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FLAME-RESISTANT TEXTILE FABRICS

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ABSTRACT OF THE DISCLOSURE

A flame-resistant textile article comprising filaments, staple fibers or yarns from natural and/or synthetic material and a homogeneous mixture consisting of a halogen phosphoric acid esters, a phosphorus-containing plasticizing agent, a polyalkyl halide, antimony oxide, and a polyurethane.

This is a continuation of Ser. No. 776,773, filed Nov. 18, 1968, now abandoned.

The present invention relates to flame-resistant textile material and to a method for flameproofing textile fabrics, e.g. floor coverings, upholstery cloth, wall coverings, curtaining material, and industrial textile material of synthetic, regenerated and natural fibers, as well as mixtures thereof, which contain mixtures of phosphorus-containing compounds, polyalkyl halides, antimony compounds, and polyurethanes as flameproofing agents.

The use of many compounds for flame-resistant synthetic moulded materials of fibers and filaments has already been proposed. For example, halogen-containing polymers or/and polymers with antimony trioxide additions as well as salts of phosphoric acid have been used for this purpose. These known products do not, however, satisfy all the requirements which arise in connection with the flameproofing of textiles. The use of flameproofing agents must not affect detrimentally the physical properties of the textiles, in particular their mechanical strength. The texture and the appearance of the goods, e.g. the suppleness and pliability of the handle is not to be impaired. Moreover, the use of flameproofing agents must not increase the affinity of the textile to dirt or produce a superficially visible covering, and the finish they produce must be resistant to washing, cleaning, in particular using vacuum cleaners, shampooing, abrasion, vacuum cleaning and aging.

The object of the present invention is to provide textile materials, in particular upholstery cloth, floor coverings, heavy curtaining material, tent cloth, fleeces and industrial fabrics being efficiently flameproofed, with a homogeneous mixture of halogen-containing phosphoric acid esters, phosphorus-containing plasticizers, polyalkyl halides, antimony oxides, and polyurethanes. The surprisingly good flameproofed effects obtained with these mixtures result from the synergistical properties of the individual components and cannot be achieved by using the individual compounds alone or are achieved by the individual compounds alone to a minor degree only. At least one surface of the textiles to be treated, e.g. the reverse side, is coated or finished with the homogeneous mixtures according to the invention. The homogeneous mixture can also be applied as an intermediate coating by the application of a lining fabric, subsequently to the doctor blade finishing procedure and can thus simultaneously serve as a lining material or adhesive. Another advantage of the homogeneous mixtures according to the invention when used as flameproofing agents resides in improved rebound properties of the textile fabrics treated therewith and, in connection therewith, a lower tendency of forming creases and wrinkles during processing and when worn.

The flameproofing agents to be used according to the invention are applied in quantities between 5 and 150% by weight, preferably 40 to 70%, based on the weight of the textile fabric to be treated. The quantities to be applied are substantially lower than the quantities conventionally used for obtaining normal coatings, e.g. for awning cloth and waterproof cloth. The textiles being treated according to the invention are permeable to steam and air. The individual components are preferably employed in quantities (phosphoric acid compound:polyalkyl halide:antimony oxide:polyurethane) of 1:0.2:0.05:0.1 to 1:1:0.5:1.

As halogen-phosphoric acid esters tris-halogenalkylphosphate are used. Suitable phosphorus-containing plasticizing agents are triaryl phosphate or trialkyl phosphate. Stabilizing agents for polyvinylchloride such as triphenyl phosphite or diphenyl thiourea may be added.

Examples of phosphorus-containing compounds which are effective both as flame-inhibiting agents and as plasticizers are phosphoric acid esters, such as tritoluyl phosphate, tricresyl phosphate, triphenyl phosphate, diphenylcresyl phosphate, trioctyl phosphate, diphenyloctyl phosphate, tris-(chloroethyl)-phosphate, tris-(dichloroethyl)-phosphate, tris-(dichloropropyl) - phosphate, tris - (dichlorobutyl)-phosphate, tris-(bromoethyl)-phosphate, tris-(dibromoethyl)-phosphate, tris-(dibromopropyl) - phosphate, bis-(dichloropropyl)-propylphosphate, and mixtures thereof.

