

Egyptian Journal of Chemistry

http://ejchem.journals.ekb.eg/



Flame Resistance of Cotton Fabrics and Their Natural Dyeing Using Plant Waste of Banana Pseudostem (BPS)



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THE present study has demonstrated the flame reatardancy effect of Banana pseudostem (BPS) as plant wastes at different concentrations on bleached and mercerized Egyptian cotton fabric made of Giza 90. Alkaline method was used to produce the extract BPS. The cotton fabric after the treatment was found to produce stable natural semi kaki color. Flame retardant properties of both the untreated and the treated cotton fabrics were analyzed in terms of limiting oxygen index (LOI), horizontal and vertical flammability. The control and the treated cotton fabrics were performed using FTIR, thermo-gravimetric analysis, mechanical properties, and color components analysis. The results revealed that the treated cotton fabrics were found to have good flame retardant property of LOI at BPS concentration 10% compared to the control fabrics. In the vertical flammability test, the treated fabric showed flame for a few seconds and then got extinguished. In the horizontal flammability test, the treated fabric showed no flame, but was burning with a propagation rate of 7.5 mm/min, which was almost 10 times lower than that noted with the control fabric. The results revealed that there was no significant degradation in mechanical strengths. Based on the results, the mechanisms of formation of natural color on the cotton fabrics using the proposed BPS treatments have been postulated.

Keywords: Egyptian cotton, Fire retardant, Ecofriendly, Plant waste, Banana pseudo stem sap (BPS).

Introduction

Egypt is the largest producer in Africa and worldwide of long (LS) and extra-long staple (ELS) cotton varieties [1]. Petrilli, pointed out that Cotton is 100% cellulosic in nature, catches flame readily and it is quite difficult to extinguish the same, resulting in serious health risk and damage to textile products, [2]. Application of flame retardant products on cotton is an important textile issue, especially for protection of consumers in military and the airline industry, [3]. Many researches have been made to improve the flame retardant property of cotton textile using various synthetic chemicals. The more effective fire retardant chemicals available in the market are inorganic salts, borax and boric acid mixture, di-

ammonium phosphate, urea, etc [4]. When such formulations are applied on cotton fabric, its tear and tensile strengths are reduced and the fabric becomes stiff as it's applied in acidic condition. Besides, the treatment is toxic, hazardous, expensive, and also time consuming due to involvement of high quantity chemical and high temperature curing processes [5]. Development of ecofriendly natural products fire retardant is still under investigation, and challenge for the researchers. To maintain cotton fabrics its quality to a great extent, there is the need to develop more cost-effective, natural, environmentally friendly and sustainable fire retardant products. To prevent the quantity of formaldehyde release from fire retardant fabric, Butane tetra carboxylic acid (BTCA) binding formulation was used [6].

Recently, polycarboxylic acid and nano zinc oxide was used to make environmentally friendly fire retardant cotton fabric [7]. Fire retardant was produced from natural products such as natural dyes for coloration, enzyme for bio-polishing [8]. Neem, and aloe vera is getting attention in research and development [9]. Due to recent awareness about human health and hygiene, very few researches have been reported regarding the fire retardancy of cellulosic fabric using biomacromolecules. The main target of present study made to provide the flame retardancy and natural dye to cellulosic cotton textiles by using banana pseudostem sap (BPS), a plant waste extract as it contains phosphorous, nitrogen, chlorine and other metallic constituents. According to the official report issued by the Ministry of Agriculture and Land Reclamation represented in the Central Administration of Horticulture, the annual cultivated area from banana plant in Egypt was about 1800000 feddans in 2018 and it gave about 20 tons per Fadden. Banana waste materials are rich in nutrients and minerals, [10]. According to our previous work, the alkaline fractions of banana peel (Musa, cv. Cavendish) of fruits have been used as a natural dye for cotton fabrics. In our study, banana peel was evaluated as a multi-functional antibacterial and UV protective agent on the cotton substrate, [11]. Banana peel can also be used in wine [12], ethanol production [13], as substrate for biogas production [14] and as base material for pectin extraction. Peel ash can be used as fertilizer for banana plants and as source of alkali for soap production [15]. Ethanol extract of Banana peels can be used as an inhibitor for mild steel corrosion [16]. Banana peel can also be used in wastewater treatment plants [17]. Our previous work also aimed to use the extracted solution from banana leaves as natural waste source to dye some Egyptian cotton fabrics. Both alkaline and acetone extracted solutions were analyzed by high performance thin layer chromatography (HPTLC) analysis technique, [18]. The application of Banana pseudo stem sap (BPS) in cotton textile for coloration and functionalization will give the advantages of value addition using natural products. It is also ecofriendly and produced from renewable source. Chips, fig, ready to serve drink, flour, jam, confections, dehydrated slices, and pickles and various products such as Paper board, tissue paper, etc., can be made and prepared from Banana pseudo stem sap (BPS). Due to its higher specific strength modulus and

