

## Eco Friendly Flame Retardant Finish For Cotton Fabric

Saminathan Ratnapandian<sup>1\*</sup>, Nafil Rehman J<sup>2</sup>, Surya C<sup>2</sup>, Vaithiyanathan B<sup>2</sup>

<sup>1</sup>Associate Professor, Department of Textile Technology, Kumaraguru College of Technology, Coimbatore, Tamilnadu-49, India.

\*Corresponding author: saminathan.r.txt@kct.ac.in

<sup>2</sup>Final year Students, Department of Textile Technology, Kumaraguru College of Technology, Coimbatore, Tamilnadu-49, India.

### ABSTRACT

Functional finishes enhance the natural appeal of cotton fabrics while providing value-addition. The use of eco-friendly natural ingredients to obtain the functionality opens up niche markets for such products. In line with this concept, our paper reports the combination of spinach leaf juice (SJ) and coconut shell extract (CSE) being employed to impart flame retardant properties to cotton fabric. Researchers have investigated the two components separately for similar activity and therefore we are seeking a synergistic effect. Vertical flammability and Limiting Oxygen Index (LOI) were used to compare the flammability characteristics of treated and untreated fabrics. The results indicate an increase by 1.25 times of the LOI values of the treated fabric. This was also higher than the LOI values of fabrics treated individually. Similar results were also obtained in vertical flammability tests. The capacity of the treatment to withstand soap washing and rubbing has also been investigated. Application of SJ and CSE on cotton fabric sample produced natural green and yellowish brown colors respectively while the combination yielded a darkish green color. Other physical properties were not altered significantly. The importance of the work is that a common bio-waste and easily procurable spinach can be applied by a simple cost-effective process to impart a desirable finish to cotton fabric. An additional advantage is that the treated fabric can also be considered as a natural dyed material. Further work is needed to optimize the process and to determine additional effects of the treatment.

**KEYWORDS:** Cotton fabric, Flame retardant, sustainable, spinach, coconut shell.

### 1. INTRODUCTION

Cotton, “the king of natural fibres”, is a popular and versatile textile fibre. It is the fibre of choice for apparel due to the superior wearer comfort accorded by the breathability and moisture management characteristics. Further, cotton finds applications in a variety of areas ranging from simple apparel to home furnishings and upholstery to hospital linen and many other industrial uses. However, cotton has inherent drawbacks such as high water absorbency, easy soiling and

*Received:* 12 Feb. 2023

*Revised:* 23 Feb 2023

*Final Accepted for publication:* 8 March 2023

Copyright © authors 2023

wrinkling, prone to microbial attack and readily flammable. In order to address these, functional finishes that impart wrinkle resistance, water repellency, anti soiling; anti microbial properties color resistance and fire retardance to cotton textile have been developed. Among these, fire retardance is considered important for materials whose intended use is in military, home furnishings, hospital, and railways.

### 1.1 Fire Hazard:

Cotton, being 100% cellulosic, catches fire readily, and is difficult to extinguish after ignition. This results not only in damage of the textile product but also poses serious and at times fatal health risk. Cellulosic materials produce smoke and toxic gases such as carbon dioxide and carbon monoxide during burning. The catastrophic effects were highlighted during hospital fires in the recent Covid-19 pandemic in India. Majority of the hospital materials such as mattresses, bed sheets, bedspreads, etc. being made of cellulose burnt readily during fire accidents causing serious injuries and many casualties. Similar situations are seen when cotton textiles are used in railways and home furnishings. The means of avoiding this danger is to treat cotton with flame retardants.

### 1.2 Flame Retardants And Their Action:

Treatments that make materials fire proof, that is the material will not ignite, are used for applications such as military and fire fighter uniforms where the risk of exposure to flames is high. Under normal circumstances it is sufficient to treat materials with flame retardant chemicals that delay and or inhibit the ignition and spread of flames. However, such treatments should not disturb the desirable physical characteristics of the fabrics.

