This invention relates to an improved process for the purification of cotton linters. After the long staple fibers, which are used in the textile industry, are removed from cotton seeds by the ginning operation there remain clinging to the seeds shorter fibers which may in turn be separated by passing the seeds between a series of knives having very close clearance. The fibers thus removed, known by the term linters, are mainly cellulose and when freed from foreign impurities, such as seed hull particles, oils, waxes, and grease, serve as a basic raw material in the manufacture of many cellulose derivatives and products prepared therefrom, i.e., lacquers, rayon, plastics, and smokeless powder.

The principal method used for the purification of cotton linters is by digestion of the same with solutions of caustic soda, generally under pressure.

This invention has as an object an improvement in the penetrative power of dilute caustic soda solutions into crude cotton linters. Another object of this invention is an improvement in the efficiency of the digestion process by means of which cotton linters are purified from non-cellulosic materials such as seed hull particles, natural oils, waxes, and grease. A further object of this invention is an improvement in the quality of linters cellulose in respect to color, both in the unbleached and bleached condition, and general purity.

These objects are accomplished by digesting the crude cotton linters in dilute caustic soda solution containing agents of the type of the sodium salts of sulfuric acid esters of saturated aliphatic alcohols having 6-18 carbon atoms or of sodium salts of saturated mono-carboxylic acids having 6-18 carbon atoms.

In the practice of my invention crude cotton linters are digested with 5-10 parts of a 1-5% (preferably 2%) solution of caustic soda containing between 0.05 and 1.0% (preferably 0.25 to 0.50%) based on the dry weight of the linters) of one or more agents taken from groups comprising either sodium salts of sulfuric acid esters of saturated aliphatic alcohols having 6-18 carbon atoms or sodium salts of saturated aliphatic carboxylic acids having 6-18 carbon atoms. With either class of agents, mixtures of compounds are found to be more effective than individual compounds and in general also the efficiency of these agents in dilute caustic solutions is greater for higher molecular weight substances. Other digestion conditions, temperature and pressure, etc., may be varied at will depending on the purpose for which the purified cellulose is to be used, viscosity desired in the cellulose, etc.

The following examples are illustrative of the method of practicing my invention:

**Example 1**

Five hundred grams of crude second cut cotton linters were placed in an autoclave and covered by 5000 grams of a 2% caustic soda solution containing 2.5 grams of sulfite of normal 10 dodecyl alcohol. It was noted at this point that the crude linters were wet almost instantly by the caustic solution although a caustic solution of similar strength containing no wetting agent wet linters only after several minutes contact. The autoclave thus charged was closed and heated to 150° C. and maintained at this temperature for six hours, the pressure throughout being about 80 pounds per square inch. At the end of this period the autoclave was cooled, emptied, and the digested cotton washed free of alkali black liquor. The cotton was next bleached in a 3% alurite using 0.4% (based on dry weight of cellulose) of chlorine in the form of calcium hypochlorite. The resulting bleached product was 25 possessed of a high white color which was definitely superior to that obtained from a control experiment in which the cotton linters were digested under similar conditions except for the wetting agent and were then bleached with 0.5% 30 chlorine.

In the above example the active wetting agent is the sodium salt of the half sulfuric acid ester of dodecyl alcohol. In place of the dodecyl alcohol salt I may use with similar results the corresponding sodium salt of the sulfates of other saturated aliphatic alcohols of 6 to 18 carbon atoms. These alcohols may be either primary or secondary and may have either straight or branched carbon chains. Furthermore, I may use in place of the dodecyl alcohol of Example I the so-called "technical lauryl alcohol" which may be obtained by the carboxylic hydrogenation of an essentially saturated natural oil such as coconut oil, or palm kernel oil, or by the hydrogenation of the acids obtained by the hydrolysis of these oils. This hydrogenation product may be further treated if desired, for example, by distillation in order to separate the more desirable alcohol fractions. One alcohol mixture which is particularly desirable for use in this invention is obtained by the hydrogenation of coconut oil and contains substantially all the alcohols corresponding in...
chain length to the C6, C8 and C10 acids, and about half the C12 and C14 acids present in the oil.

