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Banana pseudostem sap and boric acid— A new green intumescent for making self- extinguishing cotton fabric

S Basak[a], Kartick K Samanta[b], S K Chattopadhyay, S Saxena & R Narkar

Department of Chemical and Biochemical processing Division,
ICAR- Central Institute for Research on Cotton Technology, Adenwala Road, Matunga, Mumbai 400 019, India

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The flame retardant functionality has been imparted in cellulosic fabric using mixed formulation of banana pseudostem sap (BPS) and boric acid (BA). The extracted sap is mixed with different concentration of BA and applied onto the pre-mordanted bleached and mercerized cotton fabrics at elevated temperature. It is found that BA acts as a strong afterglow and smoke arresting agent, when applied at the concentration of > 2% (w/v). Flame retardant characteristics of both the control and the treated fabrics have been analysed in terms of limiting oxygen index, vertical flammability and temperature generation profile during burning. The (BPS+3% BA) treated cotton fabric sample shows the LOI value of 42 and the specific char length of 14cm after vertical flammability test. The thermal degradation and pyrolysis mechanism are also studied, using both thermogravimetric analysis and fourier transform infrared spectroscopy. Besides, the charring morphology and mechanism of both the control and the treated fabric is analysed in detail by scanning electron microscopy and FTIR analysis. A char structure model and the mechanism of char formation have also been proposed in the paper.

Keywords: Banana pseudostem sap, Boric acid, Cotton, Flame retardant fabric, Self-extinguishing fabric

1 Introduction

Cellulosic cotton textile is flammable as it catches flame readily and poses a serious risk to health and life of a living being. Significant efforts have been made from the past to improve the flame retardant property of cellulosic textiles using various synthetic chemicals and many of them are available in the market. The most simple and common ecofriendly formulation used is borax and boric acid mixture[1]. However, larger quantity of chemicals, used in this formulation, detoriates the quality of the treated fabric. Phosphorous based flame retardants along with nitrogenous and sulphur compound are the most-effective formulation reported so far, due to their synergistic effect. Consequently, from the last fifty years, different flame retardants based on the composition of phosphorous, nitrogen, and sulphur has come into the market for imparting flame retardancy. Among all these chemicals used for flame retardancy, Tetrakis phosphonium salt (Proban process) and N-alkyl phosphopropionamide (Pyrovatex process) derivatives are widely dominate commercially[2]. However, as such formulations need to be applied in an acidic condition, the cotton fabric loses its tensile strength and becomes stiffer. Besides, such a treatment is expensive and non-ecofriendly due to the involvement of larger quantity of chemicals, high temperature curing process and toxic formaldehyde emission during treatment if the process is not properly controlled[3]. Antimony in combination with halogen, though could impart good flame retardant property, but still is not very successful due to the negative impact of halogen compounds in the environment[4]. Therefore, as now-a-days sustainability and eco-friendliness are the major concerns for the researchers and textile industries, some intumescent based flame retardants, which work by charring and foaming mechanism have come into the market. For example recently researchers have used sodium metasilicate nonahydrate for making a fire retardant cellulosic jute textile[5]. A composition of nano zinc-oxide and polycarboxylic acid[6] have also been developed by the researchers to make the fire retardant process environment friendly, However, such treatment cannot satisfy the handle, strength and the fire resistant durability requirements of the fabric. Recently, researchers have also used plasma treatment with various polymerisation gases to impart the fire retardancy property to the cellulosic fabric. Though the plasma process is water free and ecofriendly, but it is very costly and also the imparted flame retardant

[a] Corresponding author.
E-mail: shantanubasak@gmail.com
[b]Present address: National Institute of Research on Jute and Allied Fibre Technology, 12, Regent Park, kolkata 700 040, India