Examples of polyalkyl halides are polyvinyl chloride and polyvinyl bromide compounds of the molecular weight in the range of 30,000 to 150,000. The products must be capable of being processed in the form of pastes and must contain at least 25 to 30% by weight of a plasticizer. Copolymers thereof, e.g. with vinylacetate, vinylidenechloride, maleic and fumaric acid esters, styrene, acrylic esters and acrylonitrile can also be used.

Suitable polyurethanes for use according to the invention are compounds which are prepared from aliphatic and aromatic polyesters and/or polyethers having a molecular weight of about 500 to 10,000 with aliphatic and aromatic polyisocyanates. Examples of suitable polyisocyanates are toluylene-diisocyanate-2,4, toluylene-diisocyanate-2,6 or mixtures thereof, hexamethylene-diisocyanate-1,6 and the triisocyanate of one mol of trimethylolpropane and three mols of toluylene-diisocyanate-2,4. Suitable polyesters and polyethers are polyesters of adipic acid/hexanediol-1,6 having a molecular weight of about 2,000 or of terephthalic acid/ethyleneglycol or/and polypropylene oxide ether. The polyurethanes used as prepolymers being prepared by reaction of a polyester and/or polyether with a polyisocyanate in a molar ratio of 1:1. Reaction products from ethanolamines and isocyanates, e.g. from N-methyldiethanolamine and phenylisocyanate, are used as catalysts. They are added to the coating material together with polyisocyanates, the ratio of the NH groups of the polyurethanes to the NCO groups of the polyisocyanate being in the ratio of 1:0.5 to 1:1. The antimony compound is used as antimony trioxide in pulverized or suspended form. The textiles to be treated are made of fibers and filaments of natural and synthetic origin, e.g. of polyamides, polyacrylonitriles, polyesters, polyalkylenes, polycarbonates, polyurethanes, halogenated polyolefines, regenerated cellulose, cotton and wool or mixtures thereof.

The moulded fabrics, e.g. coated fabrics of fibers and filaments as described above or textiles containing an in-

termediate coating of the herein described flameproofing material are distinguished over non-treated material by a substantially reduced flammability. The herein described method and the mixtures to be used as flameproofing agents according to the invention are especially suitable in the treatment of textile fabrics of polyacrylonitrile fibers. A specific advantage of the herein described process consists in that the action of flames does not effect a dropping melt.

The technological data of the textiles treated according to the invention are affected either not at all or to an immaterial degree only. The resiliency of the materials treated according to the invention is increased. A further advantage is the resistance to washing and solvents of the textile fabrics treated according to the invention.

Textile fabrics can be rendered flame-resistant by coating the textile material with the mixtures according to the invention by means of doctor blades (a doctor arrangement) and subsequently drying the textile material thus treated in a drying chamber at 70 to 160° C. The blades are to be adjusted in such a manner that the coatings of the dry coating material, referred to the weight of the textiles, range between 5 to 150% by weight, preferably between 40 and 70% by weight. The coating is to be accomplished so that the textile material treated substantially retains its permeability to air and steam.

The invention is now illustrated by the following examples without being restricted thereto.

EXAMPLE 1

Tufted carpets of polyamide-6-fibers (weight 1,300 g./m.²) are coated on the back by means of a doctor arrangement, the blades being adjusted so that a coating of 65% by weight is obtained. The coating paste consisting of two mixed components (A and B) is prepared in form of a homogeneous mass by vigorously mixing immediately prior to its application. The paste has the following composition:

Part A

	Percent by weight
Polyvinylchloride (molecular weight of about 40,000)	30
Tricresyl phosphate	42
Tris-(dibromopropyl)-phosphate	10
Diphenyl thiourea (stabilizer for polyvinylchloride)	1
Antimony trioxide	2.5

Part B

	Percent by weight
Polyurethane (consisting of an aliphatic polyether with a molecular weight of 2,000 and toluylene-diisocyanate-2,4)	11.6
Triisocyanate (consisting of trimethylolpropane and toluylene-diisocyanate-2,4) 75% in ethylacetate	2
Reaction product of N-methyldiethanolamine and phenylisocyanate (1:2) (catalyt)	0.9

The components of Part B are dissolved in ethylacetate in the weight ratio 1:1 and mixed with Part A.

