Patented Jan. 9, 1940 2,186,034

UNITED STATES PATENT OFFICE

2,186,034

PULP BLEACHING AND REFINING PROCESS

Harold R. Murdock, Canton, N. C., assignor to The Champion Paper and Fibre Company, Canton, N. C., a corporation of Ohio

No Drawing. Application August 24, 1937, Serial No. 160,720

4 Claims. (Cl. 92—16)

This invention relates to an improved process of refining and bleaching fibrous material, and is concerned particularly with the preparation of a refined and bleached paper pulp in a form adapted for direct use in the manufacture of paper, fiber board, and like manufactured products.

In the manufacture of bleached pulp from raw fibrous materials, such as, for example; wood and the like, it is usually the practice to digest the raw material with a suitable chemical in such quantities and for such periods of time and at such elevated temperatures and pressures that practically complete solution of the interfibrous material is effected.

The raw fibrous material used in the production of such chemical pulps rarely is uniform in chemical and physical properties. As a consequence, the interfibrous material in various parts of the raw material usually is dissolved at different rates. According to the desired character of the pulp to be produced, the digestion treatment may be, and usually is, continued until even the more resistant portions of the raw material are substantially and thoroughly digested—in which case the less resistant portions of the raw material suffer an excessive digestion with a consequent low yield. In the increasingly common practice of developing pulps of high strength characteristics, however, the said treatment is discontinued when all but the more resistant portions of the raw materials have yielded to the solvent action of the digestive agent. Particularly in the latter case, the resulting pulp contains bundles of fibers which do not disintegrate into separate fibers under the subsequent mild mechanical treatment commonly given pulps preliminary to bleaching. Before chemical wood pulps can be converted into high quality paper or the like, however, it is absolutely necessary that each individual fiber be liberated from other fibers adjoining it in the cellular structure. Therefore in order to make unusually strong white pulps it is not only essential that the digestion be "on the raw side" but that all the fibers be liberated. This seems to be a paradoxical condition but nevertheless must be realized. Consequently, somewhere in the process must be interposed a step for liberating individual fibers from the fiber bundles.

It has been common practice to complete this liberation of individual fibers at a point following the steps of bleaching or whitening of the brown pulp, and just prior to the formation of the bleached pulp into a paper web. This refining treatment is usually effected by means such as, for example, the beater, the jordan, and the refiner. In addition to bringing about fiber liberation, this same refining treatment coincidentally results in that physical processing of the fibers which is classed as "hydration," and which is well known to be a requisite to the formation of a web of paper.

In the usual series of operations which has just been described, the step of bleaching precedes that of final fiber liberation, and consequently the unbleached pulp when treated with the customary bleaching agents contains fiber bundles, slivers, or shives. These fiber bundles are not readily and uniformly penetrated by the usual bleaching agents, and a satisfactory evenness in bleaching is not obtained. If the major portion of the pulp is correctly bleached, then the fiber bundles are insufficiently bleached and appear in the white pulp as yellowish slivers or discolorations. On the other hand, if the bleaching treatment is made sufficiently drastic to remove the color from these fiber bundles, the major portion of the pulp will have been excessively bleached and the resultant bleached pulp will possess relatively low strength characteristics.

It recently has been recognized that the so called refining treatment can be done prior to the bleaching process with the advantage that the fiber bundles are separated into individual fibers before bleaching so that a more uniform bleaching operation thereby is made possible. This process was disclosed in Helder's U. S. Patent No. 1,890,179, wherein the step of mechanical refining is interposed between the digestion step and the first step involving bleaching or refining by chemical means. While this process makes possible some improvement over the prior art process, it does not fully realize the benefits which are possible by the applicant's process.

The applicant's process represents a further departure from, and improvement over, prior art processes. It makes recognition of two points familiar to those well versed in the art, and more particularly so since the development and perfection in recent years of chlorination steps in the bleaching process: Namely that bleaching is essentially delignification in the early stages and oxidizing or whitening in the later stages, and that little or no fiber deterioration occurs in the earlier or delignification stages. I have found that certain definite benefits are achieved by placing the step of mechanical refining in that sequence of bleaching operations near the end of the delignification stage, yet preliminary to

oxidation or whitening to a high degree. This mechanical refining may be done after the first hypochlorite stage in processes where hypochlorite treatment follows treatments by chlorine, gaseous or liquid, and by alkaline solutions. However, I may, and preferably do, perform the refining step at a point following treatments with chlorine, gaseous or liquid, and with alkaline solutions, but preliminary to treatment with hypochlorite or like oxidizing material.

