

# Self-extinguishable ligno-cellulosic fabric using banana pseudostem sap

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**Flame retardancy was imparted to ligno-cellulosic jute textiles using banana pseudostem sap (BPS), an eco-friendly natural by-product obtained during the extraction of fibres from the banana pseudostem. The extracted sap was made alkaline and applied to a pre-mordanted Greige fabric. The flame-retardant properties of both the control and treated fabrics were analysed in terms of limiting oxygen index (LOI), horizontal and vertical flammability, and the total heat of combustion. The treated jute fabrics showed a far better flame-retardant property compared to the control fabric. By 1.9 times increase in the LOI after application of the alkaline BPS, the treated fabric (1 : 4) showed no flame and got self-extinguished within a minute. Based on thermal degradation, pyrolysis and dehydration studies, and analysis of the chemical composition of the flame-retardant finish prepared from the BPS, the mechanism of imparting flame retardancy to jute textiles has been postulated. The imparted finish was found semi-durable in soap wash, and did not cause any significant loss in tensile and tear strength of the fabric.**

**Keywords:** Banana pseudostem sap, flame retardancy, jute fabric, ligno-cellulose, pyrolysis.

CONSUMERS all over the world are not only demanding newer textiles with improved aesthetic feel, but also emphasizing concern for human health, hygiene and safety. Some of the examples of improved functionality in textiles nowadays are: wrinkle-free, easy soil-release property, water-repellent, anti-microbial and resistant to colour fading and fire. Among these, fire-retardant or resistant textile is considered to be the most important for application in home furnishing and products like curtains, sofa covers, etc. In all such applications, jute fabrics are used traditionally, because of their advantages of biodegradability, eco-friendliness and aristocratic-look with better moisture management property. However, being 50–60% cellulosic in nature, jute fabric catches fire readily and it is difficult to extinguish the flame, thus posing serious risk of health hazards to humans and damage to textile products. Significant research efforts have been made in the past to improve the flame retardancy of jute

textiles using various commercially available synthetic chemicals. The common chemicals used are borax and boric acid mixture<sup>1</sup>, di-ammonium phosphate, urea, etc.<sup>2</sup>. In the mid 1980s, researchers reported the use of sodium potassium tartarate for making a fire-retardant jute textile<sup>3</sup>, besides, antimony-based halogen-containing formulations as the back-coating<sup>4</sup>. Though antimony in combination with halogen could impart a good flame-retardant property, it was not commercially successful due to the negative environmental impact of halogen compounds<sup>4</sup>. At the beginning of the 20th century, researchers used sulphur–nitrogen-based thio-urea and phosphorous-based tetra sodium pyrophosphate to improve the fire-retardant property of the jute fabric<sup>5</sup>. Jute was also made fire retardant by treating with metal oxide like sodium silicate, etc.<sup>6</sup>. However, these finishes, in general, are not eco-friendly and durable. It has been reported that by far the phosphorus-based flame retardants along with nitrogenous compounds are the most effective due to their synergistic effect<sup>7</sup>. Consequently, flame retardants based on the composition of phosphorous, nitrogen and halogen like tetrakis phosphonium salt and *N*-alkyl phosphopropionamide derivatives widely dominate the commercial applications<sup>4</sup>. However, such formulations are found to reduce the tensile strength of the jute fabric, as they are applied in acidic condition. Besides, the treatment is toxic, hazardous, expensive and time-consuming due to the involvement of a huge-quantity of chemicals and a high-temperature curing process<sup>8</sup>. Hence, there is a need to develop cost-effective, environment-friendly and sustainable flame-retardant formulations that, when applied, will maintain the jute fabric quality to a great extent. Towards this direction, researchers have tried to reduce toxic formaldehyde releases from the fire-retardant fabric using a binding formulation, such as butane tetra carboxylic acid (BTCA)<sup>9</sup>. Some researchers are working to develop eco-friendly sulphur–nitrogen-based flame-retardant formulations using ammonium sulphamate, in order to create more char forming ligno-cellulosic substrate. However, development of easily applicable and environmental-friendly, yet cheaper and effective fire retardants for lingo-cellulosic textiles still remains a challenge. Due to recent awareness on human health and hygiene, and their stringent associated norms, the demand for textile finishes originating from natural products, such

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as natural dyes for colouration, enzyme for bio-polishing<sup>10</sup>, neem and aloe-vera extract for antimicrobial finishing, etc. are receiving greater attention in research and development<sup>11</sup>. Only limited work has been reported recently on the fire retardancy of cellulosic fabrics with bio-macromolecules. DNA from herring sperm has been applied to cotton fabric to make it thermally stable. DNA consists of phosphate, deoxyribose units, nitrogen containing bases and blowing agents, which help in carbonaceous char formation and ammonia release, thus making the cellulosic cotton thermally stable<sup>12</sup>. Researchers have also tried to make fire-retardant cotton fabric using milk protein, a by-product of the cheese industry, due to their phosphate, disulphide and protein content that can influence the pyrolysis by early char formation<sup>13,14</sup>.

However, till date, the application of plant extracts (e.g. plant biomolecules) for flame-retardant finishing of any textile and/or polymeric material has not been reported to the best of our knowledge. As some of the plants contain potassium, phosphorus, silicon and other minerals and their salts, there exists good scope of research for their effective utilization to improve the flame-retardant properties of cellulosic and non-cellulosic textiles. In this direction, we have already studied the flame retardancy of cellulosic cotton fabric treated with alkaline solution of spinach juice (SJ)<sup>15</sup>. Continuing the effort, we have now attempted to improve the flame retardancy of ligno-cellulosic jute textiles using BPS, as it contains elements like potassium, magnesium, silicate, phosphorus, chlorine and other metallic constituents<sup>16</sup> and salts of magnesium nitrate and potassium nitrate<sup>17</sup>, potassium chloride, sodium chloride and metal phosphate<sup>18</sup>. Further, BPS is abundantly available in India and normally considered as a waste material. The present study reports an extensive investigation conducted to assess the effect of different concentration of BPS on the flame-retardant properties of jute textile in terms of limiting oxygen index (LOI), burning rate and total heat production. The possible mechanism of imparting flame retardancy to jute textiles by application of BPS has been postulated based on the inferences made from thermogravimetry analysis (TGA), differential scanning calorimetry (DSC), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX) and wash durability studies.

