ТРУДНО ГОРИМА АПРЕТУРА ЗА ПАМУЧНИ И ПАМУЧЕН ТИП ТЪКАНИ

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FLAME RETARDANT FINISH FOR COTTON AND COTTON BLEND FABRICS

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ABSTRACT

Cotton is the one of the most important textile fibre because of its comfort and worldwide consumption. However, it ignites easily and is frequently implicated in fire. Since, considerable efforts have been made to develop flame-retardant cotton. The flame-retardant cotton fabrics must be durable to washing cycles, eco-friendly and have a good physical mechanical properties. This article summarizes an overview of the factors related to the requirements of flame retardant cotton fabrics such as chemical agents and technological parameters.

This section also introduces some of the new chemicals and techniques used in the fire retardant treatment for cotton fabric. They allow flame retardant treated fabrics to have more durable fire resistance and better mechanical properties while still being environmentally friendly.

In the 2 and 3 sections of this article, the experimental results of study on flame retardant treatment for cotton and cotton polyester blend fabrics of the authors are presented. The experimental results show that flame retardant cotton and cotton polyester blend fabrics, which was received from this research have a good flame retardancy, but it has limited durability to wash, moreover, after treatment, mechanical strength of fabric was reduced. In order to have the durable flame retardant, eco-friendly cotton fabric, the study should be continued in the direction of using environmentally friendly chemicals and in treatment process, it should avoid the conditions that may adversely affect the mechanical strength of the fabric. Plasma application in flame retardant treatment of fabric may be a good solution to could meet all requirements of fabric.

Keywords: *Cotton fabric, cotton polyester blend fabric, flame retardant agent, cross-link agent, Organophosphorus flam retardant agent.*

1. Literature review

1.1. Overview about flame retardant agents (FR) for cellulose fibres

Cotton is the one of the most important textile fibre because of its comfort and worldwide consumption. However, it ignites easily and is frequently implicated in fire. In fires cotton products, such as apparel, curtains, bedclothes, and upholstered furniture, become quickly engulfed and are difficult to extinguish, because Cotton is one of the most flammable textile fibres. It has Limiting Oxygen Index (LOI) less than 19% i.e. 18.4% [2]. Since, considerable efforts have been made to develop flame-retardant cotton.

In order to meet fire safety regulations and expand the use of cotton in textile applications that require flame resistance, a significant number of flame retardant treatments for textiles were developed in the last century. The majority of these flame retardant agents can be classified into four distinct groups: inorganic, halogenated organic, organophosphorus, and nitrogen based [3]

Inorganic salts which melt under the impact of high temperature and create a layer that protects the polymer surface from the heat source, thermally unstable inorganic carbonates and hydrates which yield carbon dioxide when heated and/ or water to cool down the polymer, forming a layer to protect the polymer surface from the heat source, heat conductors (metals) and phase-change materials (PCM), which absorb huge amounts of heat when decomposing or evaporating, thus removing the heat from the polymer before the conditions for ignition are reached. But, A chief disadvantage of these agents is their poor resistance to washing [9]

The use of halogen-based flame retardants to reduce the flammability of cotton is one of the most efficient ways of reducing the fire hazard. However, because of their corrosivity, the presence of dioxin, a carcinogen, and suspected smoke toxicity by products such as HBr and HCl, many European countries such as Sweden and Germany have legislated regulations to restrict halogen-based flame-retarded textile products [1], [3], [6]

Phosphorus-based flame retardants have been a major source of interest to replace halogen compounds because of their environmentally friendly by-products and their low toxicity. Also their low production of smoke in fire furthers their appeal. They are highly effective flame retardants for cellulose and cellulose derivatives. These compounds promote dehydration and char formation [1].

Phosphorus based flame retardants are used mainly for the cotton textiles. These flame retardants act by condensed phase mechanism in cellulose. Cellulose pyrolysis occurs by depolymerization or dehydration. In case of depolymerization, cellulose produces levoglucose which decomposes into flammable gases. On the other hand, dehydration produces char and non-flammable gases. Depolymerization reaction occurs in the flame retardants that do not contain phosphorus. With phosphorus containing compounds, dehydration is dominant. Char formation enhances the flame retardancy of cotton. Char acts as thermal barrier. This char hinders the oxidation of carbon to carbon dioxide. Phosphorus flame retardants also coat char and prevent burning of the surface [2, 3]

A major problem with phosphorus based flame retardant finishes is that these require high amounts to get soft hand feel and to eliminate unpleasant odour. For the flame retardant cotton, the research should be on the finishes that are durable with low formaldehyde release [2]

