UNITED STATES PATENT OFFICE.

JOHN A. JUST, OF NEW YORK, AND FRANK A. FLETCHER AND CALLIE F. GIBBS, OF WATERTOWN, N. Y.; SAID JUST AND GIBBS ASSIGNORS TO SAID FLETCHER.

PROCESS OF BLEACHING MECHANICAL WOOD PULP AND OTHER RAW VEGETABLE FIBERS FOR THE PURPOSE OF MANUFACTURING PAPER.

SPECIFICATION forming part of Letters Patent No. 348,165, dated August 24, 1886.

Application filed October 9, 1885. Serial No. 179,430. (No specimens.)

To all whom it may concern:

Be it known that we, JOHN A. JUST, a citizen of Germany, and a resident of the city, county, and State of New York, and FRANK A. FLETCHER and CALLIE F. GIBBS, both citizens of the United States, and residents of Watertown, in the county of Jefferson and State of New York, have invented a new and Improved Process of Bleaching Mechanically-Prepared Wood Pulp and other Raw Vegetable Fibers; and we do hereby declare the following to be a full and exact description thereof.

Our invention consists in an improved process of bleaching wood fibers which have been reduced to pulp by mechanical means, and other raw vegetable fibers, for the purpose of manufacturing paper therefrom. Hitherto these fibers have not been extensively used for this purpose, both because of previous inability to bleach them and on account of their hardness and brittleness. By our improved process the fibers are quickly and economically bleached without materially injuring the most delicate fibers, and at the same time are rendered soft and pliable. In the processes hitherto tried, and in which heating or boiling has been resorted to, chlorine, which is always contained in the bleaching agent, is left in solution in the water in such quantities as to render the atmosphere very injurious to the health of the workmen and others who chance to inhale it, on account of the irrespirable character of chlorine gas. Substances containing chlorine are also used in our process; but since it is conducted without any heating or boiling all possible escape of chlorine is prevented, and no injurious effects result from its practice.

The old and well-known process which has hitherto been used in endeavoring to bleach wood pulp consists in preparing oxygen and applying the same, the oxygen being evolved by heating a metallic oxide in a clear solution of chloride of lime. This process, however, fails to bleach raw vegetable fibers and mechanically-prepared wood pulp, although it does bleach all chemically-isolated fibers, such as unbleached chemically-prepared wood pulp, rags, cotton, and wastes of the same.

It has long been known that chloride of lime or water saturated with chlorine not only refuses to bleach mechanically-separated wood or vegetable fibers, but, on the contrary, produces intense colors; and strong acids turn mechanically-prepared wood pulp or raw vegetable fibers red in the sunlight, and often without sunlight in the presence of moisture, and the slightest trace of iron contained in any of the materials or vehicles employed in the bleaching, as in the water or lime, is sufficient to blacken the entire mass in a very short time.

The object of our invention is to bleach to a pure white mechanically-prepared wood pulp and other raw vegetable fibers, and to obtain the same in a soft and pliable condition and without the application of heat. Our improved process achieves this result in a practical, feasible, economical, and efficient manner.

In carrying out our process the usual beater-engine of the paper-mill is preferably employed, and water is introduced into it in sufficient quantity to carry out the process—say enough to fill the engine about half full. An emulsion is then introduced into the water in the engine, consisting of a mineral oil and a basic alkali or alkaline salt or solution thereof. These latter principles are at the same time preferably oxidizing agents, and may therefore be termed "alkaline oxidizing agents." The mineral oil employed is preferably petroleum or other oil obtained from the distillation of coal, and the alkaline oxidizing agent is preferably chloride of lime, or caustic lime and chlorine solution. Proper proportions of the ingredients of the emulsion are as follows: Four (4) pounds of petroleum and six (6) pounds of chloride of lime to each one hundred (100) pounds, dry weight, of the wood pulp or other fiber. After the emulsion has been added to the water and diluted thereby the wood pulp or other fiber is then introduced into the engine. The entire mass is then thoroughly agitated until the emulsion is brought into uniform and intimate contact with the fibers throughout. The oily nature of the oil having disappeared in the preparation of the emulsion, the emulsion acts as a favorable