Flame retardants refer to a variety of substances that are added to combustible materials to prevent fires from starting or to slow the spread of fire and provide additional escape time. The term “flame retardant” refers to a function, not a family of chemicals. They interfere with combustion at various stages of the process e.g. during heating, decomposition, ignition of flame spread. In last fifty years, flame retardants based on the composition of phosphorous, nitrogen and halogen, like Tetra phosphonium chloride salt and N-alkyl phosphon propionamide derivatives are widely dominating the commercial scenario<sup>3</sup>. However, as such formulations need to be applied in an acidic condition; cotton fabric loses its tensile strength and becomes stiffer. Besides, such treatments are expensive and not eco-friendly due to the involvement of a large amount of chemicals, high temperature curing process and possible emission of formaldehyde if the process is not controlled properly<sup>4</sup>. In 1990s antimony in combination with halogen, was found to impart good flame retardant property, but still not very acceptable due to the negative impact of halogen compounds in the environment. Today proban process and pyrovatex process are widely dominating the

market. However, such formulations need to be applied in an adverse acidic condition and more add-on is required for flame retardancy.

Apart from this, nitrogen and phosphorous based flame retardants are also used to make fire retardant cellulose. Diammonium phosphate and ammonium sulphate are the predominant chemicals used for fire retardant paper over the past 10 years in the European market. Borax diammonium phosphate mixture is gaining acceptance as it has no adverse effects on the physical properties of the treated materials. Researchers have also evaluated mixed formulations of aluminum trihydrate, sodium borate and resin as fire retardant chemicals via an impregnation method. Other researchers have used N-hydroxymethyl-3-dimethyl phospho-propionamide as a durable fire retardant on cellulosic material by using citric acid as binder and phosphoric acid as catalyst. The drawbacks are the large quantities of chemicals used lower strength and increased stiffness after treatment. Besides, the treatment is hazardous and is not friendly to the end users.

Eco friendly alternates such as sodium metasilicate nanohydrate, polycarboxylic acid and nano zinc oxide cannot give satisfactory handle, strength and durable fire resistant to the fabric. Reports on the use of plasma treatment with various polymerization gases to impart fire retardance to the cellulosic fabric are also available. The plasma process is water free and eco-friendly but this process is much costlier and the imparted flame retardant property is not wash durable. Hence efforts have been made to prepare more environment-friendly, cost effective fire retardant products, that when applied to cellulosic or ligno-cellulosic fabrics, will maintain its quality and flame retardant durability to great extent.

The trend today is to utilize natural materials for a variety of purposes. Flame retardants based on whey, milk and DNA proteins have been proposed. Their action is attributed to the phosphate, disulphide and protein content, that influence pyrolysis by early char formation. Some plants are rich in phosphorous, silicate and other mineral salts and offer immense potential as flame retardants to cellulosic and ligno-cellulosic textiles. Banana pseudo stem sap, spinach juice and coconut shell extract have been assessed individually as flame retardants for cotton by researchers. In this work, we investigated the synergistic effect of coconut shell extract and spinach juice as flame retardant for cotton fabrics. The use of bio-waste and common spinach would not only provide desired functional finish but also clean up the surroundings.

## **2. MATERIALS AND METHODS:**

### **2.1 Materials:**

A 350 GSM plain woven bleached cotton fabric of 44 EPI (ends/ inch) and 22 PPI (picks/inch) with 10/2s warp and 8/2s weft count was used in this study. Spinach juice (SJ) and coconut shell

