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MILDEW RESISTANT TEXTILE PRODUCTS AND PROCESSES FOR PREPARING THEM

Arthur C. Weick, Bronxville, N. Y., assignor to Pacific Mills, Boston, Mass., a corporation of Massachusetts

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This invention relates to the protection of textile materials against mildew, rot and similar bacteria and fungi, and to textile products which are so treated as to be highly resistant to mildew and other destructive fungi and bacteria without having appreciable toxic effects upon intimate contact with a person's skin.

Much work has been done on anti-mildew treatments for various textile materials but the big problem has been to find a process or material that will have a more or less permanent effect and that is not toxic or irritating when kept in direct and close contact with a person's skin for a considerable time. Numerous copper compounds have been tried for this purpose with more or less success. Some of these are very effective for mildew proofing if the textile materials are impregnated with a fairly high concentration of the copper compound. However, such high concentrations are expensive to use and are apt to cause irritation upon contact with a person's skin. Furthermore, the only known way of applying them heretofore has been in solution in one or more inflammable solvents which involves a considerable fire hazard.

It is an object of this invention to provide a process of treating textiles with organic copper compounds such as copper-8-quinolinolate to produce adequate impregnation of the textile materials without using dangerous amounts of highly inflammable solvents or other materials.

Another object of the invention is to impregnate textile materials with a combination of such a copper compound and an organic mercury compound which combination makes possible a substantial reduction of the amount of copper employed without sacrificing the resistance of the product to mildew or other fungi and bacteria, and at the same time reduces or eliminates any tendency of the impregnated textile material to cause irritation upon contact with a person's skin.

These and other objects of my invention are accomplished by treating the textile material with a solubilized form of copper-8-quinolinolate or similar type of oil soluble organic copper compound applied to the textile in the form of an oil-in-water emulsion or dispersion. After the cloth or other textile material is thoroughly impregnated with such an emulsion or dispersion, it is washed to remove excess compound. For this purpose it may be treated in a machine known as a "soaper" in which the cloth is subjected to washing and thorough rinsing. If desired, the cloth may then be further treated with a suitable organic mercury compound applied in solution or in the form of an emulsion to supplement the action of the copper compound. If needed or desired, a stiffening agent, such as polyvinyl acetate, methyl cellulose, carboxymethyl cellulose, starch, polyvinyl chloride, various melamine resins, or the like, can be applied to the cloth along with the mercury compound. The textile material or cloth may be finished in any desirable fashion as by hot or cold calendering.

The following description of a preferred form of processing textile materials in accordance with my invention is given with the understanding that the details of the process are not limited to this example but may be varied in various ways as will be explained and understood by those skilled in the art.

Any of numerous kinds of cloth or other textile materials can be impregnated or treated according to my process, but I have found it particularly valuable in preparing a shoe lining material to start with a grey shelter

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tent duck. A 9 ounce duck is suitable although other weights and types of cloth may, of course, be used. If the finished material is to be brought into intimate contact with a person's skin, it is better to use a cloth which is unbleached and does not contain any residual chemical that might oxidize or otherwise react with the copper compound.

The cloth selected is first put through a padder containing an emulsion of the copper compound. This emulsion may be prepared in a copper kettle provided with a large beater or agitator. Thirty parts of water are brought to a boil in a kettle and 1 part of morpholine and 1 part of monoethanolamine then added. The beater is started and 10 parts of the solubilized copper-8-quinolinolate dissolved in a suitable animal glyceride such as a hydrogenated fish oil are added with constant agitation. The temperature of the liquid is preferably maintained between 150–190° F. until the oil solution of the copper compound is completely melted and dispersed. Fifty-five parts water and 3 parts of "Triton NE" (a 33⅓% water solution of iso-octyl phenyl ether of polyethylene glycol) heated to 150° F. is then added to the contents of the kettle with continued agitation until a uniform emulsion is obtained.

In going through the padder containing this emulsion, the pickup of emulsion is about 25–50% based on the weight of the dry cloth. The cloth thus impregnated is then dried on a tenter frame or in some other suitable manner and the dried cloth is washed or soaped off with a detergent such as soap and soda ash on a continuous soaper, a machine that combines washing and thorough rinsing.

Soaping and rinsing at this stage not only removes any of the copper compound that might clog but also makes the produce safer for contact with the skin so far as toxicity is concerned, removes excess fatty oil and other extraneous materials to make possible a greater pickup in subsequent treatments, and also opens up the fabric somewhat so as to improve its "breathing" ability. Surprisingly enough, cloth which has been impregnated in this manner and washed or soaped actually has a higher tensile strength after a soil burial test than cloth which has been impregnated but not soaped and rinsed.

The cloth impregnated with the copper-8-quinolinolate is then padded through a finish mix or dispersion consisting of 10% of a polyvinyl acetate emulsion, about 0.08% of phenyl mercuric triethanol ammonium lactate, 3% of "Triton NE" and the balance water.

