Enhancing the Flame Retardancy of Cotton Fabric Using Sustainable Tree Gum

By

GAYATHRI J.J

(20PTF008)

A thesis submitted to

Avinashilingam institute for home science and higher education for women

Coimbatore-641043

In partial fulfilment of the requirement for the

Degree of master of science

In

Textiles and fashion apparel

May, 2022

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Certified as Bonafide Research work

Signature of the Head of the Department **Signature of the Supervisor**

DECLARATION

I declare that the dissertation entitled **"Enhancing the Flame Retardancy of Cotton Fabric Using Sustainable Tree Gum"** submitted by me for the degree of Master of science (M.Sc.,) is the record of work carried out by me during the period from 2021 to 2022 under the guidance of **Dr. (Tmt.) K. Kalaiarasi,** M.Sc., M.Phil., Ph.D. (Avinashilingam), Associate Professor, Department of Textiles and Clothing, Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore-642 043 and has not formed the basis for the award of any Degree, Diploma, Associate ship, Fellowship, Titles in this University or any other similar institution of higher learning.

Signature of the Candidate

CERTIFICATE FROM THE SUPERVISOR

I certify that dissertation entitled **"Enhancing the Flame Retardancy of Cotton Fabric Using Sustainable Tree Gum"** submitted for the degree of Master of science (M.Sc.,) Textiles and Fashion Apparel by Gayathri J.J. is the record of project work carried out by her during the academic year 2021 to 2022 under my guidance and supervision and this work has not formed the basis for the award of any Degree, Diploma, Associate ship, Fellowship, Titles in this University or any other similar institution of higher learning.

27/5/22

Signature of the Supervisor with Designation

27/5/22

Signature of the HOD

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INTRODUCTION

1. INTRODUCTION

The flame resistance has always been a matter of interest since the ancient Egyptians (Hindersinn,1990). 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. Additionally, 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 (Bajaj,1984).

The characteristics of flame retardant clothing include high degree of insulation, slow flame speed, abrasion resistance, strength, higher dimensional stability, low heat generation, and it should not melt or form chars under flame (Sengupta,1992). A variety of factors influence the flammability behaviour of fabrics such as cloth cover, porosity, mass/unit, finishing effects like napped or lofty construction and surface properties (Tesoro,1970 & Sello,1969). Even the choice of fibre/fabric has a fundamental effect on the flammability considering the parameters like production of char/ molten dripping, ease of ignition, toxic gases, production of smoke, heat of combustion of the material, rate of flame speed, extent of damage to adjacent skin and afterglow (John,1989).

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. In addition to that, cotton is being used in the field of technical textiles as it is eco-friendly and biodegradable (Dutta,2008).

Cotton is a versatile starting material for a new product development as it is abundant, more appealing to consumers and low in cost. Since the cellulosic material is highly flammable, (Horrocks,1986) the flame retardant cotton textiles have received significant attention from both academia and industry (Alongi2015; Wang,2016). However, the untreated cotton cannot withstand the flammability and difficult to extinguish leads to severe health hazards including damage to textile products (Petrilli,2008).

Remarkable efforts are been made to improve the flame retardancy of cotton. Among the inorganic salts, urea, phosphate, di-ammonium, (Charuchinda,2005) borax in addition to that boric acid are the simple and non-durable flame retardant chemicals (Kandola,2006). And

‘Karvin’ is used to make cheap wear clothing’s, it consists of 30% Nomex, 5% Kevlar and 65% FR-viscose. Apart from Karvin many fire-retardant fibres like Basofil, Teijin-Conex, Kermel, TP11, Kynol, Lenzing’s P84 and Philene were developed to meet its requirements (Peter,1997 & anon,1989). 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, furanes, the toxic treatment is banned in European countries (Horrocks,2011).

However, when cotton is treated with inorganic substances, it reduces the tensile strength and the processing using chemicals are time consuming. The chemically developed substances are highly harmful to the environment and to all living organisms. There is a high need to develop sustainable eco-friendly flame retardant so that the quality of the cellulosic material is also maintained. Due to the high expenses of chemical substances the organic bio-based processing is drawing attention in the research and development. The research and development team have reported about different bio-macromolecules like PRE, banana pseudo stem sap, DNA, whey protein, spinach leaves etc, (Basak,2016; Samanta,2015; Carletto,2013; Bosco,2013).

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. Despite that, the application of tree gum resin as flame retardant finish for textile materials or polymeric materials has not been disclosed to the best of our knowledge.

In this study the mesquite tree gum which is also known as *Neotea Prosopis Juliflora* is used to improve the flame retardancy of the cellulosic material. The mesquite gum also contains polyphenol (tannins), Polyphenols are another naturally occurring class of compounds with good char-forming properties. Tannins, in particular, have a high char-forming capacity and can be employed as a flame retardant (Tributsch,2008).

It is also highly composed of polysaccharide which makes it as best alternative for synthetic flame retardants. The functional properties of the mesquite gum include intrinsic viscosity, water solubility, encapsulating capacity, emulsifying ability, surface activity etc. these unique properties make it as a functional hydrocolloid (Vaile,2016). It has been investigated that the glass transition and moisture sorption properties also suggest its functional behaviour.

Considering the above facts, the investigator selected the research work on the topic "**Enhancing the Flame Retardancy of Cotton Fabric Using Sustainable Tree Gum**" With the following objectives;

- To select a natural source for flame retardant finish on cellulosic fabric
- To finish the selected fabric with selected flame retardant
- To evaluate the finished fabric for flame retardancy

REVIEW OF LITERATURE

2. REVIEW OF LITERATURE

The review of literature pertaining to the study on **"Enhancing the Flame Retardancy of Cotton Fabric Using Sustainable Tree Gum"** is discussed on the following headings;

2.1 Flame retardant finishing

2.1.1 Introduction

2.1.2 Need

2.1.3 Advantages

2.1.4 Types of chemical flame retardants and its side effects

2.1.5 Toxicity of chemical flame retardant

2.1.6 Application of flame retardants

2.2 Importance of natural flame retardant

2.3 *Neotea Prosopis Juliflora*

2.3.1 Taxonomic classification

2.3.2 Chemical constituents

2.3.3 Properties

2.4 Cotton

2.5 Recent studies in flame retardant materials

2.1 FLAME RETARDANT FINISHING

2.1.1 Introduction

The flame retardants are the treated materials which can minimize the risk of ignition and it is also a critical element of fire safety. Due to the fact that the word "flame retardant" refers to a function rather than a chemical class, a wide range of compounds are utilised for this purpose. The flame retardants can be any chemicals or organic substances which are applied to materials to prevent or to slow down the fire and it is a signature of performance of a polymer.

When flame retardants are present, escape periods are expected to be up to 15 times longer, increasing the chances of survival (NIST,1988). Flame retardants acquired popularity in the 1970s, and they were being used on flammable materials to stop the combustion cycle by influencing chemical or physical processes in one or both of the gas and condensed phases (Stapleton & Cooper,2016). Flame retardants work by interfering with the combustion cycle in the solid, liquid, or gas phase during specific stages of combustion through chemical and/or physical means (Bourbigot,2007). During specific stages of the fire process, such as heating, decomposition, ignition, or flame propagation, flame retardants should block or suppress the combustion process. Flame retardant modes of action are classified according to whether the mechanism is physical or chemical, as well as the phase in which these mechanisms occur (that is condensed or gas phase) (Suoware,2017; Wang,2015; Zenet,2011; Malvar,2001; Kozlowski,2008). All bio-based flame retardant methods work by inhibiting flame by physical action, primarily through the charring effect (Li,2001; Fouad,2018; Jimenez,2016).

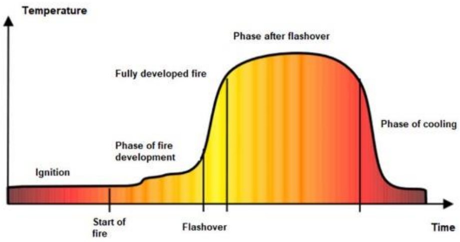


Plate 1 - The basic phases of fire

2.1.2 Need for the flame retardants

It is reported that someone is dyeing every three hours in fire accident and some are being injured every 37minutes. Among those accidents, 401,000 accidents are house fires, and the residential fire accidents can cause property damage. Because of the expansion of electrical and electronic equipment, as well as increased levels of comfort (furniture, carpets, toys, magazines and papers), both our homes and offices include an increasing potential "fire load" of combustible elements. The people most at risk are the very young and the elderly because they are least able to escape in the event of a fire. It is estimated that the escape times can be increased 15times longer when the flame retardants are present, providing high chance of survival.

The flame retardant textile material benefits the wearer with a critical layer of flame protection and this can prevent from fire hazards and it can prevent modern materials from igniting and spreading fires, such as technical plastics, building insulation, circuit boards, and wires. If a fire starts in one room of a house and spreads to items other than those that were initially sparked, it can spread quickly. When a number of items begin to burn, the temperature in the area rises to the point that "flash over" occurs, the hot burning gases cause the entire room to catch light, typically violently. When this happens, it's hard to get out of the room, and the fire will very certainly spread to nearby rooms. Flame retardants work by delaying the spread of a fire and preventing "flash over" by the initial commencement of a fire by limiting ignition (EFRA, 2009). The need for enhanced flame retardancy performance has prompted researchers and industry to improve existing flame retardant agents, replace existing compounds when safety is a problem, or use known chemistry in creative ways (Horrocks,2005).

In a fire situation, the permissible or extra time is mostly determined by the expected temperature growth of the fire, which is determined by the type and number of flammable materials present, as well as the ventilation condition (Weil,2011; Bourbigot,2007; Mariappan,2016). Although infinite fire protection by the application of coatings is unachievable, it can delay the spread of fire or preserve a structure intact against fire for a period of time, providing time for safety precautions to be performed.

2.1.3 Advantages of flame retardants

Flame retardant textiles are now required in the manufacture of uniforms for firefighters and emergency personnel to protect themselves from flame while doing their responsibilities (Schindler, 2004). Other applications for flame retardant finishes include high-performance sports, home furnishings, office/commercial infrastructure and transportation, and sleepwear for youngsters and the elderly (Gaan, 2011).

Textiles are an integral aspect of the interior design of homes, businesses, and cultural and social institutions. The majority of commercially available fabrics used in the above-mentioned facilities are extremely flammable and combustible. For many years, significant losses of life and property have been a concern owing to unwelcome fire hazards. As a result, systematic improvements in terms of textile quality, performance, and safety have become increasingly important in recent years (Vladimirtseva, 2016).

Most people are unaware that their television, sofa, mattress, and computer are all made primarily of plastics (which were originally made from crude oil), and that without flame retardants, many of these products can be set alight by a simple short circuit or cigarette and turn into a burning mass in minutes. By disrupting or slowing the combustion process, flame retardants can be added to a wide range of flammable materials to prevent or delay the initiation and spread of a fire. As a result, people, property, and the environment are all safeguarded. Flame retardants help combustible materials and final goods meet stringent fire safety requirements set forth by legislation and tests. Although non-combustible materials or design and engineering approaches can be used in some circumstances to achieve fire safety, flame retarded materials often meet the functionality and aesthetic criteria of the consumer while also being the most cost-effective option.

2.1.4 Types of chemical flame retardants and its side effects

Among hundreds of different flame retardants, Chemical structure and characteristics are frequently used to categorise them. Flame retardants are classified according to their content of bromine, chlorine, phosphorus, nitrogen, metals, or boron (Gaan, 2011).

1. **Bromine/ Chlorine Flame Retardant**

   The bromine and chlorine come under halogenated flame retardants. The 50% 0f BFRs applications are mostly used in electrical and electronical equipment's to prevent the growth of fire. The bromine/ chlorine flame retardants include many side effects like disruption of the endocrine system (Gosavi, 2013).

   Polybrominated diphenyl ethers (PBDE's) – In 1970s the PBDE was used in industries to meet flammability standards to consumer products. It has been found that impair of neurological development, lower birth weight and length of children is caused by the PBDEs (Eskenazi, 2013**).** Several PBDEs have been demonstrated to cause cancer in animal experiments conducted by NTP. PBDEs have been outlawed or phased out of manufacture, yet they are still present in the environment persist in the face of adversity (NTP. 2016 & NTP. 2015).

   Tetrabromobisphenol-A (TBBPA) – It is one of the brominated flame retardants it has been used as a raw material for printed circuit boards and also used as plastic casing in electrical/electronical compounds. It is recorded that the TBBPA is the most produced BFRs (Knudsen, 2015). It's been detected in human tissue and household dust, as well as soil, water, and fish, among other places in the environment. TBBPA appears to alter the endocrine system (Hamers, 2006). NTP undertook the first-ever two-year cancer study of this flame retardant in mice and rats, discovering that it causes uterine cancer in female rats and liver cancer in male mice (NTP. 2014). The goal of NTP research is to find compounds that could cause cancer in people. TBBPA exposure in the early years of life is also being studied by NIEHS in-house researchers.