property is not wash durable[7]. Hence, there is the need to develop more cost-effective, environment friendly and sustainable fire retardant products, which when applied to cotton fabrics, will maintain its quality and flame retardant durability to a great extent. A very few researches have so far been reported on imparting fire retardancy to cellulosic fabric using natural products[8-12]. Recently, the researchers reported that DNA from herring sperm and solomon fishes can be applied to the cotton fabric to make it thermally stable[10]. Attempts have also been made to impart fire retardancy in cotton fabrics with whey proteins, casein and hydrophobins due to their phosphate, disulphide and protein content, as they can influence the pyrolysis by an early char formation[10,11]. However, a limited application of waste plant bio-molecules has been made for imparting flame retardant finishing to any textile and/or polymeric material till date to the best of our knowledge. As some of the plants contain phosphate, phosphite, chloride, silicate and other minerals, metal oxides and mineral salts, they offer immense potential to be utilized to impart flame retardancy to cellulosic and non-cellulosic textiles. In our earlier publications, we have reported a detailed study of imparting fire retardant to cellulosic cotton fabric, using spinach juice[13] and wastage banana pseudostem sap (BPS)[14-16]. However, the main drawback of that finish is the presence of smoke and afterglow, during burning, which is very dangerous for the users of the textiles. Therefore, for arresting afterglow, and smoke, and for making the developed process more effective and user friendly, an attempt has been made in the present research to use a mixed formulation of BPS and boric acid. The treated fabric has been evaluated by different flammability tests, besides thermal and chemical characterization.

2 Materials and Methods

2.1 Materials and BPS Application

A 200 GSM (areal density) plain woven bleach cotton fabric of 30 EPI (ends/ inch) and 40 PPI (picks/inch) procured from the local market was used for the flame retardant finishing. Banana pseudostem sap (BPS) was supplied by Navsari Agricultural University, Gujarat, India for the treatment. The supplied sap has a *p*H of 7 (neutral). This sap was mixed with 2-4% boric acid (BA). The bleached cotton fabric was first mordanted with 5% tannic acid and 10% (ω/ν) alum. Thereafter, the mordanted fabrics were impregnated in the mixed formulation of BPS and BA, maintaining material-to-liquor ratio of 1:10. The fabric was then treated for 30 min, followed by drying at 110ºC for 5 min. Another two cotton fabric was treated separately with only BA and BPS in same condition for comparison of flammability behaviour.

2.2 Determination of % Add-on

Before any physical and chemical characterization, both the treated and the control cotton fabrics were conditioned at 65% RH and 27ºC temperature for 48 h. After the application of BPS formulation on cellulosic cotton textile, the add-on, i.e. the increase in sample weight, was determined by gravimetric principle from the bone dry weights of the sample before and after the treatments. The results are expressed in percentage over the initial weight of the sample, as shown below:

$$\% \text{ add-on} = [M_2\text{-}M_1/\ M_1]^* 100$$

where M_1 and M_2 are the oven dried weight of the control and the BPS treated samples respectively. The reported results are average of 5 readings.

2.3 Measuring BPS Concentration

In the study, banana pseudostem sap (BPS) as it is extracted from the pseudostem of the banana plant has been used for experiment. BPS showed peak absorbance at 220 nm. BPS was dried at 100°C for 5 min. Thereafter, different concentrations (0.1, 0.2, 0.3……0.9, 1 g) of BPS solution were made by mixing and dissolving the dried BPS with water in a 100mL volumetric containers. The absorbency of those solutions was measured at 220nm in UV-VIS spectrophotometer (Model: Lambda 25, Perkin Elmer). By following the absorbency and concentration data of different solutions, a curve was plotted. The absorbency of the BPS used for the treatment was also measured in spectrophotometer at 220nm and the value was pointed in the curve. It has been found that the concentration of the as prepared BPS solution extracted from the banana pseudostem is 23.4g/ L.

2.4 Thermal Characterization

2.4.1 Flammability Assessment

The burning behaviour of both the control and the treated samples was evaluated by standard methods. For the limiting oxygen index (LOI) analysis, IS 13501 test method was used. In vertical flammability, different parameters were measured as per IS1871 method A. The maximum temperature produced during the burning of a sample was measured using IR thermometer (Model: 15077968 FB61354 225PE Fisher Scientific) in non-contact mode. Based on the

data, the temperature generation profile curve was plotted for both the control and the treated fabric.

2.4.2 TG Analysis in N_2 Atmosphere

TG curves of both the control and the treated fabrics were obtained by using a Thermo gravimetric analyser (Mettler Toledo TG-50/ MT5) at a heating rate of 10°C/min and N_2 atmosphere.