Nitrogen and sulphur improve the performance of phosphorus based flame retardants by increasing the char formation [2, 3]. Flame retardant finishes required phosphorus nitrogen synergistic effect. There are some nitrogen compounds that have the synergistic effect with phosphorus compounds. The flame retardancy depends on the chemical structures of the nitrogen compounds. Nitrogen compounds enhance flame retardant effect of Phosphorus compounds as given below:

1 - Nitrogen in P-N synergistic retardants creates polymeric compound with P-N bonds which are more polar than already present P-O bonds. This increases electrophilicity of P-atom and hence the phosphorylation of cellulose,

2 - The formation of levoglucosan is stopped. The formation of P-N bonds accelerates the char formation and prevents volatilization of phosphorus. Nitrogen compounds like urea, melamine increase the synergistic effect and increase the action of phosphorus in cellulose. It depends on the nitrogen compound and polymer system. The presence of ammonia enhances the flame retardancy. A combination

of nitrogen compound such as urea, methylol melamine with acid such as phosphoric acid produces char. Due to the combined action of phosphorus and nitrogen, thermal decomposition of cellulose decreases and dehydration increases. This mechanism increases the amount of solid char and improves the flame resistance of treated cotton [2]

1.2. Organophosphorus FR

Durable organophosphorus flame retardant finishes of cotton are of two types depending on the way to covalently bond flame retardant to cellulose. One is the reactive finish that reacts with cellulose hydroxyl groups and forms covalent bond. These are mainly based on N- methylol dimethylphosphonopropionamide (MDPA). The other is non-reactive finish that forms insoluble crosslinked polymer network inside the cellulose fibre. These finishes are tetrakis (hydroxymethyl) phosphonium derivatives such as tetrakis (hydroxymethyl) phosphonium chloride.

Reactive finishes are based on N-methylol dimethylphosphonopropionamide (MDPA). MDPA has methyl group which reacts with cellulose. Crosslinking agents like trimethylolmelamine (TMM) are used as co-reactants. Melamine resin TMM is used to provide nitrogen to enhance the flame retarding efficiency of the treated fabric through synergism with phosphorus. Moreover, crosslinking agents are used to improve the durability of the finish and to improve the phosphorus nitrogen synergistic effect. Formaldehyde based crosslinking agents are used like dimethylolurea (DMU), dimethyloldihydroxyethyleneurea (DMDHEU), trimethylol melamine TMM. Due to the use of formaldehyde based agents there is free formaldehyde release and treated fabrics are stiff. A drawback of this finish is that they release free formaldehyde during condensation, or impregnation of textile material, as well in use [2], [9].

In order to reduce the formaldehyde contents, formaldehyde free crosslinking agents are used. For example, N, N-dimethyl-4, 5-dihydroxyethylene urea (DMeDHEU) which is also called DHDMI 1, 3 Dimethyl 4, 5-Dihydroxy 2-Imidazolidinone (DHDMI) gives zero-level formaldehyde release. It has limited durability to laundering. It gives unpleasant odor. Polycarboxylic acids such as 1, 2, 3, 4- Butanetetracarboxylic acid (BTCA) and citric acid (CA) can also be used to get formaldehyde-free finish. These require catalyst to promote crosslinking of cellulose. Sodium hypophosphite (SHP) is most effective catalyst [2], [4], [9], [10].

But, Butanetetracarboxylic acid (BTCA) has limited laundering durability and it is expensive than DMeDHEU [2]. In general, the chief disadvantages of polycarboxylic acids were considerable strength reduction of treated fabrics. Moreover, polycarboxylic had a detrimental impact on the colouring shade of color fabric. It also causes yellowing of the white fabric, as well as on the pH value of the treated fabric. However, it is eco-friendly because it has no influence on formaldehyde release, which is a major issue in the usage of conventional N- methylol compounds. [2], [4], [9], [10]

Non-reactive durable flame retardant finishes are based on tetrakis (hydroxymethyl) phosphonium salts (THPX). THPX is made by the reaction between formaldehyde and phosphine in the presence of acid. THPC and THPS are two types of THPX but THPC is preferred because THPS has larger size with less penetration. THPC is made from phosphine, formaldehyde and hydrochloric acid at room temperature [2], [9].

