UNITED STATES PATENT OFFICE.

ALFRED O. TATE, OF TORONTO, ONTARIO, CANADA.

ART OF WATERPROOFING FIBROUS MATERIALS.

933,861. Specification of Letters Patent. Patented Sept. 14, 1909.

No Drawing. Application filed June 1, 1907. Serial No. 376,841.

To all whom it may concern:

Be it known that I, ALFRED O. TATE, a subject of the King of Great Britain, residing in Toronto, Ontario, Dominion of Canada, have made a new and useful Invention in the Art of Waterproofing Fibrous Materials, of which the following is a specification.

My invention is directed particularly to an improved method or process of waterproofing fibrous articles, such as woven, knit, or fibrous cloths, for clothing materials, sails of vessels, tents, tarpaulins, etc., and it has for its objects, first, to effect such a result by overcoming in fabrics practically all tendency to capillary attraction in the fibers thereof; second, to effect a coating on the fibers of either or both surfaces of the material which shall prevent the penetration of moisture; third, to produce a ventilated water-proof product which has an especial utility in connection with clothing.

Prior to my invention attempts have been made to water-proof materials of the character referred to by saturating the same in an electrolytic solution containing a soluble metallic salt, such as sulfate of aluminum, and afterward placing it under a certain degree of pressure between two electrodes and passing a current of electricity therethrough. Processes of this general nature are disclosed in U. S. patents to Henry L. Brevoort Nos. 558,717 and 558,718 of April 21st, 1896; and to Jean T. Van Gestel, Nos. 653,715 and 653,716, July 17th, 1900.

It is assumed in the before-mentioned patents that the water-proofing of the material is effected by the actual transmission of the insoluble matter in the electrolyte into the inner surfaces of the fibers and is deposited thereon by virtue of the electrolytic action, but I have ascertained after a long series of experiments that such is not the case and that at best only a slight coating of the insoluble substance is deposited upon one face of the material and that face which, when exposed under the influence of the current in an electrolytic bath, is adjacent to the cathode. I have also ascertained that fabrics as thus treated, after being subjected to usage a short time, lose practically all of their water-proofing capacity, owing to the fact that the outer or water-proofed surface will become cracked or disrupted and the deposited substance in the inner surface—which substance is soluble—will leach or drain out in use.

Before proceeding with the tests which proved to me conclusively these facts I deemed it important to determine definitely the nature of the force or energy which causes a fabric to become saturated with a liquid when submerged therein, or placed in contact therewith. On laying a piece of silk or similar fabric upon the surface of a basin of water I observed that it instantly became suffused and that the water entered the same by ascending therethrough directly in the opposite direction to the force of gravity. I also observed that suffusion in this direction was quite as rapid as suffusion in the opposite direction or in the lines of the force of gravity. This clearly proved to me that such action was due to another force or form of energy than gravity, and none other than what is known as capillary attraction, most commonly made use of in connection with lamp wicks, blotting paper, and similar absorptive agencies. For the purpose of determining whether gravity performed any portion of this suffusion I procured a piece of cloth in which the meshes or pores were clearly visible when held before the light and treated it in accordance with my novel process which I will hereinafter describe, the meshes after treatment being as clearly open and visible as before. I made a bag of this treated material, as hereinafter described, filled it with water, and found that none of the water would pass through the open meshes, the reason being that the weight of such water in the minutely subdivided form in which it would have to enter these meshes was insufficient to displace the air columns with which the meshes were already filled, or in other words, the water was prevented from entering these meshes or pores by atmospheric pressure, just as water is prevented from leaving a small tube when the weight of the column of water is less than the atmospheric pressure upon the superficial area at the lower end of the tube. This experiment conclusively demonstrated to me the fact that all ordinary fabrics in contact with liquids become suffused by reason of capil-

lary action and was not due to passage thereof through the meshes, and showed further, in my mind, that if this capillary action could be suspended or overcome the fabric would be practically water-proofed.