2.4.3 SEM Analysis

All the control and the treated sample surfaces were characterized by scanning electron microscopy (SEM). In addition to it, the char analysis of both the control and the treated samples was done using scanning electron microscope (Philips- XL30). The samples were coated with a thin layer of conducting material (gold/ palladium) by using a sputter coater, and the same were examined under the SEM with an accelerating voltage of 12 kV.

2.4.4 FTIR Analysis

The FTIR analysis of the control and the BPS treated samples was carried out in Shimadzu IR analyser over the wavelength of 500 - 4500 cm^{-1} using KBr disc sample preparation method. Here, the ATR transmittance mode with DLaTGS detector, 49 scans and 4 resolutions were used to perform the FTIR analysis.

2.4.5 Durability Test

Washing durability of the flame retardant finishing was evaluated after washing the samples in a laundrometer using a standard detergent (1g/L) at 40ºC for 40 min. The fabric was then rinsed in fresh water for 5 min, followed by drying at 100ºC for another 5 min. The samples were then conditioned in a desiccator for 24 h under standard atmosphere. Rub fastness of the treated fabric was also measured as per IS 766 method. After rubbing, the LOI value was evaluated as per the standard method. Treated and untreated cotton fabric samples were also tested against dry-cleaning as per IS: 4802-1988 method. Like in the rubbing test, the LOI values of the control and the treated fabrics after dry-cleaning were also measured as per the standard method.

3 Results and Discussion

3.1 LOI and Vertical Flammability

Textiles having an LOI value of ≥26 are considered as a fire retardant material[17]. However, as far as the cellulosic textile is concerned, the textiles having the LOI of >30 can only clear the vertical flammability test conducted by standard method. It is found from the Table 1 that the control cotton fabric (A) has been burnt within one minute. It is also found that 3% BA treated cotton fabric (C) shows a high LOI value of 29; however, this treated fabric burns fully in the vertical flammability test by generating blue flame. Besides, no afterglow is observed during burning, and also a hard black colour char mass is obtained as residue after the burning. On the other hand, only BPS treated cotton

Table 1 — Flammability parameters of the control and different formulation treated cotton fabrics [Vertical flammability 280mm* 50mm for both control and treated fabrics]

Flammability parameter	Control (A)	Treated cotton fabric			
		BPS (B)	3% BA (C)	BPS+ 1% BA (E)	BPS+ 3%BA (D)
Add on %	-	5	6.5	7.4	9
LOI	18	28	29	34	42
Occurrence of flash over the surface	Yes	No	No	No	No
After flame, s	60	10	60	Nil	Nil
Burning with afterglow time (s) after flame stop	30	295	Nil	360	50
Total burning time (s) (flame time+ afterglow time)	60 + 30	10 + 295	60+0[a]	0+ 360	0+ 50
Char length, mm	-	-	> 300	>300	140
Observed burning rate, mm/min	186.6	55.8	280	46.6	-
State of fabric in contact with flame	Completely burnt with flame (light weight grey colour char mass left)	Burnt initially with flame followed by afterglow (light weight black colour char mass left)	Burnt with blue colour flame (hard black char mass left)	Partially burnt	Fire retardant

[a]Sample catch flame after 7s contact of 38mm Bunsen burner flame.

fabric (B) shows resistance against the flame, though severe afterglow and smoke are present in it during the vertical burning. As far as (BPS+ 3% BA) treated (D) cotton fabric is concerned, it shows resistance against both flaming and afterglow propagation. On the contrary, (BPS+ 1% BA) treated (E) cotton fabric shows resistance only against the flame propagation. BPS and BA individually are shown by symbols F and G. As with 3% BA incorporation in BPS, treated cotton fabric shows a self extinguishing property along with a char length of 140mm. Figure 1 shows the vertical burning behaviour of the control (A) and the treated cotton fabrics (B,C,D) after 60s of vertical burning.

