

UNITED STATES PATENT OFFICE.

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METHOD OF COOKING CELLULOSE FIBER.

SPECIFICATION forming part of Letters Patent No. 725,071, dated April 14, 1903.

Application filed March 31, 1902. Serial No. 100,709. (No specimens.)

To all whom it may concern:

Be it known that I, FREDERIC E. GORE, a citizen of the United States, and a resident of Yarmouth, in the county of Cumberland and State of Maine, have invented new and useful Improvements in Methods of Cooking Cellulose Fiber, of which the following is a specification.

My invention relates to the art of making paper or the fiber from which paper or compressed-fiber articles are manufactured; and it consists in an improved method of digesting wood, straw, esparto-grass, jute, bagasse—in short, natural fibrous substances which have cellulose for their basis of constitution. In the manufacture of fiber from wood, which is a characteristic example of the manufacture above indicated, the caustic or soda process heretofore quite generally employed involves the cooking of wood chips in a closed digester with a solution of caustic soda under steam heat and pressure. By this means the cellulose fiber is freed from the organic matter which is associated therewith in the natural wood. At the close of the cooking of a mass of wood fiber the digested pulp is discharged from the digester, washed, screened, bleached, and then proceeds to later stages in the manufacture for which it is intended. All of these practices are well known to those skilled in the art. Their details vary to suit the specific varieties of wood used, with the personal preferences of individual manufacturers, and with the qualities desired in the final product.

Incidental to the caustic process as heretofore practiced has been the initial presence, and perhaps to some extent the production, of sodic carbonate, which at all stages of the process is not only inert so far as concerns the reduction of fiber, but is in many ways a drag and hindrance. Its presence in the digester impedes or clogs the action of the active caustic liquor. It clings to the fiber after the latter has been discharged from the digester and renders the washing of the fiber more difficult, calls for more water and longer time in the pulp-washing, involving great dilution of the soda liquor thus reclaimed, and when

finally carried to the furnaces impairs the burning qualities of the organic residues, so that a large quantity of fuel is required to burn the ash. This troublesome carbonate of soda is present in the originally-prepared soda liquor in amounts varying from ten to twenty-five per cent. of the hydrate of soda actually employed, the cost of hydrate in a chemically-pure form being usually prohibitive. Moreover, as the digesting process progresses a complex of carbon compounds of soda is formed, which when added to the carbonate of soda already present in the liquor increases the difficulties and deterrent effects for which the carbonate of soda is already responsible. Thus in the digesting process as heretofore practiced the products of the process itself limit the efficiency of the liquor in its work of freeing the cellulose fiber from those substances in the wood which the paper manufacturer desires to remove. The time required to cook thoroughly a batch of wood fiber, such as poplar, with the process as heretofore practiced is from eight to nine hours and sometimes has to be continued for ten hours. By the employment of my process, hereinbelow described, I have reduced the time required to cook thoroughly a batch of poplar-wood to two and one-half hours. Moreover, by the employment of this process I am enabled to use less caustic for the cooking, less water for washing the fiber, less fuel for burning the spent liquor, and I obtain more fiber than before from a given quantity of wood.

The gist of my process lies in the employment, continuously and as part of the digesting process, of a substance which has the double effect of reducing the carbonate of soda in the liquor to a caustic condition and of reacting upon a considerable proportion of the carbon compounds of soda which are produced by the digesting process, so that they, too, are restored to a caustic condition. The reaction upon the carbonate of soda, most, if not all, of which is initially present at the beginning of the digesting process, is simple and easy of analysis. The reaction upon the complex carbon compounds of soda is in its

essence obscure, although the net result is clearly to be detected. Both divisions of my process, as above set forth, are characterized, however, by the removal from the soda of
 5 carbon in combination, and I define the process as one of decarbonation of the soda liquor continuously during the digesting process.

In order to carry out my process to its fullest advantage, I make use of an apparatus
 10 such as I will now describe, which embodies an invention which I reserve for claim in an application for patent therefor filed by me concurrently herewith in the United States Patent Office.

15 Referring to the drawing hereto annexed, A represents a wood-digester of the usual form and character. The steam connections and other parts which are incidental to the structure and operation of the digester are
 20 not shown, as they are well known to persons skilled in the art.

B represents a closed decarbonating vessel or tank capable of sustaining the steam-pressure maintained in the digester A.

