

UNITED STATES PATENT OFFICE.

TENNANT LEE AND ALLAN B. MONKS, OF DEDHAM, MASSACHUSETTS, ASSIGNORS, BY MESNE ASSIGNMENTS, TO ZALMON G. SIMMONS, OF KENOSHA, WISCONSIN, ANDREW W. PRESTON, OF SWAMPSCOTT, MASSACHUSETTS, AND BRADLEY W. PALMER, OF BOSTON, MASSACHUSETTS, TRUSTEES, KNOWN AS SIMMONS SUGAR LIMITED.

PROCESS OF TREATING SUGAR-CANE.

1,153,934.

Specification of Letters Patent.

Patented Sept. 21, 1915.

No Drawing.

Application filed August 26, 1912. Serial No. 717,096.

To all whom it may concern:

Be it known that we, TENNANT LEE and ALLAN B. MONKS, citizens of the United States, residing at Dedham, in the county of Norfolk and State of Massachusetts, have invented certain new and useful Improvements in Processes of Treating Sugar-Cane, of which the following is a specification.

This invention relates to processes of treating sugar-cane and certain products derived therefrom for the purpose of producing wax, paper-pulp, and other valuable products.

It is known that sugar-cane is now treated on a considerable scale by a process which consists essentially in shredding the mature cane in such manner that the fibrous and pithy portions of the tissue are structurally dissociated from each other, and thereafter quickly drying the mass to a condition of chemical stability. The dried product contains not only the sugar of the original cane, but also the waxy and albuminoid matters and the other constituents of the normal plant, and may thereafter be lixiviated by water for the preparation of sugar-solutions. The fibrous and pithy tissues are separated or concentrated with respect to each other at any desired stage of the treatment, as after the drying or immediately before or after the extraction of the sugar. The fibrous concentrate, after the sugar has been extracted, contains most or all of the so-called wax of the cane, and contains likewise those fibers which are chiefly of value for paper-making purposes.

A primary object of the present invention is the provision of a process of treating sugar-cane, and more especially fibrous concentrates thereof, whereby the cane wax may be recovered in a commercially available form, such recovery of the wax being a part of or incident to the preparation of paper-pulp. In this connection it should be understood that the application to this crude fiber of the ordinary soda cook is destructive to the wax, and is also injurious to the fiber, and entails a heavy shrinkage in the yield of stock.

The term "wax" is herein employed to

designate the so-called "cane-wax," a complex body containing in addition to glycerids of certain organic acids, and other organic compounds, a material proportion of myricyl alcohol or its derivatives and other bodies of an alcoholic character.

According to the preferred embodiment of my invention, the fibrous concentrate of the sugar-cane, after lixiviation with cold or hot water for extraction of the sugar, is subjected to a preliminary digestion having for its purpose the removal of gummy and resinous matters and the conditioning of the fiber. This digestion liquor is then drained off and the fiber washed, and thereafter subjected to a second digestion for the purpose of removing the wax and completing the pulping of the fiber. The liquor derived from the preliminary digestion of one batch of fiber is preferably used, after suitable reinforcement, for the preliminary digestion of succeeding batches; and similarly the liquor derived from the second digestion of one batch of fiber may if desired, after reinforcement, be utilized for the second digestion of a second batch or succeeding batches. By proceeding in this manner, there occurs a gradual accumulation of gummy or resinous matters in the liquors from the preliminary digestion, and of wax and certain albuminoid bodies in the liquor from the second digestion. At suitable periods, or if desired after each digestion, the liquors may be treated for recovery of the gummy and resinous matters and the wax, respectively.

The liquors used for the digestions are alkaline in character, and may be initially the same, diverging in composition by repeated use on successive batches of fiber. I have secured the best results by the use of a product which may be designated simply as "diluted cell liquor," and of which the origin and descriptive qualities are as follows: Cell liquor is the cathodic product resulting from the electrolysis of solutions of brine in an electrolytic cell having a porous diaphragm. As thus produced, it is a liquid having a density usually approximating 49° Tw., and contains chiefly caustic

soda, sodium chlorid, usually some sodium sulfate, and small proportions of certain oxy-chlorin compounds, as hypochlorite and chlorate of sodium. This liquor may be
5 diluted with water to about 14° Tw., and is then ready for use in accordance with this invention.