Hexabromocyclododecane (HBCD) - This flame retardant is generally found in polystyrene foam construction products. Humans are most at risk from leaching from items and inhaling indoor dust. Some food products have been reported to have low quantities of HBCD (Schecter, 2012). It has been proven to affect the brain, immunological, and reproductive systems in animals, as well as causing endocrine disruption.

2. Organophosphate flame retardants (OPFRs)

Phosphorus-containing flame retardants are widely used in standard and engineering plastics, polyurethane foams, thermosets, coatings, and textiles. Phosphate ester Phosphonates and phosphinates, red phosphorus and ammonium polyphosphate comes under phosphorus flame retardants. Some OPFRs have been discovered as potential replacements for PBDEs, which are being phased out. NTP is now developing a programme to assess and contrast the activity of these replacement halogenated and non-halogenated OPFRs with that of the phased-out PBDEs, as well as to gather data on possible dangers. NTP has undertaken in vitro and alternate animal model screening tests.

According to the findings, several of the replacement OPFRs had activity similar to that of the phased-out PBDEs (Behl, 2015). Because of structural similarities with organophosphorus insecticides, which are known to be neurotoxic, NTP is now performing developmental neurotoxicity studies in vivo on some typical OPFRs.

3. Other Flame Retardants

Other forms of flame retardants can still be found around the world. The chlorinated organophosphate tris(1,3-dichloro-2-propyl) phosphate (TDCPP), for example, has been related to cancer in rats (National Research Council, 2000) and has been found in people (Meeker, 2013) as well as dust from houses, offices, and automobiles (Carignan, 2013).

Researchers financed by the NIEHS are also investigating the health impacts of newer flame retardant options on the market. Some scientists, for example, are investigating the Fire master 550. This product was utilised as a replacement for the phased-out pentaBDE. The researchers discovered that several of the ingredients in this product had endocrine and metabolic effects in rats, which could lead to obesity and an earlier puberty start. NIH experts are continuing their investigation into these newer commercial mixes to see how they are metabolised in the body and if they have any negative health impacts.

2.1.5 Toxicity of chemical flame retardants

The potential hazardous effects of flame retardants are modest because they are either chemically reacted into the material they are used to treat or physically contained within it in most applications, making them unable to have major external effects. Furthermore, when compared to other regularly used compounds, flame retardants are not very harmful. There are many sorts and degrees of interactions with live creatures since there are so many different chemical families of flame retardants. Because minor changes to a molecule can have tremendous impacts depending on the level of molecular interaction with cells, there can be large variances in hazardous effects even within a chemical group.

Many flame retardants are exempt from having to be labelled as harmful compounds, indicating that they have a minimal toxicity or environmental impact. Others have a toxicity as a clean chemical, but the impact is lost when they react into a polymer. The principal flame retardants have been in use for many years, and hazardous effects would have manifested themselves in the workplace during production or processing, as this is where the most exposure occurs. Because of suspected or proved harmful qualities, only a few flame retardants have been pulled from the market, phased out, or controlled, frequently in conjunction with very large margins of safety and the precautionary principle.

Although flame retardants can provide benefits when used in certain products, but a growing body of evidence suggests that many of these chemicals are linked to negative health effects in animals and humans, including endocrine and thyroid disruption, immune system effects, cancer, reproductive toxicity, and negative effects on foetal and child development and neurologic function (Shaw,2010).

Children's exposure to flame retardants is increased by and-to-mouth behaviour and proximity to the floor. Children have higher levels of flame retardants in their systems than adults, according to studies. Because their brains and other organs are still developing, children may be more sensitive to the damaging effects of these toxins (Butt,2014).

2.1.6 Applications of Flame retardants

Textiles treated with flame retardants have a wide range of applications. Apparel, home, offices, transit, public buildings, uniforms, high-performance technical textiles, and other fields use flame-retardant textiles. For example, flame retardants are commonly used in sleepwear, upholstery (curtains, carpets), bedlinen, blankets, and protective apparel for firemen and industry personnel (Salimova, 2011, Neisius, 2014).

Industrial workwear, firefighter costumes, air force pilot uniforms, tent and parachute fabric, professional motor racing equipment, and other uses use fire resistant materials to protect the wearer from flames and electrical arcs. In hotels, hospitals, and theatres, they are typically employed in interior materials as fire retardant curtains.

Twaron is a material used in fabrics to withstand high temperatures in industries such as firefighting. Aluminum hydroxide, for example, is often used as a fire retardant because it provides three-way protection. It decomposes to release water vapour and absorbs a lot of heat, cooling the substance and the alumina residue and forming a protective layer. The flame retardancy of a fabric is determined by the number of times it has been used; the more times it has been used, the better (Fiber2Fashion).

The following are some examples of applications for flame-retardant finished textiles:

- Sleepwear for babies, children, and the elderly (Blum, 1978, Blum, 1977, Paek, 1975)
- Upholstery fabrics and home textiles such as curtains, carpets, bedcovers, and blankets (Stubbings,2014, Davis,1992, Babich,2006, Nazare,2012).

- Public transportation fabrics and fibre components, such as flame-resistant seats and seat covers for buses and planes (Flambard,2005), Uddin,2016).
- Flame-retardant coated architectural textiles made of PVC (Liang,2013).
- Polyamide fibres that have been chemically treated are used in sports fabrics (Strgmaier,2005).

2.2 Importance of natural flame retardant

In reality, synthetic fire retardants have received a lot of attention in recent years. Oil-derived organic compounds make up a large portion of commercially available flame retardants (e.g., organo-halogenated, organo-phosphorous, organo-nitrogen compounds). They confront the same concerns as other oil-based products: increasing petroleum scarcity, geopolitical issues, and the influence on global warming. Furthermore, several of these compounds (especially halogenated compounds) have gotten a poor rap because they're thought to create specific health and environmental problems (Tao,2016; Law,2014; Birnbaum,2004).

In order to present more environmentally friendly materials, it is required to advance the application of Green Chemistry principles (Anastas,1998) and therefore, encourage the development of bio-based polymer additives. As mentioned above there are huge effects of chemical flame retardants which are toxic to all the living organisms. Scientists throughout the world are increasingly concerned that brominated and chlorinated flame retardants are detrimental to human health and the environment, and that they should be phased out (DiGangi,2011).

However, in the last few years, the scientific community has published a large number of publications devoted to the creation of flame retardants derived from renewable resources. The ban on some halogenated compounds, as well as the hunt for alternative options, has fuelled the development of new bio-based additives (Ezechias,2014).

The optimal flame-retardant finish should not only provide better performance, but also be simple to apply, affordable in cost, and environmentally friendly. In the flame-retardant community, recent advancements in halogen-free, multifunctional, synergistic flame retardants, and nanotechnology have piqued interest. However, the environmental and health implications of these approaches have yet to be fully investigated (Neisius,2014).

2.3 *Neotea Prosopis Juliflora*

Mesquite gum is a type of exudate gum produced by Mesquite trees (Prosopis sp). Mesquite trees are members of the leguminous family and can be found in dry and semi-arid climates around the world. The genus Prosopis contains 44 species that are mostly found in North and South America, North Africa, and East Asia. Prosopis Juliflora and Prosopis laevigata are the main producers of this gum (Vermon-Carter,2000). Similar to other exudate gums, mesquite gum nodules can be classified into distinct classes based on colour, size, and impurities (bark content, foreign material) (Barak,2020). Because of the presence of tannins, its hue changes, limiting its usage in specific applications. Ultra-filtering procedures can be used to diminish the dark colour of mesquite gum nodules by reducing tannin content (Goycoolea,1998).

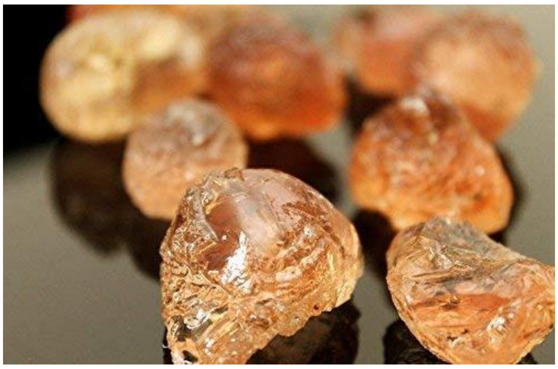



Plate -2 – Mesquite gum & tree

2.3.1 Taxonomic classification of *Neotea Prosopis Juliflora*

Table – I

Domain	Eukaryota
Kingdom	Plantae
Phylum	Spermatophyta
Sub-phylum	Angiospermae
Class	Dicotyledonae
Family	Fabaceae
Sub-family	Caesalpinioideae
Genus	Prosopis
Species	P. Juliflora

2.3.2 Chemical constituents

Mesquite gum has a slightly complicated chemical structure. It's a salt of a complicated acidic branching polymer that's neutral. It is made up of a backbone chain of (1-3)-linked -D-Galactose units and (1-6)-linked 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 (Aspinall,1970).

2.3.3 Properties

Most important properties of mesquite gum with reference to its functional applications are its solubility, intrinsic viscosity, optical rotation, emulsion stabilization interfacial tension, encapsulation capacity and emulsification ability (Vasile,2016).

Emulsifying properties

Mesquite gum is a very effective emulsifying agent because of its protective colloid functionality and its ability to form visible viscoelastic films at the oil-water interface (Molina,2003; Guerrero,2009; Aguliar,2011, Vasile,2017).

Intrinsic viscosity

Mesquite gum solution at 5% concentration in 0.1 M NaCl at 30°C has an inherent viscosity of 10.9 mLg-1, which is significantly lower than the intrinsic viscosity of gum arabic solution (21.18 mLg-1) under same concentration, temperature, and other conditions (Franco,2013).

Optical Rotation

Specific optical rotation is a property of chiral compounds that is quantified as the angle at which polarised light is rotated by the molecules under specific temperature, concentration, and wavelength circumstances. Mixed samples of mesquite gum (10 percent gum solution at 20°C) show specific optical rotation values ranging from +61.86 to +75.46, depending on nodule size and contaminants (Villafuerte,2003)

Solubility

Gums solubility is an important physical property. The tendency of a solute to dissolve in a solvent is referred to as solubility. It determines the gums' acceptability and end usage, as different uses necessitate solubility in certain solvents. Gums insolubility in various solvents can also be employed to isolate the gum. Mesquite gum is extremely soluble in aqueous solution, with a solubility of up to 50% in aqueous media (Goycoolea,1995).

Interfacial Tension

Interfacial tension is a property that can be characterised as the force that attracts molecules together at the interface of two liquids, or as the proclivity of fluid surfaces to have the smallest surface area. Interfacial tension of mesquite gum is influenced by the concentration of gum and the pH of the solution. For oil absorption, mesquite gum had higher activation energy values (46.16 kJ mol-1) (Beristain, 1996).

Encapsulation Capacity

It has been observed that mesquite gum can be employed as an encapsulating agent for orange peel oil encapsulation with good encapsulation efficiency (Barca,1997). It has been observed that a 40:60 mixture of mesquite gum and arabic gum can encapsulate orange peel oil with similar encapsulation effectiveness as pure arabic gum alone (Beristain,1995).

Emulsion Stabilization

Mesquite gum can be used as an emulsifier because it not only aids in the creation of emulsions but also gives them stability (oil-in-water). Mesquite gum is a better emulsion stabiliser than arabic gum because it provides higher stability and smaller oil-droplet mean sizes (Carrillo,2006).

Encapsulation

Food colours and flavours have been encapsulated (by spray drying) using mesquite gum as an arabic gum replacement. Mesquite gum has been studied for its potential to encapsulate essential oil of orange using the spray drying procedure alone and in combination with other encapsulating agents (Balderrama,1997, Beristain,1995). Mesquite gum has an encapsulation effectiveness of greater than 80%.

2.4 Cotton

The word Cotton was derived from an arabic word 'quotn' (Lee,2015), which belongs to Gossypium genus. Cotton is said to be the king of fibre which was utilized from four to seven years ago (Fang,2015). Cotton is the third most planted biotech crop in the world and also ranked seventh in the world cultivated area (James,2012). It is one of the fibres with demanding and advancements for the past century. The researches have initiated and performed research in every aspect of cotton sciences. These efforts have significantly advanced cotton research worldwide and aided in the resolution of important cotton production and farming concerns (Abdurakhmonov,2016).