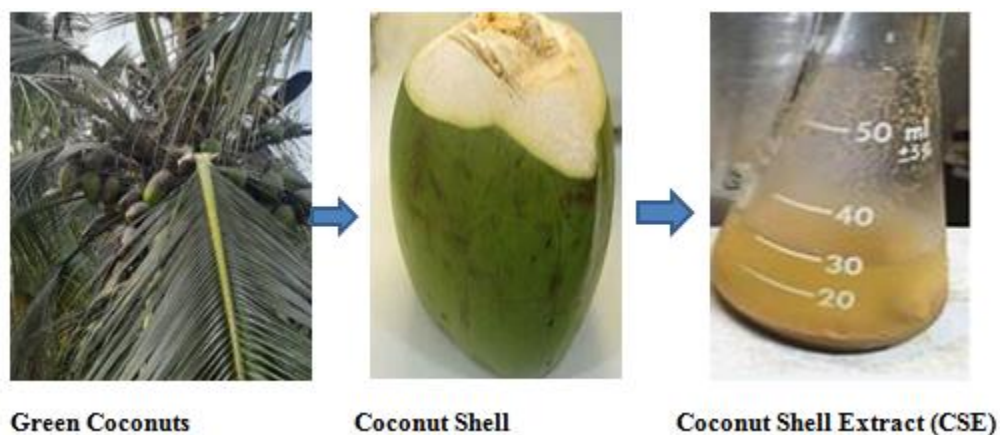
*Received: 12 Feb. 2023*

*Revised: 23 Feb 2023*

*Final Accepted for publication: 8 March 2023*

Copyright © authors 2023

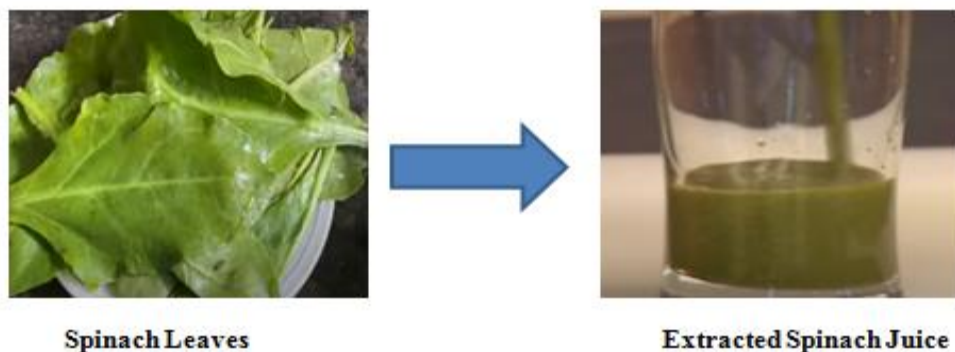
extract (CSE) were extracted from fresh spinach leaves and waste coconut shells. The above materials were procured from local market in Salem, India. Extracted juices were filtered using Whatman No 1 filter paper and prepared for application on cotton fabric. Anhydrous sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) was purchased from Sigma Aldrich and used for the treatment. Original fresh CSE solution after extraction looked yellowish brown in color and showed the pH value of 4.5. It was made alkaline (pH 10) by the addition of anhydrous sodium carbonate ( $\text{Na}_2\text{CO}_3$ ).



**Fig 1. Extraction of Coconut Shell**

## 2.2 Extraction of Spinach Juice

Spinach Juice used for study was extracted from fresh spinach leaves then filtration was carried out for further application. As shown in Fig 2, spinach leaf solution after extraction looked green in color and had a pH value of 5.5. It was made alkaline (pH 10) by the addition of anhydrous  $\text{Na}_2\text{CO}_3$ .



*Received:* 12 Feb. 2023

*Revised:* 23 Feb 2023

*Final Accepted for publication:* 8 March 2023

Copyright © authors 2023

## **Fig 2. Extracted Spinach Juice**

### **2.3 Application on cotton fabric**

Bleached fabrics were then impregnated with CSE juice, spinach juice (SJ) and mixture of CSE+SJ (1:1) at 90°C for 60 minutes with a material to liquor ratio of 1:15. After that, the treated fabric samples were air dried at room temperature. Later, samples were conditioned for at least 36 hours at 65 percent relative humidity (RH) and 27 degrees Celsius prior to testing.

### **2.4 Test Methods**

#### **2.4.1 Determination of Add-on**

The gravimetric principle was used to determine the percent add-on by measuring the bone dry weights of the sample before ( $W_1$ ) and after ( $W_2$ ) the treatments and expressing the results as a percentage of the initial bone dry weight, as shown below.

$$\text{Add-on \%} = [(W_2 - W_1) / W_1] \times 100 \dots\dots\dots(1)$$

In each example, the stated findings are the average of six tests.

#### **2.4.2 Flammability Assessment**

Standard procedures were used to assess the burning behaviour of the treated and untreated samples. ASTM D2863 test approach was applied to determine the limiting oxygen index (LOI). The different parameters in the vertical flammability test were measured according to IS: 1871 method A.