The mercury compound is preferably one with a fairly neutral pH which has been found to be best from the toxicity point of view.

The cloth is then dried on a tenter frame or in some other suitable manner and is calendered. A single pass cold calendering has been found particularly satisfactory, although hot calendering may be used if desired.

I have found that it is important in this connection to use copper and mercury compounds which do not release the copper or mercury in metallic form. In this connection, it may be possible to use other organic copper or mercury compounds than the particular ones mentioned. For example, a copper compound known as copper-3-phenyl salicylate may be substituted for the copper-8-quinolinolate.

It was found that the grey duck impregnated according to the above example contained copper-8-quinolinolate equivalent to about 0.025% copper and mercury compounds in an amount equivalent to about 0.02% mercury, was fully as resistant to the effect from bacteria or fungi as the same cloth impregnated with a considerably larger amount of copper-8-quinolinolate and no mercury compound. The cloth containing the combination of the two compounds applied as described above, however, was not only less expensive to produce but also was found to be less toxic in contact with a person's skin than cloth impregnated with larger amounts of the copper compound.

In general, in order to achieve the results of this invention it is desirable to impregnate the cloth with an amount of the water insoluble organic copper compound equivalent to from about 0.01% to 0.06% copper. The mercury compound employed should be used in quantity

to produce an amount held by the cloth equivalent to about 0.01% to about 0.03% of mercury by weight. The mercury compound apparently undergoes chemical changes so that after the cloth has been calendered, most of the mercury left in the cloth in insoluble form is diphenyl mercury and mercurous acetate together with a small amount of mercurous carbonate.

While not essential, it is highly desirable to employ a stiffening agent of some kind, such as the polyvinyl acetate resin employed in the example given. This improves the hand of the cloth and also adds to the permanence of the impregnated materials. Other heat convertible resins or even other stiffening agents such as starch can be used in place of the polyvinyl acetate although this particular resin combined with copper-8-quinolinolate and phenyl mercuric triethanol ammonium lactate has been found to be especially satisfactory from the toxicity viewpoint.

If the cloth is to be used as shoe linings or for other purposes that may involve intimate contact with a person's skin, it is important to soap, rinse or otherwise wash out excess chemicals either after the first impregnation with the copper compound or after impregnation with both copper and mercury compounds has been completed.

I claim:

1. A mildew proof textile product adaptable for use as a shoe lining that is substantially non-toxic in contact with the skin comprising textile material impregnated with small amounts of copper-8-quinolinolate and phenyl mercuric triethanol ammonium lactate, the amounts of said compounds being equivalent to about 0.01% to about 0.06% of copper and to about 0.01% to about 0.03% mercury by weight.

2. A mildew proof textile product adaptable for use as a shoe lining that is substantially non-toxic in contact with the skin comprising textile material impregnated with small amounts of copper-8-quinolinolate and phenyl mercuric triethanol ammonium lactate.

3. A woven textile fabric adaptable for use as a shoe lining having a high resistance to bacteria and fungi imparted by resin bonded impregnation with copper-8-quinolinolate in an amount equivalent to not more than about 0.06% of copper and phenyl mercuric triethanol ammonium lactate in an amount equivalent to not more than about 0.03% of mercury by weight.

4. A process of mildew proofing a woven textile fabric adaptable for use as a shoe lining which comprises impregnating the fabric with an oil solution of copper-8-quinolinolate, soaping and rinsing the fabric, and im-

pregnating the fabric with phenyl mercuric triethanol ammonium lactate.

5. A process of mildew proofing a woven textile fabric adaptable for use as a shoe lining which comprises impregnating the fabric with an aqueous emulsion of an oil solution of copper-8-quinolinolate, soaping and rinsing the fabric, and impregnating the fabric with a resin bonding agent and phenyl mercuric triethanol ammonium lactate.

6. A process of mildew proofing a woven textile fabric adaptable for use as a shoe lining which comprises impregnating the fabric with an aqueous emulsion of an oil solution of copper-8-quinolinolate, soaping and rinsing the fabric, and impregnating the fabric with an emulsion containing polyvinyl acetate and phenyl mercuric triethanol ammonium lactate.

7. A process of treating a woven textile fabric adaptable for use as a shoe lining which comprises impregnating the fabric with an aqueous dispersion of an oil solution of copper-8-quinolinolate, soaping and rinsing the impregnated fabric, impregnating the washed fabric with a dispersion containing phenyl mercuric triethanol ammonium lactate and an organic stiffening agent, drying the fabric and calendering the impregnated dried fabric.

8. A mildew proof textile product adaptable for use as a shoe lining that is substantially non-toxic in contact with the skin comprising textile material impregnated with small amounts of an oil-in-water emulsion of an oil solution of copper-8-quinolinolate, and phenyl mercuric triethanol ammonium lactate.

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