Indian Journal of Fibre & Textile Research
Vol. 42, June 2017, pp. 215-222

# Self -extinguishable cellulosic textile from *Spinacia oleracea*

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*Received 14 February 2015; revised received and accepted 24 June 2016*

Flame retardancy has been imparted to cellulosic cotton textiles using spinach (*Spinacia Oleracea*) juice (SJ). The extracted juice has been made alkaline and then applied a fresh to a bleached and premordanted cotton fabric. The flame retardant properties of both the control and the treated fabrics are analysed for limiting oxygen index (LOI) and vertical flammability. The study shows that the SJ treated fabrics have good flame retardant properties, with LOI of 30 for the SJ treated fabric, showing an increase by about 1.6 times compared to the control fabric with LOI of 18. As a result, the treated fabric does not catch flame and in the vertical flammability test, it burns with an afterglow and a propagation rate of 43.5 mm/min, which is almost 5 times lower than that observed with the control fabric. The mechanism of imparting the flame retardancy to the cotton fabric by application of SJ has been postulated and supported by SEM, EDX and char mechanism studies. The durability of the treatment to soap washing, rubbing, dry-cleaning and sunlight has also been studied. Besides, its application produces a natural green colour on the fabric, and no deterioration in other physical properties is observed.

**Keywords**: Cotton, Fastness properties, Fire retardant, Spinach juice

## 1 Introduction

Among the various improved functionalities for textiles, such as wrinkle resistance, anti-soiling, water-repellent, anti-microbial, colour fading resistance and fire retardancy, the last one is known for its paramount importance when intended to use in home furnishings, hospitals and railways. The flame retardant textiles are also important for those workers, who are directly engaged in oil, gas, and petroleum industries. Mostly the cotton textiles are used in all such application areas, due to their advantages of comfort, soft-feel, good moisture-management property, eco-friendliness and bio-degradability. But



readily that is quite difficult to extinguish. This poses a serious risk to health and life of a living being, and damage potential to textile products. Significant efforts have been made in the past to improve the flame retardant property of cotton textiles using various synthetic chemicals and many of them are available in the market[1,2] .

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In last fifty years, flame retardants based on the composition of phosphorous, nitrogen and halogen, like Tetrakis phosphonium chloride salt and N-alkyl phosphopropionamide derivatives are widely dominating the commercial scenario[3]. However, as such formulations need to be applied in an acidic condition, due to which cotton fabric loses its tensile strength and becomes stiffer. Besides, such a treatment is expensive and non-ecofriendly due to the involvement of a large amount of chemicals, high temperature curing process and the emission of formaldehyde during the treatment if the process has not been controlled properly[4]. In 1990s antimony in ... though could impart good flame retardant property, but still not very successful due to the negative impact of halogen compounds in the environment[5]. Though some intumescent based flame retardant like silicate[6], and composition of the nano zinc-oxide and polycarboxylic acid [7] have been developed to make the process environment friendly, such treatment cannot satisfy the handle, strength and fire resistant durability requirements of the fabric. This apart, the increased awareness on human health and hygiene in recent years is also driving the demand of those cellulosic textiles, which are finished with natural products, such as natural dyes for colouration, enzyme for bio-polishing, neem and aloe vera extract for antimicrobial finishing [8-10]. A very few researches have so far been reported on imparting fire retardancy to cellulosic fabric using natural products[11-13]. Recently, the researchers have reported that DNA from herring sperm and solomon fishes can be applied to the cotton fabric to make it thermally stable. They have also reported that DNA contains phosphate, carbonaceous deoxyribose units, polysaccharide dehydrate and some essential amino acids, which are helping in carbonaceous char formation and ammonia release, thus making the cotton textile thermally stable[11]. Attempts have also been made to impart fire retardancy to 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[12,13]. However, a very few applications of plant extract (bio-molecules) for imparting flame retardant finishing to any textile and/or polymeric material has been reported so far, to the best of our knowledge. As some of the plants contain phosphorous, silicate and other minerals 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 and ligno-cellulosic jute fabric, using banana pseudostem sap (BPS)[14]. In the previous paper, we have reported the application of SJ in different concentration for imparting the fire retardant property in the cotton fabric[15]. In this study, a detail investigation have been done to improve the flame retardant property of cellulosic cotton textiles using a vegetable extract, such as spinach juice (SJ), as it contains sodium, magnesium, silicate, iron, protenious nitrogen, nitrate, organophosphate and other metallic constituents[16]. In the present investigation, we report an extensive study to assess the application of spinach juice (SJ) with or without mordant on the flame retardant properties of cotton textiles. In addition, detail char mechanism and other essential fastness properties of the fabric has also been reported. This study also includes the possible mechanism of imparting the said functionality, supported by various analytical studies.

