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Flame retardancy of cotton fabric using sustainable tree gum

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Mesquite tree gum (*Prosopis gum*), an agro waste, has been investigated as an innovative flame-resistant material. The extracted mesquite gum is adjusted at three different *p*H (5, 9 & 12) and applied on selected bleached cotton fabrics. The vertical flammability test and the limiting oxygen index (LOI) have been used to analyse the flame-retardant qualities of cotton (control and finished) fabrics. The mesquite gum finished fabric at alkaline *p*H (12) shows LOI value of 27%, which is 1.5 times greater than that of the control fabric. Besides, a char length of 85mm indicates self-extinguishment. The char surface morphology indicates that mesquite tree gum preserves fibre integrity. Durability of the flame-retardant property of the treated fabric to washing and rubbing has also been studied. The results of thermal degradation show 35 % weight loss with control cotton fabric against 29% weight loss of the mesquite tree gum finished fabric sample at 900 ^{o}C. Hence, mesquite tree gum has a good potential to be a sustainable flame retardant.

Keywords: Cotton, Flame retardant, Mesquite tree gum, Self-extinguishment, Thermal degradation

1 Introduction

Cotton is a versatile starting material for a new product development, as it is abundant, more appealing to consumers and cost effective. Cotton is being used in the field of technical textiles, as it is eco-friendly and biodegradable. Since the cellulosic material is highly flammable, both academia and business have paid close attention to the flame-resistant cotton textiles[1,2].

Recent studies have been significantly featuring on the field of technical textile, which provides textiles with functionalities. In the midst of technical textiles, the fire protection clothing is more essential for industrial workers as they are prone to fire accidents and they are widely marketable for its applications in railway, hospital, home furnishing, etc. It is one of the most demanding fields among the protective clothing for both civilian and defence personnel because of the increased level of threats encountered by the service personnel[3]. The characteristics of flame-retardant clothing include high degree of insulation, slow flame speed, abrasion resistance, strength, higher dimensional stability, low heat generation, and should not melt or form chars under flame[4].

In order to select and optimise the flame retardant, it is vital to understand the chemical nature of the base material as well as its constructional aspects. A variety of factors influence the flammability behaviour of fabrics, such as porosity, mass/unit, finishing effects like napped or lofty construction and surface properties[5,6]. The flammability of clothing and textile products is affected by several factors, including fibre content, fabric weight and structure, finishes, and garment design[7].

The untreated cotton cannot withstand the flame and difficult to extinguish which leads to severe health hazards including damage to textile materials[8]. Remarkable efforts are being made to increase cotton's flame resistance. Among the inorganic salts, urea, phosphate[9], di-ammonium, borax and boric acid are the simple and durable flame-retardant chemicals[10]. For the last 50years the composition of phosphorous and nitrogen compounds has been dominating as commercial flame-retardant products. The cellulosic material was even coated with halogen for making flame retardant textiles. Because of the liberation of dioxins and furanes, this toxic treatment is banned in European countries[11].

When cotton is treated with inorganic substances, it often reduces tensile strength and is time-consuming. Additionally, these chemicals are harmful to the environment and living organisms. To preserve the quality of cellulose, sustainable, and eco-friendly flame retardants are essential. The high cost of chemical treatments has shifted attention toward organic, bio-

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based alternatives in research and development. The research and development team have reported about different bio-macromolecules like pomegranate rind extract[12], banana pseudo stem sap[13], whey protein[14], spinach leaves[15], etc as flame retardants. Considering the health benefits, safety of the environment and effectiveness, the sustainable flame retardants are at its peak of attention. The use of sustainable products can be considered as a promising solution to synthetic chemicals which are toxic in many cases.

