present better apparatus and management, and especially by prolonged contact with the lime, through the old process, better results might reasonably be expected.

PYROXYLIN, ITS MANUFACTURE AND APPLICATIONS.

By Walter D. Field.

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PART I.

By the term pyroxylin is understood the soluble nitric ethers of cellulose, namely, the di, tri, tetra, and penta-nitrates. From the date of the use of pyroxylin in photography by Scott Archer in 1851, the number of its uses has increased until, at the present time, tons of the lower nitrates of cellulose are produced yearly. With this increase of production improved methods of manufacture have been evolved.

So general has become the use of this material that to-day it is to be found everywhere. In the form of celluloid it is before us constantly. As a varnish it is used on penholders, pencils, silver and brass ware. Articles are bronzed with it as a medium. An artificial leather has been produced with it, many thousands of yards of which have found a ready market. These applications are all made, with the exception of celluloid, by the use of a solution of pyroxylin.

The first part of this paper pertains to the fibers and their selection for the purposes of nitration; the second part to the processes used for nitrating. The third and fourth parts will treat of the various methods of washing and drying the pyroxylin and of its solvents and uses.

Selection of the Fiber.—Cotton fiber, wood fiber, and flax fiber in the form of raw cotton, scoured cotton, paper, and rags are most generally used and give the best results. The fibers differ greatly in their manner of nitrating, a difference due undoubtedly to their physical structure; hence a given fiber demands a method of nitrating suitable for that particular fiber.

The cotton fiber is a flattened hollow ribbon, or collapsed cylindrical tube, twisted a number of times, and closed at one

end to form a point. The central canal is large and runs nearly to the apex of the fiber. Its side walls are membraneous.

In regard to the influence of the structure of the cotton on nitration, F. Nettlefold (*Chemical News*, **55**, 306) says: "It will be readily understood that the thin side wall tubes of the cotton fibers are readily penetrated by the mixed acids, and, consequently, the highest nitration results."

Wolfram (*Ding. Poly. Jour.*, **230**, 45) says: "Concentrated acids give with cellulose from various sources the same final product, but dilute acids nitrate under the same conditions—cotton the most readily, then hemp, paper, straw, and linen."

Hardwich (*Manuel Phot. Chem.*, 136) recognizes the varying effects produced by the same strength of mixed acid on different fibers. He ascribes this difference to a difference in composition of the fiber and not to a difference in structure. In this conclusion he was undoubtedly wrong.

In the flax fiber the walls are comparatively thick, the central canal small; hence it is to be presumed that the nitration must proceed more slowly than in the case of cotton, which has large central canal and thin walls. The New Zealand flax gives the most perfectly soluble nitrates of any of the flaxes.

Hardwich (*loc. cit.*) says: "When a rather concentrated nitro-sulphuric acid is used cotton gives a glutinous collodion and calico a fluid collodion. In another acid weaker than the first the cotton succeeds well while the calico dissolves instantly. The difference in the action appears to depend principally on the thickness of the fiber. Calico produces pyroxylin of the fluid kind and is partially dissolved because the nitric acid in acting on the outside portion of the closely twisted fiber is reduced in strength, and hence the interior of the fiber is left more nearly in the condition of zylodine." By closely twisted fiber Hardwich refers to the thread from which the fabric is woven, and with this understanding his explanation of the action of the acids is clear.

One of the largest manufacturers of pyroxylin for the industrial arts uses the "Memphis Star" brand of cotton. This cotton is an upland cotton and its fibers are very soft, moist, and elastic. Its color is a light creamy white. This color is re-

tained by the cotton after nitration. The staple is short, and the twist inferior to other grades, the straight, ribbon-like filaments being quite numerous. They use this cotton carded, but not scoured. This brand of cotton contains a large quantity of half and three-quarter ripe fiber, which is extremely thin and transparent, distributed throughout the bulk of the cotton (Monie, *Cotton Fiber*, 67). This is a significant fact when it is known that from this cotton an extremely soluble pyroxylin can be produced.

Pyroxylin of an inferior grade, as regards color only, can be produced from the cotton wastes of the trade. These wastes are scoured, which renders them clean and suitable for nitrating. Paper made from the pulps of the sulphite and sulphate processes is capable of yielding a very soluble pyroxylin. It can be nitrated at high temperatures and still yield an increase in weight. The manufacturers of celluloid in this country use a tissue paper made of the flax fiber, and two to three one-thousandths of an inch in thickness. The paper is cut into squares of about one inch before nitrating.

Mowbray (United States patent No. 443,105, Dec. 3, 1890) says that a pure cotton tissue paper less than one five-hundredth of an inch in thickness, thin as it is, takes on a glutinous or colloid surface and thus requires some thirty minutes or thereabouts to enable the immersion to take place. With a thicker paper only the surface of the paper would be nitrated and the body of the paper would be unacted upon.