#### **2.4.3 Fastness Test**

The durability of the flame retardant finishing was estimated after washing the samples in a laundrometer using a standard detergent at a concentration of 1g/L at 37°C for 45 min. The fabric was then rinsed in tap water for 5 min, followed by drying at 80°C for another 5 min. The samples were then conditioned for 24 h in a standard atmosphere. Rub fastness test was carried out according per IS 766 procedure. The LOI value was calculated using the conventional method after rubbing. A light fastness test was performed on the untreated and treated fabric samples(SJ,

CSE, SJ+CSE). In this case, materials were exposed to natural sunlight from 9 a.m. to 4 p.m. on different days.

#### 2.4.4 Char Analysis

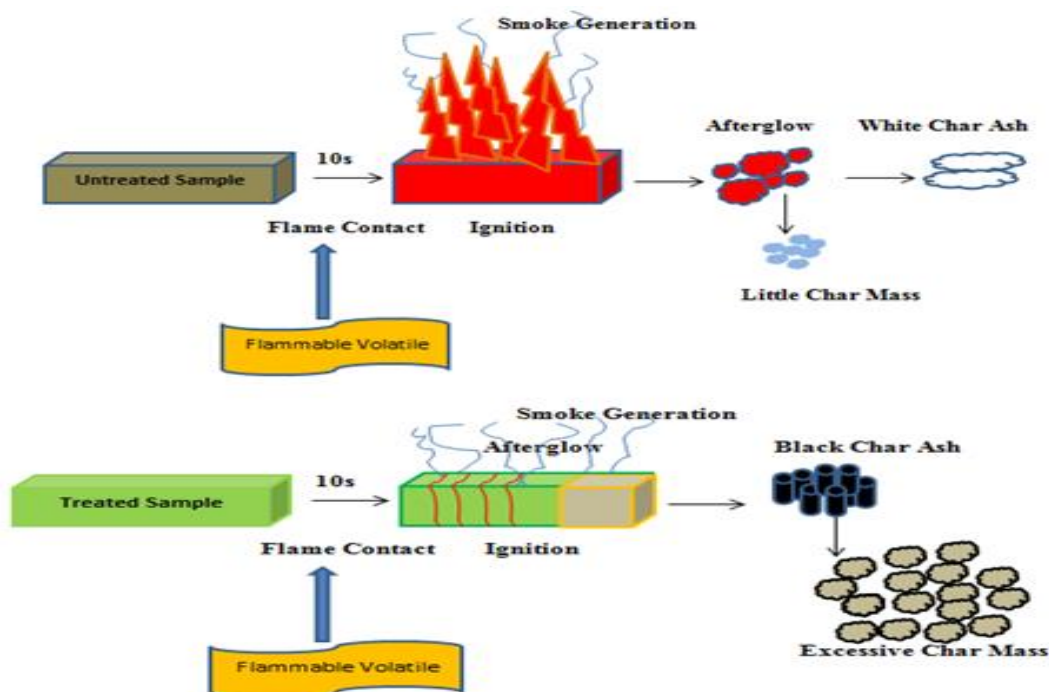
The surface morphology of the untreated and CSE+SJ treated cotton fabric was analyzed using Field-emission scanning electron microscopy (FE-SEM) (Quanta 200; Zeiss, Germany).

### 3. RESULTS AND DISCUSSION:

#### 3.1 Vertical and LOI flame retardancy analysis

Table 1 shows the flammability test results for both the control and treated samples. Textiles with a limiting oxygen index (LOI) of less than 21 ignite easily and burn quickly in the open atmosphere, while those with a LOI of more than 21 ignite but burn slowly. When a fabric's LOI value is equal to or more than 26, it is termed flame retardant<sup>20-22</sup>. The LOI of the CSE, SJ and CSE+SJ treated cotton fabric is 28, 30 & 34 respectively, which is almost 1.5, 1.6 and 1.8 times greater than the untreated fabric (LOI 18).

