Patented Oct. 28, 1941

2,260,343

UNITED STATES PATENT OFFICE

2,260,343

GELATINIZED NITROCELLULOSE AND METHOD OF MAKING SAME

Joseph K. H. Seiberlich, Brookline, Mass., assignor to Seiberlich Chemical Corporation, a corporation of Maine

No Drawing. Application June 28, 1938, Serial No. 216,285

9 Claims. (Cl. 92—68)

The present invention relates to the production of nitrocellulose derivatives, and particularly the nitrated derivatives in a uniform and physically more or less invariable state, and to an improved method for converting the derivatives into such state.

It is the general object of the invention to provide combustible or explosive derivatives of cellulose in a solvent-free and gelatinized or transparent state. More specifically, it is an object of the invention to prepare gelatinized nitrocellulose by acting on fibrous nitrocellulose without the aid of solvents or plasticizers.

It is also an object of the invention to provide a simple and safe method of converting inflammable or explosive derivatives of cellulose which are in a more or less fibrous condition into the transparent gelatinized condition.

It is a further object of the invention to provide a method for the conversion of various forms of nitrocellulose in the fibrous condition into the more or less transparent gelatinized condition which is simple and inexpensive and can be carried out completely without the aid of solvents or plasticizers.

My invention is of particular advantage in connection with the manufacture of various forms of nitrocellulose, which represents one of the most explosive or inflammable derivatives of cellulose, and I shall accordingly describe my invention in greater detail in connection with the working up of the nitrated derivatives of cellulose, but it will be understood that my invention is not restricted to these particular derivatives but may be employed with advantage in the working up of other inflammable or explosive derivatives of cellulose having a melting point considerably below the temperature at which it becomes ignited or explodes.

As is well known, the product obtained by the nitration of cotton or other cellulosic material, whether it be the more highly nitrated product employed in the manufacture of explosives or the lower nitration product employed for the manufacture of pyroxylin, celluloid, etc., is more or less fibrous in nature and retains to a considerable extent the color of the original cellulosic material. Heretofore, it has been the general practice in the preparation of gelatinized nitrocellulose to use solvents or mixtures of solvents for penetrating and dissolving the nitrated cellulose and convert the same into a more or less transparent colloidal solution. From the solution so obtained, the nitrocellulose is recovered in the gelatinized form by evaporation of the solutions. Gelatinized nitrocellulose products have also been obtained by the use of special plasticizers by kneading the nitrated cellulose with the plasticizing agents and with a solvent or a mixture of solvents. The solvents are more or less difficultly volatile or are stubbornly retained by the cellulosic mass, so that varying amounts of solvents are incorporated in the nitrocellulose sheet or film and in the powder or other products made therefrom. The plasticizers are usually non-volatile or only difficultly volatile and consequently remain altogether or in large part, in the gelatinized nitrocellulose product. A large part of the solvent is driven off on working up the material on heated rolls and some is expelled during an "aging" process which lasts for varying periods of time, but the product still retains a considerable amount of solvents. This retained solvent is highly objectionable not only because it represents waste but because, varying as it does in amount, the true cellulose content of a mass of the product cannot be exactly determined; while when plasticizers are employed, the nitrocellulose content is necessarily reduced. These factors combine not only to reduce the ballistic qualities of the guncotton, but also make the ballistic effect variable. In the case of the lower nitrated forms, the slow evaporation of the solvent and/or plasticizer frequently results in distortion of sheets or films made of the product, and in shrinkage.

According to the present invention, the fibrous cellulose derivatives are converted into the transparent, gelatinized form by heat, preferably with the aid of pressure. This conversion is accomplished by me without the aid of solvents or plasticizers. In this way, not only is the process made more economical by reason of the saving in solvent and plasticizer and in the reduced time for the conversion, but I obtain a product which is composed entirely of the cellulose derivative and hence a product which is always uniform in quality and composition, is free form shrinkage and has a maximum and at the same time uniform ballistic effect. As a guncotton prepared in accordance with the present invention can consist wholly of nitrated cellulose, being free from solvent, plasticizer, or other additions, cartridges and the like manufactured therefrom have a much higher initial velocity on firing than those made with known forms of guncotton and having a powder chamber of the same capacity. Moreover, as the product of the present invention need contain

no additions which are retained in varying amounts in the final material, by reason of slight and unavoidable changes in the conditions of manufacture at different times and in the compositions of the solvents and plasticizers, the ballistic effect of a given mass of the guncotton will always be the same, assuming that the nitration has been conducted under uniform conditions.