3.2 Temperature Generation during Burning

As far as vertical flammability is concerned, we have reported in Table 1 that the BPS treated fabric shows the flame for a few seconds and then, self extinguished exhibiting an afterglow, which helps to burn the total fabric much slowly as compared to the control fabric. On the other hand, (BPS+ 3% BA) treated fabric shows no flame and the self extinguishing behaviour of afterglow propagation. To understand this burning behaviour and the mechanism, the temperature of the fabric is measured during flaming as well as during the afterglow. The same is plotted in Fig. 2 depicting the temperature generation curves of both the control and the treated fabrics during the vertical burning in a real practical situation. It can be seen that the rate of temperature generation is higher for the control fabric as compared to the other treated fabrics (B, C and D) by 12.8, 25 and 50% respectively. The BA treated fabric shows almost an equal temperature generation like the control fabric due to the presence of blue flame during the burning. Concerning the temperature profile of the only BPS treated fabric, the results show a prolonged temperature (200- 300°C), due to the presence of afterglow during burning. As far as (BPS+ BA) treated fabric is concerned, it shows a much less initial temperature (75% less than BA and 50% less than BPS) than the only BPS and BA treated fabric due to the absence of flame. This proves the synergistic behaviour of flame retardancy of BPS and BA. Here, only afterglow is present for a few seconds that gets self-extinguished, reflecting a rapid fall of the temperature in the curve of (BPS+BA). It may be due to the fact that the intumescent coating of (BA+ BPS) on the fabric surface helps to absorb the temperature quickly by heat sink mechanism, discussed hereunder.

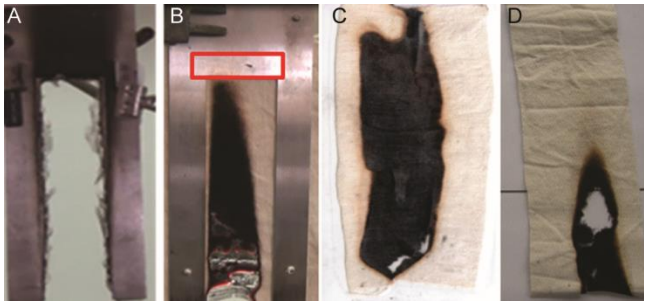

Fig. 1 — Vertical burning behaviour of control (A) , BPS (B) , 3% BA (C), and BPS +3% BA (D) treated cotton fabrics after 60s of burning [Red marked portion shows gelatine like viscous material after complete burning of the BPS treated cotton fabric]

3.3 SEM and FTIR Analysis

SEM morphology of the control cotton fibre (A) shows a smooth, clean surface whereas BPS treated cotton fabric (B) shows a uniform coating throughout the treated cotton fabric surface. 3% BA and BPS treated cotton fabric (D), depicted in Fig. 3, shows a glassy rough coating of boron trioxide on the cotton fabric surface. This glassy coating, formed around the fabric surface, prevents the sample to come in contact with the heat source. As far as FTIR analysis is concerned, BPS (F) shows the presence of phenolic –OH group and also the presence of different inorganic salts and oxides. FTIR analysis of the cotton fabric and the BPS treated cotton fabrics shows no significant changes. As far as FTIR analysis of BA (G) is concerned, it shows sharp B-O bands at 712 and 882 cm^{-1}. Peak observed at 2259 cm^{-1}, may be assigned due to the presence of a B-O band of BO_2 (BPS+ 3% BA) treated cotton fabric (D) shows clear peaks of B-O bands at 1025cm^{-1} and 1130 cm^{-1}. It proves the presence of insulating coating of boron trioxide on the treated cotton fabric surface.

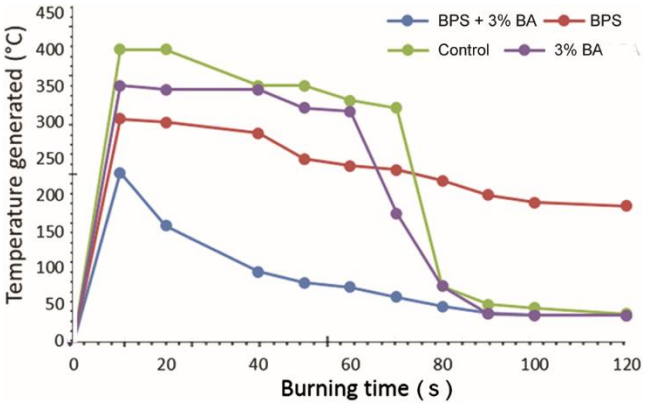

Fig. 2 — Temperature profile of control and treated fabrics during vertical burning