solvent of the resinous and gummy materials which incrust the raw fibers in small quantities, and as the operation advances it distinctly betrays its action on the fibers by assuming a slight but evident alcoholic and acetic odor. The wood pulp on becoming saturated by this solution loses its hardness and woody characteristics and becomes soft and pliable equally, as in the case of chemically-isolated fibers or rag-stock. With the aid of chlorine or any oxidizing or deoxidizing agent present at this stage of the process, the coloring-matter existing in the sap substances of the wood stuff is fully brought out, and is then bleached pure white by the following treatment: The wood pulp under treatment being now saturated with the emulsion dilute in water, three to four (3 to 4) pounds of common salt (sodium chloride) in solution are added to each one hundred (100) pounds, dry weight, of the pulp or fiber. This solution being capable of dissolving a large quantity of chlorine gas, thus prevents any possible escape of the same, which otherwise would expand and escape into the air, it being set free by the warmth which is evolved by the process of bleaching. Thus not only is no chlorine lost, but injurious effects to the workmen are avoided, since the chlorine is not mechanically suspended in the mass, but is held dissolved in the dilute salt solution, and from that is abstracted as fast as required, to oxidize in the process of bleaching. To accelerate the process of bleaching, a small quantity of acid, either vegetable or mineral—such as oxalic acid, for example—may be added, thus neutralizing alkaline principles favorable to oxidation. In bleaching some fibers—such as flax and jute, for example—the acid will be found necessary, but with the wood pulp is not absolutely essential, although preferable. The proportion of acid which is properly used is three-quarters of a pound to each one hundred pounds of the fiber.

Instead of using an acid for the purpose of accelerating the process, a small quantity of sulphate of zinc—say two (2) to three (3) pounds to each one hundred pounds of the pulp—may be advantageously substituted as an equivalent thereof. The sulphate of zinc acts very energetically to decompose the chlorides, and thus liberate the chlorine, and if the water originally introduced into the engine at the beginning of the process be warm (say from forty (40°) to fifty (50°) centigrade) on the completion of the process the pulp is left in so nearly a neutral state that little or no anti-chlorine is required, since there is at the conclusion little or no excess of chlorine.

The next stage in the process consists in the introduction into the mass of chloride of lime, chlorine gas, or water saturated with chlorine gas, together with a natron salt; or a potassium salt may be substituted for the natron salt. The carbonate or sulphate of one of these bases is to be preferred. The proper proportions of these ingredients to each one hundred pounds of pulp or fiber would be between thirty (30) and forty (40) pounds of the chloride, depending on its strength, and between twelve (12) and fifteen (15) pounds of the alkali carbonate or sulphate. Upon the incorporation of these substances with the mass by agitation in the beater-engine the wood pulp or other fiber is bleached permanently and perfectly white, owing to the decomposition of the hypochlorites formed with the above-mentioned bases. After the bleaching is completed any excess of chlorine is removed by an anti-chlorine substance—such as sodium hyposulphite or sulphurous-acid solution, for example—and the product is washed, and is then ready for being made into paper.

In case sulphate of zinc has been used to accelerate the process, there will usually be no necessity for the use of anti-chlorine, and hence the washing may be dispensed with, since washing is only essential to prevent brownish tints being given to the pulp by the anti-chlorines.

In carrying out our invention, in practice we employ a beating-engine which will manipulate about five hundred pounds of the pulp or fiber. The steps of the process carried out with the preferred ingredients are then as follows: First, the emulsion composed of petroleum and chloride of lime is introduced; second, common salt is added; third, an acid or sulphate of zinc is introduced; fourth, chloride of lime and carbonate of soda are introduced. After the introduction of each ingredient the entire mass is thoroughly agitated, so as to subject every portion of the mass to the action of the substances used.

We claim as our invention—

1. In the process of bleaching wood pulp and other fibers, treating the pulp or fiber, for the purpose of softening the same, with an emulsion composed of a mineral oil and a basic alkali, or alkaline salt or solution thereof, substantially as set forth.

2. In the process of bleaching wood pulp and other fibers, treating the pulp or fiber, for the purpose of softening the same, with an emulsion composed of a mineral oil and an alkaline oxidizing agent, substantially as set forth.

3. In the process of bleaching wood pulp and other fibers in which chlorine gas is evolved, introducing a solution of common salt, substantially as set forth, whereby the free chlorine is dissolved and its escape into the air prevented.

4. In the process of bleaching wood pulp and other fibers which have by previous stages been reduced to a soft and pliable condition, treating the same with an oxidizing agent, together with an alkali basic salt, substantially as set forth.

5. The process of bleaching mechanically-prepared wood pulp and other raw vegetable fibers, consisting of the following steps: First, treating the pulp or fibers with an emulsion composed of a mineral oil and an alkaline oxidizing agent; second, adding a solution of com-

mon salt, and, third, treating the pulp or fibers with an oxidizing or bleaching agent together with a basic alkali salt, or solution thereof, substantially as set forth.

6. The process of bleaching mechanically-prepared wood pulp and other raw vegetable fibers, which consists of the following steps: First, treating the pulp or fiber with an emulsion composed of a mineral oil and an alkaline oxidizing agent; second, adding common salt; third, adding an acid or its specified equivalent, and, fourth, treating the resulting product with an oxidizing or bleaching agent, together with a basic alkali salt, or solution thereof, substantially as set forth.

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

JOHN A. JUST.
FRANK A. FLETCHER.
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Witnesses:
GEO. H. WALKER,
C. L. ADAMS.