For the purpose of disclosing my invention more fully I will assume that any fabric or material of a fibrous or porous nature which has the power of capillary action is possessed of two systems of capillaries;—(1) an outside or surface system which may be brought directly into contact with the electrodes of an electrolytic bath—as I have ascertained that chemical changes in the treatment of such materials can only occur on the faces of such electrodes and not in the electrolytic solution, as is maintained by the inventors in the before-mentioned patents. (2) An inner system in which the original condition of the solution employed undergoes no chemical change through electrolytic action.

For the purpose of making the several tests, both as to the processes disclosed in the before-mentioned patents and as to my novel process, I organized the following apparatus—(1) An anode composed of a $\frac{1}{4}$ inch copper plate 14″x14″ square, interconnected at its under side by wires and a series of binding posts distributed at several points so as to insure an even distribution of the current. (2) A cathode consisting of a $\frac{1}{2}$ inch aluminum plate about 8″x8″, provided with like wiring and binding post connections for the purpose of insuring a like even distribution of the current. (3) A Weston voltmeter. (4) A Weston ammeter. (5) A variable resistance to regulate the current flow. (6) An electric generator having sufficient current generating capacity to make the tests.

Following the processes described in the before-mentioned patents, I prepared a solution consisting of 4 oz. of sulfate of aluminum dissolved in a half gallon of distilled water. The fabrics to be treated were then immersed in this solution and placed between the before-mentioned electrodes and current applied. After a long series of tests I ascertained that for silk fabrics the best results were obtained with a current strength of 45 ampere seconds, or 10 amperes applied for $4\frac{1}{2}$ seconds. The chemical reaction in this case which produces the waterproofing effects in the fabric is due to the precipitation at the cathode of hydroxid of aluminum which fills the outside or surface system of capillaries where such surface rests against the face of the electrode, and this substance is insoluble in water. The inner system of capillaries is filled with the solution alone in its original state and when the fabric is dried there is formed therein an extremely thin deposit or scale of soluble matter which is rapidly destroyed by absorption, if water be introduced through the meshes or pores of the fabric by pressure.

For the purpose of further disclosing the fact that processes of this nature are based upon a misconception of facts I covered the copper anode with two thicknesses of cotton cloth soaked in the before-mentioned solution. The fabric to be water-proofed was then immersed in the solution and laid on top of these. The aluminum cathode was then placed against the exposed surface of the fabric and the current applied. Better results were obtained in this manner than before. The fact that this precipitation takes place always at the cathode has been most clearly demonstrated by me by a very exhaustive series of tests. The results which I obtained with sulfate of aluminum were very unsatisfactory. The water-proof effects were easily broken down and most of the fabrics tested were only partially affected. Therefore, I prepared a solution of 4 oz. of sulfate of aluminum and potassium (alum) dissolved in $\frac{1}{2}$ gallon of distilled water. This produced marked improvement, but, nevertheless, I ascertained that from a dozen samples of silk fabric thus treated only two were water-proofed and as in the first case the results were easily destroyed when the material was subjected to mechanical pressure. The reason for this is apparent. The oxid which fills the surface system of capillaries may be described as being in the nature of an exceedingly thin film possessing no marked adhesive qualities.

When water is forced through the meshes or pores of a fabric, after the above treatment, the outside film is ruptured and the inside capillaries containing the dried soluble matter are leached or drained out. This same result will follow upon the use of the salts of any of the other metals described in the before-mentioned patents. After having made these tests from which I arrived at the above-named conclusions, it occurred to me that the correctness of my deductions might be arrived at by filling the inner capillary system of a fabric with a semi-insoluble substance possessing sufficient body to withstand casual attacks of moisture introduced mechanically through the meshes or pores and then sealing the surface or outer system of capillaries on one or both sides with a relatively tenacious insoluble substance produced by electro-chemical reaction, which sealing substance would remain permanently in place under practically all conditions of usage. To this end I devised my invention. I prepared a solution consisting of 2 oz. of palmate of soda (white castile soap) dissolved by boiling in $\frac{1}{2}$ gallon of distilled water. At ordinary temperatures this substance goes into solution very slowly. The cotton cloths or buffers