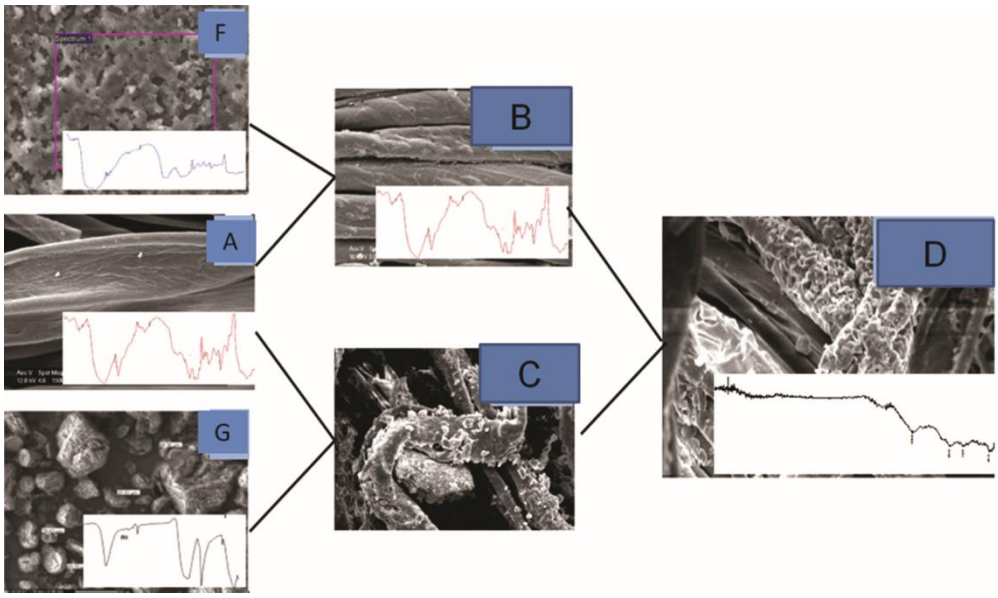

Fig. 3 — Fabric surface morphology and FTIR spectrum of the BPS only (F), cotton (A), BA only (G), BPS treated (B), 3% BA treated (C) and (BPS+3% BA) treated (D) cotton fabric.

3.4 TG Analysis

The TG curve of the control cotton fabric (A) shows a sharp fall at around 350°C due to the pyrolytic degradation of the cotton cellulose (Fig. 4). Besides, the amount of char residue left at higher temperature is also found less. In contrast, the BPS treated cotton fabric (B) shows a slow rate of degradation, and from the DTG curve of A and B it is seen that pyrolysis point shifts from 350°C to 310°C, may be due to the BPS catalyze dehydration of the cellulosic material. As a result, the amount of flammable gas like levoglucosan and pyroglucosan produced during pyrolysis is reduced as compared to the control cotton cellulose. Besides, due to the dehydration effect, the amount of the char mass generated at higher temperature is also more than in control cotton fabric. Coming to the TG curve of the only 3% BA incorporated cotton fabric (C), it shows the same lower rate of degradation and BA catalyze dehydration at around 300°C, which helps to reduce the amount of the flammable gas generation during pyrolysis. In addition to this, the thick insulated coating of BA also helps to increase the amount of graphitic char mass production (proved by char FTIR analysis) at higher temperature. On the other hand, treated fabric D shows almost a similar thermo-gravimetric behavior or pyrolytic mechanism like fabric C. However, from the vertical flammability test it is proved that the combine fire retardant action of (BPS+ BA) is better as BA is taking crucial role to stop the afterglow of the fabric. This mismatch concept of