In terms of vertical flammability, the control fabric burns in 50 sec with flame and flashing, whereas the CSE & SJ treated cloth burns in over 255 & 340 sec with after-glow. Whereas CSE+SJ treated fabric burns in 378 sec afterglow. A burning model has been created and depicted in Fig. 3 based on the vertical burning behavior of the untreated and treated fabrics. It demonstrates that no flame was noticed in the case of treated fabrics.



**Fig 3. Burning behavior of the untreated and treated fabrics**

However, there is an afterglow, which spreads slower than untreated fabric (270 mm/min) at the rate of 80mm/min (CSE), 47.5 mm/min (SJ), and 42mm/min (CSE + SJ.) And burns the cloth. In comparison to the treated fabric, the char mass formed here is more blackish and tougher. We have used a non-contact thermometer to measure the temperature during vertical burning. The control cloth is burned at 390-400°C, whereas treated samples has an initial temperature of 280°C that is gradually decreased to 150°C throughout the duration of the burning process.

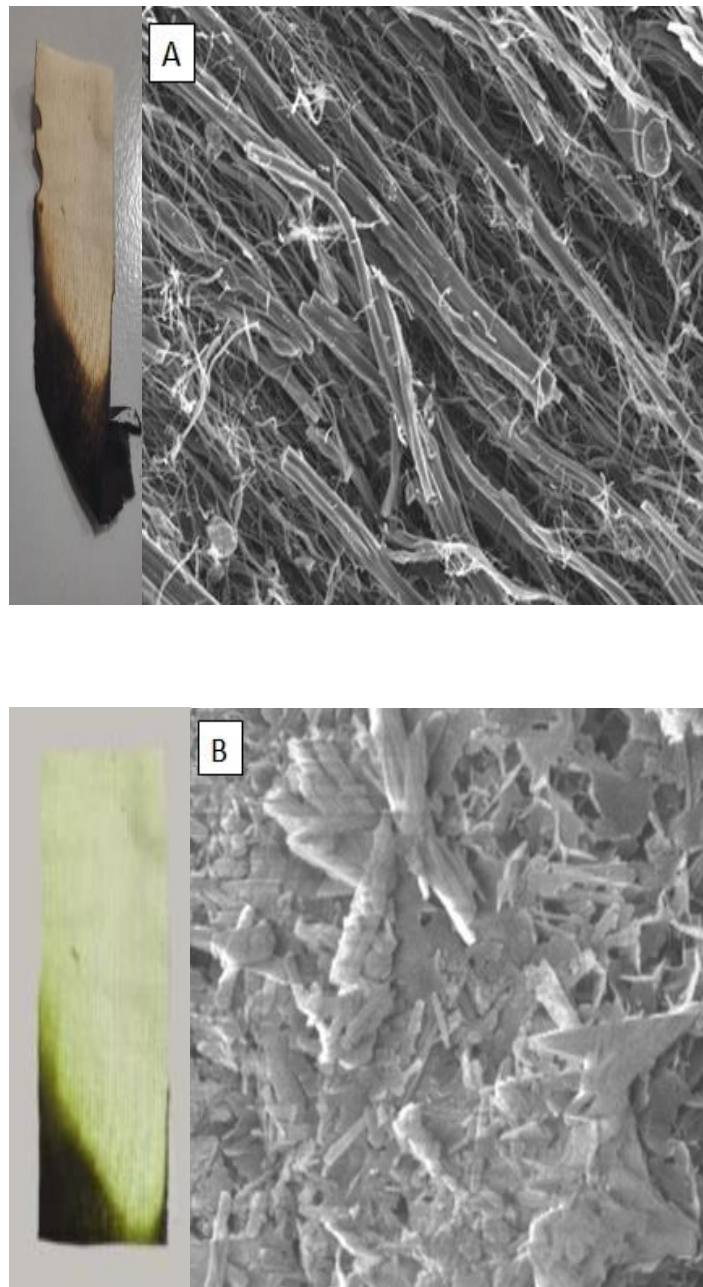
As a result, users in the real world will benefit greatly from it, as they will have significantly more time (378s) to evacuate the fire hazard zone or extinguish the flame. If the fabric isn't flame retardant, the users will only have one minute to get out of a fire hazard with a temperature of 400°C. One disadvantage of this treatment is that the treated fabric burns with afterglow and propagates at a very slow rate. It restricts the treated fabric's commercial use. As a result, 2-4 percent boric acid is used in formulation to reduce the afterglow and improve the self-extinguishing ability of the treated fabric. The cloth displays a certain char length. More research is being carried out.

