UNITED STATES PATENT OFFICE.

ISIDOR KITSEE, OF PHILADELPHIA, PENNSYLVANIA.

METHOD OF PRODUCING CELLULOSE COMPOUNDS.

No. 900,744. Specification of Letters Patent. Patented Oct. 13, 1908.

Application filed March 7, 1908. Serial No. 419,745.

To all whom it may concern:

Be it known that I, ISIDOR KITSEE, citizen of the United States, residing at Philadelphia, in the county of Philadelphia and State of Pennsylvania, have invented certain new and useful Improvements in Methods of Producing Cellulose Compounds, of which the following is a specification.

My invention relates to an improvement in a cellulose compound useful for jars and electric resistance.

The compounds consisting primarily of dissolved cellulose are mostly known in commerce as "celluloid," "pyroxylin," etc. All these compounds are, electrically considered, of low resistance when compared to rubber or gutta percha. The articles manufactured from same are to some extent brittle, and when subjected to the action of diluted acids, the resistance is usually greatly lessened, and these compounds are therefore not well adapted for electrical work.

It is the object of my invention to increase the resistance value of such compounds and to make such compounds to any degree of flexibility desired. To accomplish this object, I have carried out in the course of several years a series of experiments, and I have produced as the result of such experiments products in the shape of jars for battery cells, insulating-plates, etc., which entirely justify me in saying that the production of articles from the dissolved cellulose of different degrees of resistance values and different degrees of flexibility is not only feasible, but if the process as laid down in this my specification is carried out entirely practical.

In the course of manufacturing articles out of soluble cellulose of my own make, as well as articles made of celluloid scrap redissolved, I found that it was necessary to make these articles, mostly when used as vessels to retain some liquid, of either too great a thickness or to line the same with a coating of rubber. To obviate this, to make jars and other insulating articles from cellulose, and to be enabled to use the same for insulating-conductors in a manner so as to give it the required pliability, I have recourse to a method of compounding the cellulose as will hereinafter be fully described.

In all compounds so far used, the cellulose is intermixed with a fibrous material in its nitrated state and I have also used asbestos fibers for this purpose; but the products do not present an even surface and when a plate, made with the addition of a fibrous material in its natural state, is broken up, the inner space of same is found to be not in a homogeneous state and the particles of cellulose, surrounding the fiber, either retain—in nearly all the cases, part of the moisture of the solvent, or if entirely dry, a space between the same and the other parts of the material is discernible through a magnifying glass. In most cases, the filling material for the amorphous cellulose consists of asbestos, but it is well known that asbestos is even more hygroscopic than the amorphous cellulose itself. It is, therefore, necessary first to produce compounds which are entirely impervious to moisture and then intermix the same with the cellulose and it is also necessary to make the cellulose, with the aid of an additional material, impervious to moisture.

I will now here describe a method by which I was enabled to produce jars for secondary batteries and plates to be used as a resistance for electric devices, and I will give, so that persons versed in the art may practice this my invention, some figures as to the proper amount of the different materials to be used in the production of the compound, but it is well understood that the ratio of each of these materials may differ to suit requirements.

If it is required to produce the amorphous cellulose out of a cotton fiber, it is best to first wash this fiber in an alkali, such for instance as a carbonate of soda. If it is desired to produce the amorphous cellulose with the aid of the nitration, it is best to use the nitric acid and sulfuric acid in the proportion of ten to eight. It is supposed that it is desired to reduce one thousand pounds of raw cotton fiber to the amorphous state. To wash this fiber, about two hundred pounds of the alkali should be used. With the help of this alkali, the cotton fiber is entirely cleaned of all the grease and other foreign substances and when thoroughly washed and then dried, it is ready for the nitration. Generally in the nitration, two parts of sulfuric acid and one part of nitric acid are used, but I have found that if a quick nitration is desired, it is best to use a preponderance of nitric acid and for this one thousand pounds of cotton fiber, it is preferred to use about five thousand pounds of nitric acid, specific gravity 1.42, and four thousand pounds of sulfuric acid, specific gravity 1.84.

It has to be borne in mind that only part of

the nitric acid will be used up in the nitration and for the next nitrating bath, it is only necessary to add a comparatively small proportion of nitric acid and sulfuric acid to the solution. I have found that of the mixture aforesaid, only about eight hundred pounds of nitric acid and only about two hundred pounds of sulfuric acid are used up, so that for the purpose of nitrating a second lot of one thousand pounds, only a small amount of acid needs to be added to the nitrating bath.

The product, when once nitrated, should be washed repeatedly, so that no acid will remain therein.

In the course of my experiments, I have found that if the nitrated cotton is subjected subsequently to a water bath containing for each one hundred gallons of water one-half a gallon of ammonia water, specific gravity 0.935, the later deterioration of the product is entirely overcome. The product should then be dried at a temperature not exceeding one hundred and ten degrees Fahrenheit (43° C.).