## 2 Materials and Methods

A 200 GSM plain woven bleach cotton fabric of 30 EPI (ends/ inch), 40 PPI (picks/inch) with 25s warp and 30s weft counts, procured from the local market, was used in the study. Spinach juice (SJ) used for the study was extracted from fresh spinach leaves procured from local market. Extracted juice was filtered by Whatman (No. 1) filter paper and prepared for application on the cotton fabric. Bleached cotton fabrics were then impregnated with alkaline SJ using material- to- SJ ratio 1:15 at 90°C for 30 min. On the other hand, one bleached cotton fabric has been mordanted with 5% tannic acid and 10% alum followed by treatment with alkaline SJ by following same material-to-liquor ratio. All the treated fabric samples were then dried at 110°C for 5 min.

### 2.1 Test Methods

Before testing, the fabric samples were conditioned for 24 h at 65% RH and 27°C.

#### *2.1.1 Determination of Per cent Add-on*

The per cent add-on was determined by the gravimetric principle, from the difference of the bone dry weights of the sample before and after the treatments, and expressing the results in percentage over the initial bone dry weights, as shown below

$$\text{Add-on } \% = [M_2\text{-}M_1/ M_1] \times 100 \quad \dots (1)$$

where, $M_1$ and $M_2$ are the oven dry weights of control and treated fabric samples respectively. The reported results are the average of 5 tests in each case.

#### *2.1.2 Flammability Assessment*

The burning behaviour of the control and the treated samples was evaluated by the standard methods. For assessing the limiting oxygen Index (LOI), IS: 13501 test method was used[17]. In the vertical flammability test, the different parameters were measured as per IS: 1871 method A. The maximum temperature produced during the burning of the sample was measured using an IR thermometer, Fisher Scientific made (Model No. 15077968 FB61354 225PE) in non-contact mode.

#### *2.1.3 Fastness Test*

The durability of the flame retardant finishing was evaluated after washing the samples in a laundrometer using a standard detergent at a concentration of 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 in a standard atmosphere.

Rub fastness test was carried out as per IS 766 method. After rubbing LOI value has been evaluated as per standard method.

Light fastness test of the control, mordanted and the treated fabric samples was carried out. In this regard, fabrics have been exposed to the normal sunlight from 10 am to 3pm with different day duration. After sunlight exposure, sample colour and oxygen index values were measured. Here, reported LOI is the average of three test values. For clear understanding, light fastness test was performed as per AATCC test method 16.3-2014 colour fastness to light: Xenon-Arc and the results are presented as per AATCC grey scale ratings.

Treated and untreated cotton fabric samples were tested against dry-cleaning as per IS: 4802-1988 method. After dry-cleaning the LOI values of the control and the treated fabrics were measured as per standard method.

### 2.2 Characterization of Treated Fabric

#### *2.2.1 SEM and EDX Analysis*

The Surface of both the control and the SJ treated samples as well as washed samples were analysed using a scanning electron microscope, Philips XL-30. Char mass of the control and the treated fabric also have been analysed by SEM. Before testing, 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. The EDX analysis of the samples was carried out on a field emission gun scanning electron microscope (FEG-SEM) to determine the quantity of the elements present on the surface, and was expressed both in atomic and weight per cent [18,19].

#### *2.2.2 P and N Content Analysis*

The amount of P in the SJ treated fabrics has been measured according to the chloro-stannous blue method[18] and the amount of N by Kjeldahl method[19].

## 3 Results and Discussion

The results of the flammability test for both the control as well as the treated samples are presented in Table 1.

### 3.1 Limiting Oxygen Index (LOI) and Vertical Flammability

Textiles, having the LOI of ≤ 21, ignite easily and burn rapidly in the open atmosphere, but those with the LOI of >21, ignite but burn slowly. When the LOI value of a fabric is equal or more than 26, it may be considered as a flame retardant[20-22].

