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Applicant : **WARWICK INTERNATIONAL GROUP LIMITED**
Wortley Moor Road
Leeds LS12 4JE (GB)

Inventor : **Roche, Neil Patrick**
65 Fairway
Normanton, West Yorkshire (GB)
Inventor : **White, Susan Yvonne**
39 Fenton Street
Tingley, West Yorkshire (GB)
Inventor : **Pitt, Andrew William**
5 Leasowe Close
Leeds, LS10 2HS (GB)
Inventor : **Galek, Jan Anthony, Dr.**
26 Troy Road
Morley, Leeds LS27 8JF (GB)

Representative : **Jones, Helen Marjorie**
Meredith
Gill Jennings & Every, Broadgate House, 7
Eldon Street
London EC2M 7LH (GB)

Flame retardant composition for cellulosic textiles.

Aqueous compositions for imparting flame retardant properties comprising an inorganic halide, an oxide of antimony having an average particle size no greater than 2.0µm, a binding agent, a plasticiser and a surface modifier. The invention provides flame retardant properties without significant discolouring of fabric dyes and without causing undue stiffness to the treated fabric. The aqueous based composition provides flame retardancy throughout the material treated, requiring relatively short curing time and providing a clear finish. In a preferred process according to the invention, the composition is applied in the final stage of a fabric printing process, for example in a finishing bath.

The present invention relates to a composition for imparting flame retardant properties to cellulosic fabrics and fibres. The invention also includes a method of making such a composition and a process for applying the flame retardant composition to a textile.

Many methods of imparting flame retardant properties to fabrics are known. More recently, awareness has increased of the fire hazard of various home furnishings such as curtains and upholstery fabric and although there are many known methods for imparting flame retardant properties to textiles, various problems are associated with them. In addition, recent very high standards of performance have been set, including British Standard Nos. 5852, 5651, 7176, 4303, 1006 and 5690. Many previously known methods do not fulfil these requirements.

One of the most commonly used flame retarding materials for treating fabrics is decabromodiphenyl oxide both in its own right, and in admixture with antimony trioxide which is also a well known flame retarding material. Compositions including these compounds are generally applied to fabric as a back-coating to produce a barrier effect. However, this type of coating is not ideal because the back-coating is usually opaque and white, the treated fabric is usually objectionably stiff and dry cleaning is not usually possible without destroying the flame retardant properties of the fabric.

Such a composition is described in FR-A-2099391 where back coating flame retarding coating compositions are described for use in vehicle security inflatable bodies. Optional flame retarding materials for use in the composition comprise inorganic halides and antimony trioxide but not in combination. The preferred flame retarding materials are phosphates.

Antimony trioxide is also a well known flame retarding component. It is also known to use antimony trioxide as the only flame-retardant component in a composition for application to textile fabrics. One such composition is described in US 3,014,000. The composition described comprises antimony trioxide, a composite plasticiser, and a vinyl polymer such as vinyl chloride/vinylidene chloride copolymer. The plasticiser, which contains at least 15% by weight chlorine is included for a soft feel on the fabric and may be, for example, a chlorinated paraffin, an alkyl ester of an aliphatic or aromatic dicarboxylic acid or phosphoric acid.

One other flame retarding composition commonly used is known as "Pyrovatex" (Trade Mark) which is a dimethyl phosphonate type resin, usually cured using phosphoric acids. Although this product results in finished fabrics which have a relatively soft finish, its use has many disadvantages. During its application, toxic formaldehyde vapours are produced and therefore its use is undesirable. In order to operate a flame retarding treatment using such a composition, additional ventilation facilities are required, and must

be provided in the work place. Furthermore, the treated fabric is usually acidic which results in a much reduced tear and tensile strength of the fabric.