covering the anode were then immersed in this solution and laid smoothly in place. The fabric was also immersed in the same solution and spread on top of said buffers. A quantity of the alum solution, hereinbefore referred to, sufficient to cover it was then poured over the surface of the fabric which was immediately inclosed by the aluminum cathode plate and a current strength of 45 ampere seconds applied. Under this condition the action of electrolysis causes the palmic acid of the first solution (palmate of soda) to combine at the cathode with the aluminum contained in the second solution (alum) with the result, concerning the fabric, that the outside or surface capillaries are filled with insoluble palmate of aluminum, while the inside capillaries retain a filling of semi-insoluble matter that cannot be removed under ordinary conditions that may reasonably be anticipated. This inner filling constitutes a medium which prevents or overcomes capillary action. I ascertained that silk samples thus treated might be washed or scrubbed with tar soap, after such water-proofing treatment and after the material had dried, without in any way impairing the water-proofing efficiency.

Although I have described the preferred material for use in connection with the practice of my method or process as being palmate of soda (white castile soap) and alum, I do not limit the same to such substances; nor to the proportions of the salts which constitute the aluminum base. In fact the best results which I have obtained have been with a combination of 75 parts aluminum salts and 25 parts potassium salts, using ordinary white castile soap as before.

The essential principles of my invention lie in permeating or impregnating the interior portions of the fabric or material to be treated with a medium which tends to overcome or prevent capillary action and afterward coating the capillary surface only of either one or both sides of the fabric with a water-proof deposit—preferably electrolytic—and derived from a metallic base, and my claims are to be construed as of the most generic nature in these respects. Nor do I limit myself to the use of electrodes of copper and aluminum, as obviously various types of electrodes may be utilized and in fact I prefer in some cases to use electrodes of carbon, preferably that type of carbon known as the "Acheson graphite carbon" manufactured in Niagara Falls, N. Y.

I make no claim hereinafter to the product which results from my novel method or process, as this constitutes the subject matter of a separate application filed by me on the 25th day of February, 1908 bearing Serial No. 417,689.

Having thus described my invention what I claim and desire to secure by Letters Patent of the United States is—

1. The described method or process of water-proofing fibrous materials, consisting in permeating or impregnating the interior fibers with a substance which renders the same practically non-capillary, and afterward electrolytically effecting the coating of one or both surfaces of such material with a water-proofing substance.

2. The described method or process of water-proofing fibrous materials, consisting in permeating or impregnating the interior fibers with a substance which renders the same practically non-capillary, and afterward electrolytically effecting the deposit of a water-proofing coating, in the nature of a hydroxid, on one or both surfaces of the material.

3. The described method or process of water-proofing fibrous materials, consisting in permeating or impregnating the interior fibers with a saponaceous substance, and afterward electrolytically coating one or both surfaces of the material with a water-proofing substance.

4. The described method or process of water-proofing fibrous materials, consisting in permeating or impregnating the interior fibers with a saponaceous substance and afterward depositing palmate of aluminum upon one or both surfaces of the material.

5. The described method or process of water-proofing fibrous materials, consisting in permeating or impregnating the interior fibers with a saponaceous substance and afterward electrolytically depositing palmate of aluminum upon one or both surfaces of the material.

6. The described method or process of water-proofing fibrous materials, consisting in permeating or impregnating the interior fibers with palmate of soda and afterward coating one or both surfaces of the material with palmate of aluminum.

7. The described method or process of water-proofing fibrous materials, consisting in permeating or impregnating the interior fibers with palmate of soda and afterward electrolytically coating one or both surfaces of the material with palmate of aluminum.

In testimony whereof I have signed my name to this specification in the presence of two subscribing witnesses.

ALFRED O. TATE.

Witnesses:
C. J. KINTNER,
W. JEIN O'CONNOR.