lower strain at break, Banana pseudo stem sap (BPS) fibers can be used as natural sorbent, bioremediation agent for bacteria in natural water purifier, for mushroom production, in handicrafts and textiles when mixed with paddy straw. It is also used in production of marine cordages, high quality paper cardboards, tea bags, string thread, high quality fabric material, paper for currency notes, and good rope for tying purposes [19]. Lectins found in Banana pseudo stem sap (BPS) possessing antimicrobial properties [20]. Banana pseudo stem sap (BPS) can be recycled to be used as bio-fertilizer [21], and as color absorbent from wastewater containing textile dyes [22]. Flame retardancy was imparted in cellulosic cotton textile using Banana pseudo stem sap (BPS), an eco-friendly natural product. The extracted Banana pseudo stem sap (BPS) was made alkaline and applied in pre-mordanted bleached and mercerized cotton fabrics [23]. Fang et al, studied that the flame resistance of cotton fabric was greatly enhanced by a novel reactive flame retardant with serrated structure, ammonium salt of 1,3-diaminopropane tetra-(methylene phosphonic acid) (ADDTMPA), and the softness of cotton fabric was retained very well [24]. In the present study, the effect of Banana pseudo stem sap (BPS) on the cotton textile with regard to assessment of flammability, coloration and mechanical properties have been extensively investigated and characterized as it contains phosphorous, nitrogen, chlorine and other metallic constituents.

Materials and Methods

Materials

- 1- The fabric samples used in this investigation were long sable Egyptian cotton scoured plain weaved of Giza 90 purchased from Misr-El-Mehala Company for Spinning and Textile-Egypt. The cotton fabrics had the following specification: yarn count: 38 x 40 tex; weight: 150 g/m². Specimens of size of 40 cm x 40 cm were used.
- 2- Banana pseudo stem sap (BPS) plant waste was obtained from the research farm, Faculty agricultural, Cairo University, used as ecofriendly natural dye and fire retardant.
- 3- All chemicals used were of analytical grade using doubly distilled water.

Methods

- 1- Bleaching of the cotton fabrics were carried out according to our previous work, [25].
- 2- The bleached samples were immersed in 20%

aqueous of sodium hydroxide for two minutes at room temperature. Treatment was carried out in slack state of fabrics, and then washed with tap water, neutralized with aqueous solution containing 0.1% acetic acid followed by washing with hot water to ensure removal of residual chemicals. Finally, Samples were air dried

- 3- The bleached and mercerized cotton fabric samples were pre-mordanting with 0.2 g/l tannic acid and alum at goods to liquor ratio 1:40 for 10 min at room temperature. The samples were then dried in the oven at 130°C, for 5 min.
- 4- The dirt and impurities attached to BPS were removed using tap water and Sodium 4-(1-dodecy) benzenesulphonate (SDS) as detergent, filtered and washing successively with tap water and left to dry. Then, BPS was cut into small size, ground in a Wiley mill, and the powder produced treated with 1N Na,CO₃ for one hour at temperature 60°C.
- 5- The bleached, mercerized and mordant cotton fabrics were dipped in four different concentrations (2.5, 5, 7.5 and 10%) with the BPS extracts for 30 minutes, then, dried at 110°C for 10 min. and finally cured at 150°C for 3 min. we cannot determine the expected structure of BPS effective component, so, we need further research.