The coated goods are subsequently treated in a drying chamber at 145° C. for 5 minutes.

The flammability test according to DIN 53906 does not show a dropping melt. After 5 shampooing treatments the carpet retains its resistance to burning according to DIN 53906.

EXAMPLE 2

Cotton fabric (weight 350 g./m.²) is coated by means of a doctor blade with a flameproofing paste of the following composition specified below. The paste is applied to the back of the fabric in a quantity of 45% by weight. The coated fabric is subsequently dried on a drying frame at 150° C. for 4 minutes. The flammability test carried out at 45° C. (SWV 98896 and ASTM D 1230–52 T) shows that the treated cotton fabric is difficult to ignite and self-extinguishing.

Part A

	Percent by weight
Polyvinylchloride (molecular weight of 50,000)	28
Triphenylphosphate	40
Tris-(dichloroethyl)-phosphate	14
Triphenylphosphite (stabilizing agent)	0.5
Antimony trioxide	3

Part B

The composition used corresponds to the parts specified in Example 1. The parts of B are dissolved with ethylacetate in the weight ratio 1:1 and mixed with Part A.

EXAMPLE 3

Upholstery cloth of polyamide-6-velours (weight 320 g./m.²) is coated on the back by means of a doctor blade with the paste specified below. The paste is applied in a quantity of 70% by weight, referred to the weight of the velours fabric. The fabric is then dried on a drying tenting frame at 140° C. for 5 minutes.

The test of the flammability according to DIN 53906 was passed without the formation of a dropping melt.

Part A

	Percent by weight
Polyvinylchloride (molecular weight of 40,000)	32
Diphenyl cresylphosphate	40
Tris-(bromopropyl)-phosphate	9.5
Diphenyl thiourea (stabilizing agent)	0.8
Antimony trioxide	3.2

Part B

	Percent by weight
Polyurethane (consisting of toluylene-diisocyanate-2,4 (mol ratio 1:1) and a polyether consisting of adipic acid and hexanediol-1,6 molecular weight of 2,100)	11.8
Thiophosphoric-acid-O.O.O-triphenylether - 4,4',4''-triisocyanate	1.8
Reaction product of N-methyldiethanolamine and phenylisocyanate (1:2) (catalyst)	0.9

The parts of B are dissolved in ethylacetate in the weight ratio 1:1 and mixed with part A.

EXAMPLE 4

Curtaining material of polyacrylonitrile fibers (velours fabric) (weight 500 g./m.²) is coated on the back with the paste specified below by means of a doctor blade and sandwich-back with a perlon lining fabric. The paste is applied in a quantity of 65% by weight referred to the weight of the cloth. The fabric is subsequently dried on a drying tenting frame at 145° C. for 5 minutes.

The testing of the flammability according to DIN 53906 shows self-extinguishing non-dropping textile material. The same result is obtained after three treatments in perchloroethylene (cleaning).

Part A

	Percent by weight
Polyvinylchloride (molecular weight of about 80,000)	32
Tricresylphosphate	42
Tris-(dibromopropyl)-phosphate	8
Diphenyl thiourea (stabilizing agent)	0.8
Antimony trioxide	2.7

Part B

	Percent by weight
Polyurethane (consisting of a polyether (adipic acid/hexanediol-1,6—mol ratio 1:1) ahd toluylenediisocyanate-2,4 mixed with toluylenediisocyanate-2,6—mol ratio 1:1	11.3
Triisocyanate (consisting of trimethylolpropyl and toluylenediisocyanate-2,4) 75% in ethylacetate	2
Reaction product of N-methyldiethanolamine and phenylisocyanate (1:2) (catalyst)	1.2

Part B is dissolved in ethylacetate in the weight ratio 1:1 and mixed with part A.

