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PROCESSING OF PULP AND PRODUCT OF SAME

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This invention relates to a processing of pulp, being more particularly addressed to a processing intended to remove resinous color-generating substance from the pulp, and to produce a product having a minimum content of such substance. The resinous color-generating substance includes a variety of components, such as waxes, fats, and oils, so that the term "resin" as used herein also comprehends these components.

While not limited thereto, our invention finds important utility in producing a high alpha cellulose wood pulp intended for use as a cotton fiber substitute in the manufacture of high grade papers and derivatives. Such pulp, which for convenience of designation will hereinafter be termed "alpha pulp," is prepared by refining wood pulp, for example, sulphite, by a series of chemical treatments effective in removing non-alpha cellulose components therefrom. For instance, in refining wood pulp by a process which includes a treatment with a lignin-reactive reagent, followed by alkaline digestion and then by bleaching, as described in our patent application, Serial No. 185,019, filed April 19, 1927, substantially all the ligneous content of the pulp, together with other non-alpha cellulose components may be removed therefrom, particularly when the pulp has been well cooked in its liberation. Such refinement is hence very desirable, as lignin also tends to impart or to generate color in derivatives prepared from pulp. Even after such refinement, however, sufficient residual resin remains associated with the pulp to impart some color to or to impair the stability of derivatives and paper prepared therefrom. It is highly desirable that this residual resin be largely removed from the fiber to permit most effective use of the pulp as a substitute for cotton fiber in the manufacture of high grade paper and in the preparation of products such as synthetic silk, lacquers, films, and kindred products where purity, color, and stability of the cellulose employed as a raw material are characteristics of prime importance.

We have found that if wood pulp is digested in a saponaceous reagent, substantially all the resinous impurities may be removed therefrom. While we do not know precisely the effect of soap on such impurities, we believe that the resins are dispersed or colloidally dissolved from the fiber by the soap solution, so that they may be subsequently washed out with the reagent. Such saponaceous digestion may be practiced quite expediently and economically in a refining process such as described, by adding soap to the alkaline liquor and/or to the bleaching liquor, as no additional tanks are necessary and no additional power or time is consumed in the processing of the pulp. Preferably, deresinification is effected during alkaline digestion, particularly hot-digestion, as substantially all the resins may be removed under such conditions. Furthermore, soap-forming material may be added to produce soap in situ in the alkaline-digesting tanks. During digestion, therefore, a removal of substantially all the resinous material in the pulp may be effected along with the removal of lignin, less-resistant celluloses, and other non-alpha cellulose components.

The pulp refining process may, for instance, be carried out substantially as follows. Raw pulp produced by cooking wood chips in a suitable liquor, such, for example, as the usual calcium bisulphite liquor, may preliminarily be treated with a lignin-reactive reagent such as chlorine water, containing about 2% to 4% chlorine, based on fiber. The chlorinated pulp is then digested for five to sir hours at a consistency of about 10% and at about 212° F. in an alkaline solution containing about 8% caustic soda and about 3% soap, based on fiber. In lieu of using soap in prepared form, a saponifiable oil or fat may be used together with sufficient caustic soda in excess of 8%, based on fiber, to saponify such oil or fat. After alkaline digestion, the liquor is dark brown, containing the less-resistant celluloses, ligneous matter, and resinous impurities dissolved from the pulp. The spent liquor of digestion is washed from the pulp, preferably with warm water, and the washed pulp is then bleached at, say, 125° F. in bleach liquor containing 1% to 3% calcium hypochlorite and about

¾% caustic soda, based on fiber. The resulting pulp is of high alpha cellulose content and low copper number, and being substantially free from ligneous matter and exceedingly low in resin content, possesses a bright, snow-white color. Thus, whereas the resin content of the original sulphite pulp, as determined by extraction with organic solvents such as alcohol, ether, or chloroform, usually is about 0.5% to 0.8%, and of a pulp refined without soap-digestion about 0.2% to 0.4%, the content of such extractable matter in the resulting pulp is less than 0.1%. The elimination of resin from the pulp is reflected in the colorlessness of solutions of derivatives resulting therefrom. Thus, whereas a pulp refined without soap-digestion yields nitrocellulose lacquers of a straw-color, similar lacquers prepared from soap-digested, refined pulp are practically colorless. Rayon prepared from soap-treated stock is unusually free from coloring matter before the final bleach, and may in many instances be directly dyed even when delicate hues are sought.