## Materials and methods

### Material and BPS application

A plain woven loom-state jute fabric (Greige state with natural golden colour) of 0.2 kg/m<sup>2</sup> areal density with 12 ends/cm and 13 picks/cm and having 200 tex warp and 240 tex weft yarn counts was used in the study. The BPS supplied by the Navsari Agricultural University, Gujarat

was characterized by atomic absorption spectroscopy for the presence of traces of metals in it. It was found that it contained high amounts of potassium (9 ppm, limited by the maximum detectable range of the instrument) and manganese (4.55 ppm), followed by iron (0.49 ppm) and copper (0.043 ppm). No chromium and calcium metals were detected. The grey jute fabric was first mordanted with 5% tannic acid and 10% alum. Then it was impregnated with different concentrations of BPS solution, such as pure BPS (1 : 0) and solutions made by concentrating pure BPS to half (1 : 2) and quarter (1 : 4) by volume with applying temperature. The material to liquor ratio was kept at 1 : 15. This implies that the BPS solution was 15 times that of the jute fabric by weight. The fabric was then dipped in the alkaline (maintained by adding soda ash) BPS solution for 30 min at 90°C. The treated fabric samples were subsequently dried at 110°C for 5 min.

### Determination of per cent add-on

Before conducting any physical and chemical characterization tests, the treated and control jute fabrics were conditioned at 65% RH and 27°C for 48 h. After the application of BPS on jute fabric samples, the add-on, i.e. increase in the sample weight was determined using the gravimetric principle from the bone dry weights of the sample before and after the treatments. The per cent add-on of BPS was calculated as follows:

$$\text{Per cent add-on} = [(M_2 - M_1)/M_1] \times 100,$$

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

### Thermal characterization

**LOI, Horizontal and vertical flammability:** The burning behaviour of the control and treated samples was evaluated using standard methods. For LOI analysis, IS 13501 test method was used<sup>19</sup>. In the horizontal flammability test, the flame time and propagation rate were measured according to the FMVSS302N standard<sup>20</sup>. In the vertical flammability test, different parameters were measured according to the 1871 method A<sup>21</sup>). The maximum temperature produced during the burning of the sample was measured using an IR thermometer in non-contact mode.

**Gross calorific value analysis:** An oxygen bomb calorimeter was used to measure the gross calorific value (GCV) of the control and BPS-treated samples using the dynamic 25°C measuring mode.

**Thermogravimetry analysis:** Thermogravimetry (TG) measures the gradual weight loss of a sample with respect

to time at a constant heating rate. It also indicates the effect of flame-retardant chemical on the pyrolysis of the polymer substrate<sup>22</sup>. The TG curves of the control and treated fabrics were drawn using a thermo gravimetric analyser in a nitrogen atmosphere with 2 ml/min flow rate and at a constant heating rate of 10°C/min.

*Differential scanning calorimetry (DSC):* DSC of the control and alkaline, BPS-treated jute fabric was carried out using a DSC thermal analyser. The heating rate was maintained at 10°C/min in the N<sub>2</sub> atmosphere.

#### *FTIR analysis*

FTIR analysis of the control and BPS-treated samples was carried out in an IR analyser over the wavelength of 500–4500 cm<sup>-1</sup> using the KBr disc sample preparation method. Here, the ATR transmittance mode with DLaTGS detector, 49 scans and four resolutions was used to perform the FTIR analysis.

#### *SEM and EDX analysis*

The surface of both the control and BPS-treated samples was analysed using a scanning electron microscope. The samples were coated with a thin layer of conducting material (gold/palladium) using a sputter coater and examined under SEM with an accelerating voltage of 12 kV. The EDX analysis of these samples was carried out using a field emission gun scanning electron microscope (FEG-SEM) to determine the quantity of the elements present on the surface, and the same was expressed in atomic and weight per cent. The time of flight secondary ion mass spectroscopy (TOF-SIMS) was carried out using gallium as the primary ion surface.

#### *Wash durability of the finish*

The flame-retardant activity of the finished samples was evaluated after washing them in a laundrometer using a standard detergent of 5 g/l at 40°C for 40 min. The fabric was then rinsed in freshwater for 5 min, followed by drying at 100°C for another 5 min. The samples were then conditioned in a desiccator for 24 h, before conducting LOI and flammability tests.

#### *Mechanical strength*

Tensile strength and tear strength, the two most important properties of any textile, were evaluated in an instron tensile tester according to standards ASTM D5035 and ASTM D1004 respectively<sup>23,24</sup>. The values of the control and treated samples were compared to study the effect of BPS on the mechanical properties of the treated samples.

## Results and discussion

### *LOI, horizontal and vertical flammability*

The pre-mordanted jute fabrics were treated with different concentrations of BPS as discussed earlier. Results of the flammability test are presented in Table 1. The LOI, a measure of flammability of a sample, is defined as the minimum amount of oxygen required to support the combustion in the oxygen/nitrogen mixture. A textile with LOI of 21 or below ignites easily and burns rapidly in the open atmosphere, whereas a sample with LOI above 21, ignites but burns slowly. A sample with LOI of 26 or more may be considered as flame-retardant<sup>1</sup>.

