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PROCESS FOR THE UTILIZATION OF SUGARCANE BAGASSE

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By the term "bagasse", used throughout both specification and claims, is meant the fibrous residue of sugar cane which remains after the extraction of the sugar constituents. In present day operations, extraction of juice from the cane is entirely by means of mills, and bagasse, therefore, might be considered as the reject from a roller mill; it is a semi-dry powdery mixture of pith cells and fiber, containing but a few percent of the original sugar as a residual.

In the broader sense, bagasse is the entire residue of the cane after sugar bearing juices have been extracted. So far the use of diffusion, universal in beet sugar work, has made no inroads in cane producing territory. This is largely due to the fact that the bagasse is the all essential fuel, and therefore must be obtained in a state of sufficient dryness to burn. By means of my process bagasse becomes far too valuable to be thus utilized, so with this incentive it might well be that the roller mill might be replaced with some type of slicer and a diffuser battery. In such event, the reject from the cane juice extraction would still be "bagasse" in the sense in which the word is here used.

From the nature of the cane, bagasse must consist essentially of a mixture of woody fiber, pith cells, and the unextracted juice constituents. Today, this residual serves no purpose but to supply a certain amount of heat to the sugar making operation, and as bagasse cannot be burned with any real efficiency even this utilization is very low, rarely exceeding in heat performance the equivalent of a barrel of oil per ton of bagasse.

The cellulose fiber of the woody part of the cane, when examined under the microscope, is seen to be of excellent appearance from the paper-maker's viewpoint. The pith, of course, does not possess any real fiber, but it would have considerable merit as a filling material if paper pulp were made from the bagasse without any prior separation having been made between pith and woody fiber before submitting same to the cooking operation. In spite of this fact, and the evident suitability of the fiber to paper making, we are confronted with the economic situation that there is nowhere in the world to-day any real paper pulp operation based on sugar cane bagasse.

The reason for this anomaly is found in the peculiar sensitiveness of the cellulose fiber from cane to those chemicals to-day used universally as resolvents for lignin and lignified fiber, to-wit: highly acid derivatives of sulphur dioxide, caustic soda, and mixtures of caustic soda with subordinate amounts of sodium sulphide. As a contributing cause, we may also state that in comparison with carefully prepared wood chips bagasse, as paper pulp material, must be admitted to have been very roughly handled. Any further abuse in the cooking process therefore results in an almost absolute destruction of the strength of the fiber.

In making pulp from bagasse I have found that the two gentlest chemical agents are, 1st, a solution of sodium sulphide free from caustic soda; and, 2nd, sulphur dioxide neutralized with soda to such an extent that it corresponds to the bisulphite in chemical composition. The nature of bagasse is such that these comparatively gentle reagents will completely liberate the fiber from all incrusting matter at very low temperature, and consequently pressure, if we compare such data with the prevailing practice in pulp making based on wood. Thus in place of cooking with steam pressure of 125 lbs., I find that I can do with as low as 30, or even lower if the time be prolonged, and that the strength of cooking liquor in active ingredients can be reduced to a small fraction of that absolutely essential on wood. It follows, therefore, that bagasse so treated yields a very strong and soft fiber from which excellent paper can be made.

In the event that the best grade of pulp is required, I find it essential to separate the pith from the woody fiber before cooking is commenced, but for inferior grades both may be cooked together. After cooking it is virtually impossible to make a separation between the cellulose resulting from these two types of raw materials. However, in the uncooked state it is very easy to make an efficient separation between pith and woody fiber. It is only necessary to subject the mixed bagasse to a gentle attrition method, such as passing it through a shallow layer of pebbles in motion in a pebble mill, until the individual pitch cells have been loosened from their adhesion to woody fiber, or to one another. The product from the mill is then passed over a fairly coarse screen, say 20 mesh, when pith cells will pass through, while the clean lignified fiber, virtually free from pith, will be retained upon the screen. It will be inevitable that if water be used to aid this separation it becomes somewhat charged with sugar, and so, to prevent unpleasant fermentation, the operation had best be conducted at a temperature which will inhibit all such undesirable action. The constant reuse of such water will therefore gradually raise such sugar content, and it will be evident that to the extent to which it can be returned to the mill as

maceration water it can increase the percentage of sugar extraction. The separated pith has many uses which it will be dealt with later. To recover it from the underflow (if water be used) from the coarse screen before described, it is only necessary to pass the magma through a fine screen, say 100 mesh, when virtually all pith cells will be retained, and the water may then be reused to treat fresh bagasse, or used as maceration water as before described.