Another desirable source of saturated aliphatic alcohols suitable for sulfation and use as a wetting agent is obtained as a by-product in the catalytic synthesis of methanol from carbon monoxide and hydrogen. A satisfactory mixture of such alcohols boils above about 160° C. and contains a mixture of primary and secondary alcohols having both straight and branched carbon chains and consisting predominantly of 8 to 16 carbon atom alcohols.

Example II

In another experiment a mixture of the sodium salts of saturated aliphatic mono-carboxylic acids containing 6-8 carbon atoms was substituted for the sulfate of normal dodecyl alcohol used in Example I with other conditions of digestion and bleaching remaining the same. In this case the rate of wetting out of the linters by the caustic solution was slightly less rapid than noted with the caustic containing the sulfate of normal dodecyl alcohol, but this rate was still much faster than that obtained in the absence of any wetting agent. As in the case of the first example, the bleached product was superior in color to the control although approximately 15% less bleach was actually consumed than was the case with the control.

Instead of the mixture of salts of the 6 to 8 carbon atom acids of Example II, I may use with similar results sodium salts of other saturated aliphatic monobasic acids within the range of 6 to 18 carbon atoms, including the straight chain acids ranging in chain length from caproic acid to stearic acid, that is, from 6 to 18 carbon atoms, also saturated aliphatic monobasic acids having branched chains of 6 to 18 carbon atoms. These salts may be used separately but preferably are used in mixtures containing salts of several acids within the specified range. Desirable mixtures of acids for this purpose may be obtained by the hydrolysis of essentially saturated natural oil such as coconut oil.

Satisfactory mixtures of acids for the purpose of this invention may also be obtained by the oxidation of those mixtures of alcohols obtained as by-products in the catalytic synthesis of methanol from carbon monoxide and hydrogen. Thus, a mixture of primary saturated aliphatic alcohols containing both straight and branched carbon chains of 8 to 16 carbon atoms and boiling above 160° C. may be separated from the by-product obtained in methanol synthesis. This alcohol mixture may be oxidized by known methods to a mixture containing the corresponding acids. The oxidation must be effected by the two-step oxidation of alcohol to acid passing through aldehyde, or by the process disclosed in Patent 1,856,283.

Instead of digestion under pressure as outlined above, the crude linters may be digested under atmospheric pressure conditions with caustic solutions containing agents of the kind mentioned above. Very high viscosity linters suitable for conversion to nitrocellulose to be used in dynamite manufacture may be produced in this way. The linters so digested are considerably lighter in color and freer from hull particles than are dynamite linters prepared without the aid of the above mentioned agents.

The addition of one or more of the foregoing agents to a caustic solution to be used for linters digestion results in rapid wetting of the linters by the caustic so that digestion starts uniformly throughout the whole mass. Unbleached linters purified by means of the process of this invention are, furthermore, of better color, freer of hull particles and other impurities than are linters purified by digestion with caustic soda alone. Bleaching of such linters is easier than normal with an equivalent high white color obtained by the use of less than the normal quantity of bleach, so that under some conditions 20-25% bleach may be saved in this way.

On conversion to cellulose derivatives, bleached linters caustic digested according to the present invention are found to produce products, i. e., nitrates, acetates, xanthates, etc., of superior quality, being freer of haze producing material and of better color, etc., than normal.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that I do not limit myself to the specific embodiment thereof except as defined in the appended claim.

I claim:

A process for purifying cotton linters which comprises digesting the linters under heat and pressure in dilute caustic soda solution containing a sodium salt of a sulfuric acid ester of a saturated aliphatic alcohol containing 6 to 18 carbon atoms.

E. J. BOLTON.