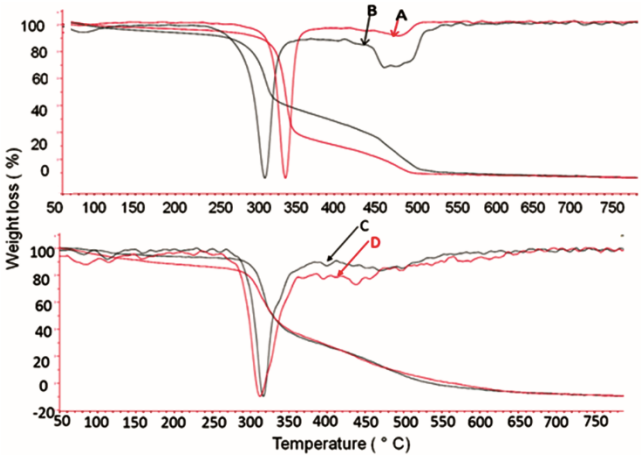

Fig. 4 — TG and DTG curves of the control cotton (A), BPS treated cotton (B), 3% BA treated cotton (C) and the mixture of (BPS+ 3% BA) treated cotton (D) fabrics

thermal stability of vertical flammability and TG analysis has been more clearly discussed in the later part of the paper, while discussing the char morphology and its characterization.

3.5 Char Morphology and Characterization

SEM morphology of the residual mass of the control cotton fabric (A) shows bird nest like open structure through which flammable gases can easily pass through and come in contact of heat source, whereas char mass of the BPS treated cotton fabric (B) shows a honeycomb like structure that restricts the flow of the flammable gases. However, still some capillary gaps

are present, that will help the flow of the gases throughout the polymeric structure. It might be one of the reasons for the lower fire retardant effect of BPS treated cotton fabric at neutral *p*H than the effect at alkaline *p*H[14]. In BA (1%) incorporation into the BPS (E), char micrograph shows a thick three dimensional glassy insulated coating over honeycomb parts, assisting to stop the afterglow. It also provides credence to the fact that during the treatment, the underlying polymeric material (cotton fabric) takes BPS first and then BA, forming the glassy coating on the BPS treated surface of the material. As far as the char mass of the fabric D is concerned, it only shows three dimensional bricks like polished coating arrangement, protecting the underlying surface to come in contact of heating and damaging. It may be due to the fact that while in contact of heat, BA expands and increases in volume and forms an intumescent like glassy insulated thick coating of boron trioxide on the cotton fabric surface. Based on the char morphology a char model has also been proposed (Fig. 5).

3.6 FTIR Analysis of Residual Char Mass

The char of the BPS treated cotton fabric (B) shows a strong peak at around 3426 cm^{-1}, assigned to the OH stretching vibration. Peaks observed at 2926 cm^{-1} may be responsible for the presence of –CH hydrocarbon. Small peak observed at 1248 cm^{-1}, may be assigned to the presence of P=O group. Small peaks are also observed at 1158 and 927 cm^{-1}, may be assigned to the polyaromatic structure and interconnecting network of P-O in P-O-C formed during the combustion[17]. Small peaks are also observed at 1081cm^{-1} , may be assigned to the presence of polyaromatic structure and interconnecting network of P-O in P-O-P. Peaks are observed at 1360cm^{-1}, may be due to the vibration of carbon atom in disordered graphite, representing unorganised carbon structure[18]. Curve D also shows almost same behaviour like curve B, as it shows the same peaks at 1430 and 1313cm^{-1}. However, the intensities of these peaks are higher than the sample B. It means that BA coating help to enhance the graphitic carbon structure after burning. Peaks are also observed at 1158cm^{-1} and 1029 cm^{-1}, may be assigned to the polyaromatic structure and interconnecting network structure formed during combustion.

3.7 Durability of Flame Retardant Finish

The durability of the imparted flame retardant finish has been determined to ensure the effectiveness of the finish against soap solution and rubbing. It is found that the LOI value of the treated (BPS+3% BA) fabric decreases from 42 to 24 after one ISO 2 washing. It might be due to the fact that after the washing, the glassy layer of boron trioxide is washed out from the fabric surface. The fastness of the treatment can be improved by the addition of lewis acid catalyst and organic acid like citric acid etc. into the BPS and BA based formulation and by performing a high temperature curing process for the crosslinking. Such process and the formulation may improve the fastness

