106-81. OR 970,643 EX 1,011 970,643 2. Silicate

STATES PATENT OFFICE.

Wood pulp + grease or wax - World articles
Bake at 240-400° F. partly carbonize (steam)
Dip in impregnate with Na_2SiO_3 or K_2SiO_3 and heat

ISAIAH L. ROBERTS, OF LOCKPORT, NEW YORK.

OIL OR GREASE RESISTANT ARTICLE.

970,643. Specification of Letters Patent. Patented Sept. 20, 1910.

No Drawing. Application filed May 6, 1909. Serial No. 494,464.

Wood pulp

same as 921,002

To all whom it may concern:

Be it known that I, ISAIAH L. ROBERTS, a citizen of the United States, residing at Lockport, in the county of Niagara and State of New York, have invented certain new and useful Improvements in Oil or Grease Resistant Articles, of which the following is a full, clear, and exact description.

This invention relates to articles for example tubes, rods, vessels, plates, sheets, etc., composed of fibrous material such as wood-pulp, strawboard, paper, cotton, and other cellulose products, and has for its object to provide an inexpensive article of the material referred to which shall be resistant to water, oil or grease, and which shall also be non-inflammable. Of the various uses to which articles having these properties may be put, I may mention in particular the case of shipping or storing vessels for containing lard or other meat-products, oil, both organic and inorganic, and other substances of a greasy character. For such purpose vessels made according to my invention are specially adapted, being light in weight, effectively resistant to the material which they contain, and strong enough to withstand the usage of shipment.

In carrying out my invention in the preferred manner I first make a water-repellent article according to the method set forth in my co-pending application Serial No. 435,130, filed May 26, 1908, now Patent No. 921,001, issued May 11, 1909 which method involves heating the article, preferably with the addition of oil, wax, or other water-repellent material in suitable amount. The most important feature of the process described in my said co-pending application resides in the discovery that on heating an article composed of fibrous cellulose the fibers of which have some water-repellent substance or substances, such as oil or paraffin, well distributed on and among them or naturally in them, in minute or attenuated quantities, so minute in fact as not to be palpable to the touch or visible to the eye, the article will repel water, acting in this respect somewhat like the feathers of a fowl. The treatment with the water-repellent substance or substances can be effected in various ways and with various materials, but the organic oils appear to give the best results. For example, in the case of wood-pulp, strawboard, etc., the oil or powdered wax may be put into the pulping machines and thoroughly mixed with the fibers. This method applies both to the original pulping and to the re-pulping of pulp-board, strawboard, etc. In the case of articles made wholly or in part of re-pulped printed paper, I have found that the oil of the printer's ink is usually sufficient for the purpose desired; but if not, additional water-repellent oil or wax may be used.

From the material treated as above or in any equivalent manner, the vessels or other articles or products are made up into the desired form by the usual methods, and are then heated. The heating is preferably effected in a closed or partially closed chamber or oven, and is carried to a temperature above that of boiling water to a point sufficient to cause the wax or oil to be thoroughly incorporated with the fibers. The temperature required for the purpose depends upon the nature of the material and the article, but in no case have I found it necessary to go much above 400° F. and in many cases a temperature as low as 240° F. has been found sufficient. The length of time during which the heating should be maintained also varies, but in general from two to eighteen hours will be found sufficient. At the lower temperature it is necessary to heat the article longer than at the higher temperature; and, similarly, thick-walled articles need longer heating at any given temperature than do those whose walls are thinner. In order to be sure of the results, the heating must be carried on for a period and at a temperature sufficient to partially decompose the fibers of the article and effect a combination of the oils, gums or waxes therewith. At temperatures between the boiling point of water and about 400° F. the heating has the effect of strengthening the articles, but at higher temperatures they are apt to be weakened. While the use of oil or other water-repellent substance is believed to be necessary for the best results, I have found that simple heating of articles made of wood-pulp, etc., will give them water-repellent properties to a marked degree,—sufficient, at least, to serve many purposes where substantially waterproof articles are desired, as for example receptacles for the storage or shipment of merchandise. Such articles, made of material of the kind referred to and heated to a temperature of from 240° to 400° F. for from two to eighteen hours or longer (the higher the

temperature the shorter the time, and vice versa), will maintain their contents dry for a long time even under adverse conditions of atmosphere or weather. If water-vapor be added to the atmosphere in the oven in which the heating is effected the results in some cases will be improved. For this purpose an open vessel containing a small quantity of water may be placed in the oven or steam may be injected into the latter. The use of water-vapor as described does not result in making the article moist or damp, since the temperature is above the condensing point of water.