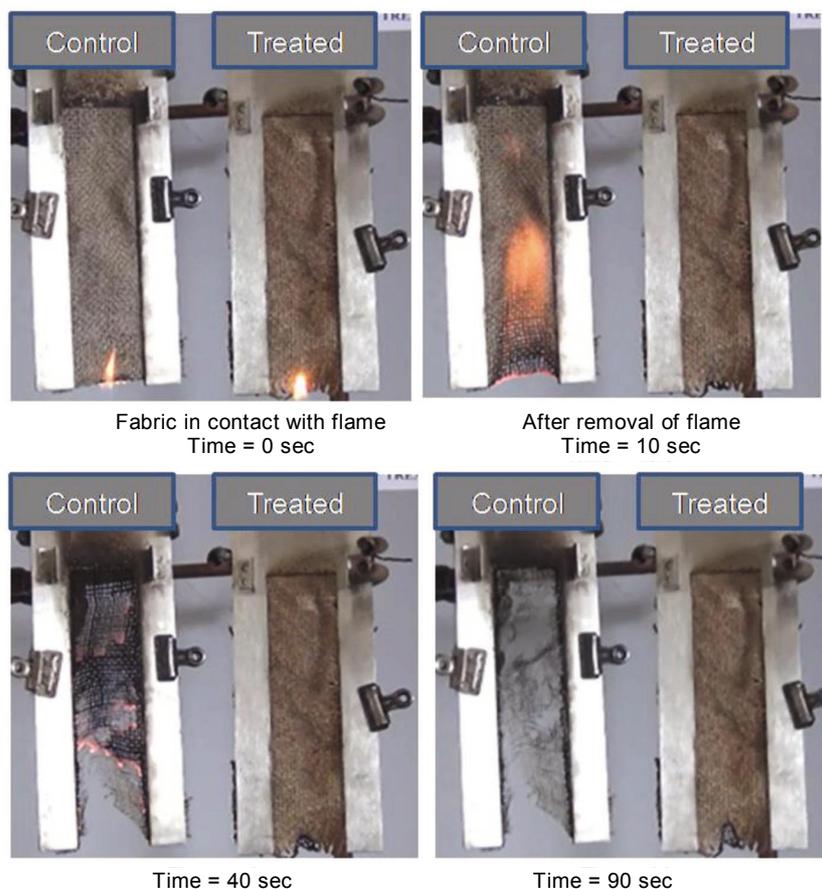
As indicated above, jute being a ligno-cellulosic fibre catches flame readily, as cellulose is its major component. Its LOI is as low as 21. In the present study, the jute sample was pre-mordanted with tannic acid and alum to increase the uptake of BPS. It was observed that there was no change in LOI of the sample due to mordanting. After application at different concentrations of BPS in the mordanted jute samples, LOI was found to increase significantly. In the mordanted fabrics treated with 1:2 BPS solution, the LOI value was found to increase to 36, which is almost 1.7 times higher than that of the control sample. With further increase in the concentration of BPS (1:4), the per cent add-on increased to 8 from 5.7, resulting in a linear increase in LOI, from 36 to 40. Because of the increased LOI, the BPS-treated samples did not catch flame easily. Whereas in the vertical flammability measurement, the control sample was found to catch flame readily and the entire sample burnt with flame within 60 sec. The BPS-treated (1:2) sample showed no after flame, followed by combustion with an afterglow in 800 sec. With further increase in the add-on percentage, the burning with afterglow also stopped within 40 sec, making the fabric a self-extinguishable one (Figure 1). Only this sample produced a 30 mm char length, indicating its superior flame retardancy.

It is interesting to note that in the 1:0 BPS-treated sample, the total burning time could be increased to 605 sec, from 60 sec merely found with the control sample. Similarly, for the BPS-treated (1:2) sample, the total burning time was 800 sec, which is approximately 13-fold more than that found with the control sample. By increasing the concentration of BPS, the burning flame time was reduced to nil, thus turning the fabric self-extinguishable within 40 sec. In a practical scenario of burning microclimate, burning with afterglow is not so severe in comparison to burning with the flame. This is because the burning flame happens at a much higher temperature compared to the burning with afterglow in a realistic vertical burning. In our experiment with vertical burning, a maximum temperature of 380°C was observed in the control sample, while in the BPS-treated (1:0) sample, it was reduced to 300°C. This indicates that the presence of

**Table 1.** Results of the flammability tests

Flammability parameters	Control	Mordanted	Alkaline BPS-treated		
			BPS concentration		
			1 : 0	1 : 2	1 : 4
Add on (%)	–		3	5.7	8
LOI	21	21	33	36	40
Horizontal flammability					
Warp way burn rate (mm/min)	55	53	15	11.5	–
Vertical flammability					
Occurrence of flashing over the surface	Yes	Yes	No	No	No
After flame (sec)	60	60	5	Nil	Nil
Burning with afterglow time (sec)	0 (as completely burnt with flame)	0 (as completely burnt with flame)	600	800	40
Total burning time (sec) (flame time + afterglow time)*	60 + 0	60 + 0	5 + 600	0 + 800	0 + 40
Char length (mm)	–	–	–	–	30
Observed burning rate (mm/min)	250	250	24.8	18.75	–
State of the fabric in contact with flame	Completely burnt with flame	Completely burnt with flame	Burnt initially with flame followed by afterglow	Burnt only with afterglow	Partially burnt with afterglow and char length observed

\*Total burning time of the fabric = burning with flame time + burning with afterglow time (after the flame has been stopped).



**Figure 1.** Comparison of burning behaviour between control and banana pseudostem sap (BPS)-treated (1 : 4) jute fabrics at different time intervals.

BPS in the jute fabric could reduce the burning temperature by 80°C. It is interesting to note that the 1 : 2 BPS treatment not only reduced the maximum burning with flame temperature, but also the afterglow temperature, which reduced gradually from 320°C to 280°C, 250°C and finally to 120°C at intervals of approximately 60 sec. In any flame-retardant finishing of a textile substrate, three time-periods, namely (i) burning with flame, (ii) burning with afterglow and (iii) total burning time are important, as they determine either the escape time or the time available to extinguish the flame. It will be beneficial if the burning with flame time could be reduced and the burning with afterglow time could be increased from the angle of fire hazards. In all the BPS-treated samples, the flame time was found almost negligible compared to the control sample with 60 sec flame time. Also, the afterglow time in the BPS-treated samples was much more, 600–800 sec, from practically nil in the control sample. In real-life situations, this longer afterglow time will help either to escape from the fire hazard zone or to extinguish the flame, which is not possible with the control sample with nil afterglow. The BPS-treated sample mostly burnt with afterglow and the burning rate was reduced significantly from 250 mm/min in the control sample to 18.75 mm/min in the 1 : 2 BPS-treated sample. Similar trends were also observed in the horizontal flammability measurement, where the burning rate reduced from 55 mm/min in the control sample to 11.5 mm/min in the 1 : 2 BPS-treated sample.