In treating cellulosic materials with a flame retardant, one other undesirable result which often occurs is that the flame retardants tend to discolour or darken the treated cellulosic substrate and/or dyes/pigments. This observation is particularly noticeable on printed metallics such as golds and silvers. The discolouring effects by the flame retardant are particularly problematic because they are often dependent upon the type of dyes/pigments used on the various fabrics. For example, the colour difference effect on pigment print may not be the same as the effect on for example vat dyes.

A water soluble composition useful as a flame retardant for cellulosic material is disclosed in US 4,888,136. The composition comprises ammonium bromide and a water-soluble aluminium salt of an organic hydroxy acid. The composition is directed in particular to the treatment of paper products although textiles are also mentioned. The invention is based on the unexpected finding that a combination of aluminium salt with ammonium bromide produces a synergistic effect.

In DD 146,069 flame proofing using an aqueous solution of ammonium bromide with borax is described. However, this use is described as only being useful for articles which are not normally washed. Although the finish obtained is flame proof and does not leave a visible deposit or matt finish, which is detrimental to the appearance of cellulosic articles, the flame retardancy is destroyed by washing the cellulosic article.

In GB 2,048,972, a PVC-free flame retardant for finishing mixed polyester/cellulose articles is described which comprises a chlorine donor and antimony trioxide in a specified ratio. The discussion of the prior art states that processes for providing a "wash resistant" flame retardant finish on cellulosic material, using halogen donors (such as chlorinated paraffins, polyvinyl chloride and bromide) in conjunction with antimony trioxide have long been known. There is no description of any particular particle size requirement and no description of specific compositions for providing a successful fire retarding treatment for cellulosic materials.

Ammonium bromide is also described as a suitable flame retardant treatment in GB 1,291,133 for producing disposable fabrics for medical use or for clothing.

Historically, ammonium bromide and other halides have been much more commonly known as flame proofing components for polymeric materials and in particular, for rubber. In JP 62112639 a flame retardant for synthetic resin is obtained by a coating of ammonium chloride or ammonium bromide with antimony trioxide. In JP 60235851 ammonium chloride

or bromide with antimony trioxide is used for manufacturing a flame resistant polyolefin.

There is still a need for a highly effective flame retarding composition which may be applied to cellulosic based substrates without destroying the natural soft handle, the strength of the fabric and which will retain the flame retardant properties after repeated dry-cleaning or after being soaked in water.

The present invention comprises a novel composition which produces particularly effective flame retardancy and which overcomes the problems of the prior art compositions.

In accordance with the present invention an aqueous composition for imparting flame retardant properties to a cellulosic substrate is provided, which comprises an inorganic halide, an oxide of antimony having an average particle size no greater than 2.0 μm , a binding agent, a plasticiser and a surface modifier.

In addition, the present invention is particularly advantageous because use of an aqueous based composition provides flame retardancy throughout the material and does not act merely as a barrier. Furthermore, the composition of the invention provides a clear finish flame retardancy treatment which does not significantly affect the appearance of the fabric, unlike the known flame retardancy treatment compositions.

The inorganic halide is preferably an ammonium halide and most preferably is ammonium bromide. The halide is generally included in the composition at a concentration of from 50 to 500 g/l and preferably from 100 to 300 g/l. The inorganic halide may be used as either a solid or as a suspension or solution.

The oxide of antimony may be antimony trioxide or tetraoxide but is preferably antimony pentoxide. It may be in a hydrated or non-hydrated form. Although antimony oxide up to a particle size of approximately 2 μm is still effective and economical, generally, the particle size of the antimony oxide is no greater than 0.5 μm as it has been found that this prevents pigmentation of the fabric surface and ensures formation of a transparent finish. Preferably the average particle size is no greater than 0.1 μm and most preferably, the particle size is from 0.01 to 0.05 μm because this ensures greater potential antimony interaction. Antimony pentoxide is preferred because it can be obtained having the very small particle size preferred for the present invention. It is well known that to produce antimony trioxide to such small particle size incurs significant manufacturing time and cost. The antimony oxide may be used as a solid powder but it is preferably used as a colloidal suspension in water. Although colloidal antimony trioxide can also be obtained, for best results, the antimony oxide is most preferably hydrated antimony pentoxide, in the form of an aqueous colloidal suspension. Although antimony pentoxide contains a lower relative amount of antimony than antimony trioxide and therefore could be expected to be

less favourable, we have found that it provides especially good flame retardant properties, especially at the preferred low particle sizes. Preferably, in order to ensure a transparent finish the refractive index of the antimony oxide is as close to 1 as possible.