Thus, the present research explores the novel use of mesquite tree gum (*Prosopis gum*) from *Prosopis juliflora* to impart flame retardancy on cotton fabric. Further, the influence of pH on flammability behaviour was assessed. The finished fabric and the mesquite tree gum were characterized using thermo-gravimetry analysis, Fourier transform infrared spectroscopy and scanning electron microscopy analyses. Mesquite gum is a neutral salt of an intricately branched acidic polymer. It is made up of a backbone chain of (1-3) linked -D-Galactose units and (1-6) connected branches that contain L-Arabinose in pyranose and furanose ring form, L-Rhamnose, -D-glucuronate, and 4-O-methyl--D-glucuronate as single monomer units or as oligosaccharide side chains[16]. Mesquite tree gum has polyphenol (tannins), which is a naturally occurring family of substances with high char-forming abilities. Tannins, in particular, can be used as a flame retardant and have a high capacity to generate char[17]. It is also highly composed of polysaccharide which makes it as best alternative for synthetic flame retardants. The LC-MS analysis of mesquite tree gum showed the presence of luteolin, a flavonoid that is widely found in natural goods with plant origins. The pharmacological properties of flavones include anti-oxidant, anti-inflammatory, anti-microbial, and anti-cancer properties. The functional properties of the mesquite tree gum include intrinsic viscosity, water solubility, encapsulating capacity, emulsifying ability, surface activity, etc. These unique properties make it as a functional hydrocolloid[18]. Additionally pointing to its functional behaviour, there are the glass transition temperature and moisture sorption characteristics. Hence, this study aims to evaluate the potential of mesquite tree gum in enhancing the flame retardancy of cotton fabric.

2 Materials and Methods

2.1 Materials

A plain, woven bleached cotton fabric (88 ends/cm and 64 picks/cm), procured from the National Textile Corporation (NTC) Coimbatore, Tamil Nadu, India, was used for the entire study. The procured cotton fabric was first desized to remove the size material from the textile. The mesquite tree gum (resin) was soaked overnight with water in 1:2 ratio and mixed thoroughly. The resin's actual *p*H was measured to be 5. The *p*H of the resin solution was adjusted at 9 and 12 using 1N sodium carbonate. The selected fabric was finished using resin solutions with three different *p*H (5, 9, and 12) individually, with material-to-liquor ratio (MLR) of 1:20 for 30min at 100^{o} C and finally used for further investigation.

2.2 Determination of Add-on Percentage

The control and finished cotton fabrics underwent a 48h conditioning period at 27 °C and 65 % RH. The add-on percentage (increase in sample weight) was calculated by comparing the dry weights of the control and finished fabrics, which are expressed as a percentage based on the initial weight of the sample using the following formula:

$$\text{Add-on \%} = [\, M_2 - M_1 / M_1] \times 100$$

where M_1 and M_2 are the weights of control and treated samples respectively.

2.3 Thermal Characterization

2.3.1 LOI Analysis

The limiting oxygen index (LOI) of control and treated fabric samples were assessed using the IS 13501 standard test procedure. For LOI analysis, a 30s ignition time that adheres to Indian standards was maintained.

2.3.2 Vertical Flammability Test

The vertical flammability test was evaluated using the IS 1871 test method, which is comparable to ASTM D 635. Flame height of 350mm was used to ignite the fabric samples for 12s. A test specimen is held vertically above a controlled flame for a predetermined period of time, while measuring vertical flammability. The flame source is turned off after the exposure. The length of time that the specimen continues to flame and the period that the afterglow persists after the flame source has been withdrawn were measured.

2.3.3 FTIR, SEM and TGA Analyses

Shimadzu's IR analyser was utilised to perform Fourier transform infrared (FTIR) analysis on the control and treated fabric samples over the wavelength range of 500-3600 cm^{-1}. To analyse the FTIR, the KBr disc sample preparation method and an

ATR transmittance mode with DLaTGS detector with 49 scans and 4 revolutions were used.

Scanning electron microscopy (SEM) analysis was done to check the surface morphology of control and treated fabrics.

Thermogravimetric analysis (TGA) calculates the rate of mass loss of a sample in relation to temperature and time. Additionally, it shows how flame retardants affect the polymer substrate's pyrolysis[19]. The TGA of the control and the finished fabrics are determined using EXSTAR/6300 at a heating rate of 10°C/min in N2 atmosphere with the standard of ASTM E1131, ISO 11358.

2.4 Fastness Properties to Washing and Rubbing

The standard test method AATCC 61 was used to measure the fastness property to washing. The treated cotton sample was washed in a laundrometer using 1g/L of detergent for 30 min at 40° C. The cloth sample was cleaned and dried for 5 min at 100° C.

The treated sample was subjected to rubbing for 30 rotations in Eureka crockmeter using the AATCC 8 standard test method. Under supervised circumstances, a white crock test cloth is used to rub the flammability along with tested specimen. The LOI of the treated sample was measured after washing and rubbing to check the durability of the flame-retardant property.

3 Results and Discussion

3.1 Flammability Assessment

The control and treated fabrics have been tested for their add-on %, limiting oxygen index and vertical flammability (Table 1).