Evaluation tests

Determination of add-on%

The treated cotton fabric samples were conditioned at 65% RH and 27°C for 48 h. The increase in the sample weight relative to the original weight was determined after the application of the BPS using the gravimetric method as follows:

Where, M1 and M2 are the oven dried weights of the control and the BPS treated samples respectively. The reported results are average of 3 readings.

LOI, and Flammability assessment

LOI, and flammability tests for the treated cotton fabric samples was carried out according to the standard methods ISO 4589 (1996), and D 1230-94 (reapproved 2001) respectively.

Fourier transformer infrared spectroscopy (FTIR) analysis

The Fourier Transform Infrared (FTIR) Analysis were carried out for samples Using FTIR Model Cary 630 FTIR spectrometer produced by Agilent technologies Company, for both Qualitative and Quantitative (for liquid samples) analysis, in spectral range (wavenumbers cm⁻¹) from 4000cm⁻¹ to 400cm⁻¹ without any treatment.

Thermo-gravimetric analysis (TGA)

The thermo-gravimetric measures the gradual weight loss of a sample with respect to time at a constant heating rate. It also indicates the effect of any flame retardant chemical on the pyrolysis of the polymer substrate. The TGA curves of the control and the treated fabrics were drawn on a Simultaneous Thermal Analysis (Model STA PT 1600- 2019) in a nitrogen atmosphere at 2 ml/min flow rate and at 10°C/min heating rate. The TGA curves of the control and the BPS treated fabrics were also taken in air atmosphere under similar flow and heating conditions to understand the thermo-oxidative decomposition.

Mechanical strengths

The tensile strength and elongation % were measured using Zweigle of model Z010, at a tension speed of 100 mm/min under the standard atmospheric conditions (temperature = 20 ± 2 °C and relative humidity = 65 ± 5 %). The testing was carried out according to ASTM D412-98a. The measurements were carried out three times, and the results indicated in this paper are the mean values

Color parameters

The color parameters were measured using a Perkin-Elmer Model 35 Lambda equipped with an integrating sphere. The color depth of the BPS treated fabrics was determined in terms of K/S from the reflectance data using the Kubelka–Munk equation as follows:

$$K/S = (1 - R)^2/2R$$

Where, K is the absorption coefficient, S the scattering coefficient and R is the reflectance of the treated fabric at the wavelength of maximum absorption. The K/S was determined at $(\lambda \max)$ of the respective dye. Other color parameters such as L* (lightness-darkness), a* (red-green), b* (blue-yellow), hue, and chroma were measured using the Win lab software CIE TC1-29. The value of Λ_{max} was around 532.

Results and Discussions

Add-on%

The results represented in Table 1 showed increase in the sample weight relative to the original weight after the application of the BPS. It has been noted that the weight increased as BPS concentration increased till the concentration reached 10%.

LOI and flammability tests

The untreated and treated cotton fabric samples were dried in horizontal position in an oven for 30 min at 105°C to discounts the effect of moisture content. A material must be considered flammable as long as the LOI value is smaller than 0.26. The cotton fabric samples have been shown to ignite in shorter exposure to ignition source, and burn with higher the time taken by a flame on a burning textile to travel specified distance and specified conditions (flame spread rate) when oven dried than when tested at higher moisture content. The LOI value is dependent on the weight, construction, moisture content and purity of the sample, on the temperature of the testing environment, and on the size and construction of the sample holder. Van and Nijinhuse mentioned that LOI of cellulose was 0.19 [26]. LOI describe the tendency of a material to sustain a flame and it is defined as the minimum fraction of oxygen in a mixture of oxygen and nitrogen that will just support combustion after ignition. The results of flammability tests were represented in

Tables 2 showed that control sample (bleached, mordanted, and mercerized) was found to 0.22. However, the application of BPS in the control samples was found to increase till it reached the value of 3.6 at BPS treated cotton fabric concentrations 10 %, which is almost 30 times than that obtained with the control fabric samples. These results might be due to that during mercerization, cellulose I converted to cellulose II, followed by recrystallization during subsequent washing and subsequently increase the chemical activity due to the free hydroxyl groups (amorphous cellulose). The presence of metal salts in BPS and alum might react with the free hydroxyl groups and lead to decrease the hydrogen to carbon ratio in the cotton samples, and lower the tendency to ignition. It has been noted that the treated fabric showed no flame, but was burning with a propagation rate of 7.5 mm/min, which was almost 10 times lower than that noted with the control fabric. The results of the vertical behavior of the control and the BPS treated samples at different concentrations showed that the control samples were completely burnet with flame, while the BPS treated samples were burnet initially with flame. From Figure 1, it has been noted that the BPS treated samples showed that ignition could be occurred after 4 sec for the control followed with BPS 2.5% treated fabric after 5 sec, and with BPS 5% after 6 and finally the ignition happened at 7 sec for the cotton fabric treated with BPS 10%.