2.5 Recent studies in flame retardant materials

Textiles play a vital role in daily life, yet they have the disadvantage of posing a risk of fire due to their flammability. The majority of house fires are caused by beds, mattresses, and clothing. Textile flame retardants, or additives that effectively suppress/delay the flame spread rate or limit the rate of combustion of textiles, have been developed as a result of their flammability. The global textile flame retardants market was estimated at USD 504.6 million in 2019, with a compound annual growth rate (CAGR) of 3.9 percent expected from 2020 to 2027. Increased demand for fire retardant fabrics from various end-use industries, such as defence, transportation, and industrial manufacturing, is driving the market. Consumer knowledge of the importance of having enough time to evacuate during a fire, as well as the effectiveness and benefits of flame retardants, is projected to boost product demand.

According to the global textile flame retardants market share the polyester application had the highest revenue share of 39.5 percent, and this is expected to continue throughout the forecast period. Polyester can be used to complete flame retardants on textile fibres since it does not easily burn. When the fabric is ignited, however, it melts and can cause severe burns. Furthermore, polyester's inherent non-flammability, combined with its widespread availability, is projected to enhance product demand in this market.

Cotton is predicted to be the market's second-largest application. Cotton is a popular fabric in domestic textiles because of its physical characteristics such as absorbency, breathability, and great softness. Cotton textiles, on the other hand, are more flammable than their synthetic counterparts. As a result, flame retardants must be applied to cotton fabrics, creating prospective growth prospects for the total market (Textile flame retardant market report.2020).

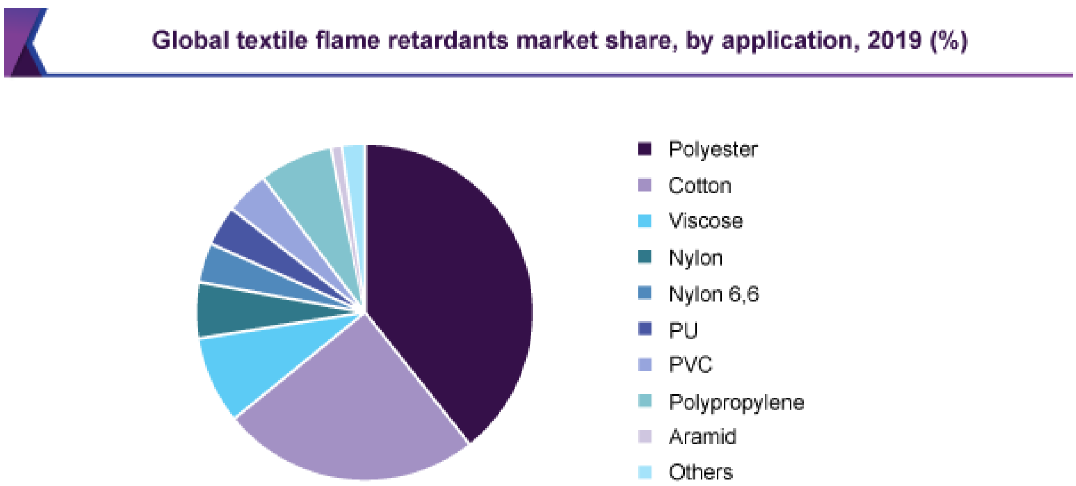


Plate - 3 - Global textile flame retardant market share

Back coating technique, which uses a variety of sources in a coating formulation depending on the type of the polymer, had the highest revenue share of 65.6 percent in 2019. Back coating is one of the most effective collective procedures for textile flame retardancy, and it is especially useful for upholstery materials. Furthermore, the segment is likely to be driven by the fast use of the back-coating technique due to its value addition to textile fibre over the forecast period.

The production of flame retardant textiles is mainly reliant on the ample availability and low cost of basic materials. Successful commercialization of ground-breaking products like metal-based flame retardants, as well as investments in production capacity, are some of the market's main factors. Companies are attempting to create ecologically friendly and sustainable products by adhering to guidelines such as WEEE, REACH, and eco-labels. As the market is leaning towards the development of chemical-free alternatives; as a result, wool and organic fabrics with built-in flame retardant capabilities have gained substantial traction, a feature that is anticipated to limit market growth in the near future. With growing application scope of such fibers and rising preference for products with fire resisting properties is expected to drive the market (Textile flame retardant market report,2020).

METHODOLOGY

3. EXPERIMENTAL PROCEDURE

The experimental procedure for the present study "**Enhancing the Flame Retardancy of Cotton Fabric Using Sustainable Tree Gum**" was carried out in the following aspects.

3.1 Selection of Fabric

3.1.1 Preparation of the fabric

3.2 Selection of flame retardant Source

3.2.1 Pre-treatment of the selected source

3.3 Finishing of cotton fabric with the selected source

3.3.1 Dip and Dry method

3.4 Fabric Evaluation

3.4.1 Subjective evaluation

3.4.1.1 Visual assessment

3.4.2 Objective evaluation

3.4.2.1 Assessment of physical property

3.4.2.1.1 Determination of add-on%

3.4.2.1.2 Fabric thickness

3.4.2.1.3 Fabric weight

3.4.2.1.4 Crease recovery

3.4.2.2 Absorbency test

3.4.2.2.1 Drop test

3.4.2.2.2 Sinking test

3.4.2.2.3 Wicking test

3.4.3 Flammability assessment

3.4.3.1 Limiting Oxygen Index (LOI)

3.4.3.2 Vertical flammability

3.4.4 SEM appearance

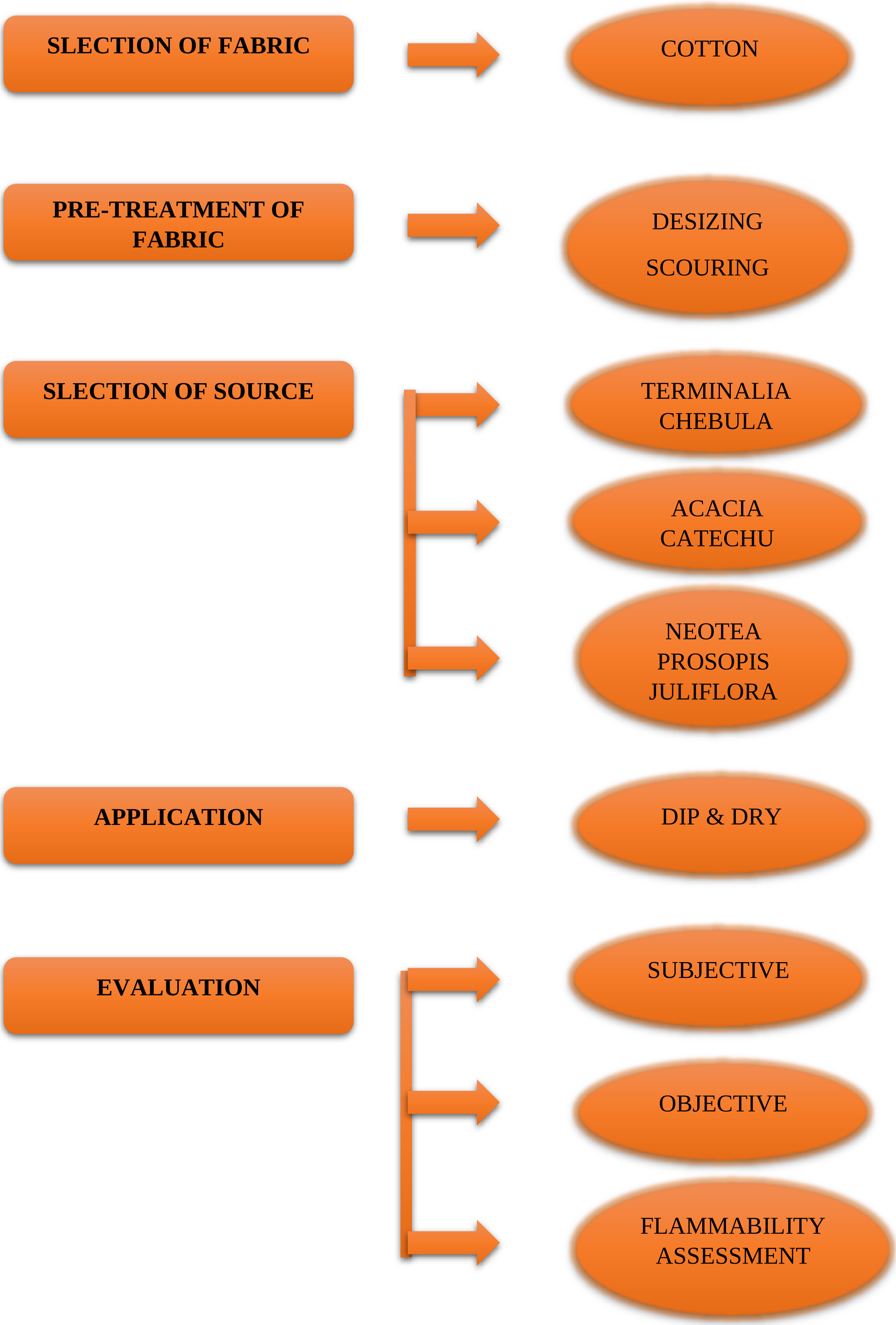
3.4.5 Thermogravimetry analysis

3.4.6 FTIR test

3.5 Statistical analysis

3.6 Nomenclature

FLOW CHART OF THE EXPERIMENTAL PROCEDURE



3.1 SELECTION OF FABRIC

A plain-woven bleached cotton fabric 88 EPC (ends/cm) and 64 PPC (picks/cm) procured from the National Textile Corporation (NTC, Coimbatore) was selected for the study.

3.1.1 Preparation of the fabric

Desizing

The bleached cotton fabric was first desized to remove the size material from the textile material.

Table II – Desizing recipe

Cotton material	**2 ms**
Material Liquor Ratio	**1:20**
Detergent powder	**3%**
Temperature	**60^{o}C**
Time	**1 hour**

Procedure

The selected cotton fabric was desized in a water bath containing detergent powder (3%), with MLR 1:20. The process of desizing was carried out at 60^{o} C for 1 hour. After desizing the cotton fabric was washed in cold water and shade dried.

Scouring

Scouring is the process of removing natural (oil, wax, gum, fat, etc.) and introduced (during the manufacture process) contaminants.

Table III –Scouring recipe

Cotton material	**2 ms**
Material Liquor Ratio	**1:20**
Wetting agent	**3%**
Temperature	**60^{o}C**
Time	**1 hour**

Procedure

Scouring was carried out on the selected cotton fabric after desizing. In a water bath containing 3% of wetting agent, the fabric was immersed at 60°C for 1hour. After the scouring process the fabric was washed in cold water and shade dried.

3.2 SELECTION OF SOURCE

Source 1

Terminalia chebula commonly known as myrobalan, it is a common medicinal herb used in Unani, Ayurveda, and homoeopathy. It is an Indian native plant whose dried fruit is commonly utilised in many home treatments (Rathinamoorthy,2014). Terminalia chebula has many medicinal properties and was commonly termed as **'King of Medicine'** in Tibet. Tannins and phenolic compounds are the major phytoconstituents identified in T. chebula. Other components of T. chebula include phenolics such as ellagic acid, chebulinic acid, anthraquinones, and polyphenols such as galloyl glucose, corilagin, triflavin A, punicalagin, and triterpene maslinic acid (Williamson,2002). T. chebula fruits contain higher tannins (32–34%), although this varies locally (Jayaramkumar,2006 & Kumar,2009). The presence of phytochemicals such as alkaloids, glycosides, phenolic compounds, flavonoids, saponin, quinine, steroids, and tannin were discovered in T. chebula fruit extracts (Baliah,2014).

Plate 4 – *Terminalia chebula*

Source 2

Acacia catechu commonly known as ***cutch tree bark (Karingali).*** The tannin content of the cutch tree bark was found to be 55-60%. The dried bark of Acacia catechu is widely utilised in India for its many pharmacological benefits. It can be used alone or in conjunction with cinnamon or opium to treat passive diarrhoea (British Pharmacopoeia,1999). Catechin, epicatechin, epigallocatechin, epicatechin gallate, phloroglucinol, protocatechuic acid, quercetin, poriferasterol glycosides, Lupe none, procyanidin, kaempferol, L-arabinose, D-galactose, D-rhamnose are the primary chemical constituents of acacia catechu. It has a variety of pharmacological properties, including immunomodulatory action, hypoglycaemic activity in rats, antifungal activity, antiviral activity, antibacterial activity, anti-inflammatory activity, and anti-oxidant activity (Lakshmi,2011).

Plate 5 - ***Acacia catechu***

Source 3

Neotea Prosopis Juliflora commonly known as **Mesquite tree gum,** Prosopis trees emit mesquite gum in the shape of round spherical balls that resemble tear drops. The mesquite gum was purchased in local organic shop. The mesquite gum is selected for this flame retardancy study as it contains polyphenol (tannins). Polyphenols are another naturally occurring class of compounds with good char-forming properties. Tannins, in particular, have a high char-forming capacity and can be employed as a flame retardant. It is also highly composed of polysaccharide which makes it as best alternative for synthetic flame retardants.

