UNITED STATES PATENT OFFICE.

JOSEPH R. FRANCE, OF PLAINFIELD, NEW JERSEY.

SOLUBLE NITRO-CELLULOSE AND PROCESS OF MANUFACTURE.

SPECIFICATION forming part of Letters Patent No. 420,445, dated February 4, 1890.

Application filed June 11, 1888. Serial No. 116,641. (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 Soluble 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 soluble nitro-cellulose, and to an improved process for manufacturing the same.

Soluble 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, causes 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 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 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 filed by me February 5, 1884, Serial No. 119,845, and in that condition subject it to the acids and to all the subsequent manipulations required to produce soluble 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 any natural cotton-dust is used each particle will have two more 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 soluble 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 nitrations, 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 soluble.

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 soluble 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 finely-comminuted 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 soluble 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 highly explosive or insoluble nitro-cellulose by the fact that it can be dissolved in the usual preparation of ethyl, or grain alcohol and ether, as used in making collodion, or in methyl or wood alcohol of ninety-five per cent. to one hundred per cent. It is distinguishable from soluble 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.

In practicing this invention I find that tak-

ing one-pound batches of finely-ground cotton, which is immersed in the mixed acids of varied proportions according to solubility required for a good soluble nitro-cellulose, a proportion of eight (8) parts nitric acid 42° Baumé, and of sulphuric acid twelve (12) parts 66° Baumé, 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 over the old methods here indicated to the use of cotton-dust as stated herein; 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 of my invention in a superior manner.

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 soluble nitro-cellulose, which consists in mechanically reducing cotton to a uniform and homogeneous dust-like condition and then subjecting it to the action of a bath of nitric and sulphuric acids in about the proportions and at the temperature stated.

2. The process of making soluble nitro-cellulose, which consists in subjecting mechanically-comminuted cotton in a homogeneous dust-like condition to the action of a bath of nitric and sulphuric acids in about the proportions and at the temperature stated.

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

JOSEPH R. FRANCE.

Witnesses:
JAMES A. SKILTON,
WILLIAM STEVENS.