The deresinification of pulp may also be effected while it is undergoing refinement by cold alkaline digestion in solutions of high alkalinity either below mercerizing strength or at mercerizing strength, as described in patent application, Serial No. 77,624, filed December 24, 1925, by George A. Richter. Saponaceous reagents may be used to great advantage with kraft, sulphate-sulphite, soda pulps, and so on, as well as with sulphite, and is of particular importance when highly resinous woods such as jack-pine are being processed, enabling high-grade pulps to be prepared from wood which would ordinarily be rejected because of its high content of troublesome resin.

Deresinification of a pulp may also be effected to great advantage while it is undergoing digestion at elevated temperature in a weakly alkaline liquor containing, say, 1% caustic soda, based on fiber, and intended more especially to develop softness of fiber rather than to increase alpha cellulose content to any great extent. For instance, wood fiber such as sulphite pulp generally yields hard, rattly papers which may be greatly improved if the pulp is digested in weakly alkaline solutions. Not only does such digestion soften the fiber, but subsequent bleaching with a moderate amount of bleach enables the attainment of a bright white color even superior to that realized if the original pulp were treated with an excessive amount of bleach. When such digestion is attempted without the use of soap, however, pitch-like specks appear in the digested pulp, utterly spoiling its otherwise snow-white character and tending to gum and clog wire-covered washing cylinders and thickeners, and doing similar mischief to papermaking machinery. It is an interesting fact that these specks are generated only when about 1% to 2% caustic soda, based on fiber, is employed in alkaline digestion, and are absent when the caustic soda usage is raised to about 6% to 8%, or greater, as in pulp refining procedure such as previously described. It is believed that the pitch-like specks generated in the stock are composed of resinous substances of varying composition, and that under the influence of heat and the motion of the fibers during digestion these substances coalesce or agglomerate to form the highly objectionable specks. When stronger alkali is used, however, it is believed that sufficient resinous impurity is dissolved to leave the residual impurities hard and with little or no tendency to agglomerate. Whether this hypothesis or picture is correct or not, it is a fact that when 1% or 2% NaOH is used in digestion, black, unsightly pitch specks are abundantly in evidence, and that when 6% or 8% NaOH is used, there are no pitch specks. By adding soap to the weakly alkaline liquor during a fiber-softening digestion such as described, the resins are evidently dispersed and are subsequently washed from the fiber, thus preventing the formation of specks and permitting the improvement of the pulp for papermaking. Soap-digestion also assists the alkali in softening the fiber and by its detergent effect upon the smaller specks usually present in pulp enhances its cleanliness and bleachability.

As stated, it is believed that the soap strips the resins from the fibers by its dispersing action, this seemingly being borne out by a simple experiment. Thus, if the dark brown, resinous residuum extracted from unbleached pulp by an organic solvent is collected and boiled up with a 1% NaOH solution, the solution assumes a deep yellow color and becomes very turbid. This reaction to produce turbidity doubtless corresponds with the formation of pitchy specks when pulp is digested in a weakly alkaline solution, as previously described. If, now, a few drops of soap solution are added to the turbid solution, it clears up, rapidly acquiring a sparkling go'len brown appearance. Evidently the soap has dispersed the suspended particles of pitch or resin.

In all the examples hereinbefore given, soap may also be added to the bleach liquor or may be added to the bleach liquor only, but where such procedure is followed it is preferable to use sodium base bleach, as calcium base bleach tends to form insoluble soaps with attendant fixation of such soaps on the fiber. Furthermore, soap may advantageously be added to bleach liquor to effect deresinification when pulp is being bleached without having previously been refined by alkaline digestion or other treatment. While our invention has been described chiefly in connection with alkaline digestion or with bleach-

ing, it is to be understood that it may be practiced as a separate step. Although this would entail the use of extra tanks, washers, and so forth, it is desirable in some cases to separate the treatments as in the preparation of a sulphite pulp low in resin content where it is inexpedient to change the usual calcium hypochlorite bleach to a soda-base bleach.

Various soaps may be used in the process of our invention, such, for example, as the straight chain, fatty-acid soaps typified by sodium oleate, sodium stearate, or sodium palmitate, or the ring-structure soaps typified by sodium resinate or the sodium soaps of the sulphonated oils.

