METHOD OF IMPROVING THE FIRE-RESISTANCE OF REGENERATED CELLULOSE FABRICS


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This invention relates to the treatment of textile materials and, in particular, to a method of decreasing the inflammability of materials, especially textile materials, having a basis of cellulose (including regenerated cellulose).

Many unsuccessful attempts have been made to provide a satisfactory process for decreasing the inflammability of textile materials of the kind referred to above. One difficulty has been that most of the substances found effective in reducing the inflammability of the materials are water-soluble. Materials impregnated with such substances are, in consequence, not suitable for use under wet conditions. A difficulty which may be even more serious is loss of tenacity caused by the action of the fire-proofing substances on the material.

According to the present invention the fire-resistance of materials, especially textile materials, comprising cellulose, including regenerated cellulose, is improved by depositing a water-insoluble oxalate, especially calcium oxalate, within the material. By a water-insoluble oxalate is meant one of solubility less than 0.01 gram per 100 ml. of water at 20°C. The water-insoluble oxalate is preferably deposited in the material by reaction in the material between oxalate ions and the ions of an appropriate metal, i.e. a metal the oxalate of which is insoluble. Thus in the preferred form of the invention the material is impregnated with two reagents one after the other, one being an aqueous solution of a water-soluble normal oxalate, e.g. disodium oxalate, dipotassium oxalate or diammonium oxalate, and the other being an aqueous solution of a normal salt of a metal the oxalate of which is water-insoluble, e.g. calcium chloride, calcium acetate or barium chloride. The term “normal salt” as used herein means a salt the acid radicle of which does not contain any ionisable hydrogen atom. “Water soluble” means of solubility at least 2 grams per 100 ml. of water at 20°C. The two reagents interact in the material to form the insoluble oxalate and the material is dried. The impregnation should preferably be so effected that equivalent proportions of the two reagents are absorbed. In selecting the two reagents it is of course necessary to exclude reagents having a damaging effect on the materials as well as reagents that in reacting would give rise to by-products having a damaging effect. A suitable weight of calcium oxalate is 2 to 5% by weight of the fabric.

By the process of the invention the fire-resistance of the material treated is greatly improved without substantial reduction in tenacity; the tenacity may even show a slight increase. By the process of the invention fabrics showing a greatly enhanced adhesion to rubber have been obtained. The process of the invention is, therefore, very suitable for the treatment of fabric for use in the construction of rubber-fabric conveyor belts intended for operation where there is a fire hazard, e.g. in coal mines.

The following examples illustrate the invention:

Example I

The fabric treated was a belting duck woven from continuous filaments regenerated cellulose yarn of tenacity 4.2 grams per denier in both weft and warp. The weight of the fabric was 20 oz. per sq. yard.

The fabric was padded with a 5% (by weight) aqueous solution of sodium oxalate at 30–40°C, the expression being 100%, and was batched for 30 minutes. The fabric was then worked for 30 minutes on a jig in an aqueous bath containing 4.14 parts by weight of calcium chloride per 100 parts by weight in untreated fabric, the bath volume being 5:1. The fabric was drained and dried.

Samples removed from the fabric showed a slightly improved tenacity in both weft and warp and a greatly improved fire-resistance. Great difficulty was experienced in burning the fabric by exposure to a flame.

The following example shows the improved adhesion to rubber obtainable by the pre-treatment of the invention:

Example II

Two layers of the fabric of improved fire-resistance of Example I were bonded to an intermediate layer of a rubber mix in a press during vulcanising of the rubber at 141°C, for 30 minutes under a pressure of 0.5 ton per sq. in. The rubber mix had the following composition by weight:

100 parts of crepe rubber pre-masticated for 30 minutes
1 part of phenyl-β-naphthylamine
3 parts of sulphur
1 part of stearic acid
25 parts of dry zinc oxide
1.2 parts of benzothiazol disulphide.

The thickness of the rubber between the layers of the fabric was 0.037 inch. In the same way a “blank” was made from two layers of the fabric which had not been given the calcium oxalate pre-treatment. Stripping tests were carried out on samples of both materials. It was found that the force required to strip apart the layers of the sample made from calcium oxalate pre-treated fabric was approximately twice that required to strip apart the layers of fabric that had not had that pre-treatment.

The following example illustrates the making of a fabric/elastomer web suitable for use as heavy duty conveyor belting, according to the invention.

Example III

A fabric of improved fire-resistance obtained as described in Example I was rubberized by friction calendering with the composition specified in Example II. Four layers of the rubberised fabric were assembled in the usual way and a cover 1/4" thick was built round the assembly from calendered sheet of the said composition. After perforating the assembly in the usual way vulcanisation was effected in a belting press.