TABLE 1. Add-on% of the cotton fabric samples before and after treatments .

Cotton Fabric (Giza 90)	Weight before/ gram	Weight after/ gram	Add-on%
Control*	75		-
Mordant sample	75	77.6	3.46 %
Sample with BPS 2.5%	77.6	79.3	2.2 %
Sample with BPS 5%	77.6	81.6	5.15%
Sample with BPS 7.5%	77.6	83.2	7.21%
Sample with BPS 10%	77.6	83.3	7.35%

Control * bleached and mercerized Egyptian cotton Giza 90

TABLE 2. Flammability parameters of control, and BPS treated cotton fabrics.

Flammability parameters —	Control*	BPS treated cotton fabric concentrations %				
Fiantinability parameters =		2.5	5	7.5	10	
LOI	0.22	0.65	1.1	3.4	3.6	
Horizontal Flammability						
Warp way burn rate (mm/min)	75	44	27	15.5	7.5	
Vertical Flammability						
State of the cotton fabric in contact with flame	C. B	B.I	B.I	B.I	B.I	

Where C. B: completely burnet with flame, B.I: burnet initially with flame

TABLE 3. Ignition parameters of control, and BPS treated cotton fabrics.

Time	Control*	BPS treated cotton fabric concentrations %				
		2.5	5	7.5	10	
3 s	NO.I	NO.I	NO.I	NO.I	NO.I	
4 s	I.O	I.O	NO.I	NO.I	NO.I	
5 s	I.O	I.O	I.O	NO.I	NO.I	
6 s	I.O	I.O	I.O	I.O	NO.I	
7 s	I.O	I.O	I.O	I.O	I.O	

Where NO.I: Ignition did not occured I.O: Ignition occurred

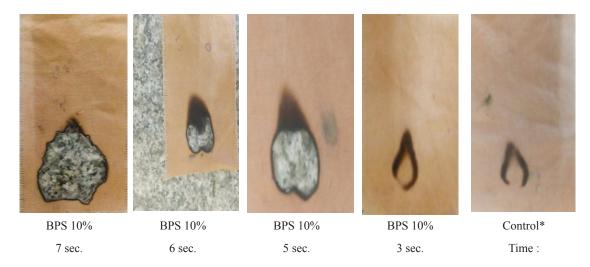


Fig. 1. Comparison in burning behavior of control and BPS treated cotton fabrics at different time interval.

FTIR assignment

Figure 2 (A, and B) showed the FTIR spectra of both cotton untreated samples and treated with BPS at 10% respectively. For the cotton fabric samples without BPS, the FTIR absorption bands observed at 1607 cm⁻¹ might be assigned to the (AlH₄)⁻. The observed peak at 2156 cm⁻¹ was mainly due to the presence inorganic salts such as Fe, and Mn, [27]. It can clearly be observed

that cotton samples with (BPS) showed a wide band from 3600 cm⁻¹ to 2600 cm⁻¹ confirming the presence of water. The observed peaks between 800 cm⁻¹ to 1300 cm⁻¹ wavelength were mainly due to the presence of (PO₄)³⁻ in the BPS [28]. The new observed peaks at 2040 cm⁻¹ wavelength were mainly due to the presence of Fe⁻ in the BPS [29].

