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# Application of Pomegranate Rind Extract for Improvement of Functional Properties of Various Materials—A Critical Review

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#### **Abstract**

Pomegranate rind is abundantly available as a waste material. Pomegranate Rind Extract (PRE) can be applied to cotton fabrics for its natural colours, as a mordanting agent and also for imparting certain functional properties such as fire retardancy and antimicrobial properties. This paper reviews the feasibility of Pomegranate Rind Extract to improve the functional properties of cellulosic fabrics. Studies show that varying concentrations and higher temperatures that were used to apply the extract on the fabric, resulted in enhanced functional properties. At a particular concentration, the treated fabric showed a 15 times lower burning rate in comparison with the control fabric. Also, antimicrobial efficacy has been observed against Gram-positive and Gram-negative bacteria. Due to the natural colouring material, it can be used as a natural dye on cotton material. The fire retardancy of pomegranate rind extract was tested on jute material under varying alkalinity. Research has indicated that pomegranate rind extract could be used to dye polyamide as well. The rubbing and wash fastness of the finished fabrics is good. The light fastness was fair, and its antibacterial efficiency against tested bacteria was good.

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# **Keywords**

Natural Polymers, Synthetic Polymers, Pomegranate Rind Extract (PRE), Dyeing, Functional Property

#### 1. Introduction

In the case of applications like home textiles, home furnishing, etc., flame-retardant textile material with attractive natural colour and hygienic antimicrobial properties has become a great need in apparel due to its fire safety and bioactive concern [1]. In recent years, interest in the investigation of natural bioactive plant extracts for the dyeing and functionalization of cotton fibers has intensified [2].

In today's realm of fire safety, one of the demanding, promising, and challenging areas is the sustainable flame-retardant properties of polymeric materials [3]. Considering various study perspective points, several researchers are attempting to attain that scientific brilliance. Over the past ten years, the field of bio-based (bio-macromolecule) flame retardancy has grown significantly and shows immense potential. It is one of the quickest-growing subfields in sustainable flame retardancy. Sustainable flame retardants are made of biodegradable material. One of the significant and main biologically active elements of the majority of plant-based bio-macromolecules is the aromatic polyphenolic chemical known as tannin. Research articles state that in the last several years, plant bio-macromolecules based on tannin have demonstrated a significant promise for flame retardancy when applied to textiles, and other natural and synthetic polymeric materials [4].

However, as of currently, the flame retardant compilation has not received any critical reviews. Researchers have been working on creating flame-retardant polymers based on tannins over the last five to ten years. The main problems in this industry, nevertheless, are the finish's poor washing durability and high add-on% on the polymer surface. Increased tannin loading (almost 40% necessary for self-extinguishment) on the polymer surface can degrade the fabric's handling as well as other physical characteristics like tensile strength, tear strength, and drapability [5]. One of the primary challenges of the textile, polymer, and research sectors in today's world is sustainable flame retardancy. In order to overcome this, one of the latest and most promising methods for creating biomacromolecule-based flame retardancy is the engineering of environmentally friendly, clean polymeric substrates that guard against fire. Researchers have looked into several plant- and protein-based, starch, phytic acid, tannic acid, natural silica from rice husk, and fishbone powders in this direction [6] [7].

Flame retardancy of the cotton fabric is an important aspect for the end users as it mainly contains cellulosic backbone and is very much prone to catch up fire [8] [9] [10]. It was reported that owing to ignition of cellulosic materials, a great deal of human life has been lost and so also various expensive valuable materials

and documents have been damaged. In addition, afterglow, toxic smoke (mostly carbon monoxide) generated from the cellulosic material is also equally harmful and life-threatening to living beings [4].

Growing consumer awareness of health and environmental concerns has led to a growing demand for plant-based extracts in textile applications. This study used the exhaustion method to make aqueous extracts of onion peel, turmeric root, and pomegranate rind, which were then applied to cotton fabric separately (O, T, and P) and in combinations (PT, OT, PO, and POT). Investigations were conducted into the antibacterial activity, UV protection, antioxidant activity, and colour and fastness characteristics [11].

Organic polymeric materials of cellulosic backbone degrade on heating and produce volatile combustible products (levoglucosan, pyroglucosan, carbon monoxide, nitrous oxide, etc.) with high-temperature flaming (around 400°C) at a certain critical temperature, commonly known as pyrolysis temperature [12]. Fundamentally, pyrolysis of cellulose is an endothermic reaction involving absorption of heat energy. During combustion, with vigorous flaming, those ligno-cellulosic (like jute) textile materials also generate toxic life-killing smoke and dangerous afterglow [13]. Salts such as:

- 1) Alum (absorb moisture and acidic dehydration);
- 2) Borax-boric acid combination (glassy insulating layer of boron trioxide on fabric surface);
  - 3) Rochelle salt (absorb moisture and tartaric acid dehydration mechanism);
- 4) Tetrasodium pyrophosphate (phosphorylation of cellulose and treated jute fabric turns whitish) formulation