Plate 6 – Neotea Prosopis Juliflora

3.2.1 Pre-treatment of the selected source

The mesquite tree gum (resin) was soaked overnight in water with 1:2 ratio and used for further processing. The pH of the resin (as it is) was recorded as 5. It was made alkaline (pH 9) and (pH 12) with the addition of soda ash.

3.3 FINISHING OF COTTON FABRIC WITH THE SELECTED SOURCE

3.3.1 Dip and Dry method

A plain-woven bleached cotton fabric of 88 EPC (ends/cm) and 64 PPC (picks/cm) was used for the study. The bleached cotton fabric was first desized to remove the size material from the textile material. The cotton fabric sample was then scoured and shade dried. The dip and dry method of application was selected for finishing. The pre-treated mesquite gum resin was coated onto both sides of the fabric and drying is carried out for 1hr, same procedure is repeated for three times followed by drying in the Hot air oven for three different pH ranges and was used for further analysis.

3.4 EVALUATION

3.4.1 SUBJECTIVE EVALUATION

3.4.1.1 Visual assessment

The visual evaluation for control and treated fabric samples was carried out. It was done to obtain the opinion and preferences of the treated fabric samples. The finished fabric samples were pasted in a white sheet and it was shown to the students of the department of textile and clothing, Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore. The students were asked to score the control and treated fabric samples for general appearance, texture and evenness of the coated textile. Based on the student's opinion a score card was prepared and presented in Results and Discussion.

3.4.2 OBJECTIVE EVALUATION

For the control and treated fabric standard test method were followed to measure the add-on %, fabric weight, fabric thickness, crease recovery and absorbency (Mansdorf, 1988).

3.4.2.1 PHYSICAL PROPERTY

3.4.2.1.1 DETERMINATION OF ADD ON PERCENTAGE %

After the application of the tree gum at different pH on the cellulosic textile material, the add on percentage (increase in sample weight) was determined by gravimetric principle by taking the both dry weights of the sample of control and treated cellulosic fabric which is expressed as a percentage based on the initial weight of the sample.

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

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

3.4.2.1.2 FABRIC THICKNESS

The thickness of the treated and untreated samples was evaluated using Shirley thickness tester. The thickness test for the control and treated fabric was carried out with the loading weight of 4.14kPa. The samples with different pH were used to test the fabric thickness. Fabric Thickness is defined as the distance between lower and upper surface of the material measured under a standard pressure, using Shirley Thickness Tester with an accuracy of 0.01 mm (Stocker et al., 2005). Fabric thickness gauge are used to measure thickness of the sample. It has two parts of anvil and pressure foot. Pressure was given at the foot to make the gauge zero. The samples were placed between the cleaned pressure foot and anvil. The reading shown by the dial was noted. For each sample at five different places away from two inches of the selvedge.

3.4.2.1.3 FABRIC WEIGHT

Fabric weight is measured as mass per unit area. Fabric mass per unit area is expresses either as grams per square meter, or grams per linear meter, ounces per liner yard fabric is also sometimes expressed inversely as linear meters per kilograms (yard per pound) with the fabric width stated, ASTM D3776 96 (2002).

The control fabric was cut with GSM (Grams per Square Meter) die cutter which is $100cm^2$. The sample was weighed in an electronic balance. The weight of the sample was measured in grams and multiplied with 100 to get GSM value. Ten samples were tested and the average was calculated. The same procedure was followed for all samples.

3.4.2.1.4 CREASE RECOVERY

As for comfort property crease recovery of the control and treated samples were examined. A measure of crease resistance specified quantitatively in terms of certain parameters such as crease recovery angle (Maitra,2007).

Shirley Crease Recovery tester was used for the testing. The samples were folded face to face, end to end and held it with tweezers without touching the specimen with any other than the tweezers. The folded specimen was placed between the two leaves of the loaded device and immediately the weight was applied. After 5 seconds the weight was removed from the specimen, using the tweezers the folded specimen was transferred to the specimen holder, leaving the other end of the specimen to hang freely. Adjust the instrument to keep the free hanging end of the specimen in alignment with the vertical mark. Frequent adjustment for 5minutes is necessary to avoid gravitational effect and the degree is noted.

3.4.2.2 ABSORBENCY TESTS

Absorbency refers to the ability of a porous solid materials to take up and retain under various conditions, significant amount of water or other liquids by several distinct spontaneous processes (Choudhury, 2006).

3.4.2.2.1 DROP TEST

The drop test was carried out for control and treated samples at different pH levels. A burette filled with distilled water with a distance of 2.6” from the fabric sample is placed. The water drops are released and the time required for the sample to absorb the water completely is noted. The nozzle of the burette was opened just to allow a drop of water to fall on the sample. The stop watch was started simultaneously and it was stopped when the drop of water fully absorbed into the material. The time taken was recorded. The same procedure was carried out for the control and treated samples and the mean value was calculated and recorded.

3.4.2.2.2 SINKING TEST

Sinking is a simple test for wettability of fabric. A small specimen 1x1” is cut and dropped into the surface of water in a beaker from a standard height. The stop watch was started when the fabric struck the surface of the water and stopped when the last corner sank below the water surface. The time taken for the specimen to sink below the surface is observed. The shorter the time, the greater the wettability (Raul, 2005). The same procedure was repeated for five samples. Then the mean value was calculated for the above samples. Similarly, the mean value of the original and treated fabric material was calculated the sinking time of each material was recorded separately.

3.4.2.2.3 WICKING TEST

Liquid flow in a direction parallel to the plane of the fabric is termed as planar uptake or planar flow and referred as “wicking” (Johnson, 1996). A strip of fabric sample was suspended vertically with its lower edge in reservoir of distilled water. The rate of rise of the leading edge of water was then noted. To detect the position of water line a dye was added to the water after 30mintues and the rise in the water line was noted. The measured height of the rise in 30minutes was taken as a direct indication of the test fabric and recorded in centimetres.

3.4.3 FLAMMABILITY ASSESSMENT

3.4.3.1 LOI ANALYSIS

Standard methods were used to evaluate the control and treated samples. IS 13501 which is equivalent to ASTM D 2863 was used for determining limiting oxygen index (LOI). As per the Indian standard ignition time of 30s was maintained for this LOI analysis.

3.4.3.2 VERTICAL FLAMMABILITY TEST

The IS 1871 test method an equivalent to ASTM D 635 was used to evaluate the vertical flammability test. The fabric sample was ignited with 350 mm flame height for 12s. In vertical flammability a test specimen is held vertically above a controlled flame for a predetermined amount of time. The flame source was 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 both measured.

3.4.4 SCANNING ELECTRON MICROSCOPY (SEM) ANALYSIS

The control and treated fabric samples was used to analyse the surface morphology. SEM (Scanning Electron Microscopy) is a test method that uses an electron beam to scan a sample and provide a magnified image for analysis.

3.4.5 Fourier Transform Infrared Spectroscopy (FTIR) analysis

The FTIR was analysed in the control and treated fabric samples in Shimadzu IR analyser over the wavelength of 500-3600 cm-1 using KBr disc sample preparation method and an ATR transmittance mode with DLaTGS detector with 49scans and 4 revolutions were used to perform FTIR analysis. Infrared light was used to scan test materials and examine chemical characteristics using the FTIR analysis method.

3.4.6 THERMO-GRAVIMETRIC (TG) ANALYSIS

The thermogravimetry measures the gradual weight loss of a sample with respect to time and temperature. It also indicates the effect of flame retardant on the pyrolysis of the polymer substrate (Mostashari & Mostashari, 2009). Thermogravimetry thermal analysis (TGA) testing was carried out using EXSTAR/6300 at a heating rate of 10degree C/min in N2 atmosphere with the standard of ASTM E1131, ISO 11358. The control and treated fabric samples were used to determine the thermo-gravimetric curves.

3.5 STATISTICAL ANALYSIS

Statistical analysis of the study

The findings of the control and treated fabric samples were analysed using F- test to find the difference between the samples.

3.6 NOMENCLATURE

Table - IV

S.NO.	Sample	Name of the fabric
1	UT	Untreated cotton sample
2	TS1	Treated sample at pH 5
3	TS2	Treated sample at pH 9
4	TS3	Treated sample at pH 12

RESULTS AND DISCUSSIONS

4. RESULTS AND DISCUSSION

The results of the study **"Enhancing the Flame Retardancy of Cotton Fabric Using Sustainable Tree Gum"** are discussed in the following headings;

4.1 Subjective evaluation

4.1.1 Visual assessment

4.2 Objective evaluation

4.2.1 Assessment of physical property

4.2.1.1 Determination of add-on %

4.2.1.2 Fabric thickness

4.2.1.3 Fabric weight

4.2.1.4 Crease recovery

4.2.2 Absorbency test

4.2.2.1 Drop test

4.2.2.2 Sinking test

4.2.2.3 Wicking test

4.3 Flammability assessment

4.3.1 LOI

4.3.2 Vertical Flammability test

4.4 Scanning Electron Microscopy (SEM) analysis

4.5 Fourier Transform Infrared Spectroscopy (FTIR) analysis

4.6 Thermo-gravimetric (TG) analysis

4.1 SUBJECTIVE EVALUATION

4.1.1 Visual assessment

The ratings of visual evaluation of control and treated samples were surveyed and presented in Table-V.

TABLE V - VISUAL EVALUATION

RATING SCALE IN %	UT	TS1 (pH 5)	TS2 (pH 9)	TS3 (pH 12)
GENERAL APPEARANCE				
Excellent	**30**	**72**	**43**	**25**
Good	**20**	**28**	**57**	**75**
Fair	**50**	**-**	**-**	**-**
TEXTURE				
Coarse	**-**	**-**	**-**	**-**
Rough	**12**	**37**	**69**	**80**
Soft	**88**	**63**	**31**	**20**
EVENNESS				
Even	**34**	**100**	**58**	**100**
Partially even	**66**	**-**	**42**	**-**
Un even	**-**	**-**	**-**	**-**

From the results of visual examination, it is clear that the general appearance of (TS1) was rated 72% among the control and treated samples (TS2 & TS3). The sample (TS3) was rated highest percentage of 80 under rough texture while 63% rated soft texture for the sample TS1. The evenness of the TS1 and TS3 was rated 100%.

4.2 OBJECTIVE EVALUATION

4.2.1 ASSESSMENT OF THE PHYSICAL PROPERTY

4.2.1.1 Determination of add on percentage %

The results of add on % was presented in Table-VI.

TABLE VI – ADD-ON % OF THE FABRIC SAMPLES

Fabric Samples	Add-on percentage (%)
TS1 (pH 5)	2
TS2 (pH 9)	5
TS3 (pH 12)	8

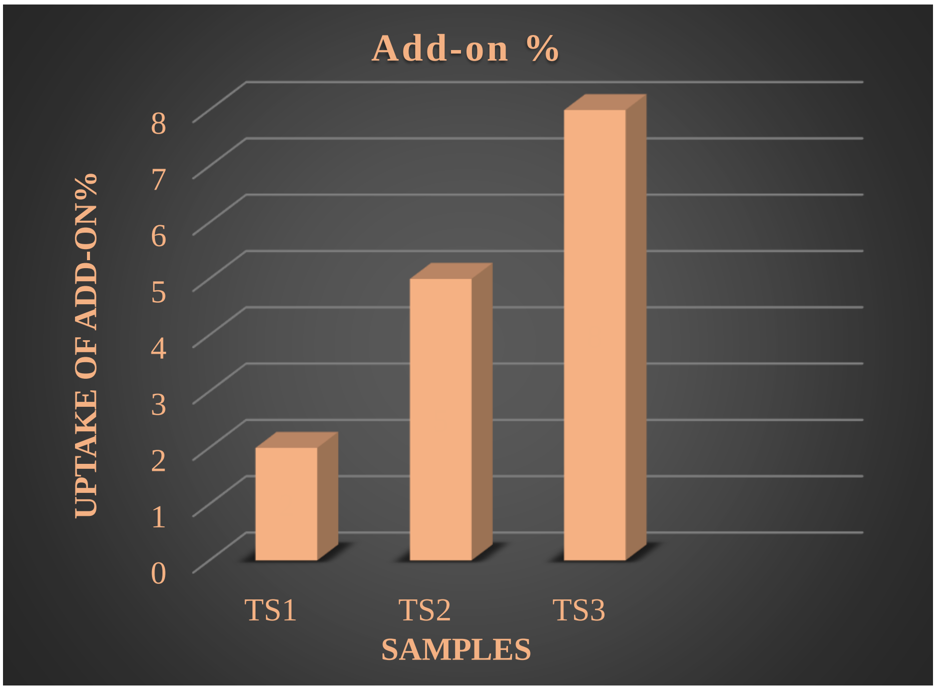


FIGURE – 1 ADD-ON % OF FABRIC SAMPLES

From Table-VI & Figure-1, it is clear that the add-on% was found to be maximum for TS3, which might be due to increased uptake at alkaline pH.