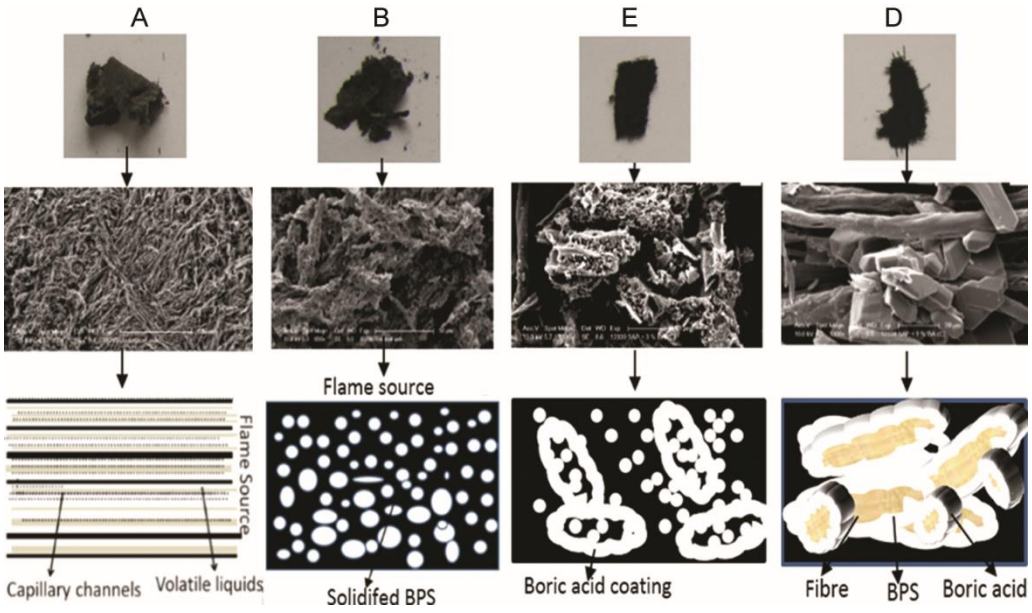

Fig. 5 — Char picture and morphologies of the control (A), BPS treated (B), BPS +1% BA (E) and BPS+3% BA (D) treated fabrics

properties of the treated fabric. Further research work is going on in this direction.

As far as the durability against rubbing is concerned, the treated fabric shows an LOI value of 42 and 41 after dry and wet rubbing respectively. From this experiment, it appears that this particular finish is only suitable for those applications where washing is not required. However, the same can be used easily in the furnishing products like sofa cover, where rubbing durability is the major concern for the end users. As far as the dry cleaning fastness is concerned, cotton fabric treated with (BPS+ 3% BA) shows a better fastness properties after washing. After dry cleaning the treated fabric shows an LOI value of 29. It may be due to the non solubility of the (BPS+BA) coating in nonpolar perchloro ethylene solution.

3.8 Mechanism of Observed Self-extinguishing Effect

The high thermal stability of the BPS based bio-molecule is attributed to the presence of various inorganic metals, metallic salt molecule (Ca^+, Mg^+ , K^+, Si^+, KCl^-, Cl^-) metal oxides, phosphate, phosphite, phenolic OH groups, etc. as is also observed from the energy dispersive X-ray (EDX), secondary ion mass spectroscopy (ToF-SIMS) and X-ray fluorescence (XRF) analysis[14,16]. The negative ToF-SIMS analysis of the BPS shows the presence of major molecules at different mass units, such as H^- (1 amu), C^- (12 amu), CH^- (13 amu), N^- (14 amu), O^- (16 amu), OH^- (17 amu), F^- (19 amu), Cl^- (35, 37 amu) , PO_2^- (62, 63 amu), PO_3^- (79 amu), KCl^- (74, 76 amu), Cl_2^- (70,71 amu), etc. On the other hand, the positive mass spectrum mostly shows the presence of various metal ions, such as Mg^+ (24, 25 amu), K^+ (39 amu), Fe^+ (55, 56), etc. indeed, it might be due to the fact that phosphate, phosphite and other positive metallic salts and oxides present in the BPS, forming an intumescent coating on the cellulosic cotton fabric, absorb heat energy and show a fire retardant effect in a condensed phase mechanism. Condensed phase mechanism can be confirmed from extensive dehydration and char formation (observed from the TG results), restricting the formation of flammable gases like levoglucosan and pyroglucosan[14]. Besides, chlorine present in the BPS might be working in the gas phase mechanism, helping to restrict the flow of oxygen in contact with the fabric. High thermal stability has been reflected by the TG and the DTG curves of the dried BPS powder and BPS treated fabric, as dried BPS powder shows four stage weight loss at 55° (5%), 200° (13%), 350° (22%) and 500°C (20%). This phenomena reflects lower rate of weight loss and thus, at high temperature of 750°C, 30% carbonaceous char mass is found to remain. However, BPS treated cotton fabric shows a severe afterglow as well as the toxic smoke formation during burning, which limits its commercial application[16].

