, be correlated with the chemical recovery in a kraft mill, soda mill, or other mill wherein an alkaline sodium base cooking liquor is employed for the pulping of wood or other raw cellulosic material and/or with the chemical recovery in a pulp mill wherein preliberated wood pulp or other preliberated cellulose pulp, such as the very sulphite pulp produced in the ammonium base cooking liquor, undergoes refinement or other chemical modification with an alkaline sodium base liquor. In the event that the present invention is being practiced in connection with a kraft mill, the smelting of the inorganic content of the mixed spent liquors is preferably accomplished under reducing conditions so as to make possible a regeneration of a fresh kraft cooking liquor, as will appear hereinafter in greater detail.

With the foregoing and other features and objects in view, I shall now describe my invention with particular reference to the accompanying drawings, wherein,—

Figure 1 is a flow sheet depicting how my invention is applied in correlating chemical recovery in a sulphite mill with chemical recovery in a kraft mill.

Figure 2 is a flow sheet depicting how my invention is applied in correlating chemical recovery in a sulphite mill with chemical recovery in a pulp-refining mill operating to refine the sulphite pulp through the use of alkaline sodium base liquor.

I shall first deal with the flow sheet of Figure 1 which relates to the simultaneous production of sulphite pulp and kraft pulp. The cooking liquor used for the production of the sulphite pulp may be a sulphurous acid solution of ammonium sulphite of the desired combined and free SO_2 content. The cooking liquor used for the production of kraft pulp may, as ordinarily, contain caustic soda and sodium sulphide as the essential fiber-liberating chemicals and be of the desired strength and sulphidity. After cooking in the sulphite digester has been completed, the digester may be blown and the spent acid liquor separated from the pulp. Similarly, after cooking in the kraft digester has been completed, its contents may be discharged into a blow-pit and the pulp then separated, as in a countercurrent washer, from the "black" or spent alkaline liquor. The spent alkaline liquor and spend acid liquor are commingled, preferably in hot condition, and the mixture passed through a tower in countercurrent contact with steam and/or hot air which

serves to accelerate the liberation of the ammonia content of the spent acid liquor. It might be mentioned at this point that sufficient spent alkaline liquor is added to the spent acid liquor to produce a distinctly alkaline mixture, preferably one wherein there is present sufficient sodium constituent to displace substantially all the ammonia constituent associated with the spent acid liquor. In view of the fact that there is considerable active or unspent alkali present in the spent alkaline liquor, there is a tendency to liberate considerable ammonia even at room temperature, but, as already indicated, the hot mixed liquors are preferably subjected to the action of steam and/or hot air to promote a rapid and substantially complete liberation of the ammonia constituent associated with the spent acid liquor. The liberated ammonia may be caused to react with water and sulphur dioxide to produce a fresh acid sulphite cooking liquor, such ammonia as is lost in the cycle being supplied to the acid-liquor-making system.

The mixed liquors from which the ammonia has been substantially completely expelled may then be concentrated, as in multiple-effect evaporators, to a stage where furnacing operations may be satisfactorily and economically performed thereupon. The concentrated mixture is then delivered into a furnace or kiln maintained under reducing conditions wherein its organic content is burned and its inorganic content smelted to form a mixture of sodium salts including essentially sodium sulphide and sodium carbonate. Such sulphur dioxide as is generated in the furnace and passes out as part of the stack gases may be recovered from the stack gases and used in the acid-liquor-making system. The smelted sodium salts may be dissolved in water to form a solution, whereupon the sodium carbonate component of the solution may be causticized with lime, thereby producing a fresh kraft cooking liquor.

The process hereinbefore described possesses various advantages among which are the following:—

(1) The alkali used for the recovery of the ammonia does not represent an added cost, as it is alkali associated with spent alkaline liquor that must undergo recovery operations anyway;

(2) The "black" or spent alkaline liquor may as ordinarily be separated at elevated temperature, say, at a temperature as high as about 180° F. and be commingled in such hot condition with the spent acid liquor, wherefore, appreciable steam saving is possible in the recovery of the ammonia;

(3) The sulphur constituent as well as the ammonia content of the spent acid liquor is recovered, as such sulphur radical is made to combine with the sodium constituent and furnish sulphidity in the kraft cooking liquor;

(4) The sulphidity in the kraft cooking liquor may be maintained high, in consequence of which it is possible to produce a high grade kraft pulp;