#### *Gross calorific value analysis*

In the oxygen bomb calorimeter, the specimen sample was inserted in a steel cylinder (i.e. the bomb) and pressurized with the excess of pure oxygen. The cylinder was then inserted in a bucket filled with water, and the surrounding jacket was kept at a constant temperature. The burning of the specimen caused a rise in temperature of the bomb and that of water in the bucket. An ignition circuit was used to ignite the specimen. After allowing an appropriate correction factor, the rise in temperature of water in the bucket was measured to determine the GCV of the sample. It has been observed that the control cellulosic jute sample showed a GCV of 16,299 J/g, however, the 1 : 4 BPS-treated sample produced much less heat of 12,848 J/g, which was almost 21% lower than the control fabric as shown in Table 2. The total heat produced by the BPS-treated fabric could be compared with the commercially available flame-retardant chemical formulation (10% diammonium phosphate (DAP) + 5% urea) for the cellulosic substrate, as reported in the literature<sup>25</sup>. It was observed that the residues obtained after the GCV experiment had no calorific values, as it is mostly a non-combustible inorganic material. Therefore, the lesser heat production along with a lower burning temperature and

burning rate, and more afterglow time, together signify that BPS could be considered as an excellent flame-retardant green-chemical for the ligno-cellulosic jute textile.

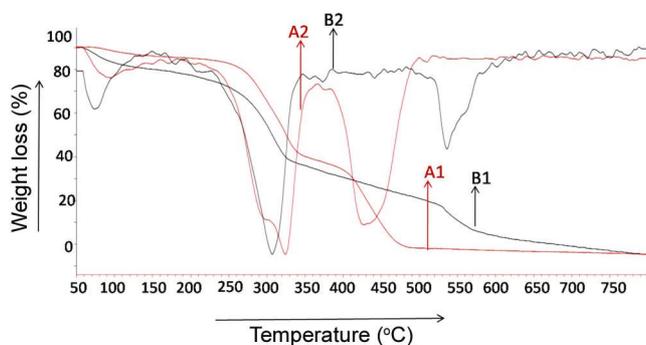
#### *Thermogravimetry*

Figure 2 shows the TG curves of the control (A1) and the 1 : 4 BPS-treated (B1) jute fabrics in N<sub>2</sub> atmosphere at a heating rate of 10°C/min. The TG curve of the control (A1) jute samples depicts three stages of mass loss. In the initial stage at temperature below 200°C, mass loss occurred mainly due to the removal of bound and unbound absorbed moisture from the cellulose polymer<sup>26</sup>. However, the main pyrolysis occurred in the temperature range 260–500°C. In this range, the major mass loss happened at around 330°C due to pyrolysis of cellulose<sup>27</sup>.

Above 400°C, dehydration and char formation occurred. In the case of dehydration, the non-oxidizable water and CO<sub>2</sub> might have been released<sup>22</sup>. It could be seen that the control sample (A1) lost approximately 98% of its mass at below 500°C. A similar phenomenon was observed in the vertical flammability measurement, where the control and mordanted fabric burnt completely with a maximum temperature of 380°C and burning rate as high as 250 mm/min. In the BPS-treated jute fabric (B1), more weight loss (10%) was observed at the initial stage (50–150°C) of the TG curve compared to the 5% weight loss in the control fabric. This may be due to the presence of more bound or unbound moisture in the BPS-treated fabric due to the presence of various inorganic salts and their hydrates, as discussed below. In the second stage, the curve B1 started to lose its mass with the onset of thermal degradation at 215°C, which is 45°C below the degradation temperature (260°C) of the control sample with a shift in temperature from 330°C to 300°C (i.e. 30°C lower) at which the peak in the main loss occurred. As 60% of the mass loss was observed, hence it can be deduced that the BPS treatment reduced the amount of mass loss in the second degradation range only. Further, it reduced the combustion temperature of the cellulosic substrate with dilution of the flammable volatiles by the generation of non-oxidizable CO<sub>2</sub>, H<sub>2</sub>O at a comparatively low temperature. It can also be observed from the TG curve that in the third stage, the treated fabric B1 started forming char and the quantity of char residue remained higher than the control fabric at any higher temperature and at 500°C, it increased from 15% to 30%. It shows that the alkaline BPS also has a positive effect on prior dehydration and char formation. With regard to alkalinity the thermal stability of uniaxial natural ligno-cellulosic fabric increased when it was treated with alkali. This might be because alkali treatment increases the crystallinity of the treated fabric. The TG curve shows that for both untreated and 3% alkali treated ligno-cellulosic fibre pyrolysis started from 250°C. However, the rate of

**Table 2.** Gross calorific value (GCV) of jute fabric

Fabric parameters	Sample mass (g)	Temperature ( $\Delta T$ ) (K)	GCV ( $H$ ) (J/g)
Control jute	0.4279	0.783	16,299
BPS-treated (1 : 4) jute	0.696	1.000	12,848

**Figure 2.** TGA curves of the control (A1) and 1 : 4 BPS-treated jute fabric (B1).

weight loss is slightly more in untreated fabric than the treated one<sup>28</sup>.

### DSC analysis

The DSC analysis was carried for both the control and BPS-treated jute fabrics to determine their thermal behaviour (Figure 3). An endothermic peak was observed in the temperature range 50–130°C in both the control and BPS-treated jute fabrics which corresponds to the heat of evaporation of water molecules absorbed in the fabric<sup>29</sup>. Similar behaviour could be seen in the TG curves for all the samples. The observed endotherm was found to be steep in the case of the treated fabrics. This might be attributed to the vaporization of water of BPS from the treated jute fabric, in addition to the removal of absorbed moisture from the fabric, as it could absorb 13% moisture of its own weight. For the control jute fabric (Figure 3 a), a weak broad exotherm over the range 260–370°C was observed, which is attributed to the thermal degradation of hemicellulose<sup>30</sup>. It also showed an endothermic minimum at around 376–399°C with the peak at 387°C. This endotherm corresponds to the depolymerization of cellulose with the formation of flammable gases like pyroglucosan and levoglucosan<sup>27,30</sup>. At this temperature, pyrolytic degradation taken place with a rapid cleavage of the glucosidic bond<sup>20</sup>. Further, the prominent exothermic bond observed at relatively higher temperature >400°C is attributed to the exothermic decomposition of jute lignin<sup>30</sup>. On the other hand, the DSC thermogram of the treated jute fabric (Figure 3 b) showed an exothermic bend (hemicellulose degradation) from 270°C to 340°C, followed by an endothermic peak (cellulose decomposition)

at around 365°C, which is lower than the control jute fabric. Therefore, the curve in Figure 3 b reveals that the endothermic peak related to the degradation of cellulose has shifted to a lower temperature (from 387°C to 365°C), with the BPS add-on increased to 8%. Like TG analysis, DSC interpretation also showed that BPS treatment lowered the pyrolysis temperature and diluted the formation of flammable gases, resulting in an increase in LOI and decrease in flame time and the burning rate. Besides, the areas under the endothermic peak (326°C) and exothermic peak (365°C) were also found more in the case of BPS-treated jute fabric. Similar to TG analysis, it might be attributed to the dehydration of cellulose that occurred more extensively as BPS content in the fabric was increased.