THPX finished cotton fabric has flame retardancy that is durable to 100 industrial launderings because there are no hydrolysable groups in the cross-linked polymer network. As the reaction of THPX system on cotton is in neutral to slightly alkaline pH, there is no acid degradation of cellulose. Hence there is no effect on fabric strength. THPX finishes are also used for children sleepwear. The finish contains no chlorine and phosphorus is in the form of phosphine oxide which is most stable to hydrolysis. However, the production of hydrochloric acid and formaldehyde during curing is problematic. The application process involves the use of an ammonia chamber and strict control of application conditions to obtain consistent results. The treated fabric is stiff. This treatment process requires a lot of water, energy and chemicals. Bisulphite is a reducing agent and this treatment affects the shade of dyed materials. During the storage of material, any formaldehyde adduct that remains in the fabric breaks down with the passage of time and will gradually increase free formaldehyde levels

In conclusion, Non-Reactive Finish (THPC ammonia cured) allows get the very durable flame retardant cotton fabric (Flame retardant properties of treated fabric are durable to more than 100 hospital washes (75°C)) with moderate emission of formaldehyde. The fabric can be used for children's sleep wear. But, complex application method due to the use of special ammonia chamber and it is not compatible with sulphur dyes

For Reactive Finish (MDPA), flame retardant properties of treated fabric are also durable to more than 100 hospital washes (75°C) but only with detergents without bleach. This is simple application method and compatible with all dyes. But, there is high formaldehyde release during application and during storage and use. So the treated fabric cannot be used for children's sleep wear.

Despite these restrictions, THPX- and MDPA-based FRs have been used until present days with no substantial alternative and are applied to textile materials by pad-dry method

1.3. The new flame retardant agents

1.3.1. Requirements for The new flame retardant agents

A good flame retardant for cellulosic should ideally have following properties:

- Prevent ignition
- Delay the spread of fires
- Delay the time of flashover to allow people time to escape
- Longer durability
- Prevent loss of physico-mechanical properties of the substrate
- The cross-linker and or binder copolymer should also eco-friendly
- Ease of application
- Prevention of immediate local pollution to air and water
- Prevention of lesser-known long-term environmental effects
- Cost-effectiveness

1.3.2. Alternative flame retardants

Biomacromolecules

The biomacromolecules most frequently used in fire-resistant and fire-retardant treatments for textile materials in laboratory conditions are chitin derivatives, casein proteins, wheys, hydrophobins and DNA [9].

Recently the application of bio-macromolecules, such as proteins (whey proteins, caseins, and hydrophobins) and deoxyribonucleic acid (DNA), is gaining major momentum as a research topic because they have shown appreciable results of flame retardancy for cellulosic as well as synthetic substrates [7]. The major advantages can be outlined as follows [7, 9]:

1. Flame retardancy obtained is quite significant and cab be on par with conventional phosphorus-based flame retardants in some cases. However, the complete understanding of the mechanism by which to confer flame retardancy to fabrics is still under investigation.

2. Ease of application such as impregnation/ exhaustion/layer-by-layer depositions are commonly practised for the chemical finishing of textiles.

3. Being bio-macromolecules, the process imposes a low environmental load and toxicity because they are usually dissolved or suspended at low concentrations in aqueous media; no volatile organic carbon (VOC) species are produced.

4. Some of these bio-macromolecules, such as caseins (whey proteins), can be considered as by-products/waste products from the agro-food industry.

5. The recovery and subsequent use of bio-macromolecules as flame retardants may be useful in the valorization of agro-food crops, thus avoiding their landfill.

6. Although bio-macromolecules are currently expensive, their availability has become more competitive with diminishing cost because a large-scale method of production was recently developed. They proposed a new large-scale method of extraction and purification of DNA from salmon milt and roe sacs

7. Chief disadvantage of all of these environment friendly treatments is their poor fastness to washing, even at 30°C with no detergent used.

1.4 New processes of applying FRs to cellulose textile fabrics

1.4.1. Nanoparticle Adsorption [7, 8, 9]]

Nanoparticle adsorption is a simple, fast and cheap, but not permanent process. This is one of the easiest way of surface modification with nano-particles just by simply immersing the fabric into an aqueous suspension of nanoparticles, while bonding is based on ionic interaction of negatively charged textile substrate and positively charged nanoparticle to enhanced the flame retardancy properties of both cellulosics, polyester fibres and their blends as well as the possibility of application to other fibres. The mechanism developed is an inorganic shield that protects underlying polymer from heat, oxygen, and flames because these nanocoatings can act as thermal insulators. This type of coating can entrap volatile species produced by the substrate, thus reducing the fuel for further combustion; rather the substrate tends to pyrolyse instead of burn. Nanoparticle adsorption is classified as a single-layer coating. It includes natural and synthetic zeolites (montmorillonite, klinoptilolit), nanoclays (carbonate hydrotalcite, sulphonate bohemite), nanoparticles (zinc oxide, titanium dioxide, silicon dioxide, octapropylammonium polyhedral oligomeric silsesquioxane [9]. The efficacy depends on the length of immersion time, the pH of the nanoparticle dispersions, and the textiles.