As far as the (BPS+1%BA) treated cotton fabric is concerned, it forms an additional insulated glassy coating of boron trioxide on the BPS coated underlying layer of the treated cotton fabric surface. This insulated boron trioxide coating might be helping to arrest the after-glow by thermal insulation (poor conductivity) technique. It may also be due to the fact that some amount of BA reacts with the acidic phosphate and phosphite present in the BPS at the high temperature during burning, forms an insulating white amorphous microcrystalline boron phosphate, and helps to stop the afterglow. Higher amount of BA (3%) used in this BPS formulation further enhances the insulation property and reduces the char length of the treated fabric. Another interesting observation is that, only BA (3%) coating cannot sustain this 10s flame contact time in vertical condition. It may be due to the fact that boron trioxide cannot penetrate into the interstices of the fabric, but forms an intumescent coating only on the upper surface of the fabric, which has less capability to restrict the burner flame. It means that by using more flame contact time, heat might breaks the three dimensional bricks of boron trioxide shown in char morphology and entered into the underlying material (cotton) surface, helping to burn it freely. However, BA in a combination of the BPS acts as a thick physical insulated barrier to the heat and mass transfer. It also hinders the diffusion of the oxygen towards the burning surface of the cellulose polymer, resulting in a very good afterglow arresting agent, whereas BPS is only acting as flame arresting agent. Here, BA may catalyze dehydration and also the isomerization of the newly formed polymeric materials by forming aromatic structures. This also may be one of the reasons of the stoppage of the afterglow. Therefore, we can conclude that the combine fire retardant action of both BPS and BA make the cotton fabric to exhibit the self-extinguishing property and help to get desired char length, following the condensed phase mechanism of intumescent fire retardant system. Char morphological pictures and char FTIR analysis presented in Figs 5 and 6, also provide a valid support behind the proposed condensed phase intumescent mechanism.

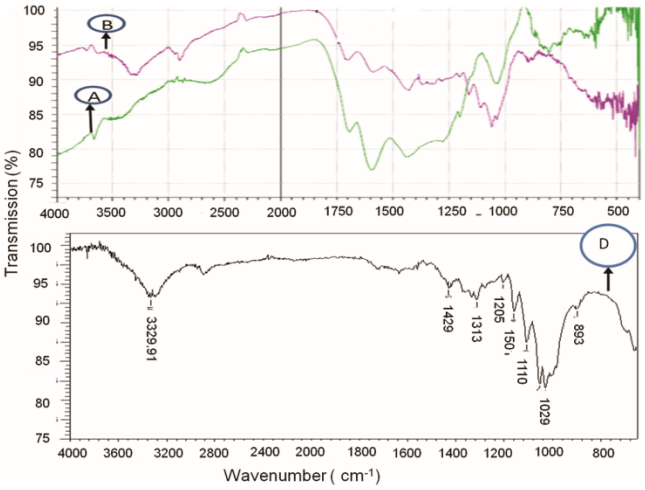

Fig. 6 — Char FTIR analysis of the control (A), BPS (B) and BPS+3% BA (D) treated cotton fabrics

4 Conclusion

The present study has enlighted about the flame retardancy effect of the mixture of BPS and BA. It has been found that the mixed formulation of BPS and 3% BA is the most suitable for imparting flame retardancy in the cellulosic products in a condensed phase intumescent mechanism. The mixture after application, forms an intumescent layer on the fabric surface, which has a self extinguishing effect that lowers the temperature and heat generated from the textile during heating. The char morphology of the treated cotton fabric shows a thick intumescent coating with the presence of long chain aromatic compounds. This reported process can be used beneficially for imparting flame retardancy to household table lamp cloth and also, as a covering material of non permanent structures like those used in making tents for pilgrims, militaries, book fair, festival, religious purposes and such on, where a large quantity of textile is used and the wash durability is not a major concern.

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