4.2.1.2 FABRIC THICKNESS

The results of the fabric thickness of the control and treated samples are presented in Table-VII & Figure-2.

TABLE VII – FABRIC THICKNESS

Samples	Mean (gsm)	S. D	Gain/Loss	%Gain/Loss	F-Value
UT	0.28	0.01	-	-	2.61^{ns}
TS1	1.79	5.19	-1.51	539.28	
TS2	2.38	1.71	-2.1	750	
TS3	2.41	0.72	-2.13	760	

ns – not significant

The mean calculation of the thickness test shows that, the thickness of the cotton fabric was found to be increased after the application of the mesquite gum in different pH levels. The control sample showed a mean of 0.28 whereas, the treated sample at pH12 showed 2.41 mean value.

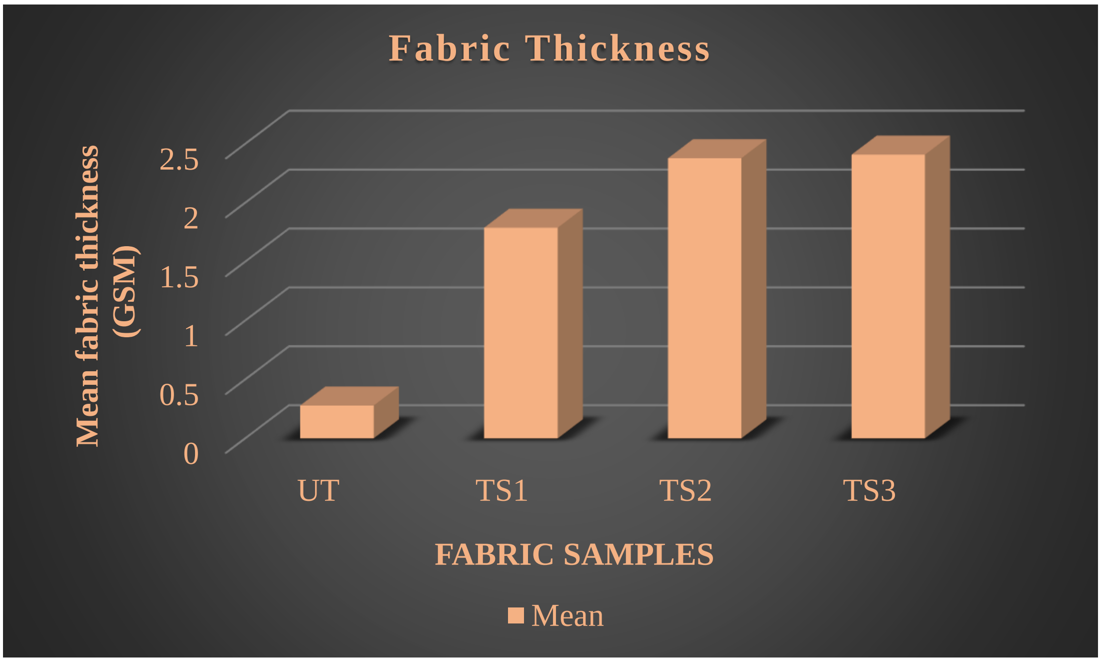


FIGURE 2 - FABRIC THICKNESS OF UNTREATED AND TREATED SAMPLES

From Table-VII & Figure-2, it is evident that the fabric thickness increased in all the samples finished with mesquite gum when compared to the control sample. The increase in fabric thickness was found to be maximum in (TS3) followed by TS2 & TS1, which indicates that pH influences the uptake of the finish. The statistical analysis proves that there is no significant difference between the fabric samples.

4.2.1.3 FABRIC WEIGHT

The fabric weight of the control cotton fabric and all the treated fabric samples are presented in Table-VIII & Figure-3.

TABLE VIII – FABRIC WEIGHT

Samples	Mean (grams)	Gain/Loss	%Gain/Loss	F-Value
UT	1.246	-	-	0.7256^{ns}
TS1	1.885	-0.639	51	
TS2	2.248	-1.002	80	
TS3	2.466	-1.22	97	

^{ns} – not significant

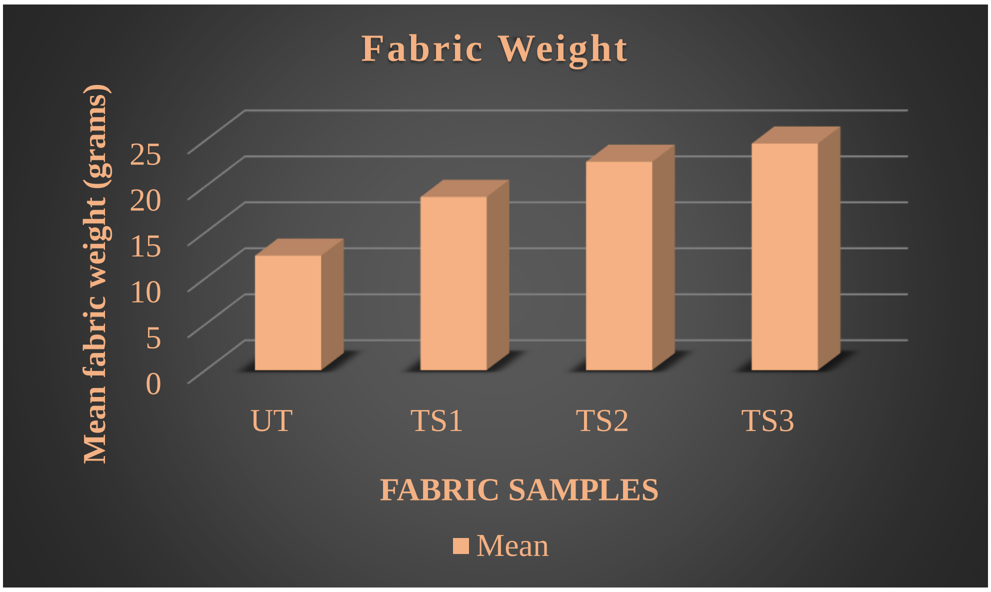


FIGURE 3 – FABRIC WEIGHT

From table-VIII & figure-3, it is evident that the weight of the fabric was found to be increased in all the treated fabric samples when compared to the control fabric. Hence, it could be concluded that the weight of the treated fabric samples increased after the application of mesquite gum. The statistical analysis indicates that the increase was not significant among the finished fabric samples.

4.2.1.4 CREASE RECOVERY

The results of the crease recovery of the control and treated fabric samples are presented in Table-IX & Figure-4.

TABLE IX – CREASE RECOVERY

Samples	Mean (degree)	Gain/Loss	%Gain/Loss	F-ratio
UT	63.4	-	-	10.922^{ns}
TS1	89.5	-26.1	41	
TS2	95.1	-31.7	50	
TS3	103.4	-40	63	

ns – not significant

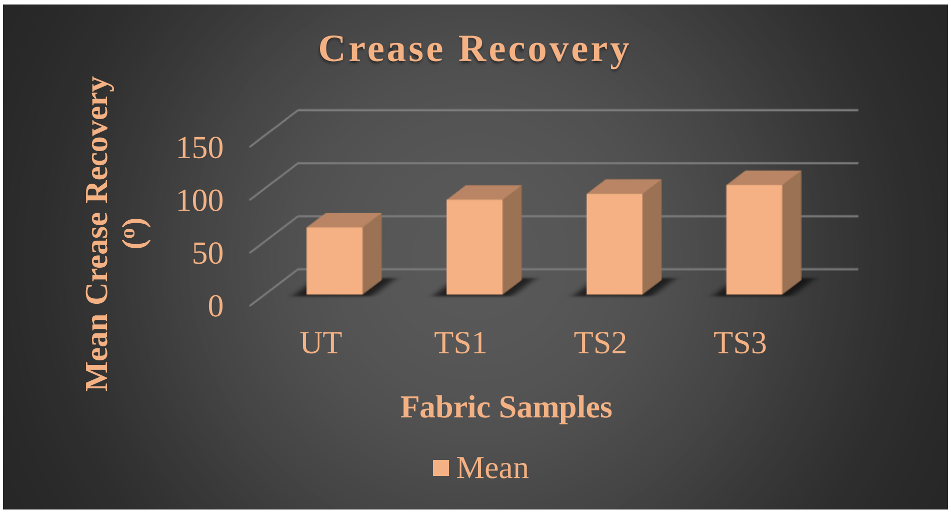


FIGURE 4 - CREASE RECOVERY

From the Table-IX & Figure-4, the crease recovery was increased in all the treated sample and when compared to the control fabric sample. Hence, it could be concluded that the crease recovery was gradually increased in the treated samples. The increase in crease recovery was not significant as there is not much difference between the samples.

4.2.2 ABSORBENCY TEST

The absorbency tests were carried out on the control and treated fabrics and the results of drop test, sinking & wicking test are recorded.

4.2.2.1 DROP TEST

The results of drop test of the control and treated samples are presented in Table- X & Figure-5.

TABLE X – DROP TEST

Samples	Mean (sec)	Gain/Loss	%Gain/Loss	F-Value
UT	11.58	-	-	2.073**
TS1	18.34	-6.76	58	
TS2	23.8	-12.22	105	
TS3	35.81	-24.23	67	

** - 1% significant

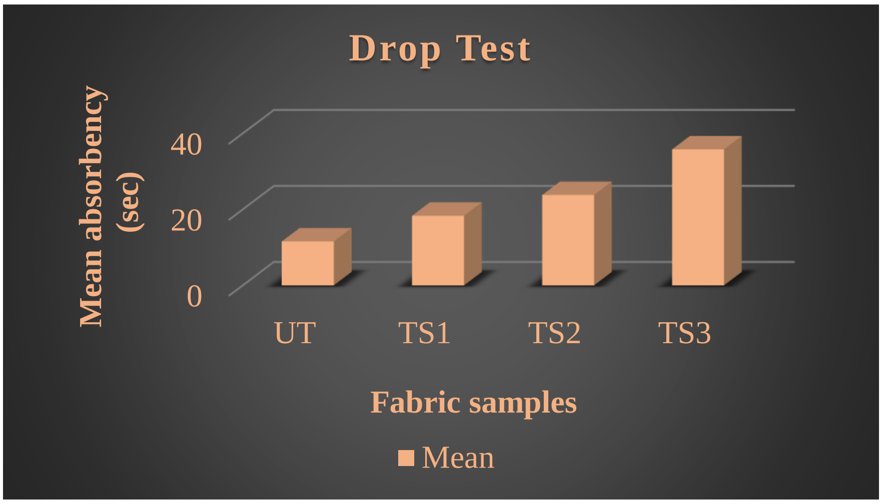


FIGURE 5 - DROP TEST OF UNTREATED AND TREATED SAMPLES

From Table-X & Figure-5, it was evident that the absorbency of the treated and un treated samples showed that the absorbency was high in the control sample. The absorbency was found to be decreased in all the treated samples and the decrease was found to be maximum in TS3. The statistics analysis also proves that there was 1% significant difference between the variables.

4.2.2.2 SINKING TEST

The results of the sinking test of control and treated fabric samples are presented in the Table-XI & Figure-6.