SJ treated cotton fabric shows LOI of 30 which is approximately 1.6 times higher than that of the control fabric (LOI 18). As far as the vertical flammability is concerned, control fabric is burnt within 1 min with flame and flashing, whereas the SJ treated fabric shows no flashing and flame and it takes more than 5 min for complete burning with after-glow. Depending on the vertical burning behaviour, of the control and the treated fabric, burning model has been proposed and represented in Fig. 1. It shows that

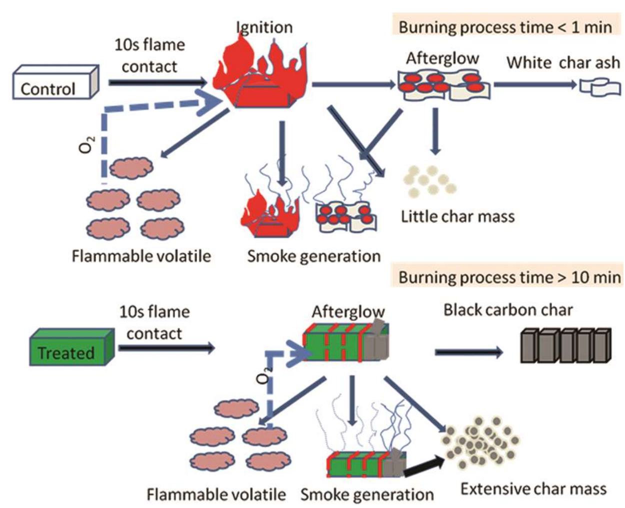


Fig. 1 — Comparison of burning model between control and SJ treated cotton fabrics

Table1 — Flammability test results of control and SJ treated cotton fabrics

| Flammability parameters | Control | Mordanted (A) | SJ treated (B) | Mordanted+ SJ treated (C) |
|---|---|---|---|---|
| Add on,% | - | 2 | 8 | 8 |
| LOI | 18 | 19 | 30 | 25 |
| Vertical flammability | | | | |
| Occurring of flashing over the surface | Yes | No | No | No |
| Burning with flame time, s | 60 | 60 | Nil | 100 |
| Burning with afterglow time, s (after flame stop) | 15 (after complete burning of the fabric) | Nil ( No afterglow after complete burning) | 345 | Nil |
| Total burningtime, s (flame time + after glow time) | 60 + 0 | 60+ 0 | 0 + 345 | 100 |
| Char length, mm | Nil | Nil | Nil | Nil |
| Observed burning rate, mm/min | 250 | 250 | 43.5 | 150 |
| State of the fabric | Completely burnt | Completely burnt | Completely burnt | Completely burnt |

in case of SJ treated fabric no flame has been observed. However, afterglow present and propagates slowly at the rate of 43.5mm/min and the fabric is burnt. It is also observed that here char mass produced is more blackish and harder rather than the control fabric, which will be discussed later in this paper. Here, during vertical burning we have measured the temperature of the control and only SJ treated fabric by non-contact thermometer. It shows that the control fabric is burnt with 395-400°C temperature, whereas the sample B shows the initial temperature of 270°C which is reduced gradually to 150°C during further burning course. Therefore, it will be very much helpful for the users in the real life situation, as they will get much longer time (345s) to escape from the fire hazard-zone or to extinguish the flame. In contrast, the user will be getting hardly any time (60s only) to escape from a fire hazard environment with temperature of ~400ºC, if the fabric is not flame retardant. One disadvantage of this treatment is that, the SJ treated fabric is burnt with afterglow and it propagates with very slow rate. It limits the application of the treated fabric commercially. Therefore, for stopping the afterglow, 2-4% boric acid is used in the SJ formulation which can stop the glow and help in enhancing self-extinguishing property of the treated fabric. As a result, the treated fabric shows the specific char length after vertical flammability test. In this regard, the further research is going on.

