This invention relates to a process for treating cellulose and particularly to a process for changing its activity towards dyestuffs and for altering other properties thereof. It also relates to products resulting from such treatment.

Esterification of cellulose and reprecipitated cellulose (hydro-cellulose) with higher fatty acids has been heretofore described.

Generally heretofore esterification of cellulose and hydro-cellulose was carried out with acid chlorides in the presence of organic bases such as pyridine or quinoline while heating to a greater or less degree whereby highly esterified products soluble in organic solvents were produced. Cf. British Patent 201,510 of September 6, 1922; H. Gault and P. Ehrmann (Bl. Soc. Chim. 1923, 1285); P. Karrer and Zorka Zega (Helv. Chim. 5, 833 (1922), 9, 622 (1923); Grün and Wittka (Zt. f. angew. Ch. 34, 645 (1921); British Patents 283,181 and 284,288; G. Kita and collaborators (Kunstoffe 16, 41 (1926)).

All these experiments and processes deal with the production of esters from loose cotton, cellulose, cellulose dust, paper, and the like, and result mostly in amorphous products devoid of structure.

It has been found in accordance with the present invention that changes in vegetable yarns and fabrics of all kinds can be produced while preserving their fibrous structure by superficial esterification thereof by means of higher fatty acids.

An object of the present invention accordingly is to provide a simple, efficient process for carrying on such surface esterification and to produce a series of products having their activity towards substantive dyestuffs effectively altered along with alterations in other properties such as their appearance and feel.

Further objects and advantages of the invention will more fully appear from the following description and the preferred methods of carrying out the invention.

Vegetable yarns and fabrics of all kinds are superficially esterified by means of higher fatty acids, such as caprylic, capric, lauric, palmitic, stearic, oleic, and similar acids, as well as mixtures of such acids. By this process one obtains a yarn or fabric which has a wooly soft texture, and which, while possessing an undiminished or hardly altered strength has excellent immunity to substantive dyes and is difficult to moisten and repels water. These valuable properties constitute a hitherto unknown novel technical effect which was not to be foreseen on the basis of previous knowledge.

The process of producing such effects comprises acting on a vegetable yarn or fabric which has previously been immersed in aqueous sodium or potassium caustic solution or in alkaline caustic solutions, after expression of the excess caustic solution, with the chloride (or bromide) of one of the aforesaid acids in an indifferent solvent such as carbon tetrachloride, chloroform, nitrobenzol, benzol, etc. The reaction starts even at room temperature and may, if desired, be accelerated by heating. The quantity of the acid chloride as used may vary within wide limits according to the effect that is to be obtained, and may for example, amount to 0.5 mol. or 2 mol. or 3 mol. to 1 mol. of C₆H₅0₂Cl.

In addition to yarns and woven materials, all other kinds of structures for example knitted goods, embroidery, and the like composed of or including material containing cellulose may be enabled in this manner. By the expression “material containing cellulose”, in accordance with the usual usage in this art, is included regenerated cellulose or other form of artificial silk.

Examples of procedure

1. 16 parts by weight of cotton cloth are immersed for 2 hours in a 20-percent aqueous caustic soda solution, are then pressed free of excess caustic solution, and are introduced into carbon tetrachloride in which there has previously been dissolved 28 parts by weight of the acid chloride mixture obtained by treatment of commercial stearin with thionyl chloride and which therefore consists principally of the chlorides of palmitic and stearic acids. The cotton fabric remains for some time, for example 1 hour...
in the carbon tetrachloride solution, which is expediently heated to 35-55° C. The fabric that has been treated in this manner possesses, with undiminished strength, a fatted feel, has a soft woolly texture and is difficult to moisten with water. It is relatively immune to substantive dyestuffs.

2. 32 parts of cotton yarn are immersed for 1 hour in a 12-percent alcoholic caustic soda solution, are thereupon pressed out, introduced into a solution of 25 parts of stearic acid chloride in chloroform, and kept therein for 2 hours at 45° C. Thereafter one washes with alcohol and water, and dries.

3. After the alkalizing of the cotton fabric with aqueous caustic soda solution, in conformity with Example 1, it is pressed out and dried. Thereupon it is caused to react with a solution of oleic acid chloride in carbon tetrachloride (1 part of acid chloride to 2 parts of alkalized fabric), for 2 hours at 50° C.

