UNITED STATES PATENT OFFICE.

HILAIRE DE CHARDONNET, OF BESANÇON, FRANCE.

MANUFACTURE OF PYROXYLINE.

SPECIFICATION forming part of Letters Patent No. 455,245, dated June 30, 1891.

Application filed April 25, 1890. Serial No. 349,412. (No specimens.) Patented in England April 8, 1890, No. 5,376.

To all whom it may concern:

Be it known that I, HILAIRE DE CHARDONNET, a citizen of the Republic of France, residing in Besançon, Doubs, France, have invented certain new and useful Improvements in the Manufacture of Pyroxyline, of which the following is a specification.

This invention is patented in England by Patent No. 5,376, dated April 8, 1890.

This invention introduces certain improvements in the manufacture of nitro-cellulose of pyroxyline, which improvements pertain to the processes of nitration and washing and the recovery of the acids.

Notwithstanding the great number of the processes proposed heretofore for the industrial preparation of pyroxyline no known method of manufacture gives uniform results and pure pyroxyline. The processes which form the subject of my present invention permit of reducing to a minimum the waste of acids and the obtaining of pure pyroxyline, in which the nitration of the separate fibers differs only in a very small percentage.

Nitration.—Cotton fiber or any other cellulose, (ramie, hemp, purified wood pulp, rags, &c.,) previously well dried by heat, is introduced into large pots previously filled about three-quarters full with the acid mixture, prepared in the ordinary proportions and kept at a fixed temperature by a steam-jacket. The concentration of the acids and the temperature are determined, as usual, by the degree of nitration that it is desired to obtain. (For example, if it is desired to obtain a soluble pyroxyline, to one kilogram of dry cotton use twelve liters of nitric acid at the density of 1.34 and eighteen liters of sulphuric acid at the density of 1.83.) After leaving to soak for a time, which may vary from one to twenty-four hours, or even more, the pots are raised and poured into a centrifugal machine lined with lead or caoutchouc. The acid is extracted by this machine and run off into a reservoir, after which the communication with the reservoir is shut off and the material is washed.

Washing.—The washing is effected by the use of a large quantity of water, and either by removing the acid fiber to a separate vat or by leaving it in the centrifugal machine, in either case taking care to prevent any increase of temperature.

The nitric acid left in the mass by the centrifugal machine may be recovered in the following manner: The first rinsing-water may be neutralized either by adding each time an alkaline carbonate or by placing at the bottom of the vat some fragments of limestone. A new quantity of pyroxyline may then be rinsed in the same water without inconvenience, and this may be repeated successively until this water is sufficiently charged with nitrate to be advantageously evaporated. The nitrate of lime, if desired, may be transformed into alkaline nitrate by sulphate of soda, (which always exists in abundance in the manufacture of nitric acid,) and the nitrate of soda, after being revivified, may serve anew in the manufacture of nitric acid. After this first rinsing the material is deposited in a centrifugal machine so constructed that it may be filled with water. The first centrifugal machine may serve the purpose if thus constructed. The material is then successively dried by the centrifugal action and washed with a large quantity of water while turning the machine slowly. This succession of drying and washing permits a perfect cleansing to be rapidly effected by twelve or fifteen alternations. All the washings should be made with pure water as cold as possible. For wetting the fiber between the centrifugal drying operations the machine may be turned slowly and the water thrown on the mass of pyroxyline; but the water must be very pure in order not to leave any deposit in the mass.

I claim as my invention the following defined novel features or improvements, namely:

1. The described improvement in the manufacture of pyroxyline, consisting in the successive steps of nitration, centrifugal extraction of spent acids, washing of the pyroxyline, and neutralization of the wash-water by an alkaline or basic material for the recovery of the residue of nitric acid left in the pyroxyline by the centrifugal action.

2. The described improvement in the manufacure of pyroxyline, consisting in the successive steps of nitration, centrifugal extrac-

tion of acids, washing with water to remove the acid left after the centrifugal extraction, neutralization of the acid in this water and its reuse with successive quantities of pyroxyline, and successive alternations of washing with water and centrifugal dryings of each quantity of pyroxyline.

In witnesss whereof I have hereunto signed my name in the presence of two subscribing witnessess.

HILAIRE DE CHARDONNET.

Witnesses:

R. J. PRESTON,

MICHEL COQUORT.