The antimony oxide is generally included in the composition in an amount of from 25 to 400 g/l and preferably from 25 to 150 g/l.

As the binding agent, one or more conventional binding agents which are well known throughout the industry can be used. The binding agent can be one or more of a wide variety of polymers and copolymers. However, we have found vinylidene chloride based polymers and admixtures thereof, or for example copolymers of vinyl chloride and ethylene, are eminently suitable. In addition to their primary role in binding other active flame retardant components to the cellulosic material, they also contribute towards the fire retardant effect of the composition by virtue of their halogen content. Other binders may be used, for example acrylate based polymers, which may act solely as film-forming agents and bind the active flame retardant components to the cellulose. Generally, a 10-70% by weight aqueous emulsion of the binder is used for ease of handling, but solid materials may be used if so desired. The proportion of binder used in the composition would typically be in the amount of from 200 to 600 g/l and preferably in the range 300 to 500 g/l.

The plasticiser, may be for example as described in US 3014000. It is preferably a halogenated paraffin, and most preferably a chlorinated paraffin. Again, by virtue of their halogen content, these chlorinated paraffins contribute to the flame retardant effect. Particularly suitable chlorinated paraffins contain between 25 and 75% by weight of chlorine, and more preferably between 35 and 50% by weight. Paraffins having a carbon chain length of from C_8 to C_{14} have been found to be particularly suitable.

On curing, the binding agent binds the antimony oxide and inorganic halide to the cellulosic substrate, thereby providing the substrate with flame retardant properties.

The surface modifier can be any of a number of known compounds and is included in the composition to impart a water resistant elastomeric finish to the substrate. Many suitable elastomeric materials exist, for example, silicones and polyurethanes which can be cross-linked. Preferably, the surface modifier comprises a blend of compounds, for example based on polysiloxane(s) in which the silicon is substituted with two groups which may be the same or different, chosen from alkyl, aralkyl, aryl or hydrogen with the exception that where hydrogen is one substituent, the two groups must be different. The blend may be based on polydialkyl siloxane(s). The alkyl, aryl or aralkyl groups themselves may or may not be substituted. Preferred silicones are the compounds where

the alkyl group is either methyl, ethyl, propyl or butyl groups, which may be substituted with one or more groups such as amino, hydroxy, alkoxy or cyano. Most preferably, the surface modifier is a reactive amino siloxane in which the amino substituent is positioned on the alkyl group i.e. the silicon is substituted with at least one aminoalkyl group, in which the amino group(s) may be primary and/or secondary amino groups, for example N(2-aminoethyl)-2-ethylamine groups.

It has been found that the addition of such a surface modifier significantly improves the resistance to dry-cleaning and water soaking, of the treated cellulose. The addition of a surface modifier also ensures that the final treated cellulosic substrate retains a soft handle. Generally, the total amount of the surface modifier may be from 5 to 250 g/l, but more preferably in the range 5 to 150 g/l. Where the surface modifier comprises more than one compound, for example, more than one type of siloxane, compounds in the modifier may be in any ratio. Preferred ratios are dependant on the type and amounts of the other ingredients in the flame retardant composition.

Other optional ingredients, in the composition, include afterglow inhibiting agents such as zinc borate, aluminium trihydrate and boric acid esters, handle modifiers such as silicone and non-silicone softeners, perfumes, lubricants, brightening agents, resins, bleaching compounds, thickening additives, anti-bleed agents, anti-leaching compounds, viscosity modifiers, water/oil repellant compounds such as fluorochemical additives and colouring agents etc. These types of additives are well known in the industry and thus are used routinely.

