UNITED STATES PATENT OFFICE.

JAMES McINTOSH, OF NORRISTOWN, PENNSYLVANIA, ASSIGNOR TO DIAMOND STATE FIBER COMPANY, OF ELSMERE, DELAWARE, A CORPORATION OF DELAWARE.

PROCESS OF MAKING FIBER PRODUCT.

1,236,959. Specification of Letters Patent. Patented Aug. 14, 1917.

No Drawing. Application filed March 10, 1916, Serial No. 83,350. Renewed July 5, 1917. Serial No. 178,787.

To all whom it may concern:

Be it known that I, JAMES McINTOSH, a citizen of the United States, and a resident of Norristown, Montgomery county, Pennsylvania, have invented Processes of Making Fiber Products, of which the following is a specification.

In the attempt to render vulcanized fiber and similar products waterproof or water-repellent, the chief and in most cases, the prohibitive difficulty has been to introduce the water-repelling material so that it uniformly impregnated the fiber structure. If it was attempted to introduce such material during the manufacture of the fiber, it was found to prevent the complete removal of the chlorid of zinc or other parchmentizing reagent employed, and it could not be successfully introduced after the manufacture of the fiber was complete, since the latter was then of such a hard, compact membraneous nature and contained such an amount of water, that it was a practical impossibility to make the waterproofing material enter it.

Again, while it has hitherto been possible to so treat parchmentized fiber as to render it comparatively flexible, it was then none the less useless in many arts, since the substance imparting the flexibility could not be retained, particularly if the fiber was exposed to moisture or was actually immersed in water;—the reason being that the only ingredients capable of imparting flexibility to the fiber, which could be commercially introduced into the same, were soluble in water. It was therefore either an impossibility to introduce waterproofing material into the fiber, or where in a certain case such introduction was possible, the fiber was rendered, if anything, more inflexible and harder than before.

One object of my invention therefore is to provide a commercially practical and relatively simple process whereby vulcanized fiber, vegetable parchment, and similar chemically treated or mechanically hydrated cellulose products may be made both flexible and waterproof, the invention being of such a nature as to be conveniently carried out in a relatively short time, depending upon the manner of carrying out the process and also upon the thickness of the fiber under treatment.

I further desire to provide a form of vulcanized fiber which, while being highly water-repellent, shall at the same time be relatively flexible as well as durable, in order that it may be advantageously used as a leather substitute and also for purposes of electrical insulation, especially where the hygroscopic characteristics of untreated vulcanized fiber would ordinarily prohibit such use.

In carrying out my invention I preferably operate on vulcanized fiber while it is in what is known as the "green" condition;—that is, after its manufacture has been completed except for the final drying, although it is to be noted that the treatment may be given while the fiber still contains the chlorid of zinc or other parchmentizing reagent, and before it has been washed. Under either of these conditions, the fiber contains a relative high percentage of water, and in the simplest form of my invention, I immerse the fiber to be treated in a vessel containing an organic solvent such as acetone or alcohol;—for example, methyl or ethyl alcohol, leaving it in this bath for a period depending on its thickness, the temperature of said bath, etc. If for instance, the fiber sheets operated on are one-eighth of an inch thick, they should remain in the alcohol for a period of about twenty-four hours, though obviously this time may be widely varied without departing from my invention. Under these conditions the alcohol acts to replace the water or other liquid throughout the body of fiber and it is obvious that other liquids soluble in or miscible with water may be used in place of alcohol without departing from my invention.

At the end of the above noted time the fiber contains a liquid in which there is but a small proportion of water and it is thereafter removed to two or more baths, preferably of the same liquid as that first employed, in which it is allowed to soak for another period of about twenty-four hours;—it being noted however that this as well as the other times specified, will depend on a number of factors such as the temperature, whether or not the liquid is quiet or circulated, the pressure on the liquid, etc. At the end of this time practically all of the water within the fiber structure has been replaced by the alcohol or other solvent liquid and the fiber may then be placed in a bath of the waterproofing compound.

This latter is characterized by the fact that it is soluble in the liquid which has been used to replace the water in the fiber, and while many liquids or liquid bodies may be employed for this purpose, I have found that vegetable oils, such as castor oil, rape seed oil or poppy oil give good results, as well as mineral and animal oils, fatty acids, waxes, esters, rubber, gums such as shellac, dammar, and the like.

In treating fiber of the thickness above indicated, I have found that four days immersion in castor oil or oleic acid suffices to cause the alcohol to be practically completely replaced. Said fiber, after such treatment is as flexible as leather of the same thickness, is uniformly impregnated with waterproofing compound throughout its entire structure and is wholly or partially repellent to water. Moreover it is a better electric insulator than ordinary vulcanized fiber and it is to be especially noted that it retains its flexibility and waterproof qualities for an indefinite period. At the same time it may be cut with the utmost ease, although its structural strength is not appreciably diminished.

If found advisable, in order to replace the water or other substance originally in the fiber, I may immerse the green or partially finished fiber or other material in a succession of baths of alcohol or equivalent solvent or I may place the fiber in a single bath containing both the alcohol or other water replacing liquid and the waterproofing agent. In this case the alcohol or other solvent first withdraws the water from the fiber by osmotic action, after which the oil or other waterproofing material permeates the body of the fiber by a similar action.

In some cases it may be necessary to treat the fiber with more than two liquids in order to put into it the waterproofing or flexibilizing material, particularly in view of the fact that most oils are not easily soluble in cold alcohol. In such a case, after subjecting the fiber to one or more alcohol baths, it may be placed in a bath of a liquid in which both the alcohol and the waterproofing and flexibilizing liquid is soluble, such for example as ether, which thus replaces the alcohol as before. The latter may then be placed in the olein liquid for rendering it flexible and waterproof, which thereupon displaces the ether, giving a product having the qualities desired.

