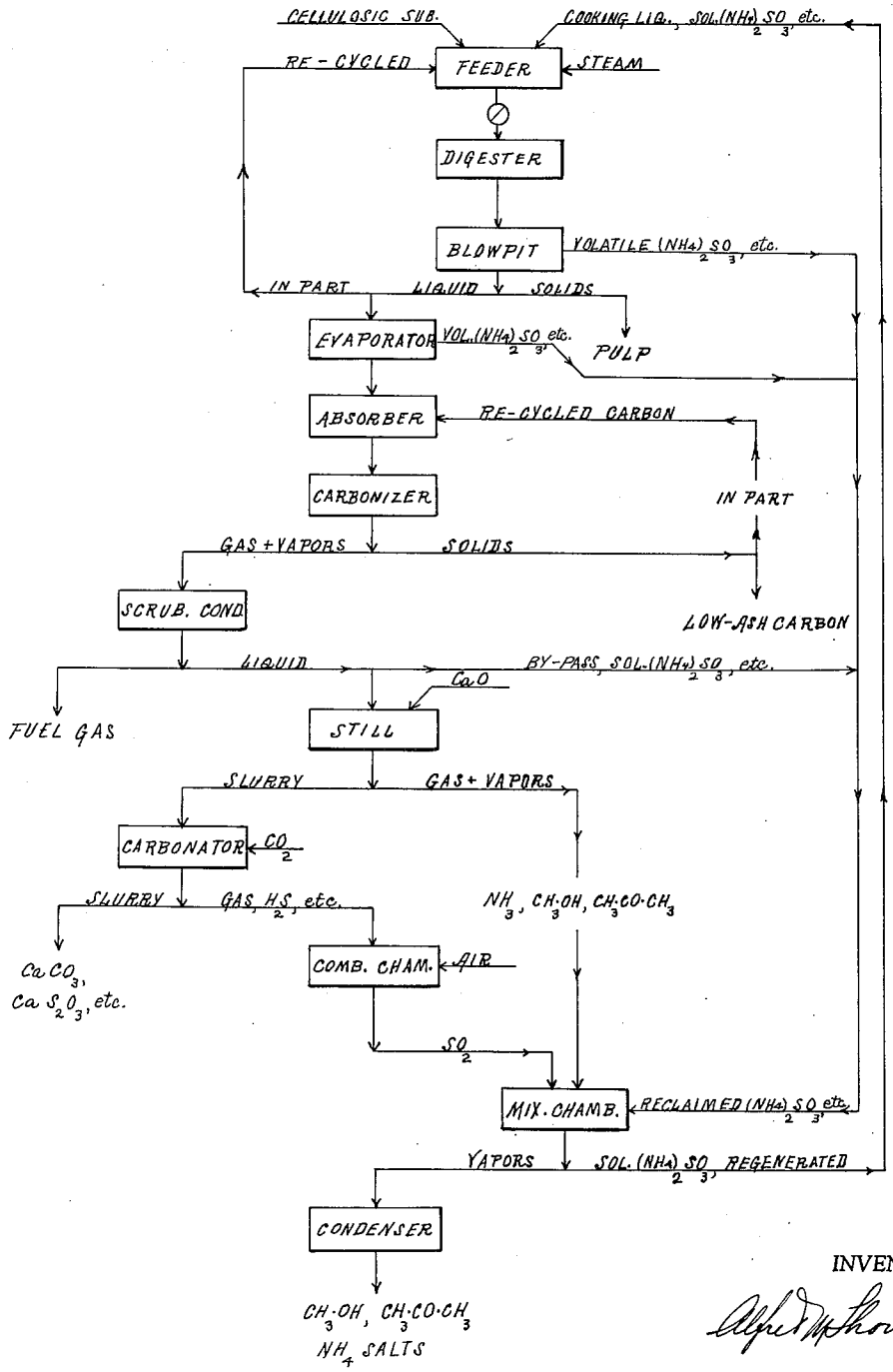
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METHOD OF PULPING CELLULOSIC SUBSTANCES

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METHOD OF PULPING CELLULOSIC SUBSTANCES

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In general, when the term "pulp" is used in connection with cellulose the inference is that the subject discussed is to produce something suited to paper making. Contrariwise, the field in which this process belongs is in the culture of micro-organisms, so the object of my work is to produce a pulp suited to conversion into sugars, the almost universal food for such micro-organisms as have an economic status.

It is also an object of my process to obtain, in some commercial form, various other derivatives of the non-cellulose portion of the cellulosic raw material on which I operate and thus to cheapen the cost of the entire operation. The character of such material is most varied. It could, of course, be high grade wood, but the lowest form of that mixture of bark and saw-dust which is the ultimate end of the combined lumber and paper mill will be equally acceptable. Virtually any collected vegetable "waste," such as sugar cane bagasse, fit only for fuel is however my prime objective. On such "woody" material the cellulose fraction will be more than half, while methanol, acetone and a low-ash carbon will represent the non-cellulose portion.

My pulping agent is the normal ammonium sulphite. I am aware that the conventional "sulphite pulping" with an ammonia base, instead of lime, soda or magnesia, is old and produces an excellent pulp for paper making. This version is, however, entirely different. Presumably this is due to the higher temperature employed, something that in conventional practice would result in a "burned cook." I find it necessary with my sulphite operation to reach a temperature approximately 375° F., which corresponds to a gage pressure of 175 pounds. At this temperature there is no sign of carbonization but a rapid pulping action, but the liberated cellulose has but little strength and is unfit for paper making. This very attack on the fiber itself is a gain in my next step, that of saccharification, shortening the time and thus increasing the yield of sugar. Variations in my process is virtually confined to minor adjustments as to temperature and time, so I will call the steps set forth on the drawing as representing my preferred version. In thus explaining said drawing I will, therefore, have fully described my process and I will give at the same time full instructions for the operation so that any one familiar with the language of pulping can duplicate my work.

At the top of the drawing I have shown a "feeder" which is merely an old-fashioned "acid egg" worked with steam. In this device I commingle the cellulosic material with cooking liquor and with some re-cycled spent cooking liquor. Of course, the raw material must be in a suitable state of division. I employ approximately 40% of the weight of the raw material in ammonium sulphite and then turn on steam. It is of course to advantage that all material be pre-heated. When the pressure in the "egg" exceeds that in the digester the valve between both is opened and the charge in the "egg" is projected into the digester where no extra steam is needed. From the digester the pulped material flows continuously into the blowpit, with attendant evolution of steam, through a "nozzle type" of discharge opening.

The diameter of said nozzle and the volume in the digester are so proportioned that the time of residence in the digester at approximately 175 pounds' gage pressure shall be enough for complete pulping. This depends

largely upon the type of raw material processed, say 2 hours, more or less as may be found advantageous by actual performance. As the charge from the "egg" is in super-heated condition with reference to the digester no steam and no "circulation" is needed or desired in this device.

In the blowpit separation is made between the pulp, which is now ready for saccharification, and the spent liquor. As much of this as possible is re-cycled direct to the "feeder" and the density of the accepted fraction passed on the evaporator is increased thereby. A convenient source of steam for the evaporator is the low-pressure steam continuously evolved from the digester discharge.

After evaporation to a density of approximately 50% solids, this concentrated liquor is absorbed in the pores of a pre-formed spent liquor coke which is, in part, re-cycled from a subsequent "carbonizing" stage. The carbonizer may be an outside heated retort or a Stafford type with inside heating in the shape of circulated, re-heated gas. In either case a mixture of gas and vapors is evolved and coke remains. This coke is, in part, re-cycled to the absorber as previously described and in part constitutes an accepted fraction. It is very low in ash and well suited for many purposes. Most of this ash will, in general, be removed if said coke be washed with a solution of sulphur dioxide in water, and the dissolved calcium bi-sulphite can then be neutralized with ammonia, the calcium separated and the ammonium sulphite added to the cooking liquor to make up for unavoidable losses. It forms a valuable by-product and, of course, avoids any "waste liquor" disposal problem.

The "gas and vapors" leaving the carbonizer is next sent to a "scrubber-condenser," i.e., a vessel in which cooling is effected by a liquid spray, said liquid passing through a cooler before being used once more as a spray. From this device, only a fuel gas should emerge in addition to the re-cycling liquid which, of course, is augmented in volume by the condensed liquids yielded by the gas and vapors from the carbonizer.

Inasmuch as ammonium sulphite is not quite stable at temperatures near boiling, I have shown some recoveries of such volatilized material from both the blowpit and from the evaporator. All such material becomes, ultimately, a part of the regenerated cooking liquor and to this I now add a part of this condensate obtained from said scrubber-condenser. However, as non-volatile ammonium compounds are also formed in the digestion I send a part of said condensate through a still with the addition of enough lime to decompose all such ammonia salts, the resulting "gas and vapors" going to a mixing chamber near the bottom of the drawing. The still thus gives two products, said volatile material and a slurry composed of insoluble calcium salts and a solution of soluble calcium salts.

In the event that said slurry contains enough calcium sulphide to justify the operation I have shown it entering into a carbonator where it is commingled with carbon dioxide, setting free such resident sulphur as hydrogen sulphide. Such gas is, in turn, burned and the resulting sulphur dioxide sent to the mixing chamber. I have then shown said device as yielding the re-cycled mother liquor and some volatilized material which passes to a condenser. The condensed material is chiefly methanol but some acetone is also present, as well as some ammonia salts in solution. The latter is, of course, re-cycled and the former refined for the market. Collectively, they represent a derivative from the methoxyl content of the raw material.

This narrative, in conjunction with the drawing, will enable anyone familiar with conventional pulp operations and such additional matters as ammonia distillation and

charcoal making to operate my process. Subsequent refinements will, of course, only be possible after such a person has had some experience with the specific method herein disclosed.

Having thus fully described my process, I claim:

1. The method of pulping cellulosic material with a cooking liquor that consists essentially of normal ammonium sulphite which comprises; commingling said cellulosic material with a solution of ammonium sulphite and digesting same at the approximate temperature 375° F. until said material shall consist essentially of cellulose fibers and spent cooking liquor; separating said cellulose from said spent cooking liquor; evaporating the cooking liquor and absorbing the concentrated material in a preformed, recycled spent liquor coke; carbonizing the resultant liquor-saturated coke to obtain coke for recycling, coke as an accepted product, and volatile substances; condensing said volatiles to produce liquid and gaseous products; separating said liquid products into a solution of ammonium salts and methoxyl degradation products; and re-cycling said solution of ammonium salts as a regenerated cooking liquor.

2. The method of pulping cellulosic material set forth in claim 1, with the added step that the digestion of said material be performed on a continuous basis, the mixture of cellulose fibers and spent cooking liquor issuing continuously from the confining vessel in which said digestion takes place, essentially in the form of a jet with simultaneous evolution of steam, said steam being thereafter utilized in the evaporation of spent liquor.

3. The method of pulping cellulosic material set forth in claim 1, with the added step that a portion of the condensed liquid products from carbonizing be commingled with lime and heated to decompose resident non-volatile salts of ammonia, the ammonia thus volatilized being afterwards absorbed in and becoming a part of the regenerated cooking liquor.

4. The method of pulping cellulosic material set forth in claim 1, with the added step that the accepted coke-fraction produced therein be further refined by leaching same with a water solution of sulphur dioxide, the lime salts thus solubilized as calcium bi-sulphite being freed from calcium by precipitation with ammonia, the resulting ammonium sulphite being re-cycled as a component of the regenerated cooking liquor.

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