The cellulosic substrates which are to be treated by the composition of the present invention include cotton, viscose and all other cellulosic materials, including fibre blends. Blends including non-cellulosic material are also suitable substrates. For example, polyester-cotton and/or polyester viscose blends comprising from 5 to 95% by weight cellulosic material, preferably at least 20% cellulosic, most preferably at least 50% cellulosic material. Typically blends contain up to 80% cellulosic material. The flame retardancy treatment of the invention is particularly directed to treatment of woven or other formed fabrics, but may also be used on suitable non-woven substrates. In particular, it is intended for use on fixed and loose upholstery fabrics and covers.

The process for the manufacture of the flame retardant composition generally requires high-shear mixing to intimately blend the individual ingredients. It has been found that the order of addition of the ingredients can affect the performance of the flame retardant effect, but is generally governed by ease of handling on the production scale. However, pH control is an important factor during the manufacture.

The preferred method of manufacture of the com-

position according to the invention comprises firstly, mixing a suspension of small particle size antimony oxide, preferably antimony pentoxide with water. Secondly, the pH of the mixture is monitored and adjusted to between 7.5 and 9.5, preferably approximately pH 8 to 9, using a base.

The inorganic halide is then mixed into the composition.

Following addition of the inorganic halide and mixing of the composition until a substantially homogeneous composition results, the pH of the composition is preferably once again monitored and adjusted to the final pH of 6.0 to 9.0, if necessary.

In choosing the base for adjusting the pH, the main consideration is whether the particular base chosen will have an adverse effect on the dyes or resins in the cellulosic substrate to be treated. For example, it has been found that sodium hydroxide may produce a yellowing cast on the fabric and is therefore not preferred. The most preferred base for adjusting the pH at one or stages is ammonia which may be in the form of ammonia gas, by bubbling through the solution or, preferably, in the form of aqueous solution of ammonia (ammonium hydroxide). Ammonia is preferred because it produces substantially no discolouration on the final fabric.

The antimony oxide/ammonium halide mixture formed is then mixed with the binding agent and plasticiser and surface modifier in any order of addition. Preferably, the plasticiser and inorganic halide/antimony oxide mixture are mixed vigorously to obtain a substantially homogeneous composition. This is because the halide/antimony oxide mixture is viscous and addition of the plasticiser dilutes the mixture to promote ease of handling of the composition. Because the aqueous antimony oxide/halide mixture is not generally miscible with the plasticiser, an emulsion is formed. Additional dispersants/surfactants may be included to further increase the stability of the emulsion, if desired.

Preferably the binder is then added to the composition, preferably followed by high shear mixing. As the binder is mixed in the composition some reaction occurs with the plasticiser and the viscosity of the composition increases.

After addition and blending of all the ingredients, including any optional textile performance auxiliaries, as described above, the composition will generally be a white, slightly viscous liquid, having a pH preferably in the range 6.0 to 9.0, and most preferably between 7.0 and 8.0. The application of the composition having a pH outside the range of 6.0 to 9.0, to cellulosic substrates will have an adverse effect on the substrate. This is usually exhibited in a reduced tear and tensile strength of the substrate.

The present invention also includes a process for imparting flame retarding properties to a cellulosic substrate comprising contacting the cellulosic sub-

strate with a composition comprising an inorganic halide, an oxide of antimony having particle size no greater than 2.0 μm , a binding agent, a plasticiser and a surface modifier.

At present, the commonly used flame retardant compositions are usually applied to fabrics in a separate process step, often following the printing, dyeing, drying or finishing stages. Obviously, this increases manufacturing costs and in many instances, the final dried fabric will be taken to a separate location for flame retardant treatment. The present invention is highly advantageous in that it is compatible with the usual finishing bath additives. Thus a separate treatment stage can be avoided as the flame retardant composition can simply be included in the finishing bath, if so desired. Following application, the cellulosic material is dried at temperatures normally associated with textile flame retardant treatments, typically between 90°C and 160°C, depending on fabric throughput speed. The drying step can also effect curing of the flame retardant onto the fabric.