By breaking up the fiber bundles before oxidation is brought about, the process set forth in this invention achieves a desired evenness in bleaching and attains high degrees of whiteness with treatments less drastic than hitherto considered necessary. Economy of bleaching agent is a coincidental advantage. The novelty of this invention, however, lies in an improvement in the means of attaining these known benefits. In the delignification steps, particularly chlorination and the subsequent alkaline treatment, the interfibrous material remaining after digestion is softened to a considerable degree. Fiber liberation then can be accomplished without undue power consumption and without undue physical degradation of the fibers, such as often results from the drastic treatment necessary for refractory, highly lignified fiber bundles. As further advantages, this process is suitable for processing pulp which is digested more "on the raw side" than usual, and which consequently has high strength and yield characteristics.

An object of the present invention is to produce, in an economical manner, a final refined and bleached pulp product having uniform whiteness. Another object of the invention is the provision of a refining and bleaching process wherein fiber degradation is minimized, so that the product thereof has improved properties as contrasted with prior art processes. Another object is to provide a process in which an economy of bleaching agent may be effected over those processes heretofore employed. A further object of the invention is to produce a refined and bleached pulp of higher yield, based on the equipment used, than those available in the earlier processes.

I have found that the above, and other, objects are effected by the carrying out of the following operations in the sequence named:

(1) Digesting the wood (or its equivalent) in a solution of the usual digestion chemicals—preferably terminating the digestion while the product is still on the "raw side";

(2) Washing the digestion chemicals and spent cooking liquor from the resulting pulp; preferably followed by the screening of the washed pulp;

(3) Treating the pulp with chlorine (either chlorine gas or chlorine water) in an amount sufficient to chlorinate the lignin content of the pulp;

(4) Treating the pulp with mild caustic alkali solution under conditions and in an amount sufficient to solubilize the chlorinated compounds content thereof;

(5) Washing soluble ingredients from the pulp;

(6) As an optional step which may be omitted, treating the pulp with hypochlorite solution in an amount insufficient to cause appreciable whitening or oxidation;

(7) Refining and hydrating the pulp to a degree just short of that desired in the manufacture of the required paper product from the pulp;

(8) Bleaching the partially refined and hydrated pulp with hypochlorite solution to the desired standard of whiteness;

(9) Finally hydrating the bleached pulp, in a much milder manner than at stage (7) above, to the exact degree of hydration appropriate to form the pulp into the desired paper product.

By this process it is possible to effect a better yield, of a stronger and otherwise superior pulp of an arbitrary standard of whiteness, with simultaneous improvement in economy of chemicals.

In accordance with this process, wood is prepared and digested in a manner common to any of the conventional chemical pulping processes. I need not, and preferably do not, carry the digestion to the point at which substantially all of the fiber bundles have been opened up, but rather may and preferably do stop the digestion while the pulp is still slightly undercooked or "on the raw side". The digestion mixture, after discharge from the digester, and after or coincident with separation of the pulp from the liquids content of the mixture, may if desired be passed through the conventional equipment for the removal of knots, large portions of undercooked wood, dirt and the like. While this latter step usually is desirable it may not be necessary in all cases.

The pulp at this stage is distinctly brown in color, and contains a relatively large content of lignin or lignous compounds. For the safe removal of lignin or lignous compounds I treat the pulp with gaseous chlorine or with chlorine water in an amount sufficient to react with (i. e., chlorinate) the lignin content of the crude pulp. In carrying out the chlorination I prefer for economical reasons to react the chlorine with the pulp while the latter is in a relatively concentrated state (i. e., a "high-density" pulp), and to use only enough chlorine to effect the aforesaid chlorination of the lignin compounds content of the pulp.

The product of the chlorination step is a decidedly brown pulp—darker in color than was the pulp at the time of its discharge from the digester—due to the brown color of the chlorinated lignin compounds. These latter are not soluble in water as to be removable from the pulp by washing. Consequently, I solubilize the same by a treatment of the pulp with a suitable amount of dilute caustic alkali (e. g., caustic soda) solution, with agitation of the pulp and solution to effect satisfactorily thorough permeation of the pulp with the base. Thereafter, the so-treated pulp is thoroughly washed with water to free the pulp from soluble ingredients.

If desired, the pulp at this point may be subjected to treatment with hypochlorite (e. g., calcium hypochlorite) bleaching powder solution or suspension, in an amount insufficient to cause appreciable oxidation or whitening. This treatment may be made at room temperature or at elevated temperature, and the pulp thereafter may be washed with water. It will be recognized that little or no fiber deterioration through oxidation has occurred up to this point. However, this step may be, and preferably is, omitted at this point in the sequence of operations.