Many of plants and their parts contain phosphorous and other minerals. The flame retardant functionality imparted by two such plant extracts of Banana pseudo stem sap (BPS) and Spinach juice (SJ) for FR finishing of cellulosic and lignocellulosic textiles. BPS, being an agro-waste plant extract, and SJ, being a vegetable extract, are rich in phosphorous, nitrogen, chlorine, silicate and other many metallic compounds. BPS coating may act as an intumescent that swells on heating to protect the underlying cellulose polymer from heat or flame. Unlike BPS, SJ can be applied to cotton textile directly without any premordanting. Even an 8 % add-on showed increased LOI to 30 from 18; the fiber neither caught flame nor presented afterglow for 400 s (the control sample burned completely within 60 s at 400-450°C). However, a reduction in LOI from 30 to 22 after soap washing was noted. Both BPS and SJ, so therefore, can be regarded as semi durable finishings [8]

1.4.2. Sol-gel process

Sol-gel process is a simple, inexpensive and environmentally friendly procedure of synthesizing homogenous metal oxides, or organic-inorganic hybrids (dual-cure sol-gel) of good mechanical, optical, electric and thermal properties, at the temperatures below 100 °C. The procedure includes hydrolysis and condensation reactions of metal alkoxides (precursors), which changes the colloidal solution (sol) into s solid gel with uninterrupted three-dimensional metal oxide network, with an acid or alkali as a catalyst. The process of applying FR by sol-gel technique onto a textile material starts with precursor hydrolysis. Hydrolysed precursor is then added into the bath with organic FRs, which is then used to impregnate the textile fabric to be treated. Drying follows after impregnation, together with condensation, creating a solid gel on the fabric [9]

1.4.3. Plasma treatment [7, 9]

Plasma treatment is a process in which functional groups and macromolecules are synthesised by grafting onto the surface of textile fabric, with no internal modification of the textile, through [9]:

1. etching fabric surface and/or functionalisation with the help of nonpolymerizing gases (N_2, H_2, O_2, Ar, NH_3, CO_2 etc.)
2. polymer synthesis with the help of shoots from non-volatile kinds of phosphorus in cold plasma,
3. deposition of organosilicon compounds with the help of plasma polymerisation,
4. using cold N_2 plasma technique,
5. using acrylic monomers for graft polymerisation. Cold plasma in flame retardant treatments of cellulosic fabrics offer satisfactory values of limiting oxygen index, even after 50 washing cycles (27%). However, the process has not been widely accepted by the industry, primarily due to high necessary investments, as compared to the conventional commercial FR processes, which results in too high a price for the final product.

Plasma treatment has been reported either as a pre-treatment to increase the uptake of fire-retardant chemicals, for graft polymerization of acrylate phosphate and phosphonate derivatives, or as a posttreatment for better reaction. In such cases, organosilicon compounds are deposited by plasma polymerization. The use of nitrogen by cold remote plasma technique has also been studied as the use of acrylic monomers for grafting reactions. Atmospheric pressure plasma has become easily and efficiently applied to improve the functional properties of cotton fabrics as reported by Bourbigot and Duquesne [7].

SiO_2-atmospheric pressure plasma (APP)-coated flame-retardant cotton textiles with enhanced thermal properties and improved flame retardancy have been obtained in a process where a dense, thin film of tetramethyldisiloxane (TDMS) monomer (premixed with oxygen) forms an SiO_2 network armor through hydrolysis and condensation of the precursor TEOS, which becomes crosslinked on the surface of the cotton fibre. The SiO_2 network was found to be stable on fabric surfaces irrespective of intense ultrasound washing. These nanocomposite structure polymers form a surface protective layer the presence of carbonaceous and silica-like layers act as a barrier, which also slowed down toxic gas formation [7]

1.4.4. Layer-by-layer deposition

Layer-by-layer deposition (LbL) is a surface adsorption of long-chain polyelectrolyte molecules of one charge (+) on a solid substrate of the opposite charge (-), followed by rinsing with deionised water. The second phase consists in linking positively charged polyelectrolyte to negatively charged polyelectrolyte. The process is alternately repeated. It is thus possible to arrange a few layers of the same or completely different electrolytes one on the other as a bilayer (BL), trilayer (TL) or quadlayer (QL). LbL coating has been experimentally tested for FR treatments of textile fabrics, using various FR agents. Chief disadvantage of this method is again poor wash-fastness, as polyelectrolyte links are based on electrostatic bonds or H-bonds. Somewhat better results have been achieved by post-treatments of UV cross-linking. The advantage of this process is its simplicity, possibility to control the number, thickness and homogeneity of individual layers (which depends upon the choice and concentration of polyelectrolytes, solution pH, additional ultrasonicating etc.), as well as the usage of green solvent - water. LbL coating is implemented in laboratories using the following techniques: dipping and horizontal or vertical spraying. Only a few papers could be found dealing with possible commercial approaches to a continuous industrial LbL process of coating textile materials by dipping technique. LbL coating has until now been applied to the following cellulosic fibres: cotton, ramie and sisal [7]