TABLE XI – SINKING TEST

Samples	Mean	Gain/Loss	%Gain/Loss
UT	6.9	-	-
TS1	7.8	0.9	13.04
TS2	6.2	0.7	10.14
TS3	3.6	2.2	31.88

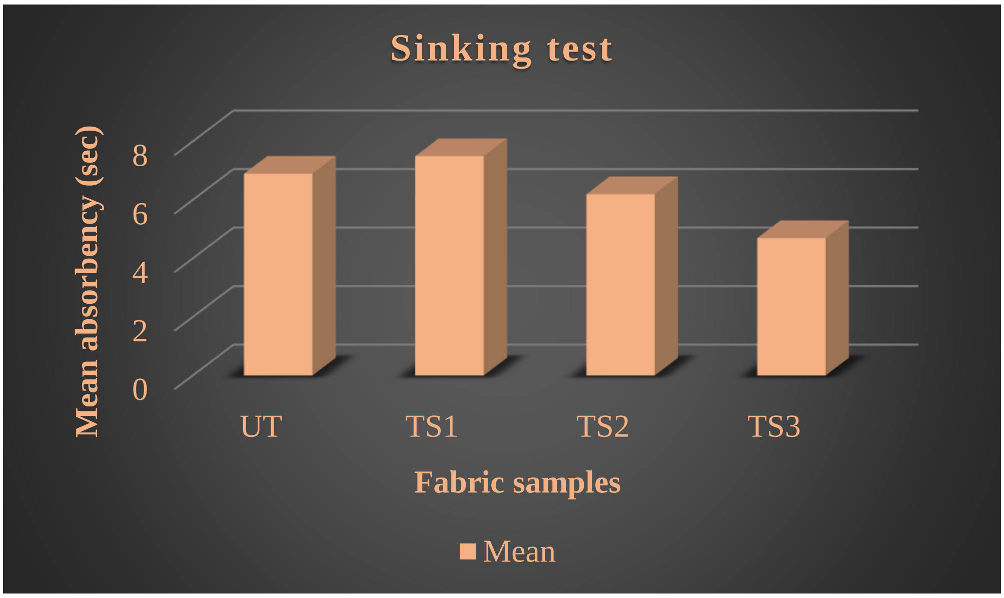


FIGURE 6 - SINKING TEST

From Table-XI & Figure-6, it is clear that the sinking property of the sample increased inTS1 and found to be decreased in all other treated samples (TS2 & TS3).

4.2.2.3 WICKING TEST

The results of the mean wicking time of control and treated fabric samples are presented in Table-XII & Figure-7.

TABLE XII – WICKING TEST

Samples	Mean absorbency (cm)	Gain/Loss	%Gain/Loss
UT	4.35	-	-
TS1	6.17	1.82	41.8
TS2	5.64	1.29	29.6
TS3	4.20	0.15	3.44

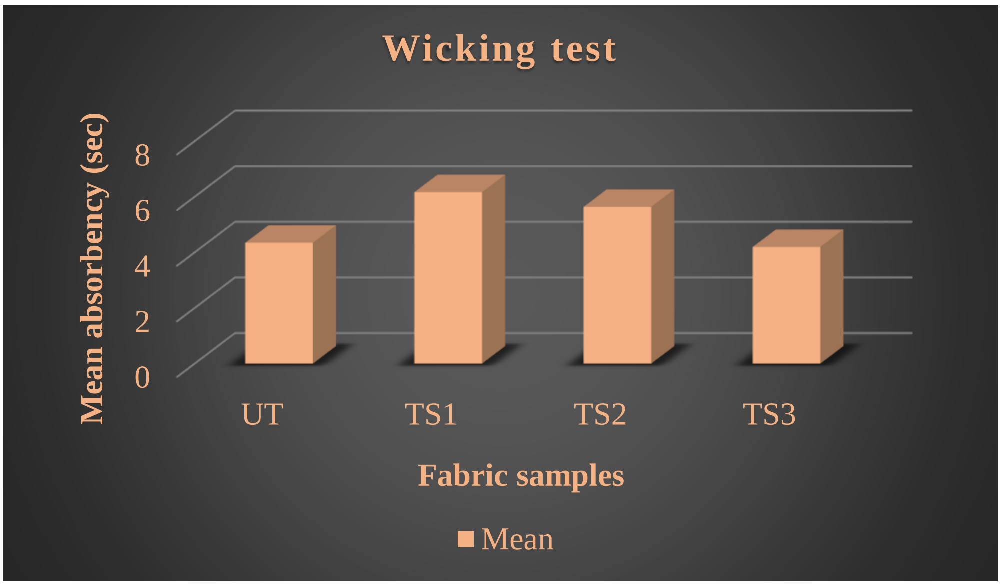


FIGURE 7 - WICKING TEST

From the Table-XII & Figure-7, it was observed that the wickability of the treated samples was increased in TS1 and found to be decreased in the treated samples (TS2 & TS3). The Gain/loss percentage varied from 41 to 3%. Hence it could be concluded that the wicking property was decreased in the finished samples when compared to the control fabric sample.

4.3 FLAMMABILITY TEST

4.3.1 Limiting Oxygen Index

The Limiting Oxygen Index (LOI) was determined in the control and treated samples and the results are presented in Table -XIII.

4.3.2 Vertical Flammability test

The results of the Vertical flammability test of the control and treated samples are summarized in Table-XIII.

Table XIII - Flammability parameters of control and treated samples of cotton fabric

Flammability Parameters	Control Cotton (A)	Treated samples at different pH		
		pH 5	pH 9	pH12
LOI	18	25	26	27
***Vertical Flammability test* - (Sample size: - 350 x 70) mm**				
Occurrence of flashing over the surface	Yes	No	No	No
Burning with flame time (s)	12s	97s	112s	24s
Burning with afterglow time (s) after flame stop	0s (As completely burnt with flame)	147s	793s	470s
Total burning time (flame time + afterglow time)	12+0s	97+ 147s	112+793s	24+470s
Char length (mm)	Nil	Nil	Nil	85
State of the 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

The result of the flammability testes is presented in Table-XIII. The mesquite gum treatment has increased the thermal stability of the treated fabric in terms of LOI and Vertical flammability assessment, irrespective of the pH used. The LOI is the parameter which is frequently used to characterize the improvements in the fire retardancy of protective textiles. LOI indicates the minimum oxygen concentration in the test atmosphere to support ignition and flaming combustion of the material. Textiles having LOI values of 21 or below, ignite easily and burn rapidly in the open atmosphere. The LOI value of above 21, ignite but burn slowly and the LOI value equal or above 26 it may be considered as flame retardant (Petrilli, 2008).

As cotton being 100% cellulosic material, it catches flame easily and showed LOI value of 18. The LOI value has been increased with increase in pH of the treated fabrics from 18 to 27, which is greater than the control cotton fabric.

In the vertical flammability, due to the increase in the pH of the mesquite gum, the intensity of smoke generation was found to be decreased. The burning with afterglow was increased in pH 5 and 9. As a result, the afterglow was completely arrested and showed self-extinguishment in the treated sample with increased pH 12 Table 9. The burning rate of pH 12 was found to be less when compared to pH 5 and 9. The burnt portion was observed as greyish with continuous smoke generation. Moreover, the flame time of the treated samples showed a great reduction to 23.6 sec on pH 12, as the flame time is less, the escape time from the flame will be increased automatically, which will be more helpful in real life situation as the wearer gets a longer time to escape from the fire zone or to extinguish the flame. Hence to conclude, the mesquite gum finished fabric at pH 12 showed complete self-extinguishment.

Cotton (Untreated Fabric)

TS1 – Flame time

TS1 – Afterglow time

TS1 – Ash

TS2 – Flame time

TS2 – Afterglow time

TS2 – Ash

TS3 – Self extinguished

Partly burnt by afterglow

Plates – 7 - Vertical flammability

4.4 SURFACE MORPHOLOGY AND ELEMENTAL ANALYSIS

The SEM analysis was carried out on the control and treated samples Fig-4 & 5, represents the SEM observations of the control and treated samples. The images of control cotton sample showed rough surface without any deposition (Fig.8). On the contrary, the treated sample showed thicker and smooth surface, which indicates that the tree gum coating has been uniformly distributed over the entire surface (Fig.9). Sample B (treated) showed a thicker surface, as they are tightly secured when compared to sample A (control) with loosely woven surface. The tight structure might be due to the presence of the tree gum in the alkaline formulation. Thus, the structural integrity was maintained well in the treated fabric than the control fabric with distorted structure. The wicking process of flammable gases inside the polymer is due to the distorted structure and it also assist to burn continuously while the mesquite gum treated sample with tightly bonded structure helps to prevent the continuous burning.