The limiting oxygen index and vertical flammability assessment have shown that the mesquite tree gum finishing has increased the thermal stability of the finished fabric. The LOI is a parameter that is widely used to describe advancements in protective fabrics fire retardancy. LOI denotes the amount of oxygen that must be present in the test atmosphere in order for the material to ignite and burn in a fiery manner. Textiles having LOI values of 21 or below, ignite easily and burn rapidly in the open atmosphere. The textile material with LOI value of 21 or above, burns slowly but ignites and when the LOI value is equal to or higher than 26 it may be regarded as flame retardant[8].

As cotton being 100% cellulosic material, it easily catches fire and displays a LOI value of 18. After finishing the fabric with mesquite tree gum, the LOI value is found to increase from 18 to 27, which is 1.5 times higher than that of control cotton fabric. The add-on % of the treated sample shows increase from 2 to 8, which results in increase in LOI.

With regard to the vertical flammability test, the control cotton fabric is found to burn completely at high temperature. However, all the treated fabrics display a reduction in burning during the course of the flame test (Fig. 1). A considerable increase in afterglow time is observed from 0s (control) to 147s at *p*H 5, 793s at *p*H 9 and 470s at *p*H 12 (Table 1). Afterglow of the mesquite gum treated fabric at *p*H 12 is found to be completely arrested and shows self-extinguishment (Fig.1). The finished fabric burnt area is found to be grey and continuously produces smoke. The flame time of the treated samples is found to be reduced. Hence, the escape time from the flame

Table 1 — Flammability parameters of control and treated samples of cotton fabric

Flammability parameters	Control sample (A)	Treated samples at different *p*H		
		*p*H 5	*p*H 9	*p*H 12
Add-on, %	-	2	5	8
LOI (O_2 %)	18	22	25	27
Vertical Flammability Test (sample size - 350 × 70 mm)				
Occurrence of flashing over surface	Yes	No	No	No
Burning with flame time, s	60	15	Nil	Nil
Burning with afterglow time after flame stop,s	0 (As completely burnt with flame)	147	793	470
Total burning time (flame time + afterglow time), s	60+0	15+ 147	0+793	0+470
Char length, mm	-	-	-	85
State of fabric in contact of flame	Completely burnt with flame	Completely burnt with after glow	Completely burnt with after glow	Self – extinguishment observed as partly burnt with after glow