1.4.5. Microcapsule Technique

Melamine-formaldehyde polymer-wall microcapsules with a triphenyl phosphate core were applied to both a cotton woven and a polyester nonwoven fabric using impregnation and screen printing. The results show that the microcapsules with triphenyl phosphate can be successfully applied to cotton and polyester fabrics using screen printing and impregnation methods, but the fire retardation was successful only at the highest concentration of the microcapsules. At this concentration, the mechanical properties of the starting materials appear to deteriorate. But the chief disadvantage of this technique is also poor wash-fastness [12]

1.5. Conclusion of literature review

The review of literature show that Phosphorus based flame retardants (THPX- and MDPA-based FRs) are the most effective for cellulose textile until present days with no substantial alternative. Their chief disadvantage is that they release free formaldehyde during condensation, or impregna-tion of textile material, as well in use. As an alternative to formaldehyde-free flame retardants cross-linking agents based on polycarboxylic acids were developed. However, their chief disad-vantages were that considerable strength reduction of treated fabrics. Moreover, they had a detrimental impact on the colouring shade, as well as on the pH value of the fabric treated. Acidic environment and high temperature during curing process are the main causes of the fabric strength reduction. So it need to use formaldehyde-free flame retardants cross-linking agents, apply the new technologies which can support the cross-linker to form covalent bond between substrate and FR but don't need to carry out at very high temperature.

The new processes of applying FRs to cellulose textile fabrics that have been presented above shown that their main advantage is that they are environmentally friendly and do not affect the mechanical properties of the material. Their main disadvantage is poor washing performance (except plasma treatment). Plasma treatment can support to increase the uptake of fire-retardant chemicals by grafting onto the surface of textile fabric, with no internal modification of the textile. For these reasons, plasma treatment have been considerably studied to develop durable flame-retardant cotton.

2. Experimental

In this study, Pekoflam DPN.CN liq - a commercial FR agent from Clariant was used as FR agent for durable flame retardant finish of cotton and 65/35 polyester cotton blend fabrics. Pekoflam DPN.CN liq Pekoflam® DPN.CN is an organic phosphorus compound for the permanent flame-retardant finishing of cotton and cotton-rich blends. It provides excellent wash resistance and - depending on the cross linker system - is effective at up to 95°C. It is low yellowing and suitable for Öko-tex Class IV requirements.

2.1. Materials

2.1.1. Fabric

Mercerised 100% cotton fabric with a fabric density of 230 g/m^2.

Pre-treated cotton polyester blend fabric in which, polyester composition is of 65% and cotton composition is of 35%, fabric density is of 200 g/m^2

Both fabrics were supplied by Namdinh Co., Ltd.

2.1.2. Chemical

Pekoflam DPN, Arkofix NED, Catalyst NKC, Ceraperm SFC, Nuva TTC were supplied by Clariant Vietnam.

Phosphoric acid was purchased from Ducgiang Chemical Co., Ltd.

Arkofix NED - cross-linking agent, it is a modified DMDHEU

In this study, Catalyst NKC and Phosphoric acid are compared to each other to clarify their role as a catalyst in the reaction between flame retardants agent, crosslinkers and cellulose.

Nuva TTC dispersion of perfluoroalkylacrylate, finishing product for extremely durable water-and-oil repellent finishing of textiles made of cellulosic fibres, like cotton and also for synthetics like polyester and blends. It is used in this study to enhance the water repellency of treated fabric

Ceraperm SFC liq, which has been developed for use with fluorocarbon products (Nuva TTC). In contrast to most other softeners, and especially existing silicones, it has no negative influence on water and oil repellency. Ceraperm SFC provides a very soft and pleasing hand on finished knitwear goods and also improves sewing properties and helps to avoid stitching damage.

2.2. Methods

2.2.1. Chemical composition of padding solution for fire retardant finish of cotton fabrics

Mercerised cotton fabric was treated with various recipes composition of FR argent, cross-linking agents, catalysts (from PK 1 to PK 7) as shown in ***Table 1*** to find the most effective recipe.

Table 1

Recipes of padding solution

Chemical agent	Concentration g/l	Recipe						
		RK 1	RK 2	RK 3	RK 4	RK 5	RK 6	RK 7
Pekoflam DPN	400	x	x	x				
Arkofix NED	60	x	x	x				
Pecoflam DPN	525				x	x	x	x
Arkofix NED	80				x	x	x	x
Catalyst NKC	15	x			x		x	
Phosphoric Acid	30		x	x		x		x
Ceraperm SFC	30	x	x		x	x	x	x
Nuva TTC	80	x	x		x	x	x	x
Distilled water	-	x	x	x	x	x	x	x
Time of curing (minutes)		2	2	2	2	2	2	2

The chemical composition of recipe PK 6 is similar to which of PK 4 and that of PK 7 is similar to which of PK 5, which differ only in curing time.