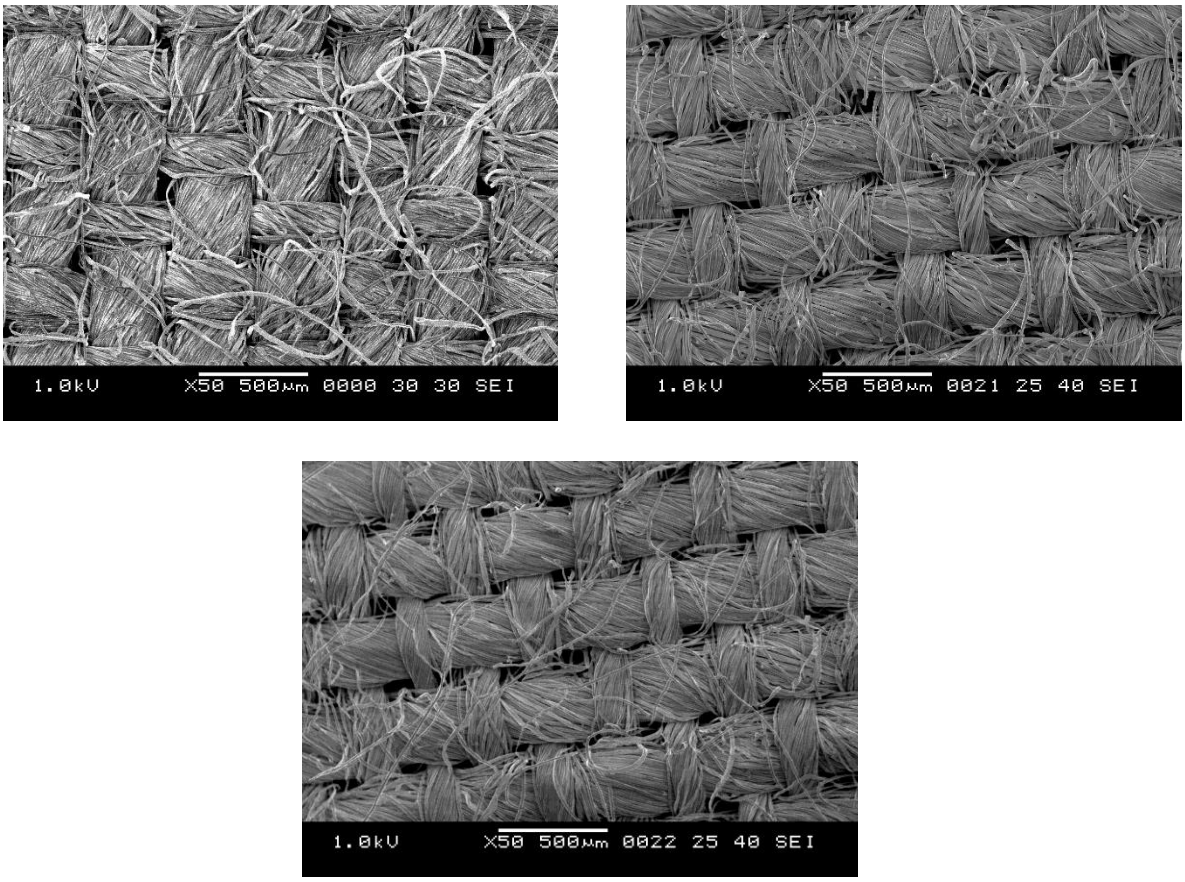


FIG. 8. SEM IMAGES OF CONTROL SAMPLE

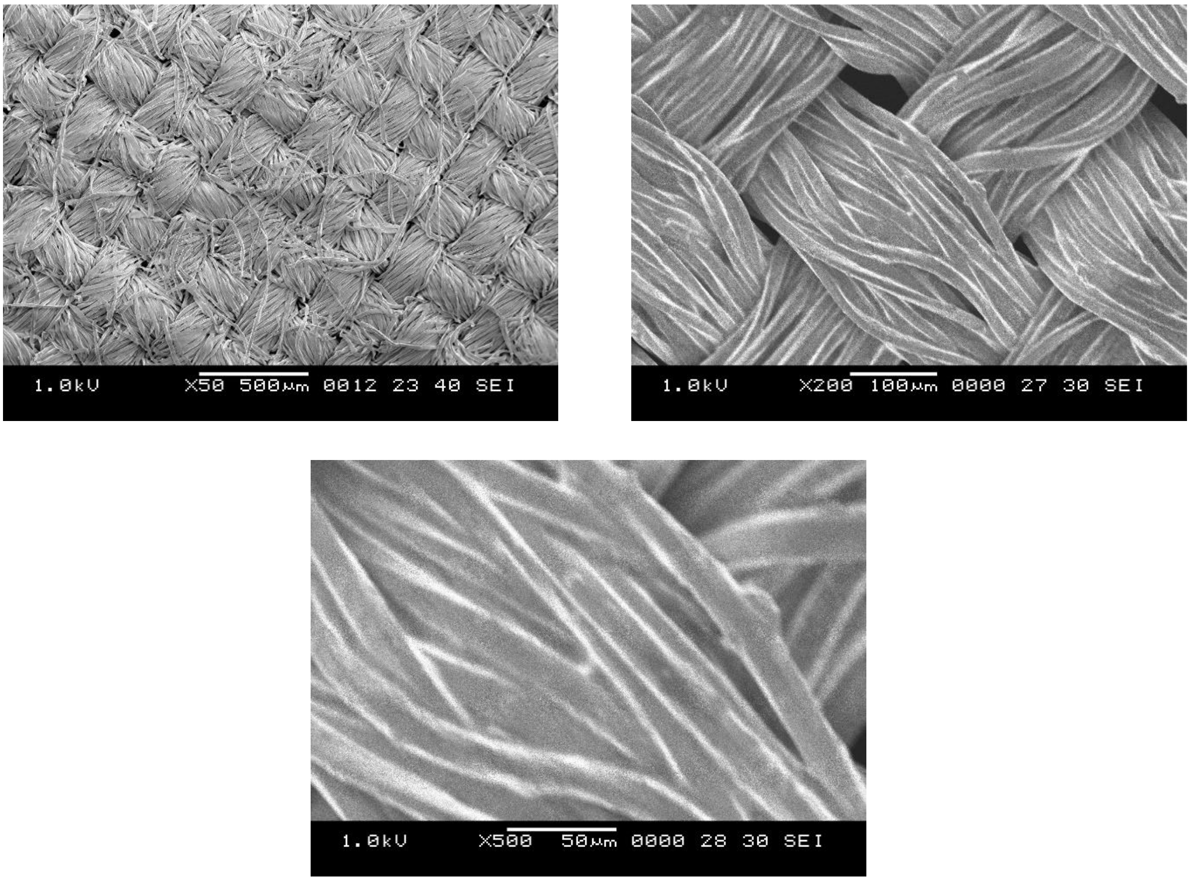


FIG.9. SEM IMAGES OF FINISHED SAMPLE

4.5 FOURIER TRANSFORM INFRARED ANALYSIS

The results of FITR analysis of control and treated samples are represented in Table.2. The treated sample showed a peak at 1002cm^{-1}, may be assigned to the C-O single bond stretching vibration of alcohols, C-F stretch of aliphatic fluro compounds and cyclohexane ring vibration of methylene >CH_2. Peak observed at 648cm^{-1}, associated with the aliphatic organohalogen compounds like C-Br, C-I, C-Cl. Another peak observed at 686cm^{-1}, associated with the aromatic mono substituted ring, very broad band of amides, C-Cl, C-Br, alcohol, CH_2-S-thioether and aryl thioethers. Small peak observed at 1095cm^{-1} represents the plane bend of aromatic ring, C-C vibration of methyne skeletal, inorganic ions (silicate, phosphate, sulphate), C-O stretch of alkyl substituted ether, silicon-oxy compounds and large rings of cyclic ether (C-F) stretch. 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 like (mono fluro alkanes, poly fluro alkanes, aryl fluorides), aromatic C-H stretch and secondary & tertiary amines (CN) stretch. Weak peak observed at

725cm^{-1}, may be assigned 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 observed at 902cm^{-1}, may be assigned 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}, represents aliphatic bromides & iodides and S-S stretch of aryl & poly disulfides. However, FTIR curves of treated and control samples are not showing any significant changes in the intensity of the peaks. In addition, there is no identifiable extra peaks in the FTIR curves on the treated sample surface. The presence of aromatic rings, phosphorous, bromide, metallic salts, oxides in the treated sample surface acts as a barrier of heat resistance. It has been observed that the gum treated cotton sample provides more thermal stability once the applicated of the finishing was maintained as alkaline (pH 12).

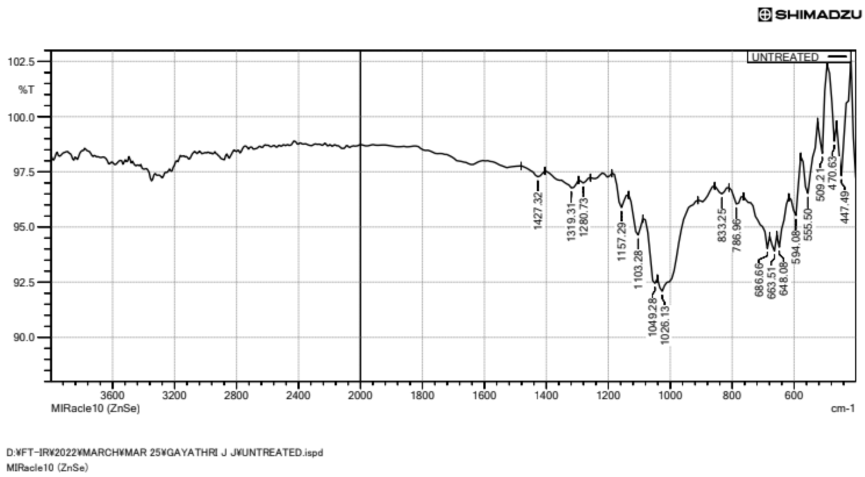


FIG. 10 FTIR SPECTRUM OF CONTROL SAMPLE

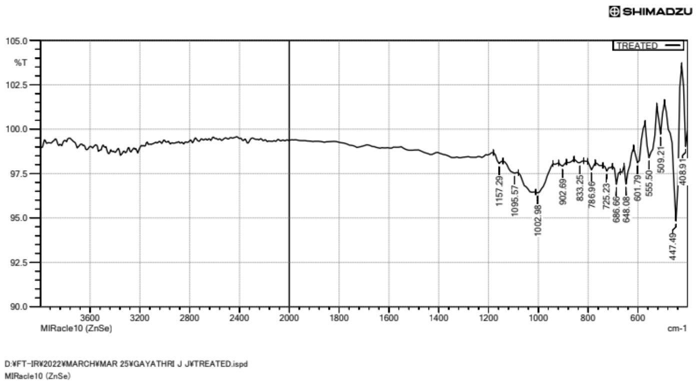


FIG. 11 FTIR SPECTRUM OF FINISHED SAMPLE

4.6 THERMOGRAVIMETRIC ANALYSIS (TGA)

The 1st, 2nd & 3rd degradation of the fabric samples in the thermogravimetric curves are presented in Table – XIV, XV & XVI.

TABLE – XIV – 1ST DEGRADATION OF TG CURVES

SI. NO.	Sample	Standard weight (mg)	1st degradation		
			Temperature (C^{o})	Weight (mg)	Percentage (%)
1.	Control sample	8.094mg	250^{o}C	8.0mg	11.5%
2.	Finished sample	7.751mg	300^{o}C	7.5mg	8.5%

TABLE – XV – 2ND DEGRADATION OF TG CURVES

SI. NO.	Sample	Standard weight (mg)	2st degradation		
			Temperature (C^{o})	Weight (mg)	Percentage (%)
1.	Control sample	8.094mg	400^{o}C	5.5mg	19.5%
2.	Finished sample	7.751mg	360^{o}C	5.8mg	16.6%

TABLE – XVI - 3RD DEGRADATION OF TG CURVES

SI. NO.	Sample	Standard weight (mg)	3st degradation		
			Temperature (C^o)	Weight (mg)	Percentage (%)
1.	Control sample	8.094mg	910^oC	5.1mg	35.7%
2.	Finished sample	7.751mg	920^oC	5.4mg	29.8%

The TG curves of control and tree gum coated fabric samples in N_2 atmosphere at a heating rate of 20^o C/min using EXSTAR/6300 Alumina pan. The gum treated fabric sample was presented in Figure 14 & 15, showed the initial peak with 8.5 % of mass loss occurred mainly due to the removal of bound and unbound absorbed moisture of the cellulosic polymer. The treated sample was stable from 0 to 250^o C, around 250^o C, the initial decomposition has occurred with the weight loss of 7.751mg on thermal decomposition. Second major weight loss with a sharp fall in the first derivative peak of 16.6% at 360 ^{o} C**,** here the depolymerization of cellulose might be occurred with the loss of 5.8mg. In addition to that, the last stage of decomposition has occurred at 920 ^{o} C with 29.8% of fabric weight reduction of 5.4mg**.** The gum coated fabric showed a lower rate of weight loss with the increase in temperature.

The TG curves of untreated sample was presented in Figure 12 & 13, showed initial peak with a mass loss of 11.5% which may be attributed with the loss of moisture content of the pure cotton. The stability of the control fabric sample was found to be maintained from 0 to 300^o C. On thermal decomposition the initial weight loss of control fabric sample was found to be reduced to 8.0 mg from the standard sample weight 8.094mg. The second weight loss was observed at 400 ^{o} C with the mass loss of 19.5% and the weight reduction was about 5.5mg in the second degradation. The final stage of decomposition happened at 910 ^{o} C which showed a drastic weight loss of 35.7% and the weight reduction was observed at 5.1mg. The TG curves of treated and untreated fabric samples revealed that the weight loss of control sample increases with increase in temperature than the treated fabric sample which showed a lower rate of decomposition with increase in temperature.