Similarly fabric/elastomer webs, especially webs suitable for use as belting can be made by bonding together alternate layers of the pre-treated fabrics, especially woven fabrics containing at least a major proportion (i.e. 50 or more parts by weight) of regenerated cellulose yarns of tenacity at least 4 grams per denier and carrying a deposit of calcium oxalate, and of an elastomer other than natural rubber. Such other elastomers may, for example, comprise a synthetic rubber such as polychloroprene or a copolymer of butadiene or isoprene with a minor proportion of styrene or acrylonitrile or with isobutylene, or may comprise a plastisised polyvinyl chloride or copolymer of vinyl chloride with a minor proportion
of another mono-olefinic monomer, such as vinyl acetate, vinylidene chloride or acrylonitrile. By a pre-treatment according to the invention, i.e., the deposition of water-insoluble oxalate in the materials, the adhesion between materials of such high tenacity regenerated cellulose and an elastomer can be substantially improved besides reducing the inflammability of the fabric. Preferably an elastomer having olefinic unsaturation such as natural rubber or the diene polymers and copolymers is used, the elastomer is applied in the form of a mix containing vulcanising ingredients and the assembly of alternate layers of pre-treated fabric and elastomer mix is subjected to heat and pressure under conditions in which vulcanisation of the elastomer occurs. Elastomers which are free from olefinic unsaturation, e.g., plasticised polyvinyl chloride, may be bonded together under such conditions of heat and pressure that the elastomer is temporarily rendered fluid. Such elastomers may be applied in the form of dispersions in a plasticiser, the elastomer being gelled by the action of heat during the bonding process. Volatile liquids may also be employed to produce the desired temporary fluidity in these elastomers.

The invention has been described with particular reference to the treatment of conveyor belting fabric made from high tenacity continuous filament regenerated cellulose yarn. It is generally applicable, however, to the treatment of textile materials, e.g., in the form of staple fibres, yarns or woven or knitted fabric, made of or containing natural cellulose, e.g., cotton, linen or jute or regenerated cellulose. The textile materials may, for example, comprise continuous filaments or staple fibres made by the viscose process or the cuprammonium process. Preferably, however, the materials comprise continuous filaments of regenerated cellulose made by the complete saponification of high tenacity cellulose acetate filaments. Such high tenacity cellulose acetate filaments can be obtained by stretching cellulose acetate filaments of normal tenacity in steam, hot water or an organic stretch-assisting agent, or by wet spinning cellulose acetate yarn and drawing off the yarn at a sufficiently high speed relative to the speed of extrusion to give the desired high tenacity.

Having described our invention, what we desire to secure by Letters Patent is:

1. The process which comprises improving the fire-resistance of a fabric containing at least 50% by weight of regenerated cellulose of tenacity at least 4 grams per denier by impregnating the fabric with two reagents one after the other, one being an aqueous solution of a normal water-soluble oxalate and the other being an aqueous solution of a normal salt of calcium, allowing the reagents to react in the fabric and drying the fabric.

2. The process which comprises improving the fire-resistance of a fabric containing at least 50% by weight of regenerated cellulose of tenacity at least 4 grams per denier by impregnating the fabric with two reagents one after the other, one being an aqueous solution of a normal salt of calcium, allowing the reagents to react in the fabric and drying the fabric.

3. A woven fabric containing at least 50% by weight of regenerated cellulose of tenacity at least 4 grams per denier, said fabric being of improved fire-resistance owing to the presence therein of 2 to 3%, based on the weight of the fabric, of calcium oxalate.

4. A flexible laminate that comprises at least one layer of a textile fabric bonded on each face to a layer of an elastomer, said fabric being composed substantially of cellulose and being of improved fire-resistance owing to the presence therein of 2 to 3%, based on the weight of the fabric, of calcium oxalate.

5. A flexible laminate that comprises at least one layer of a textile fabric bonded on each face to a layer of an elastomer, said fabric being composed substantially of high-tenacity regenerated cellulose and being of improved fire-resistance owing to the presence therein of 2 to 3%, based on the weight of the fabric, of calcium oxalate.

6. A flexible laminate that comprises at least one layer of a textile fabric bonded on each face to a layer of a vulcanized rubber, said fabric being composed substantially of cellulose and being of improved fire-resistance owing to the presence therein of 2 to 3%, based on the weight of the fabric, of calcium oxalate.

7. A flexible laminate that comprises at least one layer of a textile fabric bonded on each face to a layer of a vulcanized rubber, said fabric being composed substantially of high-tenacity regenerated cellulose and being of improved fire-resistance owing to the presence therein of 2 to 3%, based on the weight of the fabric, of calcium oxalate.

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