### 3.2 Char Analysis

Char mass analysis of the control and the treated fabric was performed and represented in Fig.2. It is observed that the treated fabric shows hard, solid, blackish char mass compared to the white, lightweight net like char of the control fabric. To understand the phenomena, the SEM analysis was performed on the residues of both the control and the treated fabric after the conduct of flammability test and the same is also shown in Fig.2 (outset A and B). It can be seen that the treated fabric shows an intact char structure (B) of closed cells containing many small pockets of gases. It is felt that SJ might be working as an additive Intumescent flame retardant. When heated, it swells and forms a protective thick honeycomb like coating on the underlying heated polymeric substrate, thereby preventing the flow of volatile liquids or vapours into the flame. On the other hand, the control fabric shows a poor char structure (A) without any closed cell, but with prominent channels through which the volatile gas or the decomposed polymeric substances can escape. Depending on the SEM pictures two char models (C and D) have been proposed and are shown in Fig. 3. Here control char structure (C) shows net like capillary formation through which volatile flammable gases can easily pass through and help to burn the fabric continuously. However, treated fabric consists of char of closed cells (D) so that flow of

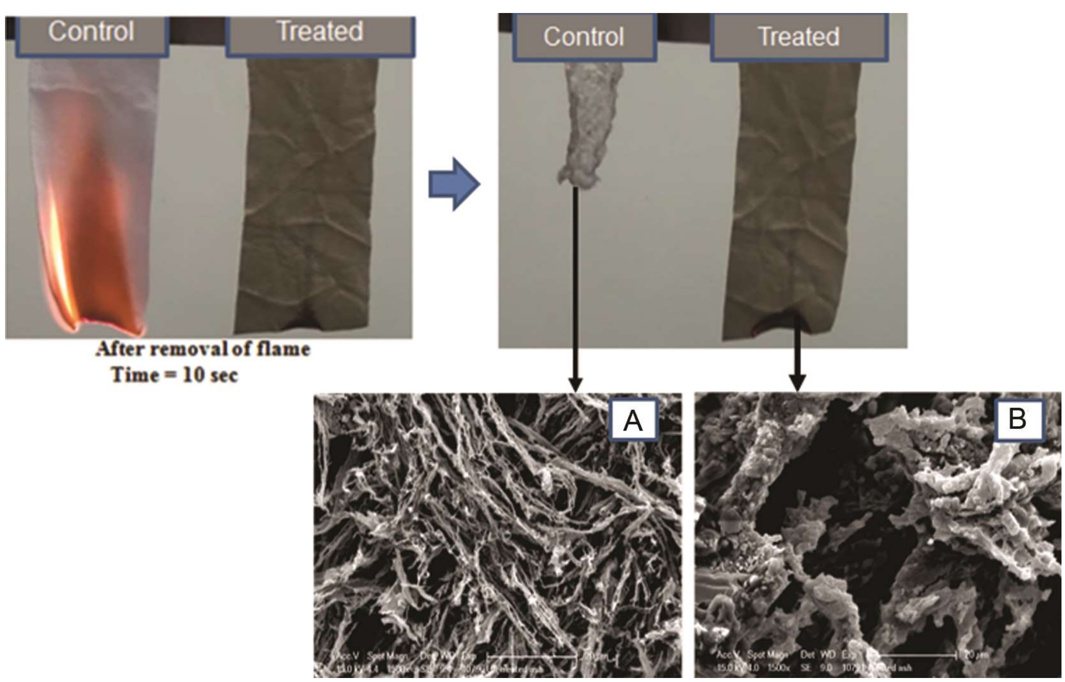


Fig.2 — Char formation and model SEM pictures of the char mass of control (A) and treated (B) fabrics

flammable gases has been interrupted inside the BPS coating and cannot come in contact of flame; and as a result fabric dose not catch flame. . Same structural phenomena have also been reported in the literature [23,24].

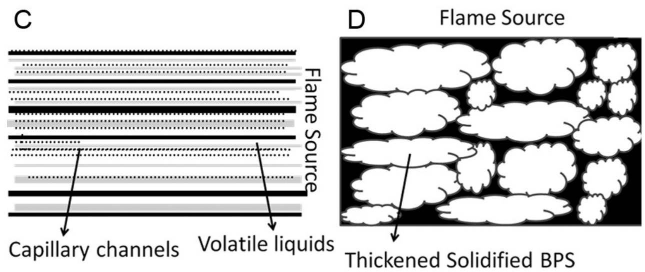


Fig. 3 — Model char structure of the control (C), and the SJ treated (D), cotton fabric

### 3.3 SEM and EDX Analysis

The SEM images of the control and the SJ treated (1: 0) cotton fabrics are shown in Fig. 4. The control sample is clean and there is no coating and deposition [Fig. 4 (A)]. However, after the SJ treatment (1: 0), the coating of SJ could easily be visible [Fig. 4 (B)], as SJ is uniformly distributed over the entire fabric surface. The energy dispersive X-ray analysis of the control and the SJ treated fabrics is reported in Table 2. As expected, the control sample shows only the presence of carbon and oxygen atom, as the technique cannot detect hydrogen atom. However, in the SJ treated sample, several elements like sodium, magnesium, silicon, and chlorine are present.

