UNITED STATES PATENT OFFICE.

ISAIAH L. ROBERTS, OF LOCKPORT, NEW YORK.

METHOD OF MAKING FIRE, WATER, AND OIL RESISTANT ARTICLES.

No. 921,002. Specification of Letters Patent. Patented May 11, 1909.

Application filed June 4, 1908. Serial No. 436,650.

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 a certain new and useful Method of Making Fire, Water, and Oil Resistant Articles, of which the following is a specification.

My invention relates to the manufacture of 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 a simple, effective, and inexpensive process of making articles of the material referred to which shall be resistant to water, oil or grease, and also 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 the vessels made by my process are specially adapted, being light in weight, thoroughly resistant to the material which they contain, and strong enough to withstand the rough 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, which involves heating the article, preferably in an atmosphere containing the vapor or oil, wax or other water-repellent material. The most important feature of the invention set forth 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 birds. The treatment with the water-repellent substance 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 original pulping or to the repulping of pulpboard, strawboard, etc. In the case of articles made wholly or in part of repulped 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 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 oil or wax 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 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 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 to 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 qualities to a marked degree,—sufficient, at least, to serve many purposes where substantially water-proof 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. Where articles already formed are to be waterproofed by the plan involving the use of water-repellent oils or waxes the desired incorporation of the oil or wax with the fiber is effected by heating the article in an atmosphere containing the vapor of the oil or wax. This may be done by placing in the oven in which the article is heated a vessel containing a small quantity of the water-repellent substance or substances, whereupon the latter are volatilized and caused to penetrate the pores or interstices of the article. If water-vapor is added to the atmosphere in the oven it will in some cases improve the results. 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 oven. The use of water-vapor as described does not result in making the articles moist or damp, since the temperature is above the condensing point of water.

A water-repellent article having been made in accordance with the method just described, the next step in my present process is to treat the article with a solution of sodium or potassium silicate. 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 nature it penetrates slowly and forms small lumps or nodules on the surface of the article when dried.

I prefer 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 a silicate solution the article is dried, and is then ready for use. I have found that better 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 at most, 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 say twenty to twenty-five degrees, 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 wall of the usual thickness, will be impregnated sufficiently in one minute's treatment. In any case, however, the time of submergence in the silicate solution, and the specific gravity and temperature of the same, can readily be ascertained by trial if necessary.

What I claim is:

1. The herein-described method of making water and oil or grease-resistant articles, which consists in forming an article of fibrous cellulose, heating the same to render it water-resistant, impregnating the article with an aqueous solution of an alkaline silicate, and then drying the article.

2. The herein-described method of making water and oil or grease-resistant articles, which consists in forming the article of fibrous cellulose, waterproofing the article as described, impregnating the article with an aqueous solution of an alkaline silicate, and then drying the article.

3 The herein-described method of making water, oil and grease resistant articles, which consists in forming an article of fibrous cellulose, rendering the same water-resistant

as described, impregnating the article with a silicate of an alkali metal by submergence in an aqueous solution of the silicate, and then drying the article.

4. The herein described method of making water, oil and grease-resistant articles, which consists in forming an article of fibrous cellulose, rendering the same water-resistant as described, impregnating the article with an aqueous solution of an alkaline silicate, and then subjecting the impregnated article to rapid drying.

5. The herein-described method of making water, oil and grease-resistant articles, which consists in forming an article of fibrous cellulose, rendering the same water-resistant as described, impregnating the article by submerging it in a hot aqueous solution of an alkaline silicate, and then drying the article.

6. The herein described method of making water, oil and grease-resistant articles, which consists in forming an article of fibrous cellulose, rendering the same water-resistant as described, impregnating the article with a hot aqueous solution of an alkaline silicate, and then subjecting the article to rapid drying.

ISAIAH L. ROBERTS.

Witnesses:
M. J. NELLIGAN,
WM. DIRFENBACH.