UNITED STATES PATENT OFFICE.

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PROCESS OF REFINING WOOD-PULP FOR NITRATING OR SIMILAR PURPOSES.

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To all whom it may concern:

Be it known that I, VIGGO DREWSEN, a citizen of the United States, and resident of the borough of Brooklyn, county of Kings, city and State of New York, have made a certain new and useful Invention Relating to Processes of Refining Wood-Pulp for Nitrating or Similar Purposes, of which the following is a specification.

This invention relates especially to the refining or purification of wood pulp produced from pine, spruce, or other suitable wood by the caustic soda or sulfate processes so as to secure a refined pulp of low cautic potash solubility with a minimum loss of the original wood pulp material, and of such color and other properties as makes it suitable for use in producing nitrated cellulose of various grades or other generally similar cellulose acetate compounds. The original soda or sulfate wood pulp is preferably partially bleached with bleaching powder, or still better with chlorin water so as to considerably change some of the colored and other compounds, thus giving the wood pulp a grayish brown or yellowish cast or tinge. This changed material is then preferably removed as by a cooking process with suitable alkaline refining reagent, such, for example, as sodium carbonate or caustic which renders these materials soluble and removes them in the cooking liquor which is highly colored. The pulp has after this treatment a considerably reduced caustic potash solubility and is sufficiently light in color to be used without serious objection for some nitrating or similar processes. If desired, however, the treated pulp may be given a second bleaching action with a relatively small proportion of bleaching powder solution or the like which still further improves its color without undesirably increasing its caustic potash solubility.

Caustic soda pulp and sulfate pulp formed from pine, spruce, hemlock or other similar wood has in many cases undesirably great caustic potash solubility for use in making nitro-cellulose, and in some instances the color of the material is too dark for use in this connection. The colored impurities or components are furthermore undesirable because combining with the nitric acid or other reagents used so as to seriously interfere with their continued action in such processes. By submitting such ordinary or unbleached wood pulp to a special refining process its caustic potash solubility may be considerably reduced and its color and other properties greatly improved for use in the production of nitro-cellulose or the like; by partially bleaching the pulp, and then dissolving or removing therefrom the reduced or changed compounds. For this purpose such unbleached chemical wood pulp may advantageously be partially bleached by the action of a relatively small proportion of bleaching material such as by use of five to ten per cent., for instance, of ordinary bleaching powder or calcium or sodium hypochlorite, the pulp being agitated with a cold water solution thereof for a number of hours. If desired in this connection the bleaching action may be hastened and improved from some standpoints by the use of carbonic acid gas during the bleaching process and incorporated by the usual agitator with the material, or sulfuric acid may be added during the bleaching action which develops more or less free chlorin. Better results can ordinarily be secured, however, by a preliminary partial bleaching of the pulp with strong chlorin water in such amounts as to contain from two to eight per cent. by weight of chlorin more or less, as compared to the dry weight of the original unbleached pulp to be treated thereby, the mixture being agitated in a suitable tank or bleaching apparatus for a couple of hours. This preliminary partial bleaching changes the color of the pulp from the ordinary brownish cast to a decidedly yellow color, the coloring matter being apparently chemically changed although not completely decolorized and the free chlorin being practically absorbed or removed from the solution when five per cent. of chlorin is used in this way.

The partially bleached pulp is then subjected to an alkaline refining treatment as by treating it for a suitable time in a hot water solution of sodium carbonate or sodium hydrate or other similar alkaline material capable of dissolving or removing from the pulp the transformed or partially bleached coloring matter and other similar

products formed during the preliminary bleaching. Sodium carbonate seems to give quite desirable results and some ten to fifteen per cent. of sodium carbonate as compared to the dry weight of the original wood pulp may be used in strong water solution to cook the partially bleached pulp under pressure for a couple of hours in a digester at, say, 60 to 100 or so pounds pressure per square inch. This refining action dissolves out the coloring matter and other transformed compounds which render the alkaline liquor a dark or coffee brown color and this liquor is drained off and the pulp is washed in the usual way until clean, after which it may be dried and used in some cases for nitro-cellulose and similar purposes where the usual somewhat grayish cast or tinge of the pulp is not objectionable. If desired, however, this refined pulp may be still further bleached or improved in color by subjecting it to a secondary light or partial bleaching action. For this purpose the usual water solution of bleaching powder, such as calcium or sodium hypochlorite may be used and only two or three per cent. or less of bleaching powder as compared to the dry weight of the pulp is necessary to secure sufficient bleaching action to give the desired light or whitish color to the pulp. This secondary bleaching action is performed in the usual way, the pulp being stirred with the bleach solution for an hour or so and subsequently washed thoroughly and dried when it can be used with advantage for production of nitro-cellulose or cellulose acetate manufacture.