In carrying out the present invention, pure fibrous nitrated cellulose is heated to a temperature of about 120–150° C., preferably under pressure. I have found that when subjected to this temperature the fibrous nitrocellulose can be converted into a uniform, transparent, gelatinized mass. I have found further that, contrary to expectations, heating of the inflammable or explosive material to these temperatures can be conducted with complete safety, and so rapidly, that the time heretofore employed for obtaining a similar conversion is very greatly reduced. In the preferred manner of carrying out the invention, the nitrated cellulose, after being thoroughly washed to remove free acids and other impurities, is suspended in water and the suspension is then employed to produce a fibrous sheet of more or less uniform thickness, in a manner well understood in the paper making art, as by means of a Fourdrinier machine. After pressing out excess water, the still moist sheet is fed between a pair or battery of heated rollers and thereby subjected simultaneously to heat and pressure. I have found a satisfactory working temperature to be about 140° C., but any temperature, not however, considerably above 150° C., may be employed at which the cellulosic material begins to soften and under the combined influence of the heat and pressure, coalesces into a coherent, transparent film. The material, however, does not become liquid, and in the softened, plastic condition, is quickly converted from the fibrous to the gelatinous condition by the pressure rollers.

The following examples will serve to illustrate more in detail, several satisfactory procedures for carrying out the invention:

Example 1

An aqueous suspension of fibrous nitrocellulose is first converted into a fibrous sheet on a paper machine and, preferably after being pressed to remove excess water, is passed in the still moist condition beneath heated rollers capable of applying considerable pressure to the sheet. For a sheet of ⅛ inch thickness, a pressure of 3,000 lbs. per square inch has been found by me to give excellent results at a temperature of about 140° C. Higher pressures may, however, be employed in conjunction with lower temperatures. Due to the combined action of the heat and pressure, the fibrous nitrated cotton is softened sufficiently to lose its fibrous character, the whole mass congealing into a uniform transparent sheet which, after cooling, can be used in the same way as known guncotton and pyroxylin.

The rollers are preferably steam heated, as in this way a very exact control of the temperature can be maintained. I prefer to employ steam as the heating medium in contrast to electrical heating means because the latter present the danger of short circuits with resultant local overheating, and of static electricity. The use of the fibrous sheets in the moist condition adds an element of safety because the water content is quickly evaporated under the rollers, whereby the atmosphere about the apparatus is maintained in a highly humid condition.

In place of steam, preferably saturated, as the heating medium, a heated brine such as a magnesium chloride solution, may be employed.

Under the conditions of temperature, pressure and sheet thickness above indicated, the feed of the fibrous sheet to the rollers may be about 1 foot per second. The rate of feed will, in general, be determined by the speed with which the sheet is softened by the rollers and the softened sheet converted into the transparent, gelatinous condition under the influence of the pressure, and can be readily determined by simple experiment for any selected set of conditions. The gelatinized sheet discharged by the machine is quite as transparent and uniform as those heretofore obtained with the aid of solvents and/or plasticizers, but differs from the latter in having been produced by partial fusion with the aid of heat, and in being free from solvents and/or plasticizers and especially from variable quantities of such additions. The sheet can be employed in exactly the same way as sheets produced by prior processes. It will be found, however, that aside from exhibiting no shrinkage on storage, the sheets will yield powders for ordnance purposes which have uniform ballistic properties and for a given volume will possess more explosive force and thus give a higher initial velocity and greater distance to a projectile than known nitrocellulose powders.

Example 2

A washed and stabilized nitrocellulose is first dried in the usual manner and is then subjected to heat in a thin layer in a suitable container or on a suitably heated support. At about 140° C., the nitrocellulose begins to become plastic, and the material is then pressed by means of heated rollers which are adjusted so as to produce the desired form and thickness of sheet. The final product is completely transparent and shows no fibrous structure.

Example 3

Dried fibrous nitrocellulose is heated in bulk to a temperature of 120–150° C., and during the heating a stabilizing substance such as diphenylamine or dephlegmatizing substance such as diethyl diphenyl urea is added in suitable amounts, the proportions heretofore employed being satisfactory, for example in a proportion of one part of the stabilizing or dephlegmatizing substance to 100 parts of the nitrocellulose. The treatment between heated rollers is then carried on to the point at which the fibrous structure of the material has disappeared. The gelatinized sheet is drawn off and is folded in any suitable manner, or it may first be allowed to cool and then rolled upon a suitable beam or roller.