While I have found that for most practical purposes, one of the alcohols is the cheapest and most satisfactory material for use in replacing the water or other solution in fiber, there are obviously other liquids such as acetone, a mixture of alcohol and ether, or the like, which while being soluble in water, also possess the quality of dissolving the substance which it is desired to introduce into the fiber. In addition to the oils, fatty acids, etc., which may be introduced as described, I may in some instances carry into the fiber other materials such as nitro-cellulose or other cellulose compounds, for which purpose the green fiber as before should be treated with a liquid capable of removing from it the water which it contains. Thereafter it should be placed in a liquid soluble in the water-replacing liquid which itself constitutes or holds in solution, the material which it is desired to deposit in the fiber.

Obviously it is by no means necessary that the fiber shall be a commercially finished article, since it may be advisable in some cases to apply my process to partly parchmentized fiber or to fiber which is not in a green condition but is more or less hardened.

Moreover, while the process is especially designed for the treatment of vulcanized fiber, it is to be noted that it may be advantageously used to make waterproof paper, cardboard, and other fibrous materials which are impervious to oils or to the water-repellant substance in question.

In some cases it may be advisable to heat, vulcanize, alter or otherwise treat the material introduced into the fiber in order to change its nature and give it certain desirable properties;—for example, I may by the process above described introduce into parchmentized fiber a solution of a phenol-formaldehyde condensation product and thereafter by the application of a suitable degree of heat, solidify or fix the same in the body of said fiber In case a drying oil is introduced, this may be altered within the body of the fiber to any desired extent by heat or other agency. If desired the fiber may be impregnated by the process described with a solution of a water soluble soap such as potassium oleate and thereafter be treated with a mineral acid such as sulfuric acid to liberate the corresponding fatty acid.

In another case the fiber may be impregnated with a fatty acid such as oleic acid which may be changed into an insoluble soap by immersing the fiber in a bath of alum and finally removing the resulting water soluble compounds.

It is noted that it might be advantageous for some purposes to make a fiber which shall contain both a hygroscopic material as well as one which is insoluble in water. For example, after treating the fiber with alcohol, it would be immersed in a liquid containing alcohol, glycerin and oleic acid, so that the two latter substances would be carried into said fiber with the effect of increasing its flexibility.

I claim;—

1. The process which consists in treating parchmentized fiber with a water soluble material and then treating it with a water-

proofing material soluble in said first material.

2. The process which consists in treating parchmentized fiber with a water-removing material and then treating it with a material capable of rendering it flexible.

3. The process which consists in treating parchmentized fiber with a water displacing substance and then treating it with a waterproofing substance capable of rendering it flexible.

4. The process which consists in removing the water from vulcanized fiber and impregnating said fiber with a waterproofing material.

5. The process which consists in removing the water from fiber and impregnating the parchmentized fiber with a waterproofing material capable of rendering it flexible.

6. The process of introducing waterproof material into parchmentized fiber which consists in impregnating it with a liquid in which said material is soluble and then treating the fiber with the waterproofing material in liquid form.

7. The process of introducing waterproofing material into a parchmentized fiber which consists in removing the water therefrom; impregnating said fiber with a liquid in which the waterproofing material is soluble; and then treating the fiber with the waterproofing material in liquid form.

8. The process which consists in removing water from parchmentized fiber and impregnating the fiber with a water-insoluble substance.

9. The process which consists in subjecting parchmentized fiber to a succession of baths of liquid capable of removing the water therefrom; and thereafter subjecting the fiber to a bath of a compound soluble in said liquid but insoluble in water.

10. The process which consists in placing parchmentized fiber in alcohol and thereafter placing it in a liquid containing a water-insoluble substance which is soluble in alcohol.

11. The process which consists in placing parchmentized fiber in a bath of alcohol and thereafter treating the fiber with an oil soluble in the alcohol.

12. The process which consists in placing parchmentized fiber in a succession of baths of alcohol and thereafter in a bath of a liquid soluble in alcohol.

13. The process of impregnating fiber with a certain compound which consists in treating the fiber with a liquid to remove the water therefrom; replacing said liquid in the fiber by a material in which said compound is soluble; and thereafter placing the fiber in a bath of the compound with which it is desired to impregnate it.

14. The process of introducing waterproofing compound into a body of fiber which consists in placing said fiber in a bath of alcohol; replacing the alcohol by a material in which the waterproofing compound is soluble and which is also soluble in alcohol; and finally in placing the fiber in a bath of the waterproofing compound.

15. The process which consists in treating a paper web with parchmentizing material; passing the resulting fiber through alcohol to wash out the surplus parchmentizing material; and then passing said fiber through a waterproofing compound soluble in alcohol.

16. The process which consists in passing a web of paper through a solution of parchmentizing material; building up a laminated structure from successive layers of said web; and thereafter immersing the laminated structure in alcohol to wash out the surplus parchmentizing material.

17. The process which consists in passing a web of paper through a parchmentizing liquid; immersing the web in an organic liquid to remove the parchmentizing liquid; and thereafter passing the web through an impregnating liquid which is soluble in said organic liquid.

18. The process which consists in treating a web of paper with parchmentizing liquid; passing said paper web through an organic compound in liquid form to dissolve out said parchmentizing liquid; and finally passing the web through a water insoluble liquid which is soluble in said organic liquid.

19. The process which consists in passing a paper web through a parchmentizing liquid; then immersing the resulting fiber in a bath or baths of alcohol to cure it; and thereafter immersing the fiber in a bath containing an oily substance soluble in the alcohol.

JAMES McINTOSH.