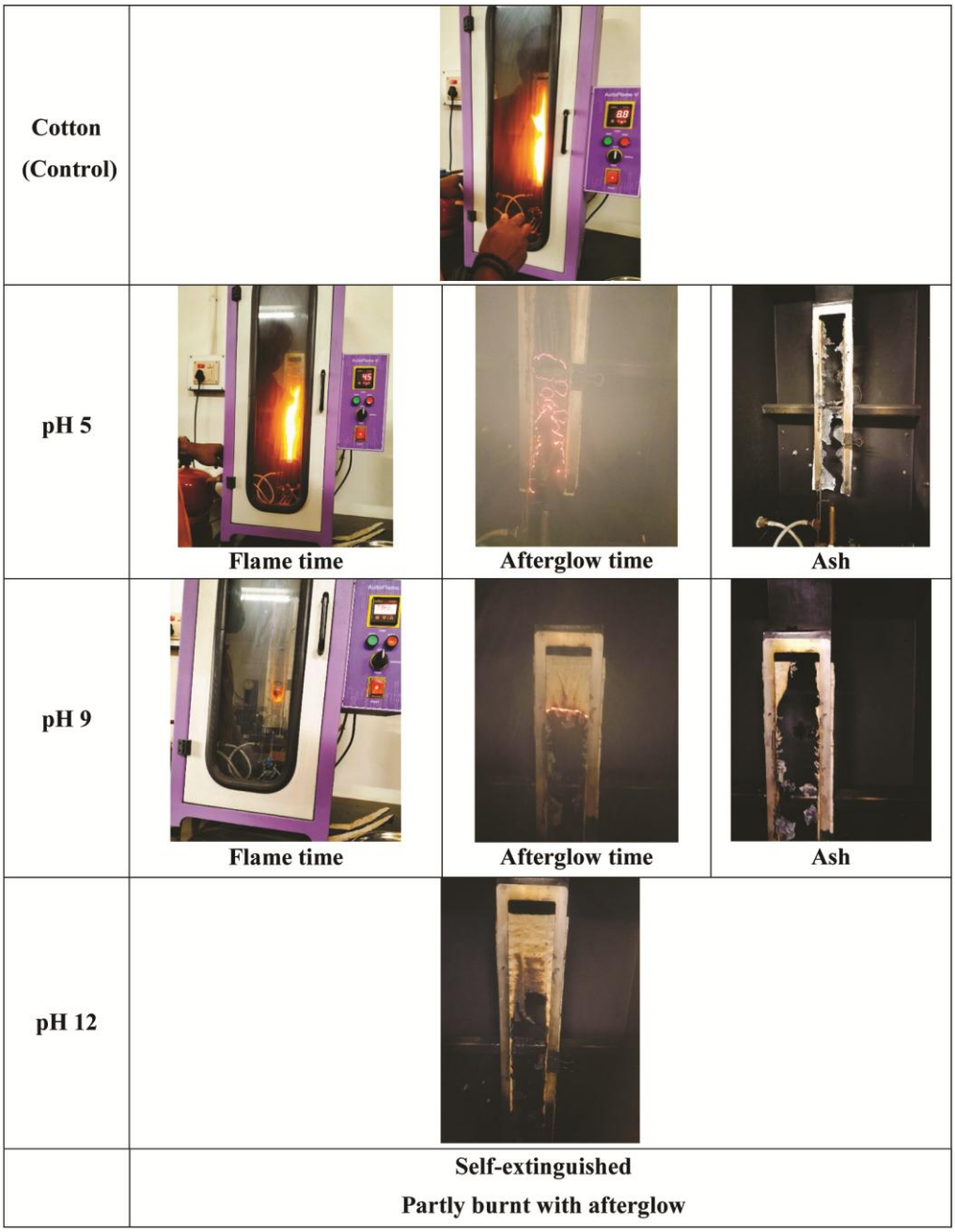

Fig. 1 — Vertical flammability of control and treated sample

will be increased automatically, which will be more beneficial in a real-life situation because it gives the wearer more time to flee a fire or put out a flame[15]. To conclude, the mesquite tree gum finished fabric at *p*H 12, which shows complete self-extinguishment, can be utilized as a flame-retardant fabric.

3.2 FTIR Analysis

The FTIR spectrum of mesquite tree gum is represented in [Fig. 2 (a)]. The tree gum shows stretching vibrations around 600-608cm^{-1}, which represents CH out of plane bending vibration and the nutrient type is clearly an organic material[20]. The small peak is detected at 686cm^{-1}, allied with the aromatic mono substituted ring, very broad band of amides, C-Cl, C-Br, alcohol, CH_2-S- thioether and aryl thioethers. Another peak is observed at 648cm^{-1}, may be associated with alkyne C-H bend and aliphatic bromo compounds C-Br stretch. The 601cm^{-1} may be representing alcohol, OH out of plane bend. Further,

