UNITED STATES PATENT OFFICE.

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PROCESS OF WATERPROOFING.

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To all whom it may concern:

Be it known that I, WALTER ALEXANDER, a citizen of the United States, residing in the borough of Manhattan, city, county, and State of New York, have invented certain new and useful Improvements in Processes of Waterproofing, of which the following is a specification.

My invention relates to the art of waterproofing either walls and like surfaces or more particularly fabrics into the interstices and fibers of which it is desired to introduce waterproof or water-repellent material.

Such materials used in the past have been waxes or waxy acid substances or metallic stearates and other metallic "soaps" and the like.

Hitherto there have been only two known methods of applying such materials:

(*a*) The alternate saturation of the material with liquids capable of producing by mutual interaction the desired substance on or in the surface. By this method the desired material was slowly formed by successive baths, and if it were desirable, as is generally the case, to use a number of such substances in order to get the advantage of their differing mechanical properties, the number of successive baths became considerable. Thus, to produce a waterproofing that combined the hardness of stearates, the flexibility of oleates, the fixing qualities of copper salts and the rubbery feeling of the aluminum salts, at least two bathings are required for each; and if a large quantity of any one material is desired, several alternate baths are required for that material alone. Furthermore the deposition by double decomposition is apt to occur largely between the fibers and to occur irregularly in different parts of the goods of different closeness of texture.

(*b*) The metal soaps are slightly soluble in certain organic solvents, and external surfaces of walls or internal surfaces of textiles, may be coated with such solution or with rubber or wax solution and thereupon dried. But the use of such solvents involves considerable fire risk and loss of valuable solvents and in case of the metallic soaps and the like, which are only very slightly soluble in the cheap commercial solvents, it again requires a number of treatments to deposit any considerable quantity of the waterproofing material in the fibers with the danger again that the material will be very irregularly distributed.

The objects of my invention are to eliminate the necessity of using organic solvents and yet to obtain all the advantages of being able to apply a homogenous mixture of various waterproofing agents; also to overcome the objection to repeated treatments in order to apply a large amount of the desired compound or compounds; also to secure these objects without a considerable number of successive or alternating treatments. Other objects and advantages will appear below.

My invention consists in a process which preferably combines most of the following steps: combining together in aqueous suspension or solution, but in a state capable of thoroughly permeating and heavily loading itself upon and within the body to be waterproofed, of the two or more components of the waterproofing composition, namely, the acidic components and the basic components or their compounds; causing this novel fluid or substantially aqueous solution to thoroughly penetrate the body, thus applying both components simultaneously; removing the excess if any; thereupon preferably further concentrating the active components by at least partial evaporation of the water; and thereafter submitting the loaded body to the action of an agent which shall fix upon and within the body the essential waterproofing elements of the solution so that they are no longer readily soluble and capable of removal by water and exert to a greater or less extent, dependent upon their nature, a water-repellent action.

As one example of my invention, I take 114 pounds of sodium stearate (soap) and dissolve this in 300 pounds water by the aid of heat. Also I take 39 pounds of aluminum sulfate and dissolve this in 100 pounds of cold water. To the latter I add 30 pounds of bicarbonate of soda dissolved or mixed in 100 pounds of cold water. A non-gelatinous aluminum hydroxid $Al_2(OH)_6$ results. This should preferably be slightly alkaline and may, but need not, be washed to remove sodium sulfate. This liquid containing the finely suspended aluminum hydroxid $Al_2(OH)_6$ is then thoroughly mixed with the warm soap solution, and may, according to

requirements of the material, then be further reduced if desired, by the addition of water.

The resulting mixture is a cloudy, soapy solution, viscous or gelatinous when cold and fairly fluid when hot. It contains both the aluminum and the stearic required to produce aluminum stearate, but apparently not combined in the insoluble or waterproofing form or condition. This liquid is applied hot and thoroughly worked into the material, in the case of textiles, for instance, by means of a jig provided with squeeze rolls. Thereafter the excess may be removed by squeeze rolls or by scraper. The next step is to substantially dry the impregnated body with the aid of moderate heat, (212° F.) if desired. Thereupon the body is submitted to the action of a hot fixing solution, for instance $Al_2(SO_4)_3$ solution, about 10% strength, which would be expected at once to react with the soap in the superficial layer of the impregnating material, whereas it actually seems to have the effect of fixing substantially all of the stearic acid throughout the mass as aluminum stearate on or in the body in waterproof form. This action is not superficial on the mass of impregnating material but is profound throughout the body of the material. In this manner a waterproofing of any desired weight can at once be fixed upon or with the body. Next if desired as in textiles, the salts of reaction and the by-products, as Na_2SO_4 and excess of aluminum sulfate may be removed as by washing out with clean water, the material dried, and the remaining fixed waterproofing compound heat-treated, *e. g.*, ironed or calendered if desired.