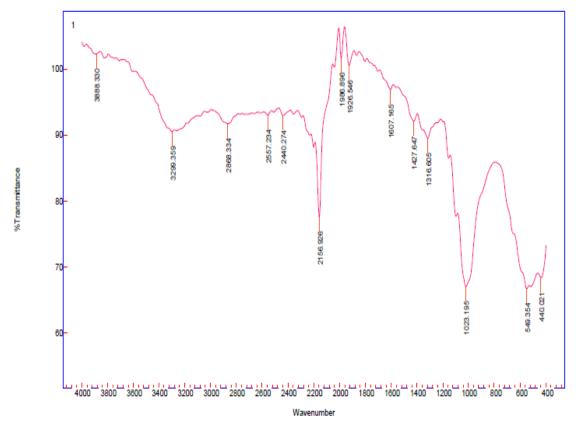


Fig. 2 (A) the FTIR spectra of cotton samples.

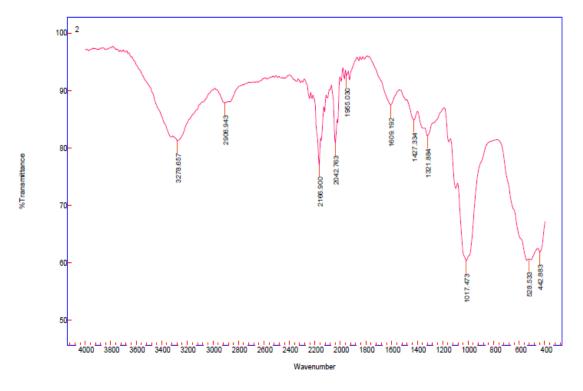


Fig. 2 (B). the FTIR spectra of cotton samples treated with BPS at 10% concentration.

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Thermo-gravimetric analysis (TGA)

Figure 3 (A and B) showed the TGA curves of both cotton untreated samples and treated with BPS at 10% respectively in N2 atmosphere at a heating rate of 10°C/min. The TGA curves of the control (A) and the BPS (B) samples evidenced three stages of progression. In the initial stage at temperature below 300°C, the little mass loss occurred mainly due to the presence of metal salts in BPS and alum might react with the free hydroxyl groups and lead to the removal of bound and unbound absorbed moisture from the cellulose polymer. These results were in accordance with the results obtained by Shen et al. [30]. However, the main thermal decomposition occurred in the temperature range of 300–360°C, where the mass of the sample sharply decreased at around 340°C. This has happened mainly due to that pyrolysis of cellulose producing the volatile compounds proceeds through two primary simultaneous reactions: (a) the initial scission of glucosidic linkages, and (b) chemical changes in anhydroglucose unit such as dehydration and scission of C-C bonds. The mechanism of formation of the volatile compounds, however, has remained to be resolved. These results

indicated that in mercerized samples, heating affected the amorphous regions of cellulose more than the crystalline. These results were in accordance with the results represented in references [31-32]. Above the temperature of 360°C, both dehydration and char formation occurred. In Figure 3 (A), degradation was observed only in the control samples where the non-oxidizable water and CO₂ might have been released. The control and the treated sample treated with 10% BPS lost approximately 98% of its mass below 500°C. In Figure 3 (B), unlike the control fabrics, the BPS treated cotton fabrics started losing more mass at the initial stage below 200°C. This might be due to the presence of the BPS that had reduced the thermal decomposition and the dehydration temperature of cellulose. The BPS also had an effect on the chemical process of incomplete combustion of certain solids when subjected to high heat (char formation). It has increased the char formation and produced nonoxidisable gases such as CO, and H₂O, while reducing the production of flammable volatile gases. As a result, the BPS treated samples showed more LOI value and lower rate of burning.

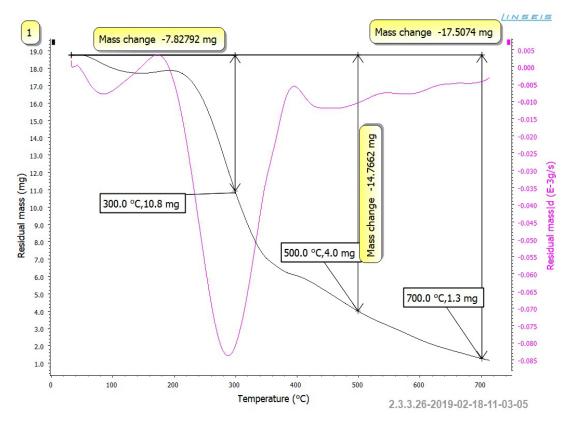


Fig. 3 (A) Thermo-gravimetric analysis of the control cotton samples.