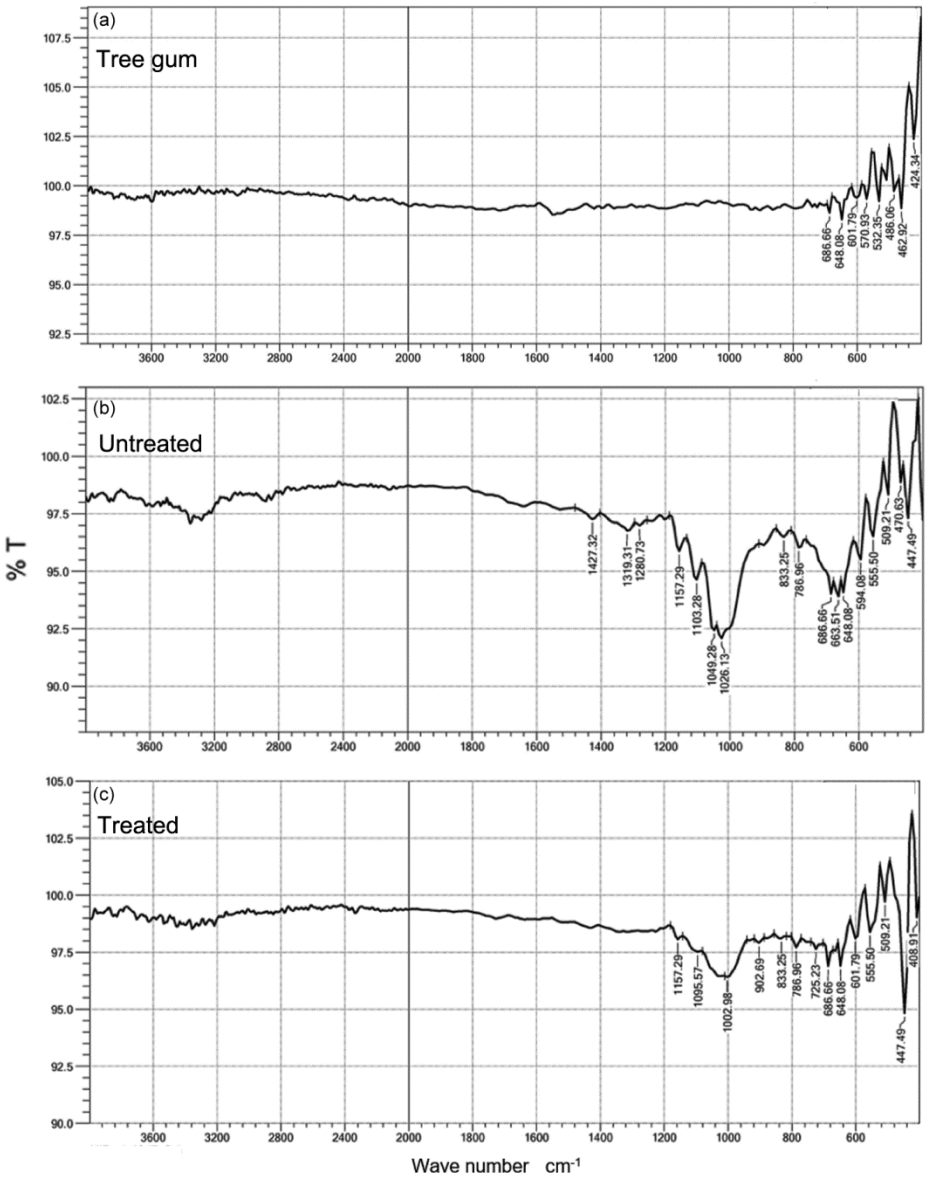

Fig. 2 — FTIR spectra of (a) mesquite tree gum, (b) control cotton fabric and (c) finished fabric

some peaks observed at 570 – 424cm^{-1}, represent aliphatic bromides and iodides and S-S stretch of aryl & poly disulfides. The flame retarding property might be due to the presence of halogens like bromide, iodides which act directly on the core of the fire.

The FTIR spectrum of control cotton and treated sample is represented in [Figs 2 (b) and (c)]. The treated sample shows a peak at 1002cm^{-1}, may be allotted to the C-O single bond stretching vibration of alcohols and cyclohexane ring vibration of methylene >CH_2. Peak seen at 648cm^{-1}, is associated with the aliphatic organohalogen compounds like C-Br, C-I, C-Cl. Another peak detected at 686cm^{-1}, is allied with the aromatic mono substituted ring and very broad band of amides. Small peak observed at 1095cm^{-1} represents the plane bend of aromatic ring, inorganic ions (silicate, phosphate, sulphate). Peak observed at 1157cm^{-1}, may represent the C-O stretching vibration of ethers and esthers, 3-methyl, 2-butanone, amines, strong and broad anhydrides, very strong band of P=O (Phosphine oxides), fluorides (mono fluro alkanes, poly fluro alkanes, aryl fluorides), aromatic C-H stretch and secondary & tertiary amines (CN) stretch. Weak peak examined at 725cm^{-1}, may be associated to C-Cl, medium band of P-O, $(CH_2)_n$ methylene, C-C skeletal vibration, long chain band of CH_2 and C-H

aromatic ring. Another peak at 786cm^{-1}, represents 1,3, - disubstituted rings (meta-aromatic ring) & 1,2, ortho disubstituted ring (secondary strong band aromatic ring), Phosphate esters and (C-Cl) aliphatic chloro compounds. The peak at 833cm^{-1}, may be assigned to strong band 1,4, disubstituted ring (para-aromatic ring) & C-H (Oop), several strong band of S-O, primary & secondary amines, medium band of P-O, and peroxides of C-O-O- stretch. Another small peak detected at 902cm^{-1}, may be allied to C-H aromatic ring, P-H phosphorous compounds, strong band of C-O anhydrides, P-O-C stretch of aromatic phosphate and silicate ion. Further, some small peaks observed from 555 – 408cm^{-1}, represent aliphatic bromides & iodides and S-S stretch of aryl & poly disulfides. However, there are no appreciable differences between the FTIR curves of treated and control samples in terms of peak intensity. The presence of aromatic rings, phosphorous, bromide, metallic salts, oxides in the treated sample surface acts as a barrier of heat.