Thereupon, the pulp is subjected to refining and beating treatment to an extent just short of that degree of refining and hydration desired for the manufacture of the eventual paper or paper product. For this purpose the pulp may be refined and hydrated, for example, in a jordan, whereby fiber bundles are opened up and reduced

to ultimate fibers and the cellulosic fibers are somewhat hydrated.

This partially, or nearly completely, refined and hydrated pulp is then subjected to an oxidizing bleach by treatment thereof with a hypochlorite (e. g., calcium hypochlorite) or bleaching powder solution or suspension, at room temperature or at elevated temperature, and thereafter may be washed with water. Because of the homogeneity and openness of the pulp at this stage, the above oxidizing bleach step requires materially less of the chemical or chemicals than that amount heretofore considered necessary to attain comparable whiteness of the product.

The so treated and bleached pulp is finally given a very mild beating in a jordan or equivalent hydrating machine, whereby to bring the already substantially refined and hydrated pulp to the exact degree of hydration indicated for best results in formation of the desired paper web, after which step the pulp may be used directly in a paper-web-forming operation.

I claim:

1. Process of preparing a bleached paper stuff, which comprises chlorinating the chlorinatable lignin compounds content of a chemically produced raw pulp by treatment of the latter with chlorine as such; solubilizing the chlorinated lignin compounds content by treating the pulp with mild caustic alkali solution and washing the so-solubilized compounds from the pulp; beating and partially hydrating the pulp to a degree just short of that required for hydrating the pulp to a degree approximating that required for paper-making; subjecting the substantially refined and hydrated pulp to an oxidizing bleach with an hypochlorite under conditions to bleach the pulp; and mechanically hydrating the bleached pulp, to the degree of hydration appropriate for paper-making, under mild hydration conditions.

2. Process of preparing a bleached paper stuff, which comprises chlorinating the chlorinatable lignin compounds content of a chemically produced raw pulp by treatment of the latter with chlorine as such; solubilizing the chlorinated lignin compounds content by treating the pulp with mild caustic alkali solution and washing the so-solubilized compounds from the pulp; treating the pulp with hypochlorite solution in amount insufficient to cause appreciable bleaching; beating and partially hydrating the pulp to a degree just short of that required for paper-making; subjecting the substantially refined and hydrated pulp to an oxidizing bleach with an hypochlorite under conditions to bleach the pulp; and mechanically hydrating the bleached pulp, to the degree of hydration appropriate for paper-making, under mild hydration conditions.

3. In the process of preparing a bleached pulp product suitable for direct use in web-forming, the sequence of operations which essentially consists of (*a*) digesting wood by a chemical pulping process, (*b*) separating liquids from the resulting raw pulp containing chlorinatable lignin compounds and screening said pulp, (*c*) chlorinating the chlorinatable lignin compounds content of the raw pulp by treatment of the latter with chlorine as such, (*d*) treating the product of step (*c*) with caustic soda solution in an amount sufficient to solubilize its content of chlorinated compounds, and washing the pulp free of the soluble components, (*e*) partially refining and hydrating the product of step (*d*) to a degree just short of the desired hydration for paper-making, (*f*) bleaching the substantially refined and hydrated pulp product of step (*e*) with hypochlorite bleach to standard whiteness, and (*g*) mildly mechanically hydrating the bleached product of step (*f*).

4. In the process of preparing a bleached pulp product suitable for direct use in paper-making, the sequence of operations which essentially consists of (*a*) digesting wood by a chemical pulping process, (*b*) separating liquids from the resulting raw pulp and screening the latter, (*c*) chlorinating the chlorinatable lignin compounds content of the raw pulp by treatment of the latter with chlorine as such, (*d*) treating the product of step (*c*) with caustic soda solution in an amount sufficient to solubilize its content of chlorinated compounds, and washing the pulp free of the soluble components, (*e*) treating the product of step (d) with an amount of hypochlorite insufficient to produce appreciable oxidation or whitening, (*f*) partially refining and hydrating the product of step (*e*) to a degree just short of the desired hydration for paper-making, (*g*) bleaching the substantially refined and hydrated pulp product of step (*f*) with hypochlorite bleach to standard whiteness, and (*h*) mildly mechanically hydrating the bleached product of step (*g*).

HAROLD R. MURDOCK.

CERTIFICATE OF CORRECTION.

January 9, 1940.

Patent No. 2,186,034.

HAROLD R. MURDOCK.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 3, first column, lines 33 and 34, claim 1, strike out the words "hydrating the pulp to a degree approximating that required for"; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 13th day of February, A. D. 1940.

Henry Van Arsdale,
Acting Commissioner of Patents.

(Seal)