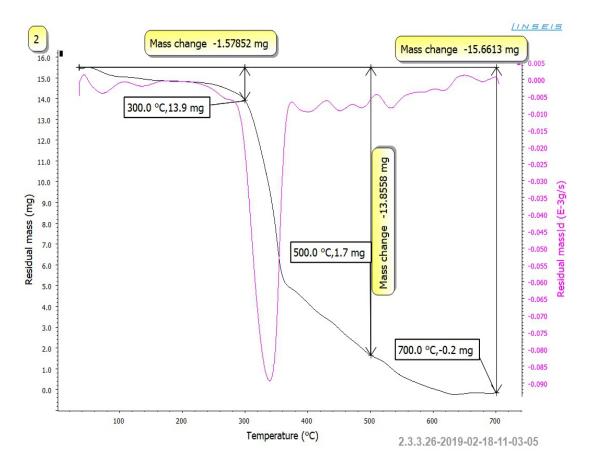


Fig. 3 (B). Thermo-gravimetric analysis of the treated cotton samples with BPS 10% concentration.

Mechanical strengths

The present application of BPS was found to have significant adverse effect on both the tensile strength and elongation percentage. In most cases of application of conventional and commercial flame retardant finishes, there occurred a significant loss of tensile strength, in the range of 10–30%, [33]. Another assumption is that in mercerization treatment, concentrated sodium hydroxide penetrates inside the fibers and reacts with the hydroxyl groups inside the macromolecule in such a way that it either produces sodium cellulosate or it links to the molecules through the pulling forces increase the amorphous region which lead to decrease the tensile strength. As shown in Table 4, there was no significant change in tensile strength. The application of BPS on the control cotton samples caused a very slight increase in tensile strength and that might be due to the linkage between the free hydroxyl groups of the cotton fabric and the metal salts present in BPS. On the other hand, an observable increase in elongation percentage in the control samples due to the Egypt.J.Chem. 63, No. 9 (2020)

effect of mercerization treatment and decrease in elongation percentage after the control fabric treated with BPS.

Color Parameters

The K/S (depth of color) changed from 0.01 for the control to 5.13 for the BPS treated samples at 10% concentration. With further increase in the BPS concentration, K/S was found to increase and L (lightness-darkness) tends to decrease. These results indicate that mercerization treatment has an influence on K/S values due to that cotton fabrics can be considerably modified during mercerization in terms of crystallinity, orientation of crystallites, as well as orientation of macromolecular chains resulting in increased amorphous and less crystalline but with improved orientation of fibers micro and macro units. Mercerization process involve partial destruction of inter molecular bonds. The fibrous transformation from cellulose I to cellulose II occurs during mercerization, which consists of a swelling of the initial fibers in alkali, followed by recrystallization during subsequent washing and subsequently increases the chemical activity due to the free hydroxyl groups (amorphous cellulose) which react with the dyes more than the un-mercerized samples [25]. The presence of alum and tannic acid would affect the dye uptake due to the formation of complex with the hydroxyl ions. According to our previous study, Tannin is not a metal salt; actually it is a watersoluble phenolic compound. When tannic acid was used as mordant, the tannin reacted forming strong complexes. On soaking the fabric into the solution, a reaction between the hydroxyl groups in the fabric surface and the charged complex carried out [34]. The (a*) values changed as the BPS concentrations changed from 2.5% to 10%. In the presence of BPS, the fabric samples were more redness in compared with the fabrics with tannic acid mordant. The (+b*) values indicated that the cotton fabrics tends to the pale yellow color, and the (+b*) values has its maximum in BPS concentration at 10%. The hue angle (h) was from 60 degree to 47 with BPS concentration at 10% and that the treated fabrics tends to the kaki color more than that of the samples BPS concentrations at 2.5, 5, and 7.5%. The results also revealed that the saturation of the color values changed with the application of BPS concentrations. It has been noted that the colored cotton have a good color fastness to light and perspiration and poor fastness for washing due to due to partial removal of the active BPS molecules such as metal salts, phosphate and silicates.