The invention also includes a process for imparting flame retardant properties to a cellulosic substrate by contacting the substrate with a composition comprising an inorganic halide, an oxide of antimony having a particle size of no greater than 2.0 μm , a binding agent, a plasticiser and a surface modifier in a finishing bath in the printing process for the substrate.

If the composition is applied as a separate process step, it must also be followed by a curing stage. Generally curing is effected by heating the treated substrate at a temperature of at least 90°C, preferably from 120° to 160°C, most preferably 120°C to 150°C.

Whether curing is in the drying stage or is a separate stage, usually the curing time is from 0.2 to 10 minutes preferably 0.5 to 5 minutes most preferably 0.5 to 3 minutes at the curing temperature. This is a significant advantage over the curing times of most known flame retarding compositions, for which longer curing times, for example up to 20 minutes or even 30 minutes are required.

The amount of the flame retardant composition required on the finished fabric, in order to give effective flame retardant properties is generally between 5 and 80% by weight of the substrate, preferably from 10 to 30%. These amounts refer to added dry weight of the composition on the substrate. The amount of the composition required depends upon the type of the substrate and the degree of flame retardancy required.

If insufficient of the flame retarding composition is incorporated into the fabric following one contact of the substrate with the composition, for example, by immersion in a finishing bath, a further quantity can be applied in a second contact stage.

The method of application of the flame retardant composition is by any of the known techniques in the

industry including, pad-mangle, foam impregnation, vacuum impregnation, spray and roller, etc. Although the composition can be used to provide a back coating, the composition of the invention is advantageous as it is particularly suitable for application by absorption into the substrate and curing to provide a flame retarding composition impregnated throughout the substrate. The application process is particularly suited to the pad-mangle method, when the composition is applied to woven cellulosic fabrics.

The amount of flame retardant on the final treated fabric can be controlled by the method of application as well as the concentration of the composition. Thus, to attain a higher % of flame retardant, a higher wet amount of composition is applied so that on drying a greater % of the flame retardant remains.

Example 1

A composition according to the present invention was made up in a stainless steel reactor fitted with a high-shear mixer, temperature control, pH monitor, and cooling facilities.

203 Kg of Nyacol A1550 (Trade Mark: a 50% colloidal suspension of antimony pentoxide in water) was charged to the vessel and high speed mixing was applied. To this was charged 57 Kg water, followed by approximately 12.5 Kg of concentrated ammonia solution, in order to adjust the pH to approximately 9.0. Once the pH had stabilised, 248 Kg of ammonium bromide (38%) was added and blended. The pH was readjusted to approximately 8.0 using ammonia solution.

The resultant pre-mix was then added to 82 Kg Cereclor 50LV (Trade Mark; a chlorinated paraffin), and mixed until homogeneous. As these two components were mixed, a change in viscosity resulted and the composition thickened. To this mixture was added 466 Kg of Emulsion CE752 (Trade Mark): this is a 50% emulsion of vinyl chloride/ethylene copolymer in water. Once intimately mixed 64 Kg of Emprimin LR28 (Trade Mark, a sodium lauryl sulphonate anionic surfactant) was added, followed by 32 Kg Silicon VP1445E (Trade Mark): an aqueous emulsion of polydimethylsiloxane. Finally there is added 54 Kg of Mykon 8430 (Trade Mark): a mixture of polyhydrogenmethylsiloxane and polydimethylsiloxane, and the mixture was mixed well. The resulting product was a white, slightly viscous liquid having pH between 7.0 to 8.0. No final adjustment of the pH was necessary.