2.2.2. Application and fixation of the solution FR agent onto cotton fabric

One bath, pad-dry-cure technique was used for the application and fixation of the chemicals onto fabric. All the process is as follow: each sample was dipped in the chemical solution prepared before with desired recipe, then the SDL D394A padder was used for the padding the chemical solution on to the fabric at the wet pick up of 80%. After that, the padded fabric was dried in Stenter SDL D398 at 110°C during 4 minutes, curing at 170°C during 3 minutes for PK 6, PK 7 and during 2 minutes for all other cases in the same Stenter. Neutralization was in solution of 30 g/l Na_2CO_3 at 80°C during 10 minutes, rinsing in the water at 80°C during 2 minutes and then, the fabric was rinsed in cold water during 10 minutes. In the end, fabric was dried in the stenter at 110°C during 3 minutes. The treated fabrics were conditioned for 24 h at 20 ± 2°C and 65 ± 4% relative humidity before conducting any testing in a controlled room by using the conditioned chamber Model M 250 - RH made by MESDAN - Italy.

2.2.3. Method of flame retardant finish for 65/35 polyester cotton blend

Pre-treated 65/35 polyester cotton blend fabric was finished with the optimum chemical recipe found by the study with cotton fabric. It is used above process for application and fixation of the solution FR agent onto fabric with the curing time of 3 minutes. This study is encoded as PK 8.

2.2.4. Testing methods

Flammability test: Standard method of ASTM D1230-94 (reapproved 2001) was used to assess the flammability of the cotton fabric before and after treatment. The ignition time, afterflame time, afterglow time, char length of the specimen were used to assess the burning behavior of fabrics. 45 degree flammability tester was used for this test.

Mechanical properties of fabrics were assessed by testing tensile strength and tear resistance of fabrics by using universal tensile tester RT- 1250A

TCVN 1754-86 reapproved 2008 (equivalent to ISO 13034-1) was used to measure the tensile strength of the cotton fabric before and after treatment.

ISO 4674-1 was used to determine the tear resistance of the cotton fabric before and after treatment.

Drape index of fabrics was used to assess their stiffness, which was determined according to the method of NF G07-109 standard.

Air permeability and water vapor permeability of fabrics were tested to assess their comfort.

Air permeability of fabric was determined according to the method of ISO 9237:1995 standard in using machine M021A SDL ATLAS.

Water vapor permeability of fabric was determined according to the method of UNI 4818 standard.

Water resistance of fabrics are evaluated by theirs hydrostatic pressure, which were tested according to the method of ISO 811 standard by using M 018 - SDL Atlas.

Formaldehyde content of fabric was determined according to the method of ISO ISO 14184 - 1 standard.

Washing (laundering) test.

The treated cotton fabric was washed according to the method of ISO 6330 standard, procedure 8A[i] in using washer type A (Electrolux washing machine), Omo Matic washing powder was used as detergent. Flammability of the treated samples after 3 washing cycle were determined to assess the durability of the flame retardant finish.

3. Results and discussion

3.1. Study on the most effective chemical composition of padding solution

All the used chemical recipes were evaluated based on the fire behavior of the cotton fabric after treatment and after 3 washing cycles. Due to the addition of water repellent agent NUVA TTC, so the water repellency of fabric after treatment and after 3 washing cycles were also assessed. The results of tests on the fire behavior and water resistance of fabric are presented in ***Table 2***.