Any other known stabilizing or dephlegmatizing substance may be employed in place of those mentioned above. Thus, any known substance which will absorb the nitrogen dioxide which may be split off from the nitrocellulose may be utilized as a stabilizing agent.

As already indicated, other inflammable or explosive derivatives of cellulose may likewise be subjected to heat and pressure to convert them from the fibrous into the transparent, non-fibrous condition. The temperature to be employed in any case, will depend, of course, upon the thickness of the sheet and the nature of the derivative,

and in any case the conditions should be such that the derivative is brought just to the softening point and pressure then applied to effect fusion of the fibers into a transparent, non-fibrous mass.

Various changes in the conditions of the treatment from those above specified will occur to those skilled in the art without departing from the principles of the invention. Thus, the fibrous sheet may be preheated before being fed to the rollers, the pressure being thus applied after the heating; or the sheet may be heated only while being subjected to pressure. Also, while I prefer to employ moist sheets, the heated rollers effectively drying the sheets in a very short time, the sheets may be dried before the heating to fusion or before being fed to the rollers.

I claim:

1. In a process for the conversion of nitrocellulose from the fibrous into the non-fibrous gelatinous condition, the step which comprises heating such a fibrous nitrocellulose in the solid condition and in the absence of a solvent and of a plasticizer to the softening point until its fibrous character has been at least partially destroyed.

2. A process for the conversion of nitrocellulose from the fibrous to the non-fibrous gelatinous condition, comprising heating such fibrous nitrocellulose in the solid condition and in the absence of a solvent and of a plasticizer to the softening point and subjecting the softened material to pressure until the fibrous character of the material has been destroyed.

3. A process for the manufacture of gelatinized nitrocellulose which comprises heating nitrocellulose in the solid fibrous condition and in the absence of a solvent and of a plasticizer to a temperature of about 120–150° C. until the material has become softened, subjecting the material to pressure between heated rollers until the fibrous structure of the material has been destroyed, and then cooling the transparent sheet so obtained.

4. A process for the manufacture of gelatinized nitrocellulose which comprises suspending solid, fibrous nitrocellulose in water, forming a sheet of the suspended fibers, heating the sheet in the absence of a solvent and of a plasticizer to approximately the softening point, and subjecting the softened sheet to pressure until a transparent gelatinized sheet is obtained.

5. A process for the manufacture of gelatinized nitrocellulose which comprises suspending solid, fibrous nitrocellulose in water, forming a sheet of the suspended fibers, heating the sheet in the absence of a solvent and of a plasticizer to a temperature of about 140° C., and subjecting the softened sheet to pressure until a transparent gelatinized sheet is obtained.

6. A process for the manufacture of gelatinized nitrocellulose which comprises suspending solid, fibrous nitrocellulose in water, forming a sheet of the suspended fibers, conducting the moist sheet in the absence of a solvent and of a plasticizer between heated rollers capable of raising the temperature of the sheet sufficiently to cause said sheet to soften, and subjecting the sheet to pressure until the fibrous structure has been destroyed and a gelatinous sheet is obtained.

7. A method for the preparation of gelatinized nitrocellulose which comprises adding a stabilizing agent to water-wet nitrocellulose fibers, drying the stabilized nitrocellulose, forming a thin layer of the said nitrocellulose upon a support, heating the fibrous material, devoid of solvent and plasticizer, to about 140° C. until the nitrocellulose becomes softened or plastic, and then passing the softened product over heated pressure rollers whereby the product loses its fibrous structure and becomes transparent.

8. A method for the preparation of gelatinized nitrocellulose which comprises treating nitrocellulose with a stabilizing agent, passing the stabilized nitrocellulose through a paper machine to form nitrocellulose sheets, passing the said sheets, devoid of solvent and plasticizer, over rollers heated to a temperature of between 120 and 150° C. until the sheets are gelatinized to a fiberless transparent mass, and thereafter cooling the product.

9. A nitrocellulose which has been converted into the transparent, gelatinous condition by heat and pressure in accordance with the process of claim 2, said nitrocellulose being devoid of all traces of solvent and plasticizing material.

JOSEPH K. H. SEIBERLICH.