3.3 Surface Morphology

The SEM images of the control cotton show rough surface without any deposition in [Fig. 3 (a)]. On the contrary, the treated sample shows smoother surface, which indicates that the mesquite tree gum (*p*H 12) has been uniformly distributed over the entire surface [Fig. 3 (b)], The treated fabric shows a thicker surface, as they are tightly secured as compared to control cotton fabric. The tight structure might be due to the presence of the tree gum in the alkaline formulation which maintains a well structural integrity in the treated fabric. The cellulosic structure of cotton fabric might cause combustible gases to wick inside the polymer and it also aid in sustained burning, while the mesquite tree gum treated sample with tightly bonded structure helps to

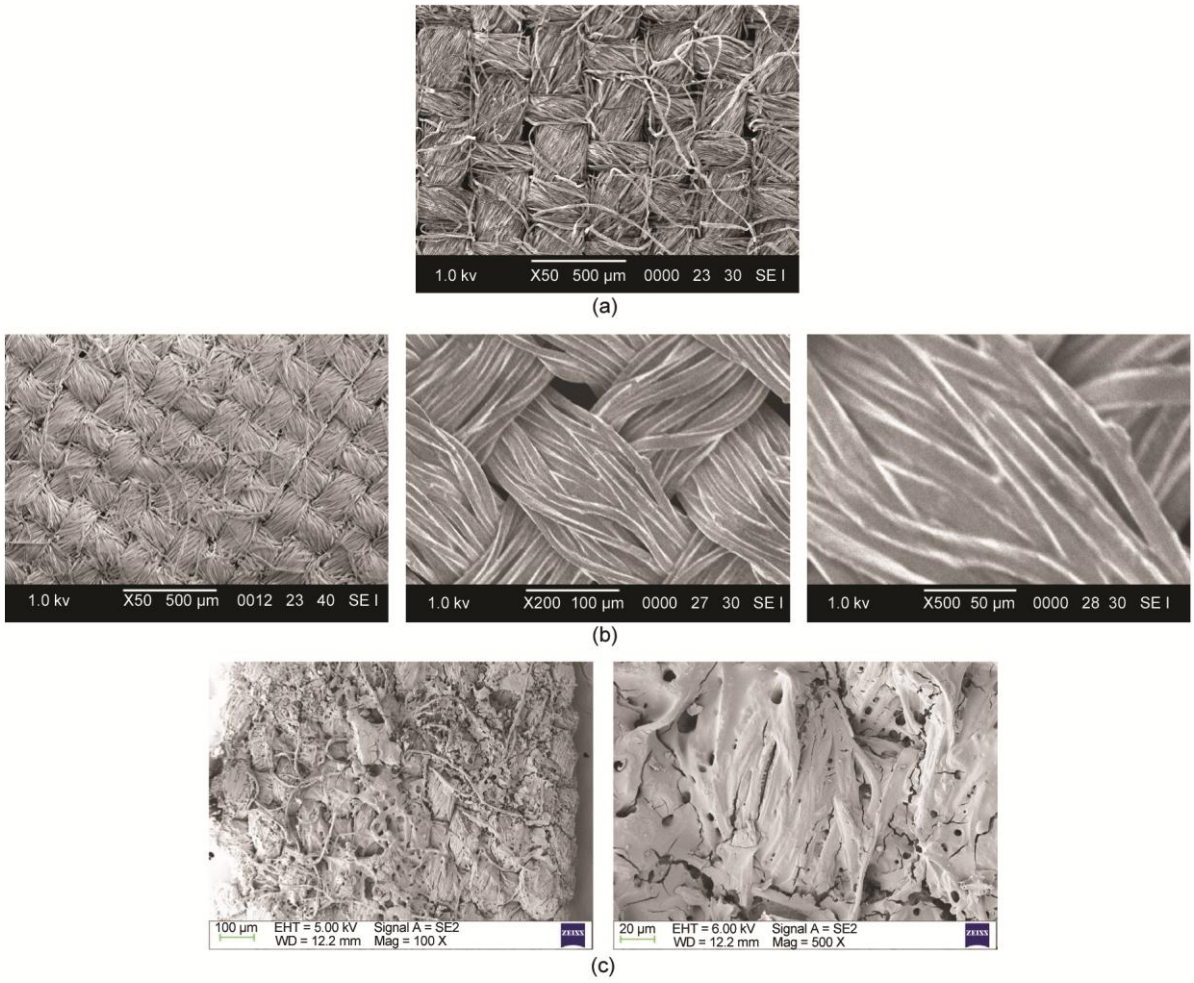

Fig. 3 — (a) SEM image of control sample, (b) SEM images of finished samples at *p*H 12 with different magnification scale, and (c) SEM images showing char surface morphology of finished sample at different magnification scale