It is apparent that while the wholly combined components of the waterproofing compound, *e. g.*, aluminum and stearic have been used before in dilute and relatively ineffective solution in organic solvents, I have produced new articles of commerce not hitherto available or applicable and never before made or used in that I have prepared the substantially aqueous solution or at any rate combination of the necessary components of the waterproofing compound in concentrated and reasonably staple form, adapted to constitute the compound by fixation with another aqueous fixing agent after application to the body to be waterproofed.

I believe that the metallic oxid or hydroxid precipitated in the presence of the liberated sodium carbonate is peptonized by the action of the substantially alkaline soap solution to produce the novel aqueous suspension or solution of the waterproofing compound in a condition of aqueous dispersion, and I believe that the resulting colloid permeates the fiber as well as adsorbs heavily on the surface of the fiber or other surface to be waterproofed perhaps 80 to 100 times the adsorption required by Gibbs law owing to supersaturation, gelatinization and possibly superinduced chemical reaction as indicated by Lewis, *Phil. Mag.* 1909, p. 490. I also believe that adherent thereto is formed a slimy layer of colloid material as the drying or concentration process proceeds. It is in this manner, I believe, that I am enabled to secure the far greater, more equable and more intimate penetration and covering of the surface by a truly colloid mixture than is possible by mere repeated production of the colloidal gel of aluminum stearate in a solution no matter how fully such solution is distributed between the fibers. Furthermore, I believe that on treatment with hot solution of aluminum sulfate or equivalent electrolyte, the gelatinization is made irreversible, possibly by fixation of the protective hydroxylions or other protective substance, or the final chemical change necessary to fix permanently the united aluminum and stearic acid in and upon the surface as aluminum stearate is brought about.

Certain it is that in this matter, with no more treatments or difficulty than is at present required by the single light "shower proofing," by my process I am able to apply any desired amount of any desired combination of mixed metallic and mixed organic acid soaps to secure any desired effect with greater uniformity and perfection than has hitherto been obtainable by the oft repeated separate and partial treatments. By using the acidic component of the waterproof compound in the form of a soluble salt and the basic component in the form of a peptonized hydroxid sol, I am able to apply both acidic and basic components simultaneously and far more effectively. In this way the two acidic and basic components penetrate simultaneouly and separately and are thereafter caused to react, thereby fixing both components together on the body to be waterproofed.

Not only am I able to use the usual oleic, stearic, palmitic, etc., acids, but also certain sulfonated oils like Turkey red oil, in which case the cuprammonium or other metallic hydrates, which are soluble in ammonia or other solvents, produce clear transparent jellies.

While I have cited numerous variations of the metallic substances and of the organic acid to secure different final effects and some to act as substantial equivalents, there are numerous other variations that will be apparent to any one skilled in the art of using waterproof soaps such as the application of resinates, etc., so that I do not limit the invention to the materials specifically named or to the precise proportions indicated by chemical equivalents, as in some instances, like that of the amount of soda used in the specific example, I se-

cure better results by using a slight excess of one reagent or the other.

Having thus described the preferred embodiment of my discovery and the best method now known to me of practising the same, what I desire to secure by Letters Patent, and therefore claim as new, is:

1. The art of waterproofing textile fabrics consisting in impregnating the fabric with an aqueous solution adapted to react as aluminum stearate, and then applying an aqueous solution of a fixation agent.

2. In the process of waterproofing, the improvement which consists in simultaneously applying the components of the effective waterproofing compound to be produced, said components being adapted separately to penetrate the body to be waterproofed, and thereafter causing the fixation of them together with the body to be waterproofed by treating with a fixing agent.