(5) There is no need for using sodium sulphate (salt cake) as the make-up chemical in the kraft mill, since such sulphidity as is desired in the kraft liquor originates in the sulphite mill and such sodium constituent as may be necessary for making up losses in the kraft cycle may be furnished in the form of sodium carbonate. If desired, the spent alkaline liquor may be mixed with the sulphite pulp while such latter pulp is still associated with its spent acid liquor so as to enable the washing of the sulphite pulp in iron equipment that would otherwise be attacked by the spent acid liquor. On the other hand, as already indicated, the spent alkaline liquor may be mixed with the spent acid liquor after the latter liquor has been drained from the sulphite pulp in the blow-pit or separated therefrom in a washer.

I shall now deal with the flow sheet of Figure 2 which relates to the simultaneous production of sulphite pulp and the refinement of the very same pulp in an alkaline sodium base liquor to higher alpha cellulose content. In such case, after the sulphite digester has been blown and the spent acid liquor separated therefrom, as by washing, the sulphite pulp is suspended in an alkaline sodium base liquor which serves to dissolve non-alpha cellulose components therefrom and thus to refine it to higher alpha cellulose content, say, to an alpha cellulose content of at least about 93%. The refining liquor may be caused to react upon the pulp at room or elevated temperature conditions, depending upon its strength or alkalinity. Thus, strong alkaline liquors may be employed at room or lower temperature, whereas weak alkaline liquors may be employed at about boiling temperature or even at temperatures attainable only under superatmospheric pressure. The alkalinity of the refining liquor may be furnished by such sodium base compounds as caustic soda and/or sodium sulphide and/or sodium carbonate. In any event, the spent alkaline liquor separated, as by washing, from the refined pulp is commingled with the spent acid liquor, which, as already indicated, has resulted from pulping raw cellulosic material in an ammonium bisulphite cooking liquor. The liquors may be mixed and the resulting mixture may be handled under conditions similar to those described in connection with Figure 1 up to the furnace or kiln. In the instant case, however, the furnace may, if desired, be operated under oxidizing conditions so as to yield a smelt inclusive essentially of sodium carbonate and sodium sulphate. The smelt may be dissolved in water and both the sodium carbonate and sodium sulphate components of the resulting solution may be causticized with lime. The causticized solution may serve as the liquor for refining the sulphite pulp. Of course, a reducing atmosphere may be maintained in the furnace, in which case, the smelt will contain essentially sodium carbonate and sodium sulphide. Such a smelt may be dissolved in water and the resulting solution used as such for the refinement of the sulphite pulp, although it may be desirable to causticize the sodium carbonate component of the solution preparatory to its use for refining pulp.

The practices hereinbefore described are subject to amplification or modification. Thus, preparatory to treating the spent acid ammonium base sulphite liquor with spent alkaline sodium base liquor, it may be desirable to remove easily releasable free sulphur dioxide therefrom as by sweeping it with steam and/or hot air in a suitable tower, thereby necessitating less spent alkaline liquor for liberating its ammonia constituent. The sulphur dioxide thus swept from the spent acid liquor may be employed in the acid-liquor-making system. The ammonia liberated and recovered from the spent acid liquor may, if desired, be employed in the production of sulphite pulp as disclosed in my application Serial

No. 747,922, filed October 11, 1934, according to which application the ammonia is caused to penetrate into the chipped wood or other raw cellulosic material prior to reacting thereupon with a sulphurous acid solution to produce an ammonium bisulphite cooking liquor.

I claim:—

1. A process of correlating chemical recovery in a pulp mill wherein a cooking liquor containing an ammonium-sulphur salt is employed with chemical recovery in a pulp mill wherein an alkaline sodium base liquor containing caustic soda is employed, those steps which comprise mixing the spent liquors produced in both such mills in such proportions as to produce a distinctly alkaline mixture and under sufficient heat to liberate to a large degree the ammonia content of the first-named liquor as ammonia gas and to substitute sodium therefor, recovering the liberated ammonia, causing reaction between the recovered ammonia, water, and an acid radical containing sulphur to form a fresh cooking liquor for the first-named mill, burning the combustible content and smelting the remaining inorganic content of the mixed liquors to produce mixed sodium salts, including sodium-sulphur salt, recovering the sodium salts in water to form a solution thereof, and causticizing at least one salt in solution to form a fresh liquor for the second-named mill.