The total amount of phosphorus and nitrogenous matter present in the control and treated fabrics are

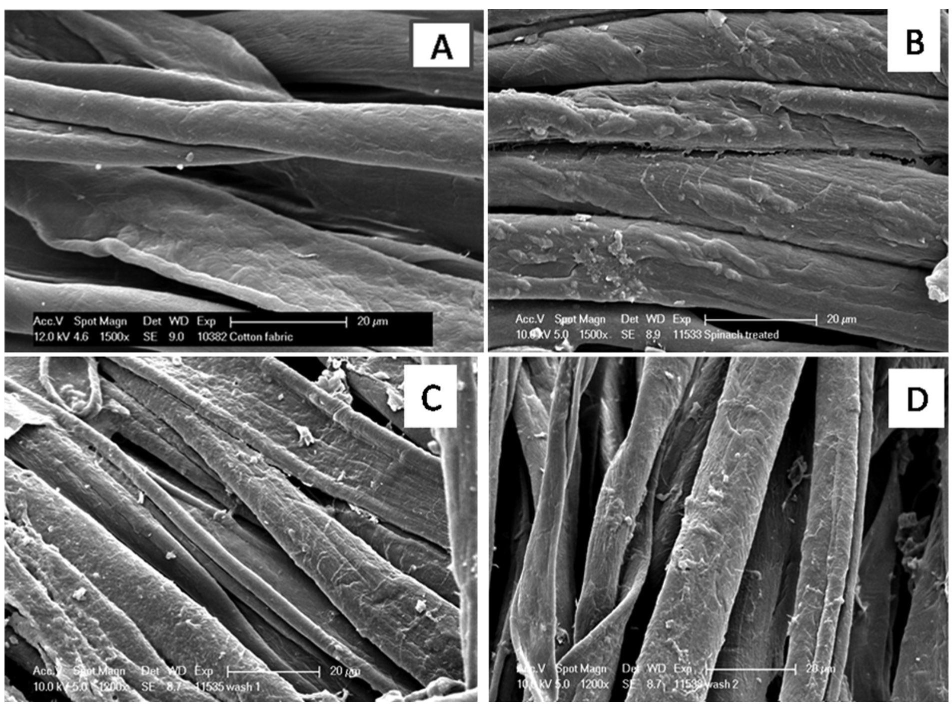


Fig.4 — SEM images of control cotton fabric (A), SJ treated (1: 0) fabric (B), treated fabric after one wash (C), and treated fabric after two wash (D)

Table 2 — Atomic and weight percentage of control and SJ treated cotton fabric by EDX

| Elements | Control | | SJ Treated | | Mordanted + SJ | |
|---|---|---|---|---|---|---|
| | Weight , % | Atomic ,% | Weight , % | Atomic, % | Weight, % | Atomic, % |
| C | 46.12 | 53.21 | 33.54 | 41.59 | 39.25 | 42.59 |
| O | 53.93 | 46.82 | 57.28 | 52.86 | 54.38 | 48.32 |
| Na | 0 | 0 | 7.78 | 5.00 | 5.75 | 4.02 |
| Mg | 0 | 0 | 0.46 | 0.28 | 0.29 | 0.21 |
| Si | 0 | 0 | 0.21 | 0.11 | 0.12 | 0.08 |
| Cl | 0 | 0 | 0.14 | 0.06 | 0.14 | 0.08 |
| K | 0 | 0 | 0.13 | 0.05 | 0.15 | 0.07 |
| Ca | 0 | 0 | 0.16 | 0.06 | 0.05 | 0.04 |
| Al | 0 | 0 | 0 | 0 | 0.34 | 0.22 |