### FTIR analysis

Figure 4 shows the FTIR spectra of various samples. It can be clearly observed that there is no significant change between the curves A and B. However, curve B shows a more intense peak of intermolecular –OH stretching at 4000 cm<sup>-1</sup>. The area under this peak curve B between 3800 cm<sup>-1</sup> and 4200 cm<sup>-1</sup> is the summation of the area shown by curves A and C in the same region. This shows that greater amount of bound and unbound moisture of both the cellulosic jute fabric and BPS enhanced the fire-retardant property of the resultant textile. Further, curve B shows one small peak at 1060 cm<sup>-1</sup> assigned to the Si–O stretching vibration<sup>31,32</sup>. Curve C pertaining to BPS showed a wide band for water from 3600 to 2600 cm<sup>-1</sup>. The peaks observed between 800 and 1300 cm<sup>-1</sup> are mainly due to the presence of inorganic salts in BPS<sup>33</sup>, and IR absorption bands observed at 1000–1100 cm<sup>-1</sup> might be assigned to the phosphate group<sup>34</sup>. The small peaks observed at 1176 and 873 cm<sup>-1</sup> might be assigned to magnesium chloride salt<sup>35</sup>. Similarly, the peak observed at 1000 cm<sup>-1</sup> is due to the presence of sodium phosphate<sup>36</sup>. Elements of inorganic salts have also been detected in EDX analysis as discussed below.

### SEM and EDX

SEM images of both the control, BPS-treated (1 : 4) jute fabric and pure BPS are shown in Figure 5. It can be seen from Figure 5 a, that the control sample is clean with no deposition and coating on the fibre surface. However,

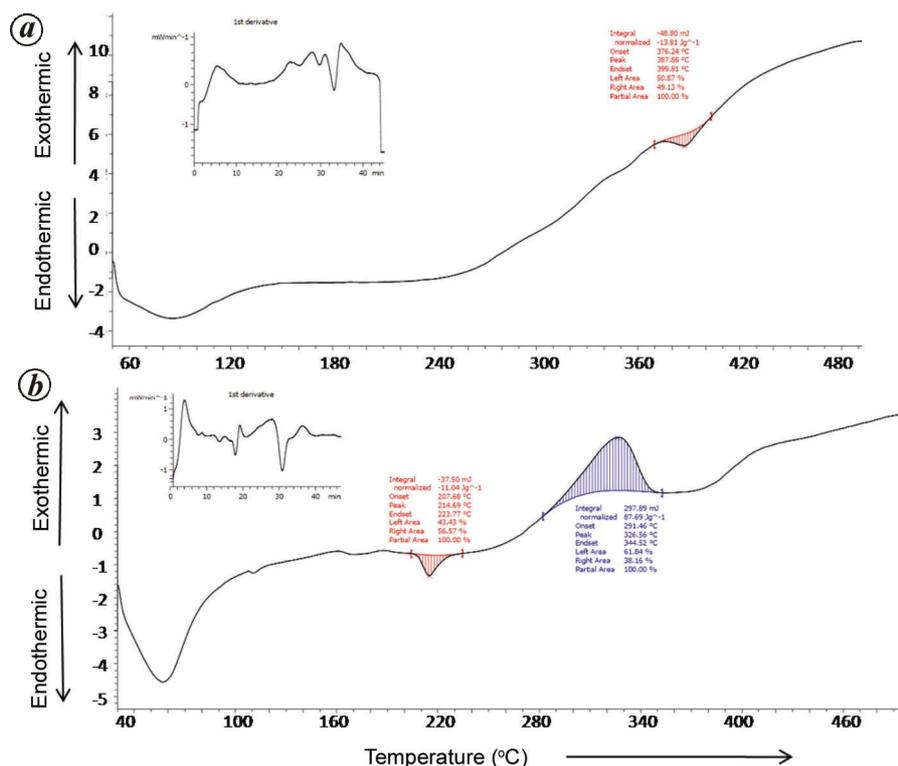


Figure 3. DSC curves of the control (a) and 1 : 4 BPS-treated jute fabric (b).

