UNITED STATES PATENT OFFICE.

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ART OF MANUFACTURING CELLULOID.

SPECIFICATION forming part of Letters Patent No. 677,012, dated June 25, 1901.

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To all whom it may concern:

Be it known that I, GEORGE HILLARD BENJAMIN, a citizen of the United States, residing at New York city, county and State of New York, have invented certain new and useful Improvements in the Art of Manufacturing Celluloid, of which the following is a specification.

My invention relates to the manufacture of celluloid, and has for its object to avoid the possibility of explosion during treatment of the materials and to bring about an intimate mechanical mixture of the materials in the required proportions unaccompanied by an excess of the solvent used, which, according to the processes now in use, has to be removed by the application of heat and hot and cold rolling before the compound sought to be obtained is ready for commercial use.

Celluloid, as is well understood, consists of pyroxylin—*i. e.*, tetra or penta nitrated cellulose—(sometimes hexa-nitrated cellulose is used. The latter, however, is seldom employed on account of its explosive properties) gum-camphor and a solvent—such, for instance, as alcohol, ether, nitrobenzene, &c.

The usual process of manufacturing celluloid consists in effecting a mechanical mixture of pyroxylin and camphor, then introducing a liquid solvent, such as alcohol or other suitable fluid, and subjecting the mixture to pressure and heat, and, if a solid is required, to hot and cold rolling to remove the excess of alcohol or other solvent employed. This process is both costly and defective, owing to the fact that explosions are liable to occur in effecting the mechanical mixture of the materials and that long-continued heating and expensive rolling and other manipulations are requisite to eliminate the excess of solvent where a solid is to be made and bring about an intimate mixture of the materials to produce a homogeneous compound.

In order to overcome the objections stated, I have devised the process which I will now proceed to describe.

I will premise by stating that I have found by experiment that if pyroxylin, camphor, and a suitable solvent—such as alcohol or ether, &c.—are subjected for a sufficient time to the action of intense cold—such, for instance as may be applied through the agency of liquid air—the liquid solvent will be brought to a solid state, and all solids may, by mechanical means and without danger of explosion, be reduced to a very fine state of division, and, further, that if the various materials are thoroughly mixed in proper proportions while in such intensely cold and divided state they will, upon regaining the temperature of the atmosphere, have combined mechanically or coacted to form a homogeneous compound having the form of a solid or a liquid, depending upon the amount of solvent employed.

To carry my invention into effect, I take a definite quantity of pyroxylin (one part) in any one of its forms—*i. e.*, tetra, penta, or hexa nitrate—and subject it to the action of intense cold. This, I find, can be conveniently done by immersing the pyroxylin in liquid air and allowing it to remain therein for from six to ten minutes. Upon withdrawing the frozen pyroxylin from the mixture it is placed in an ordinary iron mill, the metal of which is preserved at a low temperature through the means of liquid air, and ground to a fine state of division. The second solid material, camphor, (two parts,) is treated in a precisely similar manner. The solvent alcohol, ether, &c., (usually from ten to thirty per cent., by weight, of the combined mass of pyroxylin and camphor and depending upon whether a solid, semisolid, or liquid celluloid is required, although I do not limit myself to these proportions,) is introduced into an open-mouthed glass jar and the jar immersed from fifteen to eighteen minutes in liquid air. The solvent is then ground in a mill, as described for the treatment of the pyroxylin and camphor. All of the ground materials while in an intensely-cold condition are then thoroughly mixed (almost perfect mixture can thus be obtained) and set aside in a suitable vessel, preferably of stoneware, to acquire heat, or, if it is desired to hasten the process, gentle heat may be applied to the mixture by means of a water-bath. When the mixture has acquired the temperature of the atmosphere, it will be found that the various materials have combined mechanically or coacted to form a homogeneous mixture of the required physical condition.

Instead of freezing and grinding all of the ingredients I may only freeze and grind the pyroxylin and camphor. In such case the alcohol or other solvent is introduced in its normal state at the temperature of the atmosphere, or where a fluid or semifluid is required I may only freeze and grind the pyroxylin, in which case the camphor and solvent are mixed together, preferably under the influence of heat, to form a fluid, to which the frozen and comminuted pyroxylin is added in the required proportions and the mixture stirred together. This last-described process, however, requires care in manipulation and that the solution of camphor be quite cold, as otherwise there is liability of explosion. The best results and mixture, however, are obtained by freezing and grinding all of the materials and mixing while such materials are in a frozen state.

Any loading or coloring material can be advantageously introduced at the time when the mixture of frozen materials is effected. It is preferable to also chill these materials.

Having thus described my invention, I claim—

1. The herein-described process of manufacturing celluloid, which consists in reducing the materials to a frozen state, then grinding, and finally mixing.

2. The herein-described process of manufacturing celluloid, which consists in reducing the pyroxylin and camphor to a frozen state, then grinding, then adding the liquid solvent.

3. The herein-described process of manufacturing celluloid, which consists in first freezing the pyroxylin and camphor, then grinding and intimately mixing these two bodies, then introducing the liquid solvent into the mixture, and finally increasing the temperature of the mixture.

In testimony whereof I affix my signature in the presence of two witnesses.

GEORGE HILLARD BENJAMIN.

Witnesses:

J. E. PEARSON,

ARTHUR C. BLATZ.