2. A process of correlating chemical recovery in a pulp mill wherein a cooking liquor containing ammonium sulphite is employed with chemical recovery in a pulp mill wherein an alkaline sodium base liquor containing caustic soda is employed, those steps which comprise mixing the spent liquors produced in both such mills in such proportions as to produce a distinctly alkaline mixture and under sufficient heat to liberate to a large degree the ammonia content of the first-named liquor as ammonia gas and to substitute sodium therefor, recovering the liberated ammonia, causing reaction between the recovered ammonia, water and sulphur dioxide to form a fresh cooking liquor containing ammonium sulphite for the first-named mill, burning the combustible content and smelting the remaining inorganic content of the mixed liquors to produce mixed sodium salts, including sodium-sulphur salt, recovering the sodium salts in water to form a solution thereof, and causticizing at least one salt in solution to form a fresh liquor for the second-named mill.

3. A process of correlating chemical recovery in a pulp mill wherein a cooking liquor containing ammonium sulphite is employed with chemical recovery in a pulp mill wherein an alkaline sodium base liquor containing sodium sulphide and caustic soda is employed, those steps which comprise mixing the spent liquors produced in both such mills in such proportions as to produce a distinctly alkaline mixture and under sufficient heat to liberate to a large degree the ammonia content of the first-named liquor as ammonia gas and to substitute sodium therefor, recovering the liberated ammonia, causing reaction between the recovered ammonia, water, and sulphur dioxide to form a fresh cooking liquor containing ammonium sulphite for the first-named mill, burning the combustible content and smelting the remaining inorganic content of the mixed liquors under reducing conditions to produce mixed sodium salts, including sodium sulphide, recovering the sodium salts in water to form a solution thereof, and causticizing salt other than sodium sulphide in solution to form a fresh liquor for the second-named mill.

4. A process of correlating chemical recovery in a pulp mill wherein a cooking liquor containing ammonium bisulphite is employed with chemical recovery in a pulp mill wherein an alkaline sodium base liquor containing sodium sulphide and caustic soda is employed, those steps which comprise mixing the spent liquors produced in both such mills in such proportions as to produce a distinctly alkaline mixture and under sufficient heat to liberate to a large degree the ammonia content of the first-named liquor as ammonia gas and to substitute sodium therefor, recovering the liberated ammonia, causing reaction between the recovered ammonia, water, and sufficient sulphur dioxide to form a fresh cooking liquor containing ammonium bisulphite for the first-named mill, burning the combustible content and smelting the remaining inorganic content of the mixed liquors under reducing conditions to produce mixed sodium salts, including sodium sulphide, recovering the sodium salts in water to form a solution thereof, and causticizing salt other than sodium sulphide in solution to form a fresh liquor for the second-named mill.

5. A process of correlating chemical recovery in a pulp mill wherein a cooking liquor containing ammonium-sulphur salt is employed with chemical recovery in a pulp mill wherein an alkaline sodium base liquor containing caustic soda is employed, those steps which comprise mixing spent liquors derived from both such mills under conditions to liberate as ammonia gas ammonia constituent of the first-named liquor and to substitute sodium therefor, causing reaction between such liberated ammonia, water, and an acid radical containing sulphur to form a fresh cooking liquor for the first-named mill, burning the combustible content and smelting the remaining inorganic content of the mixed liquors to produce mixed sodium salts, including sodium-sulphur salt, dissolving the sodium salts in water to form a solution thereof, and causticizing at least one salt in solution to form a fresh liquor for the second-named mill.

6. A process of correlating chemical recovery in a pulp mill wherein a cooking liquor containing ammonium sulphite is employed with chemical recovery in a pulp mill wherein an alkaline sodium base liquor containing caustic soda is employed, those steps which comprise mixing spent liquors derived from both such mills under conditions to liberate as ammonia gas ammonia constituent of the first-named liquor and to substitute sodium therefor, causing reaction between such liberated ammonia, water, and sulphur dioxide to form a fresh cooking liquor containing ammonium sulphite for the first-named mill, burning the combustible content and smelting the remaining inorganic content of the mixed liquors to produce mixed sodium salts, including sodium-sulphur salt, recovering the sodium salts in water to form a solution thereof, and causticizing at least one salt in solution to form a fresh liquor for the second-named mill.

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