25 A pipe C leads from the bottom of the digester A to the bottom of the vessel B, and the pipe D, through the pump E, connects the upper part of the vessel B with the upper part of the digester. The pump E is so situated
 30 as to maintain circulation of liquid from the digester A, through pipe C, vessel B, and pipe D, to the top of the digester. Check-valves *c c c* are placed in suitable situations and serve to prevent any accidental reversal
 35 in the above-described circulation, and stop or gate valves *e* furnish means for manually cutting off the communication between the various parts of the apparatus.

In the vessel B there is provided the agitator H, which in this case is shown as a shaft,
 40 journaled at *h'* in the cross-brace *h²* and at *h³* in the stuffing-box *h⁵*. Arms *h⁶*, from which project rods *h⁴*, constitute the active portions of the agitator H, which is driven
 45 from some suitable external source of power, as J.

In the upper part of the vessel B are placed the filters F, two of these being shown in the drawing. Each of these filters consists of an
 50 outer cylindrical shell *f* and an inner cylindrical shell *f³*. Both these shells are perforated, as at *f'*. The head *f⁴* closes the filter at the top. The space between the shells *f* and *f³* is filled with closely-packed finely-com-
 55 minuted filtering material—as, for instance, quartz sand. The bottom plate *f⁵* is perforated or, if desired, consists of a spider-ring.

Brackets G are provided, secured to the shell of the decarbonating vessel B, and serve
 60 both as seats for the filters F and as conduits through which the circulation to the pipe D is maintained. The brackets G are cast with swells *g'*, wherein are located the ducts *g*,
 65 which register with perforations in the shell of the vessel B and the short pipes *d*, which are secured therein. The pipe *d'*, which extends around the vessel B, communicates with

all the short pipes *d* and at *d²* connects with the pump E. A manhole at *b* is provided, whereby the filters F may be inserted and
 70 removed and the other contents of the vessel B introduced thereto. At *b'* is situated a drain-cock for the removal of the liquid contents of the decarbonating vessel B.

The operation of this apparatus, which involves the practice of my new and improved
 75 process in one of its modes, is as follows: A charge of wood chips (or other material suitable to the manufacture of fiber) is introduced into the digester A in the ordinary
 80 manner. Fresh caustic liquor is then run in. This may be in the proportionate quantity usually employed, or, if the economies made possible by the practice of my invention are
 85 to be availed of to their full extent, less than the usual quantity of caustic liquor may be used. The ordinary caustic-soda liquor contains a considerable percentage of carbonate
 90 of soda, which remains practically unchanged during the digesting process as heretofore carried on, so that only the remainder of the caustic liquor is practically active; but by
 the employment of my process this percentage of carbonate is causticized—*i. e.*, transformed into hydrate—during the progress of
 95 the cooking, and thus becomes active. The quantity of caustic liquor first introduced may therefore be less than the usual amount in proportion to the percentage of carbonate contained and less also by an amount measured
 100 by the quantity of spent or partially-spent liquor, which is ascertained by previous experiment to be restored to its active condition after having formed the complex carbon compounds with soda, which are necessary
 105 products of the digesting process. I have found by practice of my new process that with a given weight of poplar chips for my raw material I can reduce the quantity of fresh caustic from thirty to forty per cent.
 110 below the quantity heretofore usually employed for digestion of the wood, and yet the time required for complete reduction of the wood to fiber will be reduced to one-quarter to one-third of the time required with the old
 115 digesting process. I effect this reduction of the quantity of caustic by employing liquor reclaimed from a previous cooking to dilute the fresh caustic in the digester and also to fill the vessel B when the latter is charged.
 120 Into the vessel B, I then charge a quantity of decarbonating material, such as hydrate of lime, in a finely-divided condition in an amount which may be calculated beforehand as requisite to the reduction of the carbonate
 125 of soda present at the initial stages of the process plus the other carbon compounds of soda which may be expected to develop as an incident to the cooking of the wood.

I have found in the practice of my process
 130 that for ten thousand two hundred pounds of bone-dry poplar chips, which require two thousand gallons of fresh eighty-per-cent. caustic at 1.075 specific gravity diluted with

fifteen hundred to eighteen hundred gallons of liquor recovered from a previous cooking and which is decarbonated to begin with, six hundred pounds of hydrate of lime, dry, is a quantity sufficient for the purpose.