The wood pulp which has been refined by this general method has a relatively low caustic potash solubility when tested in the usual way by cooking for three hours under atmospheric pressure in a reflux condenser with a ten per cent. water solution of caustic potash. In this test the cooked pulp is acidified with acetic acid, then washed and weighed and the reduction in weight of the dry pulp is the amount taken as the caustic potash solubility. This partial bleaching and subsequent alkaline refining of wood pulp of this character is sufficient in many cases to reduce its caustic potash solubility to four to six per cent. so that it is possible to get below the limit of seven per cent. caustic potash solubility which is the present standard for cotton used for nitro-cellulose manufacture. Furthermore, wood pulp treated in this way loses only about the same proportion of its weight as when a full or complete chlorin bleaching of the pulp is effected in the usual way which has the decided disadvantage of making some fifteen to twenty-five per cent. of the bleached pulp soluble according to the standard caustic potash test.

This invention has been described in connection with a number of illustrative processes, orders of steps, materials, proportions, arrangements, and methods of treatment and use, to the details of which disclosure the invention is not of course to be limited, since what is claimed as new and what is desired to be secured by Letters Patent is:

1. The process of refining pine, spruce or generally similar soda or sulfate process wood pulp to render it suitable for nitro-cellulose manufacture or the like, which comprises partially bleaching such ordinary unbleached wood pulp with chlorin water containing from two to five per cent. of chlorin as compared to the dry weight of the pulp to produce partially bleached yellow pulp, subjecting the same to an alkaline refining treatment by boiling under pressure for several hours in a solution to dissolve and remove coloring matter and converted products from the pulp and subjecting the resulting pulp to a supplemental weak bleaching process to decolorize the pulp and at the same time keep down its caustic potash solubility below seven per cent.

2. The process of refining chemical wood pulp to render it suitable for nitro-cellulose manufacture or the like, which comprises partially bleaching such ordinary unbleached wood pulp to produce partially bleached pulp, subjecting the same to a strong alkaline refining treatment to dissolve and remove coloring matter or converted products from the pulp and subjecting the resulting pulp to a supplemental weak bleaching process to decolorize the pulp and at the same time keep down its caustic potash solubility to not more than about seven per cent.

3. The process of refining pine, spruce or similar soda or sulfate process wood pulp to render it suitable for nitro-cellulose manufacture or the like, which comprises partially bleaching such unbleached wood pulp for several hours with chlorin water containing from two or eight per cent. of chlorin as compared to the dry weight of the pulp to produce partially bleached yellow pulp and subjecting the same to an alkaline refining treatment by boiling under pressure for several hours in a sodium carbonate solution to dissolve and remove coloring matter and converted products from the pulp and at the same time reduce its caustic potash solubility considerably below seven per cent.

4. The process of refining chemical wood pulp to render it suitable for nitro-cellulose manufacture or the like, which comprises partially bleaching such unbleached wood pulp to produce partially bleached colored pulp and subjecting the same to an alkaline refining treatment by boiling under pressure in alkaline solution to dissolve and remove coloring matter and converted products from

the pulp and at the same time reduce its caustic potash solubility considerably below seven per cent.

5. The process of refining chemical wood pulp to render it suitable for nitro-cellulose manufacture or the like, which comprises partially bleaching such wood pulp to produce partially bleached colored pulp and subjecting the same to a strong alkaline refining treatment to remove coloring matter and converted products from the pulp and at the same time reduce its caustic potash solubility to not more than about seven per cent.

6. The process of refining soda or sulfate process wood pulp for nitro-cellulose manufacture or the like, which comprises partially bleaching such ordinary wood pulp with about one-half or less the usual proportion of bleaching material and subjecting the partially bleached somewhat colored pulp to an alkaline refining treatment by boiling under pressure for several hours in a sodium carbonate water solution to remove from the pulp coloring matter and other converted products and reduce the caustic potash solubility of the pulp to less than seven per cent.

7. The process of refining soda process wood pulp for nitro-cellulose manufacture or the like, which comprises partially bleaching such wood pulp with much less than the full proportion of bleaching material and subjecting the partially bleached somewhat colored pulp to a strong alkaline refining treatment to remove from the pulp coloring matter and other converted products and reduce the caustic potash solubility of the pulp to not more than about seven per cent.

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