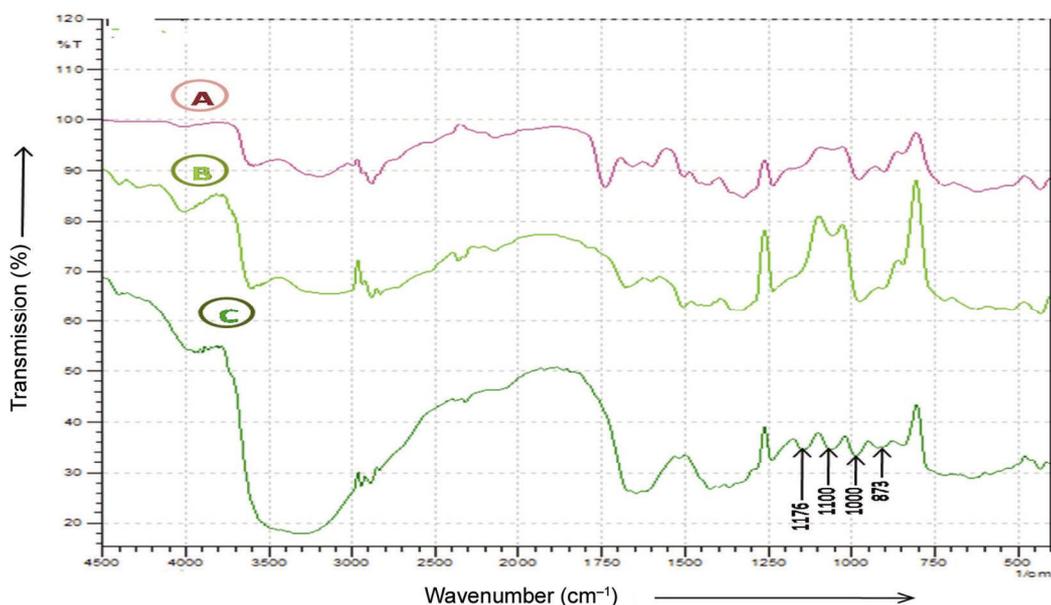
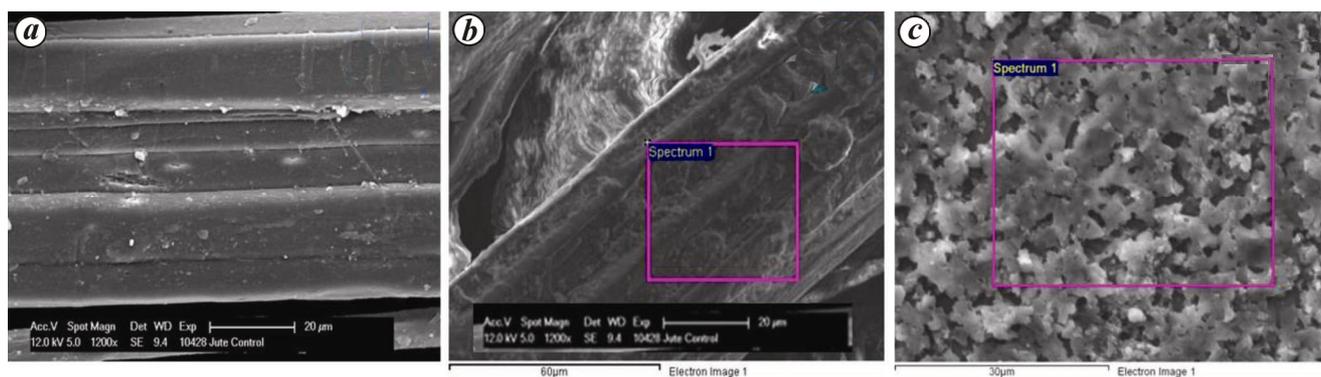


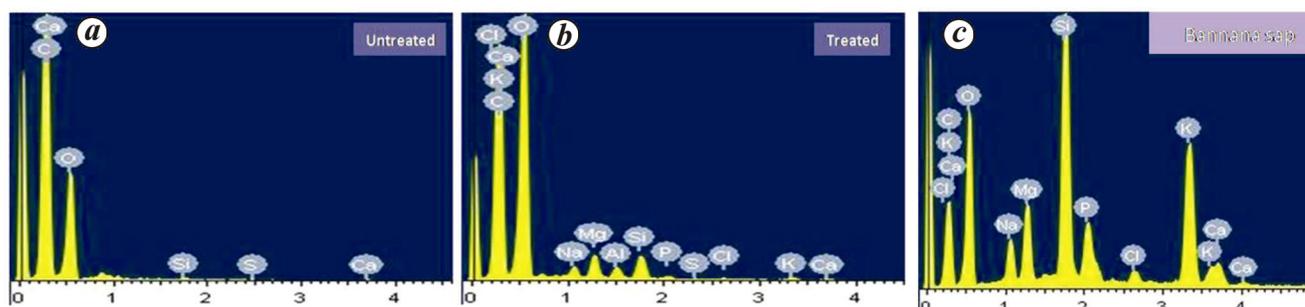
Figure 4. FTIR analysis of control fabric (A), BPS-treated (1 : 4) fabric (B) and dried BPS (C).

after BPS treatment (1 : 4), the BPS coating uniformly distributed over the entire fibre surface is easily visible (Figure 5 b). The energy-dispersive X-ray analysis of the control, BPS-treated fabric and pure dried BPS is presented in Figure 6, with the data reported in Table 3. As expected, the control sample showed only the pres-

ence of carbon and oxygen atom. However, in pure BPS, metallic substituents like magnesium, potassium silicon, etc. could be detected. Thus, in the BPS-treated samples, several atomic peaks are easily discernable as shown in Figure 6. These correspond to sodium, magnesium, aluminium, silicon, phosphorus, chlorine, potassium and



**Figure 5.** SEM images of control (a), BPS-treated (1 : 4) fabric (b), and pure dried BPS (c).



**Figure 6.** EDX images of control (a), BPS-treated (1 : 4) fabric (b) and pure dried BPS (c).

**Table 3.** Atomic percentage of control (A) and BPS-treated (1 : 4) jute fabric measured by EDX

Elements	Control		BPS-treated (1 : 4)		BPS	
	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)
C	55.68	62.72	34.74	42.47	15.26	24.27
O	43.89	37.11	58.79	53.96	39.23	47.45
Na	0	0	1.00	0.64	2.71	2.28
Mg	0	0	1.98	1.20	4.41	3.51
Al	0	0	0.76	0.41	0	0
Si	0.11	0.06	1.82	0.95	15.10	10.40
P	0	0	0.26	0.12	3.41	2.13
Cl	0	0	0.14	0.06	1.19	0.65
K	0	0	0.27	0.10	16.19	8.01
Ca	0	0	0.45	0.17	2.70	1.30
S	0.18	0.08	0.09	0.04	0	0

calcium. It can be seen that in the BPS-treated jute fabric, the magnesium percentage is more followed by sodium compared to the other atoms. Also, compared to the control sample, the per cent weight of carbon is significantly lower (by 20) in the BPS-treated sample. However, 15% more oxygen might be due to the presence of various metal chlorides and oxides. The other elements, except sulphur and aluminium, present in pure BPS as well in the BPS-treated samples might be in the form of metal oxide, metal chloride or metal phosphate. Aluminium, potassium and sulphur were found in the BPS-treated sample, mainly due to the mordant (alum:  $\text{Al}_2(\text{SO}_4)_3$ ,

$\text{K}_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$ ) and sodium from alkali  $\text{Na}_2\text{CO}_3$  and BPS used during the experiments. The presence of magnesium is due to BPS (possibly in the form of  $\text{MgCl}_2$  salt, magnesium phosphate or  $\text{MgO}$ ), phosphorus in the form of phosphate salt and silicon present in the form of silicate ( $\text{SiO}_2$  or  $\text{Na}_2\text{SiO}_3$ ).