Table 2

Parameters relating to the burning behavior and water resistance of fabric

Option	Parameters relating to the burning behavior and water resistance of fabric					
		Ignition time (s)	**Afterflame time (s)**	**Afterglow time (s)**	**Char length (mm)**	**Hydrostatic pressure (mbar)**
Control sample	Lengthwise	3	21		0	0
	Widthwise	3	20		0	0
PK1 after treatment	Lengthwise	>90	0	0	37	12.4
	Widthwise	>90	0	0	37	
PK1 after 3 wash	Lengthwise	6	52	0	140	0.3
	Widthwise	6	54	0	140	
PK2 after treatment	Lengthwise	>90	0	0	38	14.5
	Widthwise	>90	0	0	38	
PK2 after 3 wash	Lengthwise	6	57	0	140	0.9
	Widthwise	6	57	0	140	
PK3 after treatment	Lengthwise	>90	0	0	39	0
	Widthwise	>90	0	0	38	
PK3 after 3 wash	Lengthwise	10	55	0	140	0
	Widthwise	10	59	0	140	
PK4 after treatment	Lengthwise	>90	0	0	39	12.8
	Widthwise	>90	0	0	38	
PK4 after 3 wash	Lengthwise	10	57	0	140	2.6
	Widthwise	10	58	0	140	
PK5 after treatment	Lengthwise	>90	0	0	40	12.7
	Widthwise	>90	0	0	38	
PK5 after 3 wash	Lengthwise	10	62	0	140	2.9
	Widthwise	10	68	0	140	
PK6 after treatment	Lengthwise	>90	0	0	38	13.3
	Widthwise	>90	0	0	36	
PK6 after 3 wash	Lengthwise	10	58	0	140	3.3
	Widthwise	10	62	0	140	
PK7 after treatment	Lengthwise	>90	0	0	31	18
	Widthwise	>90	0	0	35	
PK7 after 3 wash	Lengthwise	12	62	0	140	4.8
	Widthwise	12	72	0	140	

Cotton fabric without any treatment could not pass the flammability test and was completely burnt, suggesting on cotton poor fire retardancy. While, Pekoflame treated cotton fabric have a good flame retardancy, and burning of the fabric was stopped immediately after removing burning source.

There was no significant difference in the char length between samples treated with Catalyst NKC as catalyst (Pk1) and sample treated with phosphoric acid as catalyst (PK2), between samples treated with NUVA TTC (PK2) and sample without NUVA TTC (PK3), between the samples treated with pekoflame DPN at the level of 400 g/l (PK1, PK2) and samples treated with pekoflame DPN at the level of 525 g/l (PK4, PK5).

Nevertheless, the sample of PK7 has the shortest char length. It means that the sample that was cured in 3 minutes demonstrated good enhancement in the flame retardancy in comparison with other samples that were cured only in 2 minutes.

The results of testing of burning behavior of the treated samples after 3 washing cycle show that flame retardancy of the samples is significantly poor. Ignition time of the sample PK1 and PK2 is smallest (only 6 seconds), that of other samples is 10 seconds and the longest afterflame time is that of PK7 sample, it is up to 12 seconds. It seems that the Pekoflam content, catalyst and curing time play important role in improvement of wash fastness of cotton flame retardant treated fabric. Ignition time of PK4 and PK5 (treated with Pekoflame at the level of 525 g/l) is 10 seconds in comparison with that of PK1 and PK2 (treated with Pekoflame at the level of 400 g/l) it is only 6 seconds. But that of PK7 (cured in 3 minutes) is 12 seconds while that of PK5 (cured in 2 minutes) is only 10 seconds. Nevertheless, ignition time of PK6 (cured in 3 minutes) is not higher than that of PK4 (cured in 2 minutes). It shows that longer curing time is effective only with the catalyst phosphoric acid.

*Therefore, it can be conclude that between 7 option that presented in the **Table 1**, the most effective flame retardant finish for cotton fabric is PK7.*

3.2. Evaluation of other properties of flame retardant treated cotton fabrics

Mechanical and physical of cotton fabric which was treated follow the process of PK7 were tested according to the methods that are described in section 2. The testing results are presented in the ***Table 3***.

Table 3

Physico-mechanical and ecological properties of cotton fabric treated by option PK7

		Properties	Control fabric	Treated fabric	Difference Δ, %
Tensile property	Force at rupture (N)	Lengthwise	650,6	467,15	- 28,19
		Widthwise	232,7	202,9	- 12,81
	Elongation at rupture (%)	Lengthwise	10,61	9	- 15,17
		Widthwise	12,44	10,17	- 18,24
Tear resistance of fabric, N		Lengthwise	24,24	11,36	-53,13
		Widthwise	27,56	13,60	-50,65
Drapery index of fabric, %			58.89	53	-10,00
Air permeability of fabric, $dm^3/m^2.s$			148	156	+5,41
Moiture regain of fabric (%)			9,3	9,6	+3,20
Water vapor permeability, $g/m^2.h$			43,32	46,67	+7,70
Formaldehyde content (ppm)			<16	<16	

The results of ***Table 3*** show that the flame retardant treatment for cotton fabric reduced its mechanical strength. The loss of tensile strength is acceptable (28% in lengthwise fabric and 13% in widthwise fabric), but, it is quite important for tear resistance of fabric (≈ 50 %). Breaking elongation of fabric also reduced (≈ 15 %). Perhaps the Chemical environment (cross-linking agent) and high temperature curing (at 170°C during 3 min) caused this loss.

The reduction in the drapery index is 10 %, that means stiffness of the fabric decreased. Air permeability, Moisture regain, Water vapor permeability slightly increased that means this flame retardant finish don't affect the comfort properties of cotton fabric.