Soap-forming substance such as palm oil, cotton seed oil, coconut oil, peanut oil, castor oil, or the like, may be employed when soap is to be produced in situ during alkaline digestion. The glycerine liberated by the saponification of the oil is frequently of assistance in the dispersion of the resinous matter, and, in some instances the glycerine content may advantageously be increased above that corresponding to the amount of soap involved. Along with these soaps or soap-forming substances may also be added peptizing agents such as sodium phosphate and borax, which are water-soluble or inorganic suspensoids such as talc and colloidal clay.

Our invention thus makes possible the removal of resinous material from wood pulp to an extent heretofore never realized, and when practiced in a refining process as in the examples hereinbefore given, makes possible the production of an alpha pulp which yields cellulose derivatives as free from coloring matter as those prepared from high grade cotton or rag fibers.

Having thus described our invention, it is evident to those skilled in the art that various changes might be resorted to without departing from the spirit or scope of invention as set forth in the appended claims.

We claim:

1. A process step which comprises digesting chemical wood pulp in raw condition in a saponaceous reagent.

2. A process step which comprises digesting chemical wood pulp in raw condition in a saponaceous reagent containing a peptizing agent.

3. A process step which comprises digesting chemical wood pulp in raw condition in an alkaline liquor containing soap.

4. A process step which comprises digesting chemical wood pulp in raw condition at elevated temperature in an alkaline liquor containing sufficient soap to effect a substantially complete deresinification of such pulp.

5. A pulp-refining process which comprises digesting chemical wood pulp in raw condition in an alkaline liquor containing soap, and bleaching the digested pulp.

6. A pulp-refining process which comprises digesting wood pulp in an alkaline liquor containing free soap, and bleaching the digested pulp in bleach liquor containing free soap.

7. A pulp-refining process which comprises digesting wood pulp in an alkaline liquor containing soap, and bleaching the digested pulp in sodium bleach liquor containing soap.

8. A pulp refining process which comprises chlorinating wood pulp, and then digesting the chlorinated pulp in an alkaline liquor containing soap.

9. A pulp-refining process which comprises treating wood pulp with chlorine water, then digesting such treated pulp in an alkaline liquor containing soap, and bleaching the digested pulp.

10. A pulp-treating step which comprises digesting chemical wood pulp in raw condition in a weakly alkaline liquor containing sufficient soap to prevent the generation of pitch-like specks in such pulp.

11. A pulp-treating process which comprises digesting chemical wood pulp in raw condition at elevated temperature in a weakly alkaline liquor containing sufficient soap to disperse the resin content of such pulp, and bleaching the resulting pulp.

12. A process step which comprises digesting chemical wood pulp in raw condition at elevated temperature in an alkaline liquor containing soap-forming substances and of sufficient alkalinity to saponify such soap-forming substances and effect a refinement of such pulp to a high alpha cellulose content.

13. A pulp-refining process which comprises treating sulphite pulp with chlorine water containing 2% to 4% chlorine, based on fiber, digesting the pulp in an alkaline solution containing about 3% soap, based on fiber, and bleaching the digested pulp.

14. A pulp-refining process which comprises treating sulphite pulp with chlorine water containing 2% to 4% chlorine based on fiber, digesting the pulp at about 212° F. for about six hours in an alkaline solution containing about 8% caustic soda and 3% soap based on fiber, and bleaching the digested pulp in bleach liquor containing 1% to 3% bleach and about ¾% caustic soda based on fiber.

15. A process step which comprises digesting pulp in a saponaceous reagent containing glycerine.

16. A process which comprises treating wood pulp with an oxidant, digesting in an alkaline liquor containing soap, and bleaching.

17. A process which comprises treating wood pulp with an oxidant, digesting in an alkaline liquor and during the digestion adding material saponifiable by the alkali to form soap, and bleaching.

18. A process which comprises treating

wood pulp in raw condition with an oxidant, digesting at elevated temperature in an alkaline liquor and during digestion adding fatty acid glycerides to form soap, and bleaching.

19. A white, wood pulp of high alpha cellulose content, containing less than 0.1% resin as determined by extraction with an organic solvent, and convertible into nitrocellulose solutions of a colorlessness substantially equal to similar solutions prepared from high grade cotton.

In testimony whereof we have affixed our signatures.

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