#### Surface ion mass spectroscopy analysis

As indicated in Figure 7 and Table 4, the negative TOF-SIMS of BPS showed the presence of H, C, N, O,  $\text{OH}^-$ ,

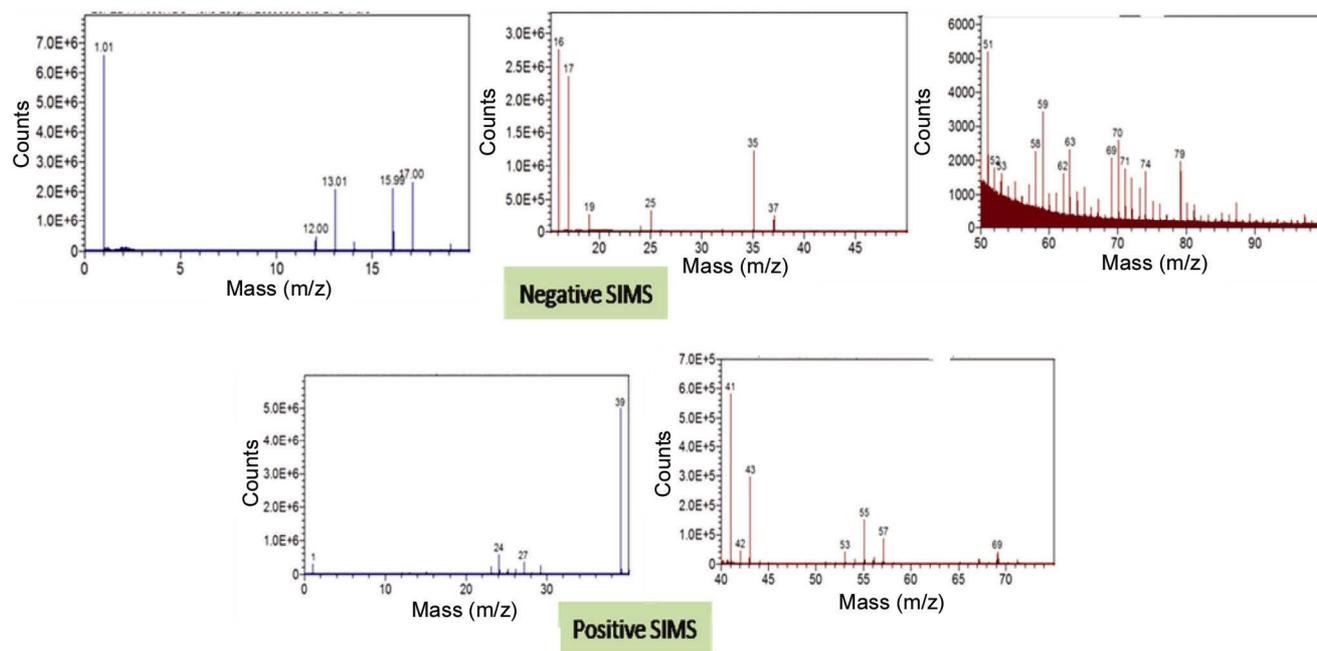


Figure 7. TOF SIMS of BPS.

Table 4. Mass spectroscopy peaks and their quantifications

Molecule	Molecular formula	Mass/charge (M/Z) ratio	Intensity/count
Hydrogen	H	1	7.0E + 6
Carbon	C	12, 13	1.0E + 6
Nitrogen	N	14	3.0E + 6
Oxygen	O	16	3.0E + 6
Hydroxyl	OH <sup>-</sup>	17	2.0E + 6
Fluorine	F <sup>-</sup>	19	5.0E + 5
Chlorine	Cl <sup>-</sup>	35, 37	1.5E + 6
Chloride	Cl <sub>2</sub>	70, 74, 72	3000
Phosphate	PO <sub>2</sub> <sup>-</sup>	62, 63	3000
Phosphite	PO <sub>3</sub> <sup>-</sup>	79	2000
Potassium chloride	KCl <sup>-</sup>	74, 76, 78	2000
Potassium	K <sup>+</sup>	40, 41	5.0E + 6
Magnesium	Mg <sup>+</sup>	24, 25, 26	1.0E + 6
Aluminium	Al <sup>+</sup>	27	1.0E + 6
Calcium	Ca <sup>+</sup>	43	3.0E + 5

F<sup>-</sup>, Cl<sup>-</sup>, PO<sub>2</sub><sup>-</sup>, PO<sub>3</sub><sup>-</sup>, KCl<sup>-</sup>, KF<sup>-</sup>, Cl<sub>2</sub>, etc. and the positive TOF-SIMS showed the presence of Mg<sup>+</sup>, K<sup>+</sup>, Mn<sup>+</sup>, Fe<sup>+</sup> ions. Therefore, the flame retardancy (i.e. more amount of char formation and higher LOI) of the cellulosic cotton fabric might be attributed to the free metal ions present in BPS, in addition to the presence of salts like potassium chloride, potassium fluoride, calcium chloride, phosphate, phosphite, etc. which also help impart fire retardancy to the BPS-treated fabric. Therefore, the mass spectroscopy results of BPS also support the conclusion drawn from the analysis of FTIR and EDX. An earlier study on GC-MS analysis of BPS has also reported that magnesium nitrate and potassium nitrate are the two major active compounds present in it<sup>17</sup>.