We claim:

1. A flame-resistant textile article comprising filaments, staple fibers or yarns and 5 to 150% by weight, calculated on the finished article, of a homogeneous mixture consisting essentially of

(a) a tris halogen alkyl phosphate in which the alkyl groups contain up to 3 carbon atoms;

(b) a phosphoric acid triester plasticizing agent in which the ester groups are aryl or alkyl of up to 8 carbon atoms;

(c) polyvinyl chloride or a polyvinyl bromide having a molecular weight of 30,000 to 150,000;

(d) antimony oxide; and

(e) a polyurethane prepared by reaction of a polyester or polyether having a molecular weight of 500 to 10,000 selected from the group consisting of the polyester of adipic acid/hexanediol-1,6; the polyester of terephthalic acid/ethylene glycol; polypropylene oxide ether; and mixtures thereof; with a polyisocyanate selected from the group consisting of toluylene-diisocyanate-2,4; toluylene-diisocyanate-2,6; mixtures thereof; hexamethylene-diisocyanate-1,6; and the triisocyanate of one mole of trimethylolpropane and three moles of toluylene-diisocyanate-2,4;

the proportions of the ingredients (a) and (b), (c), (d) and (e) in said mixture being in the range 1:0.2:0.05:0.1 to 1:1:0.5:1.

2. The flame-resistant textile article of claim 1 in which said tris halogen alkyl phosphate is tris-(dibromopropyl)-phosphate.

3. The flame-resistant textile article of claim 1 in which said tris halogen alkyl phosphate is tris-(dichloroethyl)-phosphate.

4. The flame-resistant textile article of claim 1 in which said phosphoric acid triester plasticizing agent is tricresyl phosphate.

5. The flame-resistant textile article of claim 1 in which said phosphoric acid triester plasticizing agent is triphenyl phosphate.

6. The flame-resistant textile article of claim 1 in which the component is polyvinyl chloride.

7. The flame-resistant textile article of claim 1 wherein said polyurethane is a polyurethane polymer obtained by reacting a polyester from adipic acid and hexanediol-1,6 with toluylene diisocyanate and a triisocyanate from trimethylolpropane and toluylene diisocyanate.

8. The flame-resistant textile article of claim 1 in which the ester groups are phenyl, alkyl phenyl or alkyl groups.

9. A method of preparing a flame-resistant textile article from filaments, staple fibers or yarns which comprises coating the textile article with 5 to 150% by weight, calculated on the finished textile article, of a homogeneous mixture consisting essentially of

(a) a tris halogen alkyl phosphate in which the alkyl groups contain up to 3 carbon atoms;

(b) a phosphoric acid triester plasticizing agent in which the ester groups are aryl or alkyl of up to 8 carbon atoms;

(c) polyvinyl chloride or a polyvinyl bromide having a molecular weight of 30,000 to 150,000;

(d) antimony oxide; and

(e) a polyurethane prepared by reaction of a polyester or polyether having a molecular weight of 500 to 10,000 selected from the group consisting of the polyester of adipic acid/hexanediol-1,6; the polyester of terephthalic acid ethylene glycol; polypropylene oxide ether; and mixtures thereof; with a polyisocyanate selected from the group consisting of toluylene-diisocyanate - 2,4; toluylene-diisocyanate - 2,6; mixtures thereof; hexamethylene-diisocyanate-1,6; and the triisocyanate of one mole of trimethylolpropane and three moles of toluylene-diisocyanate-2,4;

the proportions of the ingredients (a) and (b), (c), (d) and (e) in said mixture being in the range of 1:0.2:0.05:0.1 to 1:1:0.5:1, and subsequently heating the coated textile article at temperatures of between 70 and 160° C.

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