3.3. Results of study on flame retardant finish of cotton polyester blend fabric by Pekoflam DPN

As mentioned above, the cotton polyester blend fabric was treated with the most effective chemical recipe between that which were applied to cotton fabric. Follow the results of ***Table 2***, the best effective recipe is PK7. Therefore, PK7 recipe was used to cotton polyester blend fabric, process, which is described in the subsection 2.2.2 is used for application and fixation of the solution FR agent onto cotton polyester blend fabric with the curing time of 3 minutes. The testing results of burning and other properties of fabric before and after treatment are presented in ***Table 4***.

Table 4

Burning and other properties of cotton polyester blend fabric before and after flame retardant treatment

Properties			Testing results of		Difference Δ, %
			Control fabric	Treated fabric	
Burning behavior	Ignition time (s)		8	>90	
	After flame time (s)	Lengthwise	27	0	
		Widthwise	32	0	
	Char length (mm)	Lengthwise	0	38	
		Widthwise	0	46	
Water resistance	Hydrostatic pressure (mbar)		1,93	13,46	
Tensile property	Force at rupture (N)	Lengthwise	974,64	880.15	-9.7
		Widthwise	344,61	348.07	+1.0
	Elongation at rupture (%)	Lengthwise	17,18	14,62	-14.9
		Widthwise	22,9	18,9	-17.5
Tear resistance (N)		Lengthwise	23,71	15.62	-34.1
		Widthwise	31,62	17.27	-45.5
Drapery index of fabric, %			65,14	59.94	- 8
Air permeability of fabric, $dm^3/m^2.s$			24	17.1	-28.8
Moiture regain of fabric (%)			7,26	7.42	≈
Water vapor permeability, $g/m^2.h$			36,66	37,5	≈
Formaldehyde content (ppm)			<16	<16	≈

From the results of ***Table 4***, a few remarks can be drawn as follows:

- Similar to the cotton fabric, cotton polyester blend fabric without any treatment could not pass the flammability test and was completely burnt.
- Burning parameters Pekoflame treated cotton polyester blend fabric are quite good, its ignition time is over than 90 seconds and after flame time is zero second, that means it has a good flame retardancy, burning of the fabric was stopped immediately after removing burning source. However, its char length is longer than which of pekoflam cotton treated fabric.
- Same as the cotton fabric, flame retardant finish negatively affected to the mechanical properties of cotton polyester blend fabric, there were the decrease in the force at rupture, elongation at rupture and tear resistance of cotton polyester blend fabric. However, this reduction is smaller in comparison with that of cotton fabric. May be, both factors which could cause the reduction of mechanical strength of fabric are the chemical environment and high curing temperatures, could not yet affect the polyester composition of blend fabric, it make the difference between these two kinds of fabrics.
- Air permeability of Pekoflame treated cotton polyester blend fabric decreases of 28.8%. May be, flame retardant on the fiber surface is responsible for this reduction. Drapery index of fabric after treatment also decreases, that means, after treatment fabric has become softer.
- Other properties of Pekoflame treated cotton polyester blend fabric such as moiture regain of fabric, water vapor permeability and formaldehyde content are almost unchanged.

3.4. Conclusion

- The presented technology (PK7 recipe, curing at 170°C during 3 minutes) permit to have the good flame retardant cotton and flame retardant cotton polyester blend fabrics. However, It does not yet allow to have durable fire retardant cotton flame retardant cotton polyester blend fabrics, these flame retardancy is easily reduced after 3 wash cycles. May be, the hard condition of neutralization step and the use of commercial washing powder as detergent in the washing cycles could cause the deterioration of flame retardancy of fabrics. In order to enhance the durability of the flame retardancy of treated fabric, I need to improve the used technology.

- A dramatic decrease in mechanical strength of fabrics especially of cotton fabric is also a limitation of this technology. In the further studies, it also need to find solutions to control this limitation

3.5. Further studies on durable fire retardant treatment for cotton and cotton polyester blend fabrics

In order to improve durability of fire retardancy and other properties of treated flame retardant cotton fabric, the following measures are carrying out

- Combination of Pyrovatex CP New (an other organic phosphorus flame retardant agent) with various cross-linkers to obtain the optimal formulation that allows to have a durable fire retardant cotton fabric and least impact on the mechanical strength of fabric.

- Application atmospheric plasma in flame retardant process to support the bonding between the flame retardant agent and fabric. That may allow to reduce the temperature and duration of the curing process, or diminish the amount of crosslinking-agent to be used. These are two factors, which are assumed to be responsible for the mechanical deterioration of the fabric.

Acknowledgement

This research is in the framework of project KC.02.13/16-20. The authors would like to thank the Program KC. 02/16-20 for financial support.

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