I use either an acid or an alkaline agent to remove all incrusting matters, depending upon the type of pulp desired, but, with the exception of this step, all other methods employed are alike, to-wit:- the preliminary preparation of the bagasse; the type of equipment used in cooking; the cooking operation itself; the separation of the spent cooking liquor from the pulp; the evaporation of the liquor; the incineration under identical conditions; and, finally, the recovery of the same alkali salts from the ash of said incineration.

A fundamental distinction between my process and present practice is that I do not waste the cooking liquor as does the lime-sulphur-dioxide system, nor do I recover a mixture of salts for cyclic reuse as in the kraft-sulphate process, nor do I recover a mixture of salts from cooking with sodium bisulphite and use this as "make-up" alkali in the kraft process (Richter patents); but in contra-distinction to all of these processes, or of any possible combinations of them, I regard the process of paper making as the means whereby sodium sulphate is converted into commercial chemicals, to-wit, soda ash, caustic soda, sodium sulphide, and sodium thiosulphate. A joint object is attained, therefore, by the use of my process, namely; the production of pulp and simultaneously the conversion of salt cake into higher priced chemicals in an economic manner. The recovery and sale of such commodities will, therefore, constitute a means whereby the cost of pulp making can be substantially reduced.

Whether the cooking liquor represents the acid or the alkaline phase, the type of soda used is the same, namely, salt cake. When the spent cooking liquor from either phase has been evaporated and incinerated under reducing conditions, it leaves as a residue the same identical type of admixture of salts, to-wit, carbonate and sulphide of soda, and the only possible distinction between them will be in the proportion of the one to the other. The reason for this coincidence is that in either case the incrusting material from the fiber is dissolved as an organic sulpho-salt of soda, and when this is submitted to a high temperature under reducing conditions no stable salts of soda save the carbonate and sulphide can exist. An examination of the relative solubilities of these two soda salts will indicate that fractional crystallization can be employed to separate them from one another, thus yielding both the carbonate and the sulphide in sufficient purity for the market.

To illustrate: If a supersaturated solution of both salts be prepared, by acting on the incinerated residue from the spent cooking liquor with boiling water, and if the solution be then cooled, a copious crystallization of sodium carbonate (containing little sulphide) will result. The crystals are separated from the mother liquor, and this is then concentrated by evaporation until on cooling it will yield a crystallization of sodium sulphide (containing in this case a little carbonate). The crystals of sulphide are separated from the mother liquor and the latter is returned to fresh incinerated residue, with additional water, and the cycle is once more commenced. This mother liquor, which is steadily accumulating minor impurities, is periodically removed from the circuit and worked up for its constituent salts. It gradually accumulates considerable thiosulphate (by oxidation) so an excellent use is to treat it with sulphur dioxide, thus converting both its carbonate and sulphide into thiosulphate.

In the acid form of cooking a white pulp suitable for immediate use in all but the best papers is produced, while in the alkaline form of cooking a brown pulp is the result. When this latter product is bleached, it produces a pulp considerably stronger than the acid cooked variety Moreover, the consumption of bleaching material is very small, as the removal of the incrusting lignin from the fiber has been far more thorough than in the ordinary cooking methods.

The low pressure required, and the character of the cooking media, to-wit, solutions of sodium bisulphite and sodium sulphide, as well as the peculiar consistency of the mixture of bagasse and cooking liquor, combine to form a set of conditions unique in the annals of paper making. It is therefore possible to use an entirely different manipulation in cooking than that customarily employed, namely continuous cooking. It the state of the art as we find it to-day, with wood as the raw material, the cooking operation is always on a batch basis. A digester is filled with chips, the cooking liquor is added, the digester is sealed, and steam is turned on until the cooking is complete. The digester is then "blown", and the cycle repeated. In certain cases the cooking liquor is circulated, but in all cases the chips remain in the digester until they are finally discharged as pulp.