Conclusions

The present study has demonstrated the natural dyeing and flame retardancy effects of BPS on Giza 90 Egyptian cotton fabric and

postulated the scientific basis of the same. BPS is abundantly available in Egypt and is normally considered as a plant waste material, though it is an eco-friendly natural product and produced from a renewable source. After application of BPS under alkaline condition (mercerization), the LOI increased almost 30 times than that obtained with the control cotton fabric samples when the concentration of BPS reached to 10%. This will provide more safety time to a human being either to extinguish the fire or to escape from zone of fire hazards. According to the FTIR spectra, the flame retardancy in the BPS treated cotton fabric might be attributed to the presence of AlH₄, Fe, Mn, and (PO₄)³ and water molecules. Also, the presence of such inorganic salts in BPS treated cotton fabric might have aided in the production of more char and nonflammable gases. The TGA curves revealed that the dehydration and the char formation phenomena in the BPS treated cotton fabrics have been observed. The BPS application treatment is simple, cost-effective, and no costly chemicals are used. The added advantage is that the BPS treated cotton fabric could also be considered as a naturally dyed cotton fabric. The cotton fabrics acquired the khaki color which is quite attractive due to the application of BPS. The newly process can be used beneficially in coloration and in imparting flame retardant finishing of different textiles like window curtain, railway curtain, hospital curtain, table lamp, and as a covering material of non-permanent structures like those used in book fair, festival, religious purposes and such on, where a large quantity of textile is used thus, posing possible threats of fire hazards.

TABLE 4. Tensile strength (Kg/f) and Elongation % of control, and BPS treated cotton fabrics.

Elongation %	Tensile strength (Kg/f)	samples
16.10	44.67	Control*
14.39	44.84	Sample with BPS 2.5%
14.43	45.54	Sample with BPS 5%
14.36	45.93	Sample with BPS 7.5%
13.89	46.2	Sample with BPS 10%

Where Control*: bleached, mercerized and mordanted cotton samples without BPS

h	C	b*	a*	L*	K/S	Sample
		0.21	0.09	98.4	0.02	Control*
59.8	10.1	17.4	3.32	62.8	2.23	Sample with BPS 2.5%
56.2	11.8	16.76	3.5	59.1	3.25	Sample with BPS 5%
52.1	13.3	16.21	3.96	57.3	3.79	Sample with BPS 7.5%
47.2	14.4	15.9	4.3	53.8	5.13	Sample with BPS 10%

TABLE 5. Colorimetric data of Giza 90 cotton fabric samples before and after treatments.

Control*: As mentioned in Table 4

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مقاومة الاقمشة القطنية للهب وصباغتها الطبيعية باستخدام المخلفات النباتية من خلايا الموز الجزعية

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أوضحت الدراسة الحالية تأثير مقاومة الاقمشة القطنية المبيضه والمحررة المصنوع من صنف جيزة ٩٠ للهب وصباغتها الطبيعية باستخدام المخلفات النباتية من خلايا الموز الجزعية كمخلفات نباتية بتركيزات مختلفة. تم استخدام الطريقة القلوية لإنتاج مستخلص BPS .. تم تحليل خصائص مثبطات اللهب لكل من الأقمشة القطنية غير المعالجة والمعالجة من حيث الحد من مؤشر الأكسجين (LOI) والقابلية للاشتعال الأفقي والرأسي. تم تحليل وقياس الأقمشة القطنية المعالجة باستخدام FTIR ، والتحليل الحراري للجاذبية ، والخصائص الميكانيكية، وتحليل مكونات الألوان. أوضحت النتائج أن الأقمشة القطنية المعالجة وجدت أن لها خاصية مثبطة للهب (LOI) بتركيز ١٠ BPS ٪ مقارنة مع الأقمشة الغير معالجه. في اختبار القابلية للاشتعال الرأسي أظهر النسيج المعالج أي لهب ولكنه كان يحترق بمعدل انتشار ٧٠٥ ملم / دقيقة ، وهو ما يقرب من ١٠ مرات أقل مما لوحظ مع نسيج الأقمشة الغير معالجه. أوضحت النتاتج عدم وجود تدهور ملحوظ في القوة الميكانيكية.

بناءً على النتائج، تم صباغة الأقمشة القطنية باستخدام معالجات BPS ووجد ان خواص الصباغة والثيات كانت جيدة مقارنة بالأقمشة الغير معالجه.