Table 3 — Nitrogen and phosphorous content in control and treated fabrics

| Treatment condition | % Add-on | %N | %P | %N+%P | LOI |
|---|---|---|---|---|---|
| Control | - | 0.15 | 0.15 | 0.30 | 18 |
| SJ treated | 8 | 1.20 | 0.65 | 1.85 | 30 |
| Mordanted | 2 | 0.12 | 0.11 | 0.23 | 19 |
| Mordanted + SJ | 8 | 0.85 | 0.42 | 1.27 | 25 |

presented in Table 3. It can be observed that the SJ treatment results in an increase in the quantity of nitrogen and phosphorous also. Besides the total amount of phosphorus and nitrogen content, the LOI of the treated fabric is also found to increase. Thus, after SJ treatment at 8% add-on the total amount of nitrogen and phosphorous present on the fabric is 1.85%, which is translated into a higher LOI value of 30. An earlier research have reported that 3% add-on of phosphorus-nitrogen based ecofriendly 4methylpiperazin-1-yl phosphoramidate provides a total nitrogen, phosphorous percentage content of 2.15, and the cotton fabric treated with this composition provides a char length of more than 30 cm with an after-flame of 14-19s (ref. 25). It has been also observed from Table 3 that the mordanted fabric has no additional phosphorous and nitrogen content and after the treatment of this mordanted fabric with SJ total P and N content percentage is lower than in the only SJ treated fabric. It may be due to the fact that mordanting is not helping much to uptake more SJ and as a result LOI value of the fabric is also less.

### 3.4 Mechanism of Imparting Flame Retardancy

Here, cotton is exhibiting flame retardancy mainly because of the chemicals present in SJ. More explicitly, the effect of flame retardancy imparted by spinach juice (SJ) may be attributed to the presence of various mineral salts in it in the form of chloride, phosphate and silicate, such as sodium silicate, magnesium chloride, sodium chloride, etc. The presence of these salt molecules is roughly observed in the FTIR analysis, and by the peaks of the elements in the EDX analysis. An earlier research has reported[26] that dried spinach leaves are a good source of vegetable protein (11.10%) and moisture (11.17%), organophosphate, chloronitrile and phthalimide kind chemical substituents as pesticide materials in SJ as detected by mass spectroscopy. Recently, it is reported[27] that dried spinach leaf powder is rich in nitrate and nitrite content. We have also, by experimental analysis, quantified the total amount of nitrogen and phosphorous in the SJ treated fabric. As these phosphates, nitrate and chlorinated products can act as fire retardants for the cellulosic substrate[3,4], the flame retardancy effect in the spinach juice (SJ) treated cotton fabric is attributed to the combined effect of the presence of metal salts, silicate, phosphate, nitrate, and bound and unbound water molecules. Their presence has helped in the formation of more char and non-flammable gases like $CO_2$, $H_2O$, etc. As far as the flame retardancy mechanism is concerned, SJ mainly acts in the condensed phase mechanism, forming an intumescent layer on the fabric surface, which are aiding the pyrolysis of the treated fabric by forming a char at an earlier stage, as observed from the TGA curve.

Table 4 — Detail fastness properties of SJ treated cotton fabric

| Property | Control | SJ treated | Mordanted+ SJ treated |
|---|---|---|---|
| **Wash fastness** | | | |
| Grey scale rate | - | 3 (2)[a] | 4 (3-4)[a] |
| LOI (after wash) | 18 | 24(22)[a] | 24(23.5)[a] |
| **Rub fastness** | | | |
| Dry rub rate | - | 4-5 | 4-5 |
| Wet rub rate | - | 3 | 4 |
| LOI | 18 | 28 (26)[b] | 24 (23)[b] |
| **Light fastness** | | | |
| Fastness ratings | - | 1 | 1-2 |
| **Dry-cleaning fastness** | | | |
| LOI | 18 | 27 | 24 |

[a] Values after 2nd ISO washing, [b] LOI values after wet rubbing.

### 3.5 Fastness Properties

Fastness properties of the control, SJ treated and the mordanted+SJ treated fabrics are given in Table 4.

The durability of the imparted flame retardant finish in cotton textile was tested after washing the sample in the soap solution. After washing, the burning behaviour of the treated fabric is found changed. This might be attributed to the loss of mineral salts of SJ from the fabric surface during washing. However, deposition also remains on the surface of the treated fabric after washing. It can be observed from Table 4 that the LOI of the washed fabric is decreased to 24 from 30 in the unwashed fabric; still 1.3 times higher than that of the control sample. As far the rub fastness is concerned, all the treated fabric shows adequate fastness property after dry rubbing. However, wet rubbing causes decrease in the fire retardant property as the LOI value is decreased to 26 from 30 in only SJ treated fabric. Light fastness test of the treated fabric is performed as per standard method and it is found that all the treated fabric shows poor light fastness properties. As far as dry cleaning fastness is concerned, both mordanted and without mordanted fabrics treated with SJ, show adequate fastness properties. It may be due to the non-solubility of SJ in nonpolar perchloro ethylene solution.