As I operate, several digesters are connected in series, the blow-off pipe of one being connected to the top of the next digester in the series. The digesters are placed on one elevation, if desired, and hydraulic balance is produced by keeping a steam pocket at the top of each digester, so as to prevent a syphon effect being produced. If now a mixture of bagasse and cooking liquor be pumped into the first digester, it promptly induces a flow of finished pulp from the last one in the series. While this mixture of bagasse and cooking liquor will readily flow through a large pipe, it would be rather hard to pump, so I prefer to mix these together and charge them into a tall vertical pipe communicating with the bottom of the first digester. The advantage of low cooking pressure is now seen, for were 125 lbs. called for then this feed pipe would have to be at least 250 ft. in height. However, as I require but 30 lbs., and can use 15 lbs., it follows that a maximum height of only 60 ft. will be required. The handling of present day digesters entails much manual labor and supervision, while the system of cooking here described, when once brought into balance, calls for a minimum of attention with no manual labor at all, being practically automatic throughout.

I now give a preferred case of cooking in the alkaline phase. The prepared bagasse, if the highest grade of pulp is required, is saturated with a solution of sodium sulphide of such strength, and in such quantity, that the Na_2S content of the mixture equals approximately 50% of the bone dry weight of the bagasse operated upon. It is then charged into the feed pipe of

such a battery of digesters as before described, and after treatment for an hour, more or less, under the varying pressure of say 30 lbs. in the first digester, and the even lower pressure in the succeeding ones, it is discharged to a dewatering and washing filter as a finished unbleached fiber.

The separated spent cooking liquor and wash water is then in part used to make up fresh cooking liquor by the addition of sodium sulphide, and in part sent to the recovery system. Here it is evaporated to a syrupy consistency, incinerated under reducing conditions, and the resultant sinter, ashes and slag, depending on whether or not the temperature be high enough to fuse it, is discharged into a modicum of water, so as to produce a hot, concentrated solution capable of crystallization without any further operation save cooling. Before cooling, the solution will be found to consist chiefly of sodium carbonate and sodium sulphide. After cooling, the solution will consist chiefly of sodium sulphide, with a little carbonate and thio-sulphate as the chief impurities; most of the carbonate having separated as crystallized sodium carbonate containing, of course, a little sulphide as an impurity. Separation is then made by a centrifuge, or otherwise, between the crystals and the mother liquor, and the latter used "as is" to make up fresh cooking acid. The crystal product, of slightly impure sodium carbonate, is best utilized as raw material for the well known "Ferrite" process of caustic soda manufacture, and the production of caustic soda should be made an integral part of the pulp making operation.

The spent cooking liquor contains much organic material, in fact one-half of the bone dry weight of the bagasse; and it is, therefore, possible to add much sodium sulphate (salt cake) to the syrupy liquor before incineration, and yet keep a safe excess of carbon for reducing purposes. At this point the pith separated in the initial stage of preparing bagasse for cooking may be incorporated, and will thus permit of an even greater addition of salt cake. In the process of incineration, all this sulphate becomes reduced to sulphide. It not only makes up for the conversion of sulphide into the carbonate, but it supplies all the sulphide for cooking, and still leaves a surplus for the market. Such sulphide must be crystallized from the mother liquor remaining from the sodium carbonate separation to make it pure enough to sell. This will involve evaporation, cooling to produce crystallization, and separation by the centrifuge, or otherwise, between these crystals of sodium sulphide and the remaining mother liquor. The sulphide crystals will contain the customary excessive amount of water of crystallization, and should preferably be melted in their water of crystallization and boiled until the market standard of 60% Na_2S is obtained. The mother liquor from this last crystallization is best worked for thiosulphate by passing SO_2 into it, thus converting at one operation both the sulphide and carbonate contained into thiosulphate, a compound which already exists to a considerable extent in the fluid under discussion. It has been produced by the rather free access of air which is unavoidable when an extended series of manipulations is involved.