In place of the fatty acids mentioned in the above examples, other fatty acids and mixtures thereof may be employed such as capric, caprylic, and lauric acids, and instead of using the chlorides of these or the acids mentioned in the examples, other halides such as the bromides thereof may be employed.

The products resulting from the above example and in general from carrying out the invention described herein, have a fatty feel and are difficult to moisten with water. The treatments described in Examples 1 to 3 serve to alter their activity toward substantive dye-stuffs and to render them relatively immune thereto.

Thus while I have described my improvements in detail and with respect to certain preferred forms, I do not desire to be limited to such details or forms since, as will be noticed by those skilled in the art, after understanding my invention and many changes and modifications may be made and the invention embodied in widely different forms without departing from the spirit and scope thereof in its broader aspects, and I desire to cover all modifications, forms and improvements coming within the scope of any one or more of the appended claims.

Having thus described my invention what I claim as new and desire to secure by Letters Patent is:

1. A process of the kind described which comprises treating cotton with a caustic alkali and an agent including a product resulting from the treatment of a higher fatty acid with thionyl-chloride, capable of esterifying the surface of said cotton, and maintaining the treatment of cotton by said agent to form a water-repellent product having a soft fatty feel and a woolly texture.

2. A process of the kind described which comprises treating a substance containing cellulose with a caustic alkali, subjecting the alkali-treated substance to an agent including a higher fatty acid halide capable of esterifying said cellulose and maintaining the treatment of cellulose by said agent under conditions to produce a product having a soft fatty feel, a woolly texture and a repellence to moisture.

3. A process of the kind described which comprises treating cotton with caustic alkali, removing the excess of alkali, and subjecting the alkali-treated cotton to a higher fatty acid halide at a temperature and for a suitable time to produce a water-repellent product having a soft fatty feel and a woolly texture.

4. A process of the kind described which comprises treating cotton with caustic alkali, removing the excess of alkali, and subjecting the alkali-treated cotton to a stearic acid halide in a solvent for about an hour and at approximately 35-55° C. to produce a superficial reaction between the cotton and said halide.

5. Cellulose retaining substantially its original fibrous structure, superficially chemically combined with a higher fatty acid having a substantial immunity to substantive dyes possessing a fatty feel and a woolly texture and having a repellence to moisture.

6. Cotton retaining substantially its original fibrous structure and in the form of yarn or otherwise, superficially chemically combined with a substance containing stearic acid having a substantial immunity to substantive dyes possessing a fatty feel and a woolly texture and having a repellence to moisture.

7. A material containing unirey fibers of uncombined and chemically combined cellulose, retaining substantially the fibrous structure of the uncombined cellulose, said combined cellulose being made up of an ester of said cellulose and fatty acid selected from a group represented by caprylic, caprin, lauric, palmitic, stearic and oleic acids, said material having its activity toward substantive dye-stuffs relatively diminished as compared with the untreated material.

8. A water repellent material containing unitary fibers of uncombined and chemically combined cellulose, retaining substantially the fibrous structure of the uncombined cellulose, there being a relatively small proportion of combined cellulose as compared to uncombined cellulose, said combined cellulose being made up of an ester of said cellulose and fatty acid selected from a group represented by caprylic, caprin, lauric, palmitic, stearic and oleic acids, said material having its activity toward substantive dye-stuffs relatively diminished as compared with the untreated material.

9. A water repellent material containing fibers made up of cellulose with an integral superficial coating of combined cellulose, re-
taining substantially the fibrous structure of
the uncombined cellulose, said combined cel-
lulose being made up of an ester of said cellu-
lose and fatty acid selected from a group
represented by caprylic, capric, laurie, pal-
itie, stearic and oleic acids, said material
having its activity toward substantive dye-
stuffs relatively diminished as compared with
the untreated material.

10. A fabric containing cellulose retaining
substantially its original fibrous structure
chemically combined with a higher fatty acid
and having a repellence to moisture, said fabric
having its activity toward substantive dye-
stuffs relatively diminished as compared with
the untreated material.

In testimony whereof I have signed my
name to this specification.

PAUL KARRER.