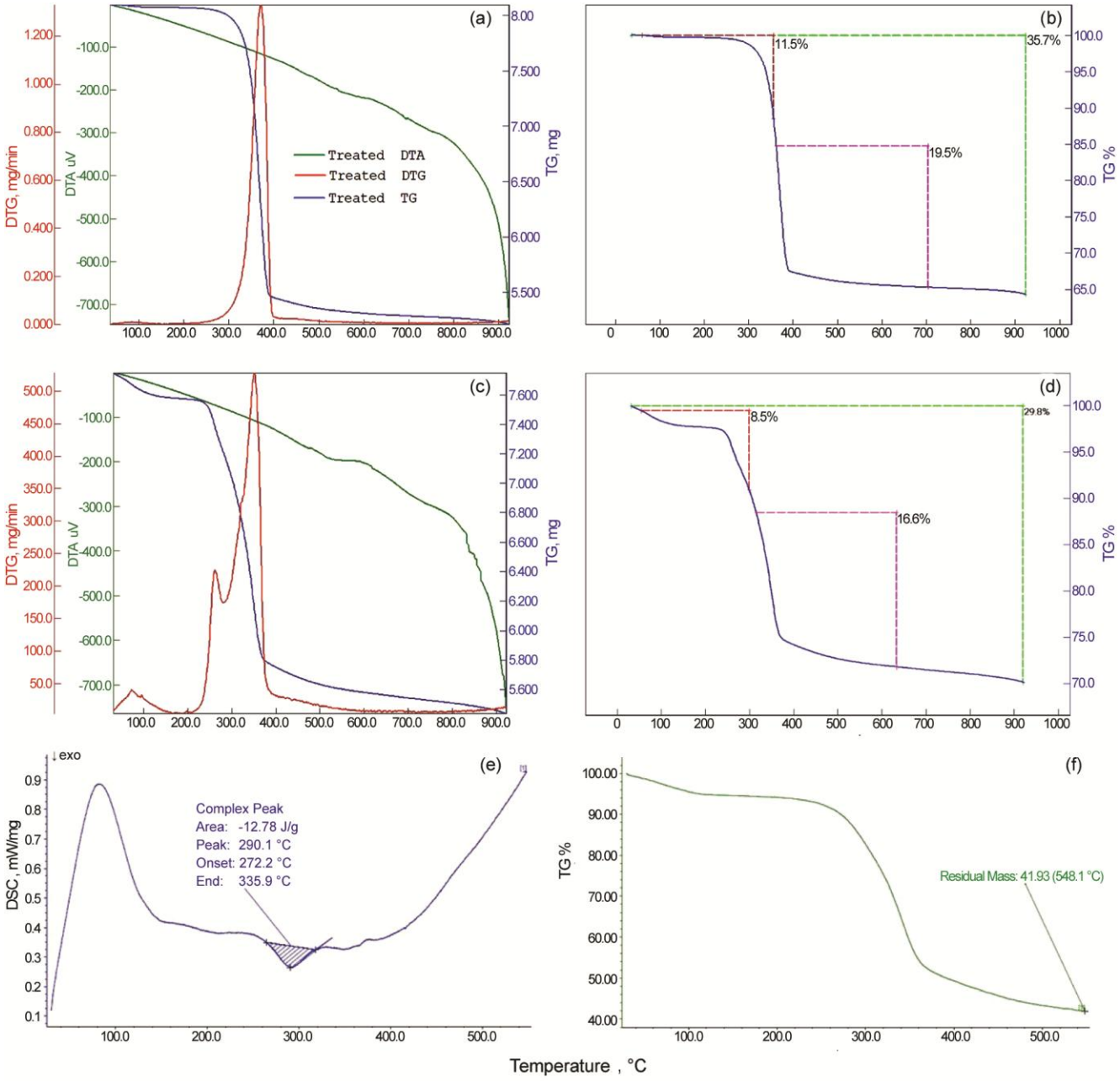

Fig. 4 — (a) TGA & (b) TG % of control sample; (c) TGA & (d) TG % of finished sample; and (e) DSC, and (f) TG % of mesquite tree gum

prevent the continuous burning. During the flammability test, the treated sample forms char, while the control cotton fabric forms ashes while burning. A very significant factor in determining a flame retardant's effectiveness could be the char's surface shape[21]. From the char surface morphology of treated sample [Fig. 3 (c)] it is clear that the fibre integrity is maintained, which might be due to the presence of mesquite tree gum, which prevents the fibre from damage.