The filters F are put in place upon the brackets G before the vessel B is charged with liquor and hydrate of lime. Then the digester A and the vessel B are closed, all valves *e* are opened, and steam is admitted to the digester in the usual manner and the cooking begins. As the pressure and temperature rise in the digester the pump E is started and the liquid contents of the closed system, which comprises the digester A, vessel B, and their pipe connections, are circulated from the bottom of the digester through the pipe C, through the vessel B, and consequently through and into reactive association with the mass of decarbonating materials, such as hydrate of lime, which is contained in the vessel B, into and through the filters F, which confine all solid matter to the vessel B, through the ducts *g*, pipes *d* and *d'*, pump E, and pipe D, back to the digester. Meanwhile power is applied to the agitator H at J, so that the circulating caustic liquor is intimately mingled with the decarbonating material, such as hydrate of lime, in the vessel B. The results of this continuous process of decarbonating the caustic liquor during the progress of the cooking of fiber are from beginning to end highly beneficial and their benefits extend to the subsequent stages and incident processes of the manufacture.

At the start, assuming that the vessel B has been charged with spent liquor and hydrate of lime or such other decarbonating material as the manufacturer may discover to be suited to his purpose, the decarbonating material reacts upon the spent liquor, converting the carbonate of soda which may be present into hydrate and separating from the soda a considerable part of the organic matter which is combined therewith, so that the entire body of liquor is placed in the most active condition possible before the circulation through the closed system causes it to pass again into the digester. As the liquor in the digester after acting upon the wood passes into the vessel B from the pipe C, charged with compounds formed by the soda with organic matter in the wood and with carbonate of soda which was either initially present or has been incidentally formed, the decarbonating material reacts upon this partially-spent liquor, converting the simple carbonates and a considerable portion of the more complex carbon compounds of soda into caustic. Thus the carbonate of soda which was present in the fresh caustic wherewith the digester was charged is hydrated and the liquor enriched in proportionate measure, while the complex carbon compounds formed during the digesting process are in large part broken down and the soda restored in pro-

portionate measure to active condition. The circulation is kept up during the entire cooking process. The decarbonating material continuously converts or reconverts the carbonates to hydrates and to a partial extent also separates the soda from the complex carbon compounds which have been formed in the digester and restores the liquor to the fiber in the digester, where it renews its action on the fiber with vigor unimpaired by the deterrent presence of simple carbonates and with the similar deterrent effect of the complex carbon compounds of soda very materially reduced.

The rate at which the circulation shall be maintained may be varied to suit the needs and notions of individual manufacturers. I have found by practice that the substantial freedom of soda liquor from carbonate and from those complex carbon compounds which are susceptible of being reacted upon by decarbonating material is properly maintained by a circulation which will entirely change the contents of the digester five times during the cooking process.

It will be found that the time required for complete reduction of the wood or other fiber to the condition of pulp when this my new process is employed may be reduced to one-quarter of the time which has been heretofore necessary with the old process, and this, too, when the quantity of caustic initially introduced into the apparatus is less than that heretofore employed. Under manufacturing conditions which demand a large production in a given time my process therefore enables the pulp-maker to run his digesters at four times their former capacity, provided he uses caustic liquor of fairly-high strength. If, however, it becomes comparatively more profitable to economize in caustic liquor rather than in time, the manufacturer is enabled by my process to use much weaker liquor than heretofore, even weaker than the liquor which I have used, as above set forth, or to dilute his fresh caustic liquor with a large quantity of spent liquor from previous cooking, for the reason that the continuation of the decarbonating process during and as part of the cooking process sustains the active efficiency of the caustic liquor, which is left free to work upon the organic matter in the fiber substantially unhampered by the presence of deterrent carbonates and other more complex carbon compounds.

By the conversion to hydrate, as above described, the carbonate of soda, whether initially introduced as carbonate or formed as an incident to the cooking process, not only becomes a useful factor in the cooking process, but by its conversion also removes elements of difficulty heretofore present as incidents to subsequent stages of the preparation of fiber. Carbonate of soda does not wash from the cooked fiber as readily as hydrate of soda, and therefore the fiber discharged from the digester after the old process required

a large quantity of water and much time for the removal of carbonate from the fiber. With my new process, on the contrary, the carbonate of soda is recovered by hydration
 5 during the progress of the cooking and substantially no carbonate is found in the fiber discharged from the digester. Consequently less time and less water are required for washing the fiber and the liquor obtained from
 10 the washing is of much higher density and causticity than heretofore, so that the usual subsequent process of recovery of soda from the liquor is thereby greatly assisted.