**Table 1.** Vertical flammability test of the untreated and CSE,SJ, CSE+SJ treated Cotton Fabrics.

Flammability parameters	Untreated	CSE	SJ	CSE+ SJ
<b>Add one %</b>	0	7	8.5	9.5
<b>LOI (%)</b>	18	28	30	34
<b>Occurring of flashing over the surface</b>	Yes	No	No	No
<b>Burning with flame time (s)</b>	50	8	NIL	NIL
<b>Burning with afterglow time (s) after the flame stops</b>	20 (after complete burning of the fabric)	255	340	378
<b>Total burning time (s) (flame time afterglow time)</b>	60+20	8+255	0+340	0+378
<b>Char length (mm)</b>	NIL	NIL	NIL	NIL
<b>burning rate (mm/min)</b>	270	80	47.5	42
<b>State of the fabric</b>	Completely burnt	Completely burnt	Completely burnt	Completely burnt

### 3.2 SEM Analysis of Char of the Untreated and the treated (CSE+SJ):

SEM was used to examine the char mass of the control and treated fabrics. Prior to testing, samples were coated with a thin layer of conducting material (gold) using a sputter coater and studied under a SEM with a 12 kV accelerating voltage. Fabric was performed and represented in Fig.4. 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 untreated and the treated fabric (CSE+SJ) after the conduct of flammability test and the same is also shown in Fig.4. It can be seen that the treated fabric shows an intact char structure (4. B) of closed cells containing many small pockets of gases. It is felt that these natural extracts might be working as an additive Intumescent flame retardant. In comparison to the white, lightweight net-like char of the untreated fabric, the treated cloth has a firm, substantial, blackish char mass. As compare to untreated fabric char (4. A), the treated cloth has an intact char structure (4. B) of closed cells containing several tiny gas pockets. SJ is thought to function as an additional intumescent flame retardant.



**Fig.4: Char formation and model SEM pictures of the char mass of (A) Untreated and (B) (SJ+CSE) treated fabrics**

However, treated fabric contains char of closed cells Fig 5. (b), which interrupts the passage(Capillary channels) of flammable gases inside as shown in 5(a)and prevents them from coming into contact with flame; as a result, the fabric does not catch fire. In the literature, similar structural phenomena have been observed.

*Received: 12 Feb. 2023*

*Revised: 23 Feb 2023*

*Final Accepted for publication: 8 March 2023*

Copyright © authors 2023

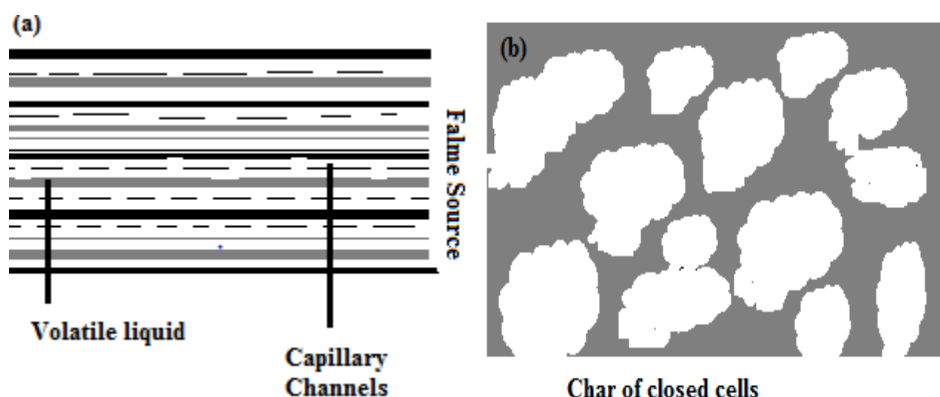


Fig 5: Illustration of char of untreated and treated samples

### 3.3 Fastness Properties:

Table 2. shows the fastness qualities of the Untreated, CSE, SJ and CSE+SJ) treated fabrics. After washing the sample in soap solution, the endurance of the imparted flame retardant coating in cotton cloth was examined. After washing, the treated fabric's burning behavior has changed. This could be due to the loss of mineral salts of CSE & SJ from the fabric surface during the washing process. When it comes to rub fastness, all of the treated fabrics demonstrate appropriate fastness following dry rubbing. Wet rubbing, on the other hand, reduces the fire retardant property. The treated fabric's light fastness is tested using a conventional method, and revealed that all of the treated fabrics have poor light fastness capabilities.