### Mechanism of imparting flame retardancy by application of BPS

From the foregoing discussion, it is seen that jute textile being ligno-cellulosic in nature, has a low LOI of 21 and does not exhibit flame retardancy. The same does not improve after mordanting with 5% alum and 10% tannic acid. This implies that the molecules present in alum and tannic acid do not have any role in the flame-retardant finishing of cellulose textiles. In our experiment, it was observed that the uptake/exhaustion of BPS by cellulose textile was very low, when the samples were treated without any mordanting agent. However, prior mordanting of jute followed by application of BPS in alkaline condition ensured a good add-on with 3–8% uptake of BPS. This implies that jute has become flame-retardant mainly because of BPS, and partially due to the interaction of BPS and the mordant. The effect of flame retardancy imparted by BPS may be attributed to the presence of phosphate, silicate and other mineral salts and metal oxides in the sap. The phosphate and silicate in the BPS-treated sample were present mainly in the form of calcium, magnesium. Similarly, other metals are present in the treated jute in the form of chloride, such as magnesium chloride, calcium chloride, potassium chloride, etc. The presence of these molecules, i.e. chloride and phosphate was also observed in the FTIR analysis and by the peaks of the elements in the EDX analysis. In this regard, recently, researchers have reported that 10.5 g inorganic salts was obtained from the evaporation of 500 ml aqueous ash extract collected by burning the air-dried BPS. It has also been reported that potassium chloride, sodium chloride and metal phosphate are the main components

**Table 5.** Flammability parameters of treated and washed fabric

Flammability parameters	Fabric parameters		
	Control sample	BPS-treated (1: 4) sample	
		After washing	Before washing
LOI	21	30	40
Horizontal flammability			
Warp way burn rate (mm/min)	55	20	–

**Table 6.** Physical properties of control and treated jute fabrics

Jute fabric	Physical properties of treated fabric			
	Tensile strength (N)		Tear strength (N)	
	Warp	Weft	Warp	Weft
Control	300	310	610	638
BPS-treated (1 : 4) sample	293	305	605	630

of the salt substitute<sup>18</sup>. It has also been reported that banana roots are able to induce silicate dissolution, thereby increasing the availability of silicon<sup>37</sup>. In the present study, it has been deduced that not only the BPS alone, but also the formation of some complex with the mordants has helped the jute textile substrate to be a better flame retardant, as a high uptake of BPS was possible only due to prior mordanting of the fabric. From the various characterizations made in the present study, it is presumed that phosphate, silicate and other mineral salts might have retarded the thermal decomposition of cellulose and contributed to the dehydration process by increasing the amount of char formation, as shown in the TG curve. Phosphate-containing molecules are known to be flame retardant<sup>3</sup>. Metal chloride has also been reported as a fire-retardant for cellulosic substrate<sup>38</sup>. Silicate has been reported to have passive fire-retardant properties<sup>6,39</sup>. Besides, the presence of water molecules in BPS as shown in the FTIR curve C (Figure 4), might have also contributed in preventing the rise in temperature of the sample by absorbing the heat of evaporation (540 cal/g). The presence of moisture peak has been observed in the FTIR spectra and the TGA curves. Therefore, the flame retardancy exhibited by the BPS-treated jute fabric might be possibly due to the combined effect of (i) phosphate compounds, (ii) metal salts (iii) silicate, and (iv) bound and unbound water molecules. These have helped in more char and non-flammable gas formation, such as like CO<sub>2</sub>, H<sub>2</sub>O, etc. Besides, researchers have also noted the presence of organometallic additives in the cellulosic cotton fabric that increased the char formation and also decreased the tar formation<sup>40</sup>. In addition, the presence of approximately 20% of the inorganic material and reduction of organic material (cellulose) in similar quantum, might have helped in the production of more char with no

calorific value, while reducing the formation of flammable gases as seen in the TGA analysis.

### *Wash durability*

The test on the durability of the imparted flame-retardant finish on jute textile was carried out with a soap solution to study the after-wash effectiveness of the finish. The changes in LOI and burning rate are presented in Table 5. It can be seen that the LOI value of the washed fabric decreased from 40 for the 1 : 4 unwashed fabric to 30 in the washed fabric sample, which is, significantly (by 1.4 times) higher than that noted with the control sample. After washing the sample, as LOI decreased its burning rate also increased. The measured horizontal burning rate (20 mm/min) in the washed sample was found to be 2.5 times slower than that of the control sample, and the decrease in the flame-retardant properties in the BPS-treated sample after washing may be attributed to the partial removal of the active component of BPS molecules such as metal salts, phosphate and silicates.

### *Physical properties*

It can be seen from Table 6 that the application of flame-retardant finish has no significant adverse effect on the tensile and tear strength of the 1 : 4 BPS-treated sample. In contrast, it may be noted that of the conventional flame-retardant finish generally causes 10–30% loss of tensile strength of the jute fabric due to application in acidic condition<sup>41</sup>. In our case, since BPS was applied in alkaline condition, the changes in either tensile or tear strength were non-significant. Here in this regard, it has been reported that alkaline treatment (4% NaOH)

improves the tensile strength of the treated ligno-cellulosic fabric<sup>28</sup>.

## Conclusions

The present study has shown the flame retardant effect of BPS on jute textiles. After application of BPS, LOI increased from 21 to 40 in the BPS-treated (1:4) samples. Due to higher LOI, the sample did not catch fire in the open atmosphere, and it demonstrated to be a self-extinguishable textile. In the vertical flammability test, the BPS-treated (1:0) fabric showed a maximum temperature of 300°C, which is much lower than that found with the control sample. As far as the heat production is concerned, the BPS-treated fabric produced 22% lower heat than the control sample. In the 1:2 BPS-treated sample, the total burning time increased to 800 from 60 sec for the control sample, and the 1:4 BPS-treated sample was self-extinguishable. Thus, the 1:4 BPS-treated sample offers more safety time for human beings either to escape from the fire hazard zone or to extinguish the fire itself. Therefore, concentrated BPS (1:4) was found to be the best in the present study for application in the mordanted jute fabric in alkaline condition. The flame retardancy in the BPS-treated jute fabric might have been attributed due to the presence of (i) phosphate compounds, (ii) metals salts, (iii) silicate and (iv) water molecules. Also, the presence of approximately 20% inorganic materials in the BPS-treated cellulose textile might have helped in the production of more char and non-flammable gases. The TG and DSC curves confirm an early pyrolysis, dehydration and char formation phenomenon in the BPS-treated jute fabrics. The application process is simple and cost-effective. Besides, BPS is abundantly available in India as well as across the globe from renewable sources, but regarded normally as a waste material. Hence, the flame-retardant textile finishing with BPS can also be considered as an eco-friendly process. The reported process can be used for imparting flame-retardant property to home furnishings such as window curtains, sofa covers, table lamps, and in the covering textiles of non-permanent structures like those used in exhibitions gatherings, etc. where a large quantity of jute is used, and there are chances of fire hazard.

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