By my new method of decarbonating while
 15 cooking and also by the reuse of a large quantity of black spent soda liquor (which my process renders practicable) the percentage of soda which combines with combustible organic material is very much increased, and
 20 therefore the part of the liquor which subsequently goes to the evaporators and furnaces is much richer in fuel properties than the residual liquors obtained by the old process. This, coupled with the fact that there is little
 25 or no soda in the residual liquor in combination with carbonic acid, enables the liquor to be burned with less fuel, in less time, and with less apparatus than has heretofore been possible. By my process also not only is the
 30 manufacturer enabled to obtain fiber with less caustic and in less time than heretofore, but he is also enabled to obtain more and better fiber from a given quantity of wood or other fibrous material than heretofore, be-
 35 cause the fiber being subjected to the action of caustic for a shorter time or to the action of weaker caustic if the time is not shortened is not depleted by the destruction of the smaller fiber-cells so much as heretofore.
 40 The resulting fiber is also stronger than that obtained from the old process, other conditions being equal.

The filtration which I have described above as part of my process will be found desirable
 45 wherever the fiber is to be used for the manufacture of the finer grades of paper. The process may be carried on without filtration, thereby allowing the hydrate of lime to be circulated along with the caustic liquors dur-
 50 ing the cooking. Thereafter the lime may be washed out of the fiber or retained as a filler or make-weight wherever the presence of the lime will not be detrimental to the uses for which the fiber is intended—for in-
 55 stance, in case of the manufacture of fiber for wrapping-paper, bag-paper, or sheathing-paper.

What I claim, and desire to secure by Letters Patent, is—

60 1. The method of cooking fiber which consists in digesting the fiber with caustic liquor, meanwhile transferring the liquor from the fiber into reactive contact with decarbonating material, and then returning the liquor
 65 to the fiber, thereby sustaining the active efficiency of the liquor during the cooking process.

2. The method of cooking fiber which consists in digesting the fiber with caustic liquor diluted with spent liquor, meanwhile
 70 transferring the dilute liquor from the fiber into reactive contact with decarbonating material, and then returning it to the fiber, thereby sustaining the active efficiency of the entire liquor during the cooking process. 75

3. The method of cooking fiber which consists in digesting the fiber with caustic-soda liquor, meanwhile transferring the liquor from the fiber into reactive contact with hy-
 80 drate of lime, and then returning the liquor to the fiber, thereby sustaining the active efficiency of the soda liquor during the cooking process.

4. The method of cooking fiber which consists in digesting the fiber with caustic-soda
 85 liquor diluted with spent-soda liquor, meanwhile transferring the dilute liquor from the fiber into reactive contact with hydrate of lime, and then returning it to the fiber, thereby sustaining the active efficiency of the liq-
 90 uor during the cooking process.

5. The method of cooking fiber which consists in digesting the fiber with caustic liquor, meanwhile transferring the liquor from the fiber into reactive contact with decarbon-
 95 ating material, agitating the liquor and decarbonating material and returning the liquor to the fiber, thereby sustaining the active efficiency of the liquor during the cooking process. 100

6. The method of cooking fiber which consists in digesting the fiber with caustic-soda liquor meanwhile transferring the liquor into
 105 reactive contact with hydrate of lime, agitating the liquor and hydrate of lime, and returning the soda liquor to the fiber, thereby sustaining the active efficiency of the soda liquor during the cooking process.

7. The method of cooking fiber which consists in digesting the fiber with caustic liq-
 110 uor, meanwhile transferring the liquor from the fiber into reactive contact with decarbonating material, then filtering the liquor and returning it to the fiber, thereby sustaining the active efficiency of the liquor during the
 115 cooking process, and excluding solid matter contained in the liquor from the fiber.

8. The method of cooking fiber which consists in digesting the fiber with caustic-soda
 120 liquor, meanwhile transferring the liquor from the fiber into reactive contact with hydrate of lime, then filtering the liquor and returning it to the fiber, thereby sustaining the active efficiency of the soda liquor during the
 125 cooking process, and excluding from the fiber solid matter contained in the liquor.

9. The method of cooking fiber which consists in digesting the fiber with caustic liquor, meanwhile transferring the liquor from the fiber into reactive contact with decarbon-
 130 ating material, agitating the liquor with the decarbonating material, then filtering the liquor and returning it to the fiber, thereby sustaining the active efficiency of the liquor dur-

ing the cooking process, and excluding from the fiber solid matter contained in the liquor.

10. The method of cooking fiber which consists in digesting the fiber with caustic-soda liquor meanwhile transferring the liquor from the fiber into reactive contact with hydrate of lime, agitating the liquor with the hydrate of lime, then filtering the liquor and returning it to the fiber, thereby sustaining the active efficiency of the liquor during the cook-

ing process, and excluding from the fiber solid matter contained in the liquor.

Signed by me at Yarmouthville, Cumberland county, Maine, this 25th day of March, 1902.

FREDERIC E. GORE.

Witnesses:

GEO. W. HAMMOND,
ALEX. H. TWOMBLY.