A water-repellent article having been made in the manner described above, the next step is to treat the article with a solution of sodium or potassium silicate, preferably in the manner set forth in my co-pending application Serial No. 436,650, filed June 4, 1908, now Patent No. 921,002, issued May 11, 1909. The object of this treatment is to impregnate the article with the silicate, the extent of the necessary impregnation being variable within rather wide limits. Thus the impregnation need not be carried farther than well below the surface, or the article may be thoroughly permeated. The preferred manner of effecting the impregnation is to submerge the article in an aqueous solution of the silicate, the said solution having a specific gravity preferably of from 20 to 25 degrees Baumé, though the specific gravity may be somewhat higher or lower without unsatisfactory results. But when the solution is more dilute than necessary to penetrate the article easily, an excess of water is absorbed which must be got rid of afterward, thus lengthening the subsequent drying of the article. If the strength of the solution is such that the bath is of a thick or viscous character it penetrates slowly and forms small lumps or nodules on the surface of the article when dry. It is better to have the solution hot when used for the reason that the time of submergence is shortened, since the solution is less viscid, and the heat acts in some way to make the fibers of the article more receptive.

After treatment with the silicate solution the article is dried, and is then ready for use. I have found that more satisfactory results are obtained by rapid drying in heated air, the reason being that when the article is wet the alkali in the absorbed solution takes up and combines with any carbonic acid gas that comes in contact with it, with the consequent production of carbonates, which impair the resistant properties of the article and also produce discoloration.

As previously stated, the extent to which the impregnation must be carried to make the article satisfactorily resistant, may be varied considerably. In some cases it is desirable not to have the solution permeate the entire article. For example, pails, tubs, and other thick and substantial articles need not be impregnated far below the surface, as this makes them sufficiently resistant to water, oil, grease, and fire, without adding the unnecessary weight which would result if the article were saturated with the solution. In fact I have found that for water-resistant articles the impregnation should not be much more than a surface treatment, so as to leave an inner unimpregnated core, so to speak. This is for the reason that the silicate is soluble, though of course slowly, in water; and hence if the article were wholly permeated the water might eventually make its way through the article by dissolving the silicate. But the unimpregnated core effectually stops the water from penetrating through the article even if the silicate in the surface portions be quite dissolved.

No exact rule can be given for the time of submergence in the silicate treatment, as much depends upon the texture and nature of the article, and also upon the temperature and strength of the solution; but if the specific gravity of the latter is about 20° or 25° Baumé, and the temperature of the bath about 180° F., a few minutes submergence will ordinarily be sufficient for good results. For example, a wood-pulp pail, with walls of the usual thickness, will be impregnated sufficiently by one minute's treatment. In any case, however, the time of submergence in the silicate solution, and the specific gravity and temperature of the latter, can readily be determined by trial if necessary.

Articles made as above described will be found admirably adapted for the purposes previously mentioned, for example as shipping or storing vessels for containing oil, both organic and inorganic, lard, and other substances of a greasy character. At the same time the articles are not only thoroughly resistant to the materials named, but are also light in weight and strong enough to withstand rough usage.

What I claim is:

1. A water and oil or grease-resistant article composed of fibrous cellulose, the article being partially carbonized and impregnated with an alkaline silicate.

2. A water and oil or grease-resistant article composed of fibrous cellulose partially carbonized and impregnated with sodium silicate.

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

ISAIAH L. ROBERTS.

Witnesses:

M. LAWSON DYER,

STURGES S. DUNHAM.

H/C