Example 2

A composition was made up in the same manner as in Example 1 with the exception that MYKON 8430 was replaced with DOW Emulsion Q2-7419, an amino-functional polyalkyl siloxane containing surface modifier.

Example 3

A composition was made up as in Example 2 and in which 20 kg of zinc borate was also incorporated.

Example 4

A sample of printed 52 x 44 (picks per inch, warp by weft) 100% cotton fabric was passed through a finishing bath, containing 650 g/l of the flame retardant composition of example 1. With the aim of producing a treated fabric having 25% dry weight add-on (a final substrate comprising flame retardant in an amount of approximately 25% of the fabric weight), the pad-mangle expression was adjusted. The combination of concentration of the composition and pad-mangle expression (determining how much wet composition remains on the substrate), by throughput speed and mangle pressure, were used to load the substrate with a wet amount of composition such that on drying, the required dry weight add-on % flame retardant would remain.

Following pad application, the fabric was heat treated at 130°C to give a flame retardant finish. No noticeable colour changes to either the fabric or the colouring agents were observed, and the fabric had retained a soft feel, no significant stiffening of the fabric having occurred.

The treated fabric was subjected to the fire retardant tests as stipulated in the BS 5852 and BS 5651 documents. No flaming was observed in cover or interior which did not cease with 120 secs of the removal of the burner tube. No externally detectable amounts of smoking, heat or glowing was observed 15 min after removal of the burner tube. This effectively resulted in a PASS certificate for BS 5852 Section 4 Ignition Source 1 and Schedule 5 of the Furniture and Furnishings (FIRE) (SAFETY) Regulations 1988.

Equally good flame retardancy results were obtained using the compositions of examples 2 and 3. The finished fabric had slightly improved feel characteristics using the compositions of examples 2 and 3.

Claims

1. An aqueous composition for imparting flame retardant properties to a cellulosic substrate comprising an inorganic halide, an oxide of antimony having an average particle size no greater than 2.0 μm , a binding agent, a plasticiser and a surface modifier.
2. An aqueous composition according to claim 1 in which the inorganic halide is an ammonium halide, preferably ammonium bromide.
3. A composition according to claim 1 or claim 2 in which the halide is in a concentration of from 50-500 g/l, preferably from 100-300 g/l.
4. A composition according to any preceding claim in which the oxide of antimony is hydrated antimony pentoxide, in the form of an aqueous colloidal suspension.
5. A composition according to any preceding claim in which the particle size of the antimony oxide is from 0.01 to 0.05 μm .
6. A composition according to any preceding claim in which the antimony oxide is present in an amount of from 25-400 g/l, preferably from 25 to 150 g/l.
7. A composition to any preceding claim in which the binding agent is a copolymer of vinyl chloride and ethylene which is present in the composition in an amount of from 200 to 600 g/l and preferably in the range 300 to 500 g/l.
8. A composition according to any preceding claim in which the plasticiser is a halogenated paraffin, the paraffin having a carbon chain length of from C_8 to C_{14} .
9. A composition according to any preceding claim in which the surface modifier is an aminoalkyl substituted polysiloxane or mixture of polysiloxanes.
10. A process for manufacture of the composition according to any preceding claim comprising mixing a suspension of small particle size antimony oxide with water, if necessary adjusting the pH to between 8.5 and 9.5 by the addition of a base, mixing inorganic halide into the composition until a substantially homogeneous composition results, followed by mixing with binding agent, plasticiser and surface modifier.
11. A process according to claim 10 in which the base is ammonia in the form of ammonia gas or preferably, aqueous solution of ammonia.
12. A process for imparting flame retardant properties to a cellulosic substrate comprising contacting the cellulosic substrate with a composition according to any of claims 1 to 9, absorbing the composition throughout the cellulosic substrate, and curing the composition into the substrate.
13. A process according to claim 12 in which contact with the cellulosic substrate is by immersion in a finishing bath in the printing process for the sub-

strate.