3.4 TGA Analysis

The TG curves of the control cotton fabric and mesquite gum finished fabric are presented in [Figs 4 (a) & (b) and (c) & (d)]. The initial decomposition temperature (IDT) has occurred at 250 ^{o}C with 8.5 % of mass loss, which may be attributed to the removal of bound and unbound absorbed moisture of the cellulosic polymer. Second major weight loss with a sharp fall at 360^{o}C temperature range with 16.6% mass loss indicates the depolymerization of cellulose.

The last stage of decomposition has occurred at 920^{o} Cwith 29.8% mass loss. The TG curves of the control cotton sample shows the initial stage at temperature below 100^{o} C, which may be attributed with the loss of moisture content of the pure cotton[19]. The main decomposition has occurred at 300^{o}C with a mass loss of 11.5%. The second weight loss is observed at 400 ^{o} C with the mass loss of 19.5%. The final stage of decomposition happens at 910 ^{o} C, which shows a drastic weight loss of 35.7%.

At the initial stage, the mesquite tree gum finished fabric [Figs 4 (c) & (d)] shows only 8.5% weight loss, whereas, the control cotton fabric shows 11.5% weight loss. In the second stage, the weight loss is 16.6% in the finished fabric as compared to 19.5% in control cotton fabric. The TG curves also show that the weight loss in the final stage is found to be 29.8% in the treated fabric and 35.7 % in the control cotton fabric. It can be detected that the mesquite tree gum finished fabric minimises mass loss across the temperature range. Hence, more masses can be retained by the mesquite tree gum finished fabric at higher temperature. The results, clearly indicate that the thermal stability is increased in the mesquite tree gum finished fabric. The TG curves of finished and control fabric samples reveal that the weight loss of control sample increases with increase in temperature than the treated fabric sample, which shows a lower rate of decomposition with increase in temperature. Hence, it can be concluded that the fabric finished with mesquite tree gum improves the treated materials overall resistance and significantly lowers the rate of cellulosic mass loss.

The analysis of the mesquite gum shows a complex peak at 290^{o}C – 335^{o}C, where the mass decomposition takes place. The TGA curves show [Figs 4 (e) & (f)] that the overall mass loss on the mesquite gum is 41 % at 548^{o}C. Whereas, the mesquite gum treated cotton sample shows a great thermal stability by minimising the mass loss across the temperature range.

3.5 Fastness Properties of Treated Sample

The wash durability of the treated sample is studied to determine the effectiveness of the flame-retardant finish after going through one wash cycle. After washing the sample, the LOI has been tested and the LOI value of the washed sample is decreased from 27 to 23. However, it is 1.2 times higher than that of control cotton sample.

The rub fastness of the treated sample has been measured using Crockmeter for 30 rotations and the LOI is tested. The LOI value is found to be 25%. According to the findings, the mesquite gum treated fabric can be used to create home textiles that do not need to be frequently washed.

4 Conclusion

For the first-time, mesquite tree gum was explored as flame retardant on the cellulosic cotton fabric. Limiting oxygen index of the cotton fabric increases from 18 to 27 after the application of mesquite tree gum. when exposed to a 38mm flame for 12s, the treated cotton fabric shows self-extinguishing behaviour. The flame retardancy of the mesquite gum may be attributed to the presence of phosphorous, aromatic rings, bromide, metallic salts and oxides, which act as a heat barrier. The TG curves show that the fabric treated with mesquite tree gum has a high degree of thermal stability. As no chemicals are used, this proposed application process is cost-effective and simple. Further, it can be advantageous to add a flame-retardant finish to household furnishings including blinds, window treatments, kitchen linens, etc. Mesquite tree gum is eco-friendly and abundantly available. Therefore, the application of mesquite gum on cellulosic cotton fabric has strong potential as sustainable flame retardant and will highlight the benefits of employing natural products to enhance value.

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