Fastness of both control and treated fabrics was assessed under sunlight and the LOI values after periodical time duration were recorded (Table 5). The status of the samples after sunlight exposure is represented in Fig.5. From the table, it is clearly observed that even after 300h of exposure, the LOI of the SJ treated fabric remains the same as of the unexposed (0h) control cotton fabric. It means that the harmful UV light of the sun is no deterrent, and does not affect adversely on the fire retardant property of the treated fabric. However, like light fastness test, the colour of the fabric has been damaged or fabric becomes colourless due to long time sunlight exposure. Mordanted and SJ treated fabric turns into permanent khaki colour after sunlight exposure. These all phenomena may be attributed with the disturbing electron stability inside the chromophore of the spinach based natural colour. However, in case of mordanted fabric, alum present in it helps to turn the colour permanent khaki even after prolong sunlight exposure.

Table 5 — Flammability parameters of control and SJ treated fabric after sunlight exposure

| Exposure period, h | LOI value | | |
|---|---|---|---|
| | Control fabric | SJ treated fabric | SJ+ mordanted fabric |
| 0 ( after treatment) | 18 | 30 | 25 |
| 100 (after 20 days) | 18 | 30 | 25 |
| 150 (after 1 month) | 18 | 30 | 25 |
| 225 ( after 45 days) | 18 | 29 | 25 |
| 300 (after 2 months) | 18 | 29 | 24 |

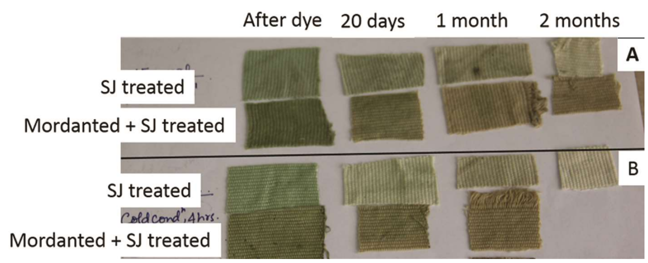


Fig. 5 — Sun light fastness of SJ treated and mordanted+ SJ treated fabrics at different time durations


Fig. 6 — Cotton fabric dyed with SJ in normal and mordanted condition

### 3.6 Physical Properties and Natural Colour

It can be seen that the application of SJ flame retardant finish has no significant adverse effect on both the tensile and tear strength of the treated sample. In most cases of conventional flame retardant finishing of cotton textiles, there occurs a high loss of fabric tensile strength in the range of 10-30%. As indicated in Fig. 5, after application of SJ in the bleached cotton fabric, the sample changes its colour from white to dark green. This can also be viewed in dyeing of cellulosic textile with a natural colouring agent. Also, a significant amount of colour is retained in the treated fabric even after washing. However, this natural colour is developed in the bleached cotton fabric under alkaline condition only. Mordanted cotton fabric after dyeing with SJ also shows green colour. However, periodical yellow and slight blackish lines are observed in the treated fabric. Treated fabric pictures are represented in Fig.6.

## 4 Conclusion

The present study has shown the flame retardancy effect of spinach leave juice (SJ) on the cellulosic cotton textile. The application process is quite simple and cost effective. This developed process can be used in tion and flame retardant finishing of home furnishing products such as home window curtain, hospital curtain, table lamp, and as a covering material of non-permanent structure like in book fair, festival, religious purpose, etc. where a large quantity of textiles is used and which poses the chance of fire hazards. The lower burning rate and the high LOI value of the treated fabric will help to use such fabrics in diversified applications like batik cloth, agro-textile, etc. Spinach leaves are abundantly available in India as well as in other countries from commonly occurring bio-source as an ecofriendly material. Therefore, the application of SJ in cotton textile for colouration and functionalization is an attractive proposition for value-addition to textiles using natural products.

**Acknowledgement**

Authors are thankful to Mr. Abhijit Paul, Mr. Rajiv and Mr. Swamisaran, of NITRA, for their cooperation in conducting the experiments and technical assistance.

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