

# UNITED STATES PATENT OFFICE.

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## PROCESS FOR PREPARING CELLULOSE ESTERS IN DEFINITE FORMS.

999,236.

Specification of Letters Patent.

Patented Aug. 1, 1911.

No Drawing.

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To all whom it may concern:

Be it known that I, LEONHARD LEDERER, a subject of the Emperor of Germany, residing at Sulzbach, Germany, have invented a certain new and useful Process for Preparing Cellulose Esters in Definite Forms, of which the following is a specification.

In esterifying cellulose by means of organic acid chlorides or anhydrides a fluid mass is generally obtained as the product of the reactions, from which the cellulose-esters must then be separated. This can be avoided and the esters obtained immediately in a solid form, if the acidifying process is carried out in the presence of a substance which does not dissolve acidified cellulose. For this purpose carbon tetrachloride has been found exceedingly well adapted, being not only cheap but also possessing the highly estimable advantage above the diluent used for similar purposes (*cf.* French patent specification 347,906) of not being combustible and at the same time having a boiling point sufficiently different from that of the fatty acids and their anhydrides to enable a separation and recovery of the various fluid substances to be effected without difficulty. The use of carbon tetrachloride was however not immediately to be expected. As is well-known, cellulose acetates dissolve in acetic acid, chloroform and tetrachlorethane. Owing to the close relationship of the two last-named substances to carbon tetrachloride the assumption was justified that carbon tetrachloride would also take up cellulose. In reality however this promising assumption does not hold good.

Example: In order to obtain say cellulose acetate in the form of cotton, one proceeds for instance, in such a manner that the cotton is first hydrolyzed with an equal weight of glacial acetic acid containing  $2\frac{1}{2}$  per cent. of sulfuric acid, then three times the weight of carbon tetrachloride is added and the cellulose acetylated with  $3\frac{1}{2}$  times the weight of acetic anhydride, which has been diluted with an equal weight of carbon tetrachloride. At ordinary temperature the hydrolyzation requires from 12 to 24 hours, and an equal space of time is occupied by the acetylation; at a higher temperature less time is required for both processes. After the re-

action is completed, the cellulose acetate, which has the structure of the starting material, is separated from the fluid mass by pressing or by centrifugal force and freed from acid in the usual way by washing with alcohol or water. In a similar manner, yarn, fabrics and other objects consisting of pure cellulose or mixed with other fibrous materials may be acetylated.

The products obtained as described are sparingly and incompletely soluble in chloroform, but easily soluble in chloroform containing alcohol. If filaments treated by the new process are woven together with other textile fibers, and the woven fabric thus produced is dyed, special color effects may be produced, because the products obtained according to the present application will not readily absorb coloring matter.

In treating cotton by my present process, the process may be conducted in such a manner, that acetylation takes place throughout the thickness of the raw material, or so as to leave a core of untreated cotton fiber.

For producing other fatty acid esters, such as the esters of propionic or butyric acid, corresponding quantities of the necessary acid anhydrides, say 4 $\frac{1}{2}$  parts by weight of propionic anhydride or 5.6 parts of butyric anhydride, are employed instead of acetic anhydride, and in other respects the process is carried out as described above.

What I claim is:—

The process for obtaining cellulose esters in the same physical form as the raw material employed, which consists in first hydrolyzing cellulose by treating it with a hydrolyzing agent, esterifying the hydrocellulose thus produced by causing the anhydride of the acid employed to act on the same in the presence of carbon tetrachloride, mechanically separating the product from the accompanying liquid and then washing it, substantially as described.

In witness whereof I have hereunto set my hand in the presence of two witnesses.

LEONHARD LEDERER.

Witnesses:

PAUL CUBASCH,  
JOHANN STROBEL.