Table 2. Fastness properties of Treated Fabrics

Fastness property		Untreated Fabric	CSE	SJ	CSE+SJ
Washing Fastness		--	3-4	3	3-4
Light Fastness		--	1	1-2	1
Rubbing Fastness	Dry	--	4	4-5	4-5
	Wet	--	4	3	4

### 4. CONCLUSIONS:

A simple and inexpensive approach was used to investigate flame retardancy effect of CSE, SJ and synergistic effect of CSE+SJ on cotton textiles. This process can be used in the flame retardant finishing of home furnishing products such as home window curtains, hospital curtains, table lamps, and as a covering material for non-permanent structures such as book fairs, festivals, religious events, and other situations where a large quantity of textiles is used and there is a risk

of fire. The treated fabric's lower burning rate and high LOI value will allow it to be used in a variety of applications, including batik cloth, agro-textiles, and so on. Spinach leaves and coconut shell are readily available as an ecofriendly material in India and other tropical countries. As a result, employing CSE and SJ to colour and functionalize cotton fabrics is an appealing proposal for adding value to textiles made from natural materials.

#### REFERENCES:

1. Basak S, Samanta K K, Chattopadhyay S K & Narkar R, *Current Sci*, 108 (2015) 372.
2. Basak S, Samanta K K, Chattopadhyay S K, Narkar R & Mahangade R, *Int J Cloth Sci Technol*, 27 (2015) 247.
3. Levchik S V & Supresta LLC, *Advances in Fire Retardant Materials*, edited by A R Horrocks (CRC Press, Washington), 2008, Chap 3, 45-48.
4. Kei S C H, *The effect of atmospheric pressure plasma on flame retardant property of cotton*, BAFT thesis, Institute of Textile and Clothing, Hong Kong Polytechnic University, 2010.
5. Horrocks A R, *Polym Degrad Stabil*, 96 (2011) 377.
6. Basak S, Samanta K K, Chattopadhyay S K, Das S, Narkar R, Desouza C & Saikh A H, *Polish J Chem Technol*, 16 (2014) 106.
7. Basak S, Samanta K K & Chattopadhyay S K, *J Text Inst*, 106 (2015) 1338.
8. Basak S, Samanta K K, Chattopadhyay S K, Narkar R & Mahangade R, *J Industrial Text*, 46, (2016) 1003
9. <http://amrita.vlab.co.in/?sub=2&brch=294&sim=1550&cnt=1>, (downloaded on 10th April 2022).
10. <http://amrita.vlab.co.in/?sub=2&brch=294&sim=1551&cnt=1>, (downloaded on 12th May 2022).
11. Sharma U, *Colourage*, 32 (1986) 19.
12. Shen Y M, Kuan F C, Kuan C H, Chen H C, Wang H J, Yip C M & Chiang L C, *J Nanomater*, (2013) 1-10.
13. Mostashari S M & Mostashari S Z, *Cellul Chem Technol*, 43 (2009) 455.
14. Miller A F & Wilkins H C, *Analytical Chem*, 24 (1952) 1259.
15. Cilurzo F, Selmin F, Minghetti P & Montanari L, *Int J Pharm*, 301 (2005) 62.
16. Nguyen T M D, Chang S, Condon B, Uchimiya M & Fortis C, *Polym Adv Technol*, 23 (2012) 1555.
17. McMichael R W, Bachman M & Huber S C, *Plant Physiology*, 108 (1995) 1077.
18. Zhao L & Stevens J, *Analysis of pesticide residues in spinach using aglient bond elent Quechers AOAC Kits by GC/MS (Agilent Technologies, Wilmington, DE 19809, USA)*, 2012