UNITED STATES PATENT OFFICE.

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INSOLUBLE NITRO-CELLULOSE AND PREPARING THE SAME.

SPECIFICATION forming part of Letters Patent No. 420,446, dated February 4, 1890.

Application filed November 12, 1886. Serial No. 116,929. (No specimens.)

To all whom it may concern:

Be it known that I, JOSEPH R. FRANCE, a citizen of the United States, residing in the city of Plainfield, county of Union and State of New Jersey, have invented certain new and useful Improvements in Insoluble Nitro-Cellulose and its Process of Manufacture, (for which I have obtained no Letters Patent whatever;) and I do hereby declare that the following is a full, clear, and exact description of the invention, which will enable others skilled in the art to which it appertains to make and use the same.

My invention relates to an improved insoluble nitro-cellulose or gun-cotton in a more highly-explosive form than the soluble nitro-cellulose, and to an improved process for manufacturing the same.

Insoluble nitro-cellulose, as hitherto made, is not uniform in its character and qualities. The object of my invention is to secure an article that is uniform in these respects, and therefore reliable when used for the several purposes to which it is adapted, and this by an easier and more certain process than that heretofore employed.

Heretofore it has been customary, according to one method, to, first free the cotton from impurities by washing it in an alkaline solution; second, wash it in pure water, and, third, dry it. It is then passed into a bath containing the mixed acids, which are kept at an even temperature of about 60° by means of ice in hot weather and hot water in cold weather, and there allowed to remain for a length of time, according to the condition and nature of the fiber, the strength of the acids, &c., until the desired chemical changes are supposed to have taken place. When it is removed, the acid is first pressed out and then washed out by repeated plunging into clear water. Some of the objections to this method of treatment are, that the action of the mixed acids upon the cotton fiber is slow, irregular, and imperfect and cannot be subjected to any uniform rule. Both expense and care are required to maintain the even temperature, notwithstanding which some lots will reach the point of "nitration" much sooner than others, necessitating constant watchfulness.

My explanation of the slow, irregular, and imperfect action of the acids in the above-mentioned process is, that however uniform the mixed acids may be in strength and proportions, and however carefully the manipulations may be conducted, there are variable elements found in different samples of cotton which defy prognosis and defeat any regular system of rules. The cotton fiber has for its protection a glazed surface, as it were enameled by nature. It is tubular and cellular in structure and contains a natural lubricating semi-fluid substance composed of characteristic oil or gum or water or other material or a combination thereof. Both the glaze and the lubricating substance vary with the soil, the climate, and other accidents of growth, as do other characteristics of the fiber. The tubes of the fiber seem to be open at one end only when the fiber is of normal length. Some or all of these elements play their parts in resisting or otherwise modifying the action of the acids upon the fiber. When the cotton is subjected to the action of the acids in its natural state and length of fiber, the line of least resistance seems to be by way of the inside of the tubes constituting the fiber of the cotton, into which they are taken in part by capillary attraction, subject to change themselves as they progress and to increased resistance from the oil or the gum, &c., in their progress, and therefore to modified action, the result of which is slower and slower and otherwise more and more imperfect chemical change. It may also well be that the power of capillary attraction is balanced in the tubes by air contained therein after a little, and sufficiently to prevent the acids from taking full effect. These objections I overcome in the manner to be shown hereinafter.

Another method consists in making the cotton up into yarn and hanks and treating it in that form with acids in the usual manner. I find that the twisting of the fibers and the disposition in the yarn form and the forming of hanks therefrom cause a certain resistance to the penetration and to the action of the acids with the result that parts of the fibers are not acted upon or acted upon imperfectly.

Still another method consists in taking paper expressly prepared from cotton fiber for the purpose, passing that through the acids, washing, drying, grinding, &c., as before described. In this last case the fibers are of course modified both by the chemical and also by the mechanical treatment to which they have been subjected in the preliminary preparation of the paper; but if the oil or gum or the glaze has been attacked by them, and if they all of them have been removed by subsequent washing, &c., which it is very difficult, if not impossible, to do, the character of the cotton fiber itself seems to have been changed chemically, mechanically, and by felting, so that the cellulose product of the paper process is not uniform or always otherwise satisfactory. In all these methods temperature is found to be an important condition.

I use the cotton fiber in a finely-comminuted condition, but otherwise in its natural state, made as pure and free from extraneous substances as possible, but cut, pulverized, or ground in advance as fine as possible, even to a dust, by the mechanical means, and to the extent set forth in an application, Serial No. 119,845, filed February 5, 1884, or by any other known means, and in that condition subject it to the acids and to all the subsequent manipulations required to produce insoluble nitro-cellulose, to be described hereinafter. The principle of my method is, that whereas in the first-named old process the acids attack the fiber, say of a half inch or an inch in length, from one end and outside, in my process, when my natural cotton-dust is used, each particle will have two mouths or openings by which the acids can enter for every additional piece into which the fiber is cut, and, in addition, the glaze of the fiber may be broken up by the cutting, rubbing, and grinding operations to which I subject it in advance, thereby giving the acid better opportunity for external attack as well. In my method the cotton fiber becomes a homogeneous mass of particles or dust, consisting of very small bits of the material, each one of which is immediately attacked by the acids and upon coming in contact with the same, the result being uniform in character in the time required for nitration, and also in the uniform equivalents of nitrogen taken up in producing the desired product.

My cotton-dust is placed in a bath containing the mixed acids in the usual well-known proportions required to produce the article at any ordinary temperature—between 40° and 90° Fahrenheit—and allowed to remain for a uniform length of time, in proportion to the strength of the acids, until the point of nitration is reached. The surplus acids may then be removed by pressure or extraction, or the nitro-cellulose may be left in the acids for an indefinite length of time, according to convenience, without change or injury, as in the process now in use.

In my process I avoid several of the operations employed in the methods previously described, and I substitute an improved base or material to be treated, having superior qualities for the purpose, which enable me to omit some of the steps required where other base material is used, as follows:

First. I do not find that it is necessary to wash either the cotton fiber or the cotton-dust in any alkaline solution. Consequently I omit that operation entirely, and find that I produce **a** superior article of insoluble nitro-cellulose when it is omitted, and this with certainty in each and every instance.

Second. The washing in pure water and the drying are therefore omitted also.

Third. The watching and constant attention to temperature I also avoid.

Fourth. I avoid the loss of material which occurs from premature or imperfect nitration, and the danger of spontaneous combustion.

Fifth. I avoid the want of uniformity in the resulting product.

Sixth. I avoid both capillary obstruction and much of that arising from the enamel or glaze of the fiber.

Among the advantages resulting from the use of my cotton-dust are the following:

First. The product is always uniform both in appearance and chemically, and will remain stable for a long period.

Second. It is always evenly insoluble.

Third. It is not liable to spontaneous combustion.

Fourth. The remaining acids are more easily and more thoroughly washed out after the point of nitration has been reached.

Fifth. My insoluble nitro-cellulose can be more cheaply produced, since waste is avoided and time is saved in washing.

Sixth. Less watching of the process of nitrogenizing is required.

The fact that the cotton is in the form of dust, and in that form is acted upon more quickly and perfectly by the acids, is important also, and has its proper effect in the washing stage above mentioned, giving more prompt and complete access to the water and egress to the acids.

The insoluble nitro-cellulose made from my cotton-dust is distinguishable from its cotton-dust base by its explosive quality, and by a certain dull, uniform, massed, and slightly-felted appearance, showing that it has not been subjected to mechanical disturbance subsequent to its subjection to the action of the acids. In other respects it corresponds in appearance to the cotton-dust from which it is made. It is distinguishable from the soluble nitro-cellulose by the facts that it cannot be dissolved by any ordinary means and is slightly harsher to the touch, and also by the fact that it is highly explosive. It is distinguishable from soluble and insoluble nitro-cellulose made by the old process, which has been reduced to dust subsequent to subjection to the acids, by its appearance, as above stated, showing that it has not been subjected to mechanical disturbance subsequent to its subjection to the action of the acids.

My gun-cotton is after washing already in a finely-divided condition and ready for compression without further manipulation, whereas when made directly from the fiber it requires to be pulped by what is known as the "paper-pulping" process before it is ready for compression and use, all of which I avoid.

The gun-cotton thus produced is reliable for its known and constant explosive power and for its superior safety.

In practicing this invention I take one-pound batches of finely-ground cotton, which is immersed in the mixed acids in the following proportions: For a good insoluble nitro-cellulose or highly explosive gun-cotton, a proportion of seven (7) parts of strong nitric acid as free from water as can be procured and of stronger sulphuric acid, or sixty-six and one-half degrees, twenty-one (21) parts is suitable. The cotton is stirred into the bath of mixed acids for fifteen (15) minutes, the superabundant acids are pressed out, and the cotton then washed in successive waters until entirely free from acids. Using cotton-dust, I can thus nitrate effectively at any ordinary temperature, say from 50° to 100° Fahrenheit. I usually prefer to keep the room in which the nitration is carried on at a temperature of about 75° Fahrenheit; but I find no perceptible difference in the nitration at ordinary temperatures, as before stated, and I attribute the advantages here indicated over the old methods to the use of cotton-dust; but I do not desire to limit my invention either to the exact proportions of acids or to the exact temperature above set forth, as by the use of my cotton-dust I am able to vary the range both of proportions and of temperature greatly, and yet accomplish the purpose in a superior manner.

In selecting the cotton for reduction to dust to be used in my process and product I seek to avoid cotton fiber in any form that has been subjected to modification or contact with unknown or not understood chemicals or other interfering substances, and in order to secure perfection in the result I prefer to begin with pure cotton—that is, cotton in its natural condition as it comes from the field—and when such cotton is reduced to dust I call it "natural cotton-dust."

I do not intend to limit myself to or by the avoidance of chemical treatment of the pure or natural cotton in known ways for known purposes, but only to exclude injurious or contaminating chemicals.

The cotton-dust described in the application, Serial No. 119,845, consists of cotton reduced to dust, in accordance with the terms of the foregoing specification and description.

I am aware that it is not new to produce an impalpable powder from cellulose by the use of chemicals and afterward treat the same for the production of pyroxyline or nitro-cellulose, and this I do not claim.

I claim as my invention—

1. The process of making insoluble nitro-cellulose, consisting in mechanically reducing cotton to a uniform homogeneous dust-like condition, then subjecting it to the action of a bath of nitric and sulphuric acids in the usual proportions and strength at a temperature of about 75° Fahrenheit, and subsequently pressing out the superabundant acids and washing the product, substantially as described.

2. The herein-described process of making insoluble nitro-cellulose, consisting in subjecting mechanically-comminuted cotton in a pure, homogeneous, and dust-like condition to the action of a bath of nitric and sulphuric acids in the usual proportions and strength at a temperature of about 75° Fahrenheit for about fifteen minutes and subsequently pressing out the superabundant acids and washing the product in successive waters until entirely free from acids, as stated.

3. As an improved article of manufacture, insoluble nitro-cellulose consisting of pure mechanically-comminuted cotton nitrated, substantially as described.

JOSEPH R. FRANCE.

Witnesses:
JAMES A. SKILTON,
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