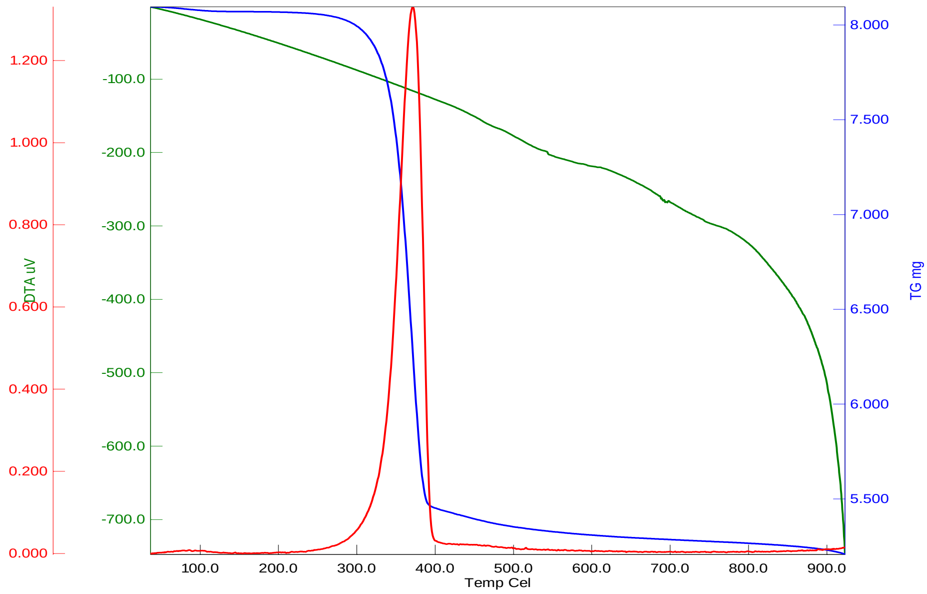


FIGURE-12 TGA OF CONTROL SAMPLE

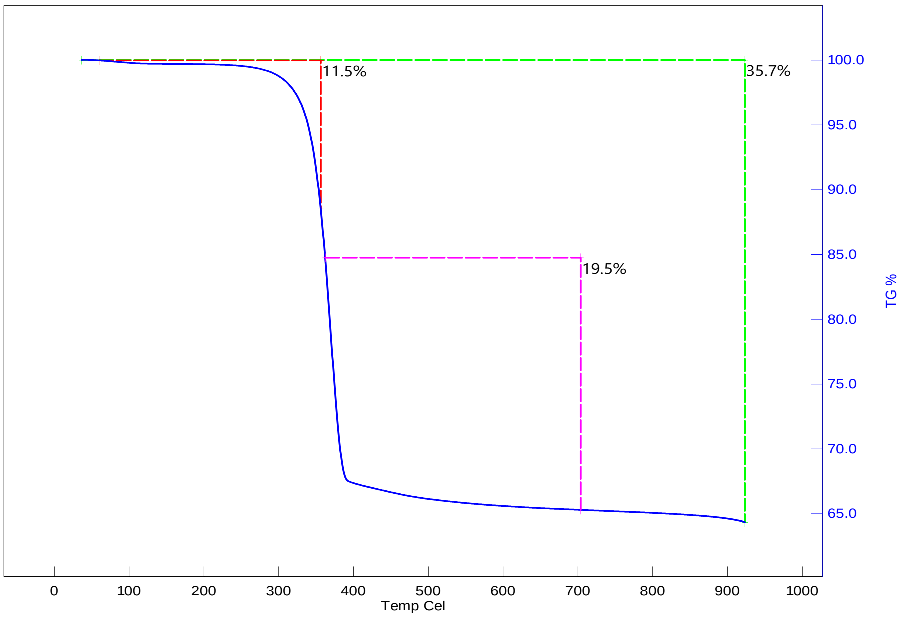


FIGURE-13 TG% OF CONTROL SAMPLE

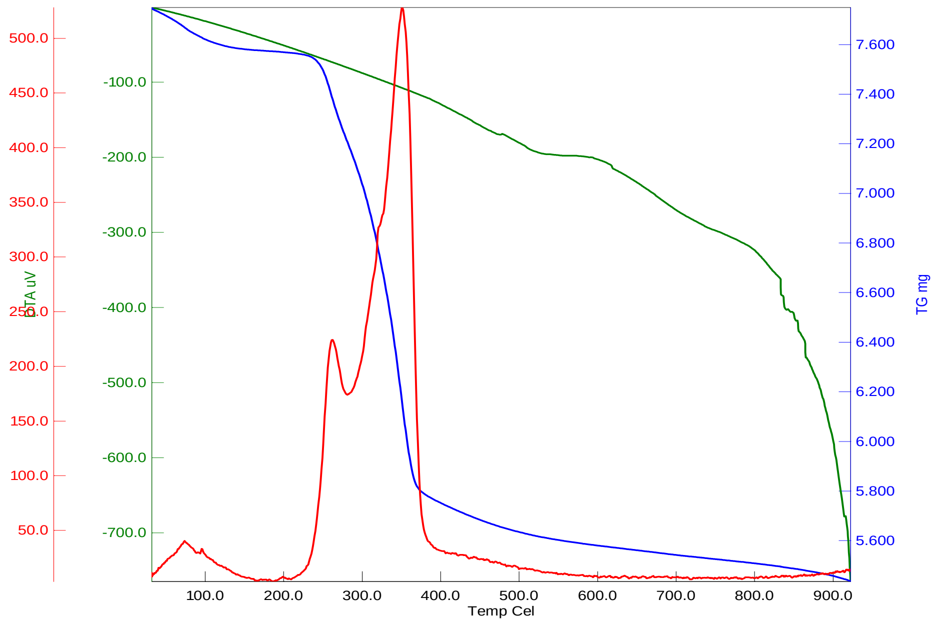


FIGURE-14 TGA OF FINISHED SAMPLE

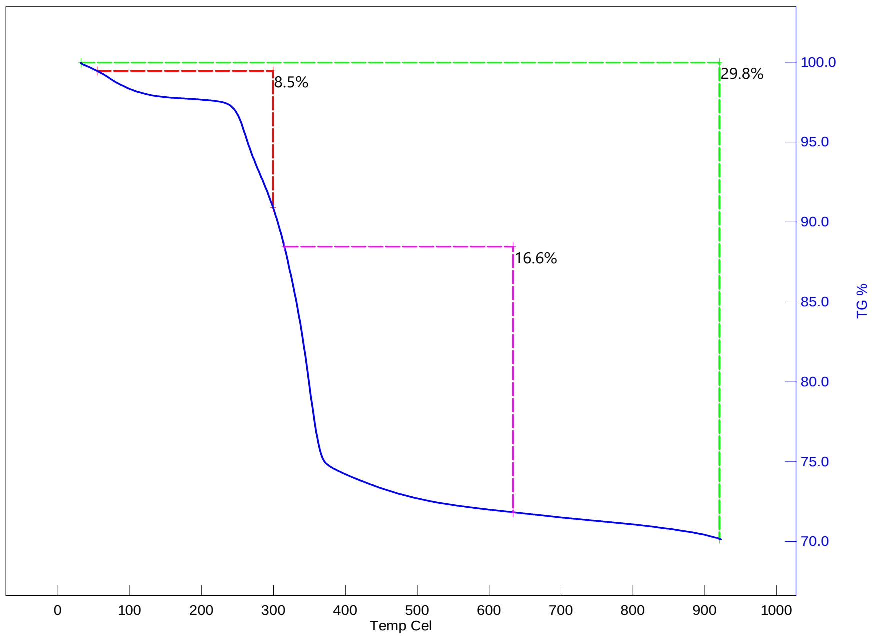


FIGURE-15 TG% OF FINISHED SAMPLE

SUMMARY AND CONCLUSION

5. SUMMARY AND CONCLUSION

SUMMARY

In this present study the cotton fabric is used to **"Enhance the flame retardancy using mesquite gum".** Flame-retardant finishing is one of the most essential types of functional finishing for textile substrates since it directly affects human health and safety. Since the textiles are used next to skin, it is called the second skin, it should be safe and skin friendly. People are becoming more concerned about the possible dangers linked with chemicals as information about their adverse effects on humans and the environment has risen fast. A vast number of flame retardants have been identified as potentially dangerous**.** With growing global awareness of sustainable eco-friendly green textile chemicals, auxiliaries, and textile products, some of the major issues related to environment friendliness, toxicity, and sustainability of various traditional flame-retardant compounds have become a serious issue since the year 2000.

Cotton is commonly used in clothes and home textiles because of its soft feel, good moisture wicking, and adequate thermal insulation. Cotton, on the other hand, is cellulosic in nature and has a low limiting oxygen index (LOI) of 18, it catches fire easily. The main challenge in flame-retardant finishing is to create an effective finish using non-toxic chemicals that does not compromise fabric comfort.

Considering the above facts, the investigator selected the research work on the topic "**Enhancing the Flame Retardancy of Cotton Fabric Using Sustainable Tree Gum**" With the following objectives;

- To select a natural source for flame retardant finish on cellulosic fabric.
- To finish the selected fabric for flame retardancy.
- To evaluate the finished flame retardant fabric.

EXPERIMENTAL PROCEDURE

The cotton fabric is selected for this study. In order to remove the size materials and other impurities from the cotton fabric, the fabric is weighed and dipped in l:20 of MLR with 3% of detergent powder at 60^{o} C for one hour. After desizing the fabric is rinsed with cold water and shade dried.

The pH of the resin (as it is) was recorded as 5. It has been made alkaline (pH 9) and (pH 12) with the addition of soda ash. The cotton fabric sample is dipped into plain water with detergent and boiling is carried out for 30mins followed by drying. The coating has been applied in the resin solutions of three different pH (5, 9, and 12). The soaked mesquite gum resin was coated onto the both sides of the fabric and drying is carried out for 1hr, same procedure is repeated for three times followed by drying in the Hot air oven and was used for further analysis.

The control and treated fabric samples were subjectively analysed by visual assessment and objectively analysed for fabric thickness, weight, crease recovery, wicking, sinking and drop test. In addition, the flammability assessment was carried out by analysing the LOI and vertical flammability test. Then SEM, FTIR and TG was also done for the samples.

FINDINGS OF THE STUDY

- The add-on percentage of the treated samples were increased gradually
- The fabric thickness increased in the treated samples when compared to the control sample. The increase of fabric thickness is due to the flame retardant finishing using mesquite gum. Statistical analysis also proved that there was a significant difference at 1% level.
- The weight of the fabric was found to be increased on the treated fabric. The values expressed that the finished fabric samples possess weight gain when compared to the control cotton fabric.
- The crease recovery of the control and treated fabric samples showed increase/ decrease in the crease recovery.
- The absorbency of the control and treated were analysed using drop test, sinking test and wicking test. The drop test shows that the absorbency time of the treated samples was found to be increased when compared to the control fabric sample.
- The difference of sinking time increases or decrease, before and after treatment of flame retardant finish with mesquite gum. The sinking time is increased after the treatment of flame retardant finish using mesquite gum on cotton fabric when compared with control and treated samples.
- The difference of wicking time increases or decrease, before and after treatment of flame retardant finish with mesquite gum. The wickability of the TS3 at (pH 12) showed

no wicking property when compared to TS1 and TS2. It is due to the finishing of the sample using mesquite gum.

- The Limiting oxygen index of the control and treated samples showed a gradual increase from 18 to 27. That is the LOI value above or equal to 26 may be considered as flame retardant.
- In the vertical flammability the treated fabric sample TS3 at (pH12) shows that the afterglow was completely arrested and showed self- extinguishment, observed as partly burnt by the afterglow.
- The SEM analysis of control and treated samples were compared and the treated samples showed much thicker and smooth surface when compared with the control fabric sample. The distorted structure in the control fabric sample assists the wicking process of flammable gases while the mesquite gum treated samples with thicker structure helps to prevent the continuous burning.
- The FTIR analysis confirmed the presence of aromatic rings, phosphorous, bromide, metallic salts, oxides in the flame retardant treated fabric sample surface which acts as a heat barrier.
- The TG curves of the fabric samples showed more than 35 % weight loss on control cotton fabric against only 29% weight loss of the alkaline treated fabric sample at 900^{o}C just after 20min of heat exposure.

CONCLUSION

In order to present more eco-friendly materials, it is required to advance the application of green chemistry and encourage the development of bio-based polymers. The ban on some halogenated compounds, as well as the hunt for alternative options, has fuelled the development of new bio-based additives. This work has proven that using the sustainable tree gum as a natural flame retardant. The mesquite gum will be a natural alternative source for flame retardants as it has more functional properties.

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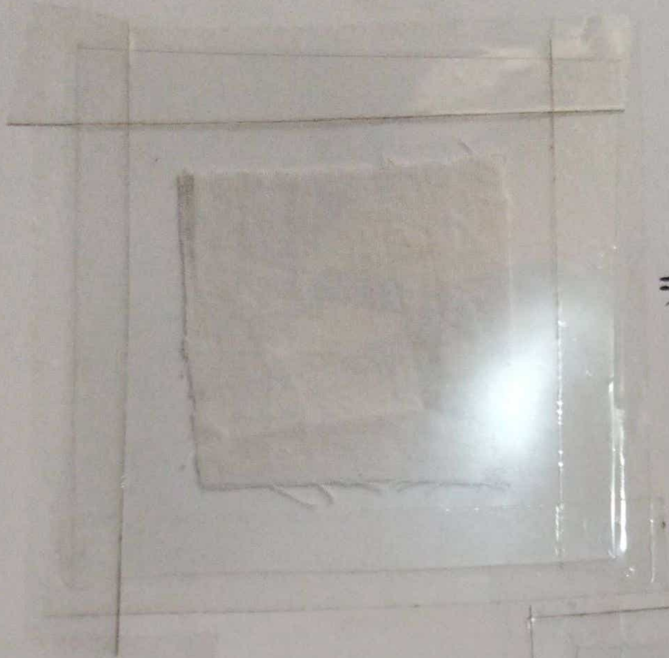
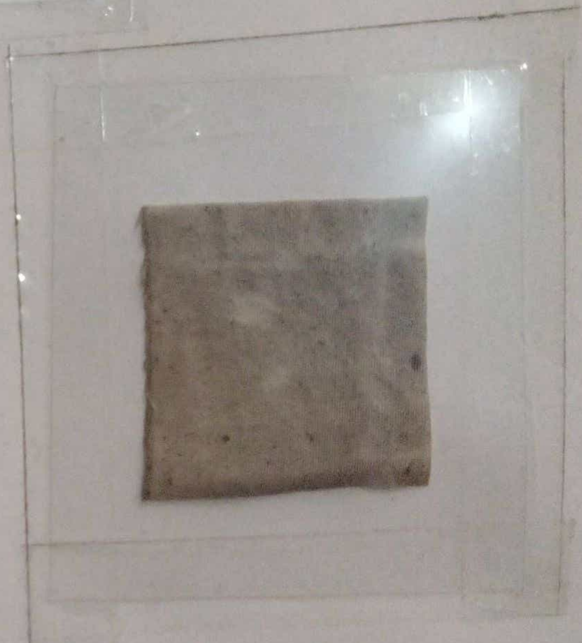
APPENDICES

APPENDIX - 1

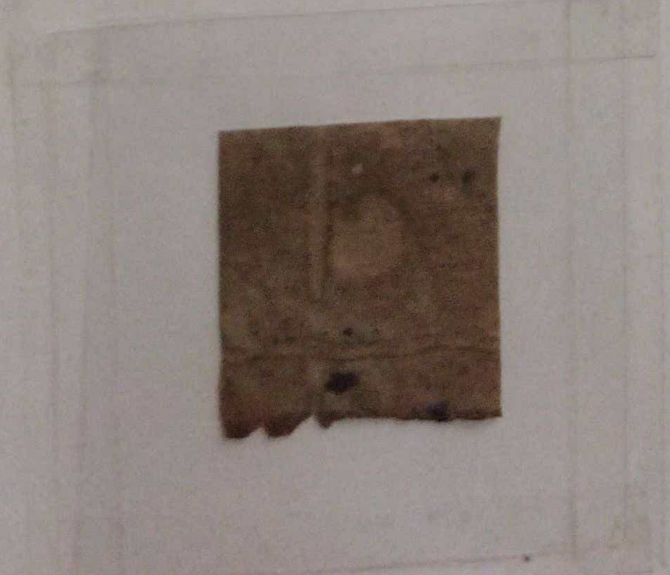


⇒ Untreated Cotton Sample

FR Treated Sample - 1



FR Treated Sample - 2



⇒ FR Treated Sample - 3