3. In the process of waterproofing, the improvement which consists in simultaneously applying an acidic and a basic component of the effective waterproofing composition to be produced by their reaction, said components being in substantially fluid form, and thereafter causing the fixation of them together with the body to be waterproofed, by treating with a fixing agent.

4. In the process of waterproofing, the improvement which consists in simultaneously applying the two components of the effective waterproofing compound to be produced, said components being in substantially fluid form, thereafter concentrating the active components by driving off a part of the fluid and thereafter causing the fixation of them together with the body to be waterproofed, by treating with a fixing agent.

5. In the process of waterproofing, the steps which consist in simultaneously and separately applying to the body to be waterproofed, components of the effective waterproofing compound to be produced by their interaction, the said components being in substantially fluid form and adapted to produce said waterproofing compound by their mutual action, and thereafter causing the fixation of said compound by subjecting the treated body to the action of a suitable electrolyte.

6. In the process of waterproofing a fluid-penetrable body, the steps consisting in simultaneously applying in substantially fluid form two components adapted separately to penetrate said body and to interact within the body to form a waterproofing compound and thereafter promoting the interaction.

7. In the process of waterproofing, the improvement which consists in applying the two components of the effective waterproofing compound to be produced simultaneously and in substantially fluid form, thereafter concentrating the active components and thereafter causing the fixation of them together with the body to be waterproofed by treating with a fixing agent.

8. In the process of waterproofing, the improvement which consists in applying the two components of the effective waterproofing compound adapted to react as stearate simultaneously in aqueous dispersion and in substantially fluid form and thereafter treating with a fixation agent.

9. In the process of waterproofing, the improvement which consists in applying the two components of the effective waterproofing compound adapted to react as aluminum stearate simultaneously in aqueous dispersion and in substantially fluid form, thereafter concentrating the active components and thereafter treating with a fixation agent.

10. In the process of waterproofing, the improvement which consists in applying the two components of the effective waterproofing compound one of which contains a metal adapted to produce a water-repellent metallic soap, and the other of which contains an organic acid adapted to produce with a heavy metal a water-repellent soap simultaneously in aqueous dispersion and in substantially fluid form and thereafter causing the fixation of them together with the body to be waterproofed by action of a suitable electrolyte.

11. In the process of waterproofing, the improvement which consists in applying the two components of the effective waterproofing compound to be produced simultaneously and in substantially fluid form and thereafter treating with a fixation agent, removing by-product salts and heat-treating the compound.

12. In the process of waterproofing, the improvement which consists in applying the two components of the effective waterproofing compound to be produced simultaneously and in substantially fluid form, thereafter concentrating the active components and thereafter treating said components together with the body to be waterproofed with a fixing agent, removing by-product salts and heat-treating the compound.

13. In the process of waterproofing, the improvement which consists in applying the two components of the effective waterproofing compound to be produced simultaneously and in substantially fluid form and thereafter fixing both components together on the body to be waterproofed by treating with a fixing agent, removing by-product salts and heat-treating the compound.

14. In the process of waterproofing, the improvement which consists in applying the two components of the effective waterproofing compound to be produced simultaneously and in substantially fluid form, there-

after concentrating the active components and thereafter causing the fixation of them together with the body to be waterproofed by treating with a fixing agent, removing by-product salts and heat-treating the compound.

15. In the process of waterproofing, the improvement which consists in applying the two components of the effective waterproofing compound to be produced simultaneously in aqueous dispersion and in substantially fluid form and thereafter treating them with a fixing agent together with the body to be waterproofed, removing saline impurities and heat-treating the compound.

16. The process of waterproofing which consists in treating the materials to be waterproofed simultaneously with the acidic and basic constituents of the desired waterproofing material, both being substantially in aqueous solution and treating them together upon the material with a fixing agent.

17. The method of waterproofing a porous body which consists in simultaneously impregnating same with acidic and basic components adapted to penetrate said body separately and to react thereon as a waterproofing compound, the said acidic component being in the form of a soluble salt and said basic component being in the form of a peptonized hydroxid sol.

18. The method of waterproofing a fluid-permeable body which consists in impregnating same with a soluble salt and simultaneously with a peptonized hydroxid sol under conditions capable of producing by synthesis a waterproofing reaction product, and causing same to react within and on the said body to yield said product.

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