To dissolve the nitrated product, different chemicals may be used, such for instance as acetone, amyl acetate, or glacial acetic acid; but to make the dissolving process more economical, I have recourse to the following method:—

I place the nitrated article in a closed chamber which, in reality, may represent the receiver of a distilling apparatus, in a manner so that the fumes, passing from this apparatus through said receiver, will have to penetrate the other part of the material placed therein. This receiver is connected with a retort filled with a solvent. On the other side, the receiver is connected, preferably, with the interposition of a worm, with a condensing reservoir. Heat is then applied to the retort and the fumes of the solvent will be carried into the receiver, will penetrate the particles of the nitrated cellulose and will dissolve the same. After the process has been carried on to a point necessary to either dissolve or only make plastic the material, the same is then taken out and is ready to be used with a compound later on to be described.

As stated above, either acetone, amyl acetate, or any of the well known solvents may be used. If a very weak solution of acetic acid is used in the retort (for the reason that the same can be bought very cheaply), it is best to moisten first the nitrated article, as the acetic acid fumes are, at the beginning, entirely void of any moisture. One hundred pounds of rosin (Burgundy pitch I found very well adapted for this purpose) is then dissolved in either acetone, or in a hydrocarbon, such as gasolene or benzene, and well intermixed with the dissolved cellulose.

The cellulose alone is not well adapted for the purpose of producing commercial articles and I prepare the same for such purpose by adding a compound which I call the "filler". To produce this filler, I preferably have recourse to the following process, taking it for granted that this filler is used for one thousand pounds of cotton, as aforesaid:—I take about five hundred pounds of asbestos in a state known as "flour of asbestos", that is, asbestos ground fine (no long fibers should be present). To eliminate the hygroscopic properties, I boil the asbestos flour with—preferably—castor-oil, but I have found that even the common linseed oil answers the purpose well. I found that for each pound of asbestos, a pound of oil is needed. After careful boiling, I melt separately about eighty pounds of sulfur in a manner, so that the same shall remain in a semi-liquid or syrupy state. The sulfur is then added to the asbestos and, by preference, I add thereto about forty pounds of ammonium phosphate so as to reduce the inflammability of the compound when intermixed with the dissolved cellulose. The compound is then very well intermixed so as to form a homogeneous paste. The gelatinized cotton is then intermixed, with suitable machines, with the compound, as stated, and then with the aid of a colander rolled into sheets of the required thickness. If the mass gets too dry, a slight amount of additional solvent will bring the same to the state of plasticity, and if worked with due care, the mass is as plastic as sculptor's clay and can be molded at pleasure.

If jars are produced of these compounds, they should be dried first slowly and then at a temperature of about from one hundred and fifty to two hundred degrees C. to complete the vulcanization. The time depends on the thickness of the layers as well as the shape of the article, but from three to four days (72 to 96 hours) are generally sufficient for all practical purposes.

It is advisable to dry at air temperature until the bulk of the solvent is evaporated (when the solvent is in liquid state and not the fumes of the solvent only are used).

In case the solvent is used in a liquid state, the evaporated solvent could be condensed and recovered with economy.

I have above given proportions which I have used with satisfactory results, but it is obvious that the various proportions may differ without departing from the scope of my invention.

It is only necessary to add that if the sulfur is added in too great a proportion, the resultant article will not have the necessary strength.

Having now described my invention, what I claim as new and desire to secure by Letters Patent is:—

1. The process of producing a compound

useful for retainer for secondary cells, which consists in first producing a cellulose compound impervious to moisture, in second, producing an asbestos compound impervious to moisture, and in third, intermixing both compounds with the addition of sulfur.

2. In the production of useful articles from amorphous cellulose, the process, which consists in boiling the filling for such cellulose in oil, and intermixing then said filling with said cellulose and a flour of sulfur and then vulcanizing the same with the aid of heat.

3. The method of curing or vulcanizing articles made of amorphous cellulose, which consists in subjecting the cellulose to a process whereby the same is made impervious to moisture and second, to a process whereby the same is vulcanized with the aid of a sulfur.

4. As an article of manufacture, a compound, consisting of an amorphous cellulose, a rosin or resin, a flour of asbestos, and oil, cured or vulcanized with the aid of sulfur.

5. Means to produce a retainer for liquid, out of an amorphous cellulose, said means comprising a dissolved rosin or resin intermixed with said cellulose, a flour of asbestos boiled in oil, and a sulfur intermixed with said compounds.

6. As a new article of manufacture, a jar for secondary batteries, consisting of a cellulose and a filling for same, the cellulose as well as the filling made impervious to moisture, said jar vulcanized with the aid of a sulfur.

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

ISIDOR KITSEE.

Witnesses:

EDITH. R. STILLEY,

MARY C. SMITH.