14. A method according to claim 12 or claim 13 in which the substrate is contacted with the flame retardant composition by pad-mangle application. 5
15. A cellulosic substrate comprising a flame retardant composition obtainable by the process according to any of claims 12 to 14. 10

**Claims for the following Contracting State :
ES**

1. A process for preparing an aqueous composition for imparting flame retardant properties to a cellulosic substrate comprising mixing an inorganic halide, an oxide of antimony having an average particle size no greater than 2.0 μm , a binding agent, a plasticiser and a surface modifier and water to form an aqueous composition. 15 20
2. A process according to claim 1 in which the inorganic halide is an ammonium halide, preferably ammonium bromide. 25
3. A process according to claim 1 or claim 2 in which the halide is in a concentration of from 50-500 g/l, preferably from 100-300 g/l. 30
4. A process according to any preceding claim in which the oxide of antimony is hydrated antimony pentoxide, in the form of an aqueous colloidal suspension. 35
5. A process according to any preceding claim in which the particle size of the antimony oxide is from 0.01 to 0.05 μm .
6. A process according to any preceding claim in which the antimony oxide is present in an amount of from 25-400 g/l, preferably from 25 to 150 g/l. 40
7. A process to any preceding claim in which the binding agent is a copolymer of vinyl chloride and ethylene which is present in the composition in an amount of from 200 to 600 g/l and preferably in the range 300 to 500 g/l. 45
8. A process according to any preceding claim in which the plasticiser is a halogenated paraffin, the paraffin having a carbon chain length of from C_8 to C_{14} . 50
9. A process according to any preceding claim in which the surface modifier is an aminoalkyl substituted polysiloxane or mixture of polysiloxanes. 55

10. A process according to any preceding claim in which a suspension of small particle size antimony oxide is mixed with water, if necessary the pH is subsequently adjusted to between 8.5 and 9.5 by the addition of a base, prior to mixing inorganic halide into the composition until a substantially homogeneous composition results, followed by mixing with binding agent, plasticiser and surface modifier.

11. A process according to claim 10 in which the base is ammonia in the form of ammonia gas or preferably, aqueous solution of ammonia.

12. A process for imparting flame retardant properties to a cellulosic substrate comprising contacting the cellulosic substrate with a composition formed according to any of claims 1 to 11, absorbing the composition throughout the cellulosic substrate, and curing the composition into the substrate.

13. A process according to claim 12 in which contact with the cellulosic substrate is by immersion in a finishing bath in the printing process for the substrate.

14. A process according to claim 12 or claim 13 in which the substrate is contacted with the flame retardant composition by pad-mangle application.



European Patent
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EUROPEAN SEARCH REPORT

Application Number

EP 92 30 8312

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
D,X	FR-A-2 099 391 (ASAHI KASEI KOGYO KABUSHIKI KAISHA)	1,2	D06M11/13 D06M11/47 D06M15/248
Y	* page 1 - page 2, line 34; claims 1-3; examples 1-3 *	3-8, 10-15	
Y	--- US-A-3 682 692 (JAMES E. LAMSON) * column 2, line 37 - line 40; claims *	1-15	
Y	--- US-A-4 536 422 (ROBERT J. HARPER) * the whole document *	1-15	
A	--- EP-A-0 038 090 (GEISER A.G. TENTA-WERKE) * the whole document *	1-15	
A	--- DE-A-3 316 197 (STÖCKERT, EDGAR) * the whole document *	1-15	
A	--- FR-A-1 481 855 (ETAT D'ISRAEL: MINISTÈRE DU COMMERCE ET DE L'INDUSTRIE) * the whole document *	1,2,3	TECHNICAL FIELDS SEARCHED (Int. Cl.5)
A	--- GB-A-1 306 838 (ASSOCIATED LEAD MANUFACTURERS LIMITED) * the whole document *	1-15	D06M

The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 20 OCTOBER 1992	Examiner BLAS V.
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document</p>			

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