Suitable operating conditions are as follows: The extracted fiber is digested for ten
10 to fourteen hours in a stationary digester, under ten to thirty pounds pressure of steam, with the diluted cell liquor. The liquor is then drained off and the fiber washed with water. The recovered liquor
15 contains the gummy and resinous matters of the plant, and may be used for the preliminary digestion of fresh batches of fiber, after being reinforced by the addition of fresh diluted cell liquor to the extent of
20 about one-third of its volume. It is noteworthy that this reinforced liquor yields a distinctly lighter-colored fiber than does the fresh liquor. The washed fiber, after being conditioned and freed from resinous and
25 gummy materials by the preliminary digestion as above described, is digested with a similarly prepared liquor under somewhat more intense conditions but for a shorter time. Suitable conditions for this second
30 digestion are four to six hours cooking under forty pounds pressure of steam. This liquor is then drained off, and the fiber, after thorough washing, may be beaten by the usual methods for the preparation of pulp.
35 For the best results, however, the fiber should be treated in a ball-mill in the manner disclosed in the copending application of Tennant Lee, Ser. No. 674,732, filed February 1, 1912. The liquor from the second
40 digestion may be reinforced and used for the second digestion of other batches of fiber, whereby the wax, in the form of alkaline compounds or saponification products, is permitted to accumulate therein; or the
45 liquor or any part thereof may be treated after each digestion for the recovery of the wax.

A simple method for recovering the wax is as follows: Liquor from the second
50 digestion is concentrated by boiling to one-third or one-fourth of its volume, and is then allowed to stand at a temperature of 70 to 100° F. for some hours or days, during which time a matter of waxy appearance
55 accumulates at the surface and may be removed by skimming. This material consists largely of alkaline compounds of the constituents of the wax, and may be treated in any suitable manner for the preparation of
60 marketable products. It may for example be dissolved in alcohol, neutralized, and reprecipitated by addition of water. The concentrated liquor from which the wax has been removed may be further treated if de-
65 sired for the recovery of other products,

notably those of an albuminoid character. During the digestion there is a considerable development of ammonia which probably arises from the decomposition of glyccoll or other amid bodies contained in the sugar-
70 cane. There is reason to believe that this ammonia, or the substances which yield it, have a material and favorable effect upon the results of the digestion, particularly in the preparation of light-colored paper pulps.
75 The ammonia may be recovered from the vapors arising from the digester by the usual methods.

While the first digestion conducted as above described is intended chiefly for con-
80 ditioning the fiber and removing therefrom those constituents which are comparatively readily soluble in alkaline solutions, it should nevertheless be understood that it may serve directly and alone for the pro-
85 duction of a pulp ready for beating or for reduction in other ways. For this purpose the conditions of treatment as above defined may be somewhat intensified if de-
90 sired, as by increasing the duration or temperature of the cook or the concentration of the liquor. Such intensification is not how-
95 ever essential. The pulp from this single cooking produces a paper of great strength and of the parchemin type. When the reduction is performed in the ball-mill, papers
100 having a strength in excess of 200 Mullins ratio are readily prepared, such papers being water-resistant, flexible, usually more or less translucent, and stronger or more du-
105 rable after moistening than when dry. Paper prepared by the double cooking process is very strong, as for example 150 Mullins ratio and upward, and possesses less or
110 none of the parchemin-like character, according to the conditions of the cook, being always more or less porous and absorbent unless sized.

While we have given certain specific conditions of operation as the best now known
110 to us, it is to be understood that our invention is not limited to these particular conditions; and it should particularly be understood that in the cooking of the fiber time
115 and temperature and concentration of the liquor are largely compensating factors, and any factor may be rather widely varied, with a compensatory variation of the others.

The invention in its broader aspects is not
120 limited to the use of the particular alkaline liquor herein called "cell liquor," for practical results for certain purposes may be obtained by means of other alkaline solutions, and it may eventually prove practical to
125 prepare the specific solution above described, or a solution capable of giving equally favorable results, by means other than the electrolysis of salt solutions, as for example by directly or indirectly compounding the
130 essential components of the mixture.

While it is always preferable to remove the resinous and gummy constituents from the fiber by a digestion preliminary to that which removes the waxy material, it is yet possible to extract all of these constituents by a single digestion with diluted cell liquor under moderate pressure of steam, as for instance forty pounds, and of sufficient duration, say six to ten hours. The waxy matter may be collected from this liquor by concentration and skimming as before described, and the gummy and resinous matters may then be separated by neutralization. Materially superior results are however obtained by the double digestion practised as above described.

The function of the several constituents of the cell liquor is not at present certainly known, but it may fairly be assumed that the caustic soda is the principal active agent in the solution of the resins and the wax, as well as in the separation of the fibers; that the sodium chlorid is active in bringing about the separation of the waxy matters from the alkaline liquor, in somewhat the same manner as it causes the separation of soaps in the ordinary processes of soap-manufacture. It is probable also that the oxy-compounds of chlorin are active in bringing about the decomposition of certain of the cane constituents, with the result that a lighter color and softer fiber is produced; and they may also be instrumental in the separation of the wax or waxy products.

The separation and recovery of the waxy material from the alkaline liquors may be aided, in certain cases, by the addition thereto of tallow, with the result that soap is formed and salted out, carrying with it the wax, which may be afterward separated by known methods; or soaps may be added directly to the liquor with similar results. Such additions are however usually unnecessary where the digestions have been properly performed and where the wax is present in considerable quantities.

The diluted cell liquor may advantageously be used for the digestion of the pith or pithy concentrates for the preparation of paper stock, but by reason of the less resistant character of the pith cells the digestion should in such case be less prolonged than in the treatment of the fiber, or should be performed under milder conditions as regards concentration or temperature of the liquor. Satisfactory results have been obtained by cooking a concentrate consisting chiefly of pithy tissue in an open vessel for one hour, the concentration of the diluted cell liquor being 12° Tw. Under these conditions the shrinkage during the cooking was approximately twelve per cent. Stock thus prepared yields a paper of the parchment type but of materially less strength than that prepared from the fiber or fibrous

concentrate. The stock derived from the pithy concentrate is also applicable to the production of paper boards or the like and for admixture with sulfite or other stocks.

We claim:

1. In a process of treating sugar-cane, the steps which consist in drying and concentrating the fibrous tissue and digesting the same in a liquor containing as essential constituents caustic soda and sodium chlorid.

2. In a process of treating sugar-cane, the steps which consist in drying and concentrating the fibrous tissue and digesting the same in a liquor containing as essential constituents caustic soda and sodium chlorid, and recovering the wax from the digestion liquor.

3. In a process of treating sugar-cane, the steps which consist in drying and concentrating the fibrous tissue and digesting the same in a liquor containing as essential constituents caustic soda, sodium chlorid, and oxy-chlorin compounds.

4. In a process of treating sugar-cane, the steps which consist in drying and concentrating the fibrous tissue and digesting the same in a liquor containing as essential constituents caustic soda, sodium chlorid, and oxy-chlorin compounds, and recovering the wax from the digestion liquor.

5. The process of treating sugar-cane, which consists in disintegrating the cane, and digesting the fibers in an alkaline cell liquor prepared by electrolyzing a sodium chlorid solution.

6. The process of treating sugar-cane, which consists in disintegrating the cane, drying and concentrating the fibrous tissue, and digesting the same in an alkaline cell liquor prepared by electrolyzing a sodium chlorid solution.

7. The process of treating sugar-cane fiber, after extraction of sugar, which consists in subjecting the fiber to an alkaline digestion under conditions to dissolve the wax, treating the digestion liquor to separate wax therefrom, and recovering the separated wax.

8. The process of treating sugar-cane fiber, after extraction of sugar, which consists in subjecting the fiber to an alkaline digestion under conditions to dissolve the wax, concentrating the digestion liquor to separate wax therefrom, and recovering the separated wax.

9. The process of treating sugar-cane fiber, after extraction of sugar, which consists in removing the resinous and gummy constituents, subjecting the partially purified fiber to an alkaline digestion under conditions to dissolve the wax, treating the digestion liquor to separate wax therefrom, and recovering the separated wax.

10. The process of treating sugar-cane fiber, after extraction of sugar, which con-

sists in removing the resinous and gummy constituents, subjecting the partially purified fiber to an alkaline digestion under conditions to dissolve the wax, concentrating the digestion liquor to separate wax therefrom, and recovering the separated wax.

In testimony whereof, we affix our signatures in presence of two witnesses.

TENNANT LEE.
ALLAN B. MONKS.

Witnesses:

CARL A. RUDQUIST,
OSCAR TOEPEL.