I now give an illustration of the acid phase of my process. I prefer to make the essential sodium bisulphite solution by running a solution of sodium sulphate down the customary acid tower, instead of using plain water. The lime stone will dissolve with equal facility and the effluent will be milky with gypsum formed in the reaction. It would be equally simple to make the ordinary cooking acid first, and then decompose this with salt cake afterwards. In either case the gypsum is readily separated just by settling, but the solution of bi-sodium sulphite formed will of course remain saturated with this salt. This is not of any significance. It is to be noted, however, in either case, that the free and combined SO_2 in the effluent from the tower must be very different in relative proportion than is customary in the preparation of the cooking acid for the present day sulphite process. Instead of having a "free SO_2" reading 3 or more times that of the "combined SO_2", it is essential that these readings be almost equal to one another, so that the compound formed shall approach the composition of the theoretical bi-sulphite. Having thus made the cooking liquor, it is next added to the bagasse prepared as before, and the same sequence of steps is repeated until the spent cooking liquor is obtained. It is, of course, desirable to neutralize this spent cooking acid before evaporation as this permits of the use of cheap iron equipment in place of costly acid proof construction, and it is providential that the sodium carbonate produced in the incineration step is adequate for the work. The impure crystals of sodium carbonate obtained later on are therefore used to effect this neutralization. Evaporation to syrupy consistency, incineration under reducing conditions, lixiviation of the resultant sinter ashes, or slag, according to the temperature employed, then follow in due course as recited in the former illustration. The sodium carbonate, crystallizing out when this hot solution is cooled, is separated as before; but instead of being used for the subsequent manufacture of caustic soda, or otherwise disposed of, it is returned to the process to neutralize the acidity of the spent cooking acid prior to evaporation. As in the former illustration, as much as possible of the spent acid is returned directly in the manufacture of new (or fresh) cooking acid, the remainder being sent to the recovery system. As there is no use in this phase for sodium sulphide, it follows that the entire yield of this chemical is available for market purposes. It is therefore crystallized out after suitable evaporation, as before described, and the mother liquor remaining from this step preferably worked for the thiosulphate.

It will be seen that the entire procedures in both cases are singularly alike, the differentiation being in relative quantity and not in kind. Also, whether or not a certain commodity is returned in whole or in part to the circuit, or whether it be marketed instead.

Parenthetically, it might be mentioned here that the poorest and cheapest forms of salt cake can be applied to this type of work, and in the production of the SO_2 any kind of sulphide ore can be utilized. In contradistinction to these facts it may be said that present processes make exacting demands upon the quality of salt cake, and that most of the cooking acid used, in this country at least, requires brimstone and not ore, or even high grade pyrite, as the source of the SO_2. It will be apparent, also, that while I have discussed my process only as applied to bagasse as the raw material used in connection with it, that it is likewise applicable to other raw materials containing fiber suitable for paper making.

Having thus described in detail my process, I claim:

1. A process for paper pulp making which comprises, separation of bagasse into two respective portions consisting of lignified fiber and pith cells; cooking the lignified fiber with active soda-salt made from salt cake for the production of paper pulp; separation of the pulp from the spent cooking liquor; evaporation of the spent cooking liquor to syrupy consistency; incineration of said concentrated liquor under reducing conditions; lixiviation of the resultant mass to form a solution of sodium carbonate and sodium sulphide; separation of these two ingredients from one another by fractional crystallization; return of a part of such recovered material to the circuit where required; and the conversion of the remainder into marketable form.

2. A process such as described in claim 1, with the added step that the pith separated in the first mentioned step be incorporated with the syrupy spent cooking liquor after evaporation and before incineration, thus permitting simultaneous addition of salt cake in greater amounts.

3. A process for paper pulp making which comprises, separation of bagasse into two respective portions of lignified fiber and pith cells; continuous cooking of the lignified fiber with active soda-salt made from salt cake for the production of paper pulp by passing said lignified fiber continuously through a series of interconnecting digesters, each of which is maintained at a higher pressure than the next one in the sequence, until cooked pulp is discharged under atmospheric pressure; separation of the pulp from the spent cooking liquor; evaporation of the spent cooking liquor to syrupy consistency; union of pith cells previously separated and said syrupy cooking liquor, thus permitting simultaneous addition of salt cake in greater amounts; incineration of said concentrated liquor under reducing conditions; lixiviation of the resultant mass to form a solution of sodium carbonate and sodium sulphide; separation of these two ingredients from one another by fractional crystallization; return of a part of such recovered material to the circuit where required; and the conversion of the remainder into marketable form.

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