Mowbray (U. S. patent No. 443,105) recognizing the importance of the structural form of the cellulose on the nitrating process, uses a fiber which has been saturated with a solution of nitrate of soda, then dried slowly, claiming that the salt crystallized in the fiber or enters by the action termed osmose and opens up the fiber to the action of the acid bath. He also says he may use "any salt crystallized, or water crystallized by freezing in the cells of fibrose cellulose." Such a preparation of the fiber is not at all necessary when using a high temperature for nitrating. It might yield with some cottons a very soluble pyroxylin, when low nitrating temperatures are used. H. De. Chardonnet (English patent No. 19,560, 1891) heats the cellu-

lose intended for nitration to about 150° C. and claims to increase the solubility of the subsequently formed pyroxylin. The heating of the cellulose could have had no other effect than that of drying the fiber before nitration, and hence a pyroxylin more perfectly soluble in the ethereal solvents he was using in his testing.

Henly and Spill (English patent No. 1017, 1870) use "esparto grass in a finely divided condition (which is the novel and indispensable condition of the operation)."

Dietz and Wayne (U. S., patent No. 133,969, 1872) use ramie, rheea, or China grass, for producing a soluble pyroxylin and say in regard to it that "Pyroxylin made from ramie will always be of uniform strength and solubility. In making collodion from this pyroxylin a much smaller quantity of solvent is required than for pyroxylin made from cotton." It is to be doubted that ramie will yield a more soluble pyroxylin than cotton. The author's experience is entirely to the contrary.

Such is the influence of the physical form of the fiber on the process of nitration that when flax fiber and cotton fiber are nitrated with acid mixtures of exactly the same strength, and at the same temperature the solution of the first is glutinous or thick and the second fluid or thin. By simply nitrating at a higher temperature than the cotton, the flax will yield a pyroxylin giving an equally fluid collodion.

The presence of chlorine in the fiber must be carefully avoided

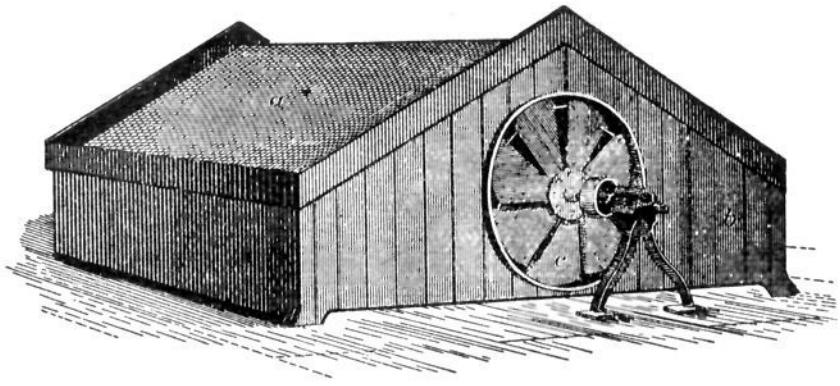

FIG. 1.

as such a fiber will yield a pyroxylin which is acid and which cannot be washed neutral.

The removal of all moisture from the fiber previous to nitra-

tion is essential to the production of a carefully prepared pyroxylin. This drying is best carried out on the form of drier used in drying wool. The plan of such a drier is given in Fig. I. The surface *a* is covered with wire cloth, having a half inch mesh, and should be galvanized.

Heater coils can be placed within the frame *b* and the hot air forced through the fiber by means of the rotary fan *c*.

If the fiber is found to need scouring to make it fit for the process of nitration, it is best scoured by those who make a business of this branch. Absorbent cotton is produced on such a large scale at the present time, that the manufacturers will sell it at a very low figure, in order to keep their plants in operation. The author obtained the fiber used by him for several years from this source.

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THE PREDOMINANT ORGANIC ACID IN SORGHUM JUICE.[1]

BY OMA CARR.

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IN the work of the Department of Agriculture during the past few years attention has often been called to peculiarly tenacious and difficultly soluble incrustations forming upon the tubes of the evaporating apparatus of the sorghum-sugar houses of Kansas. Owing to the formation of these incrustations and the difficulty of removal the processes of manufacture are greatly impeded. Dr. Wiley collected a large amount of this scale at Medicine Lodge and directed me to make a study of its composition and properties. The following paper gives the results of a brief study of the scale.

The scale examined was in pieces of varying sizes, retaining the contour of the tubes from which it had been removed. A quantity of fifteen pounds was taken from some three hundred pounds forwarded from the factory at Medicine Lodge. The scale as prepared for examination consisted of a powder sufficiently fine to pass an eighty mesh sieve.

The analyses of two samples of the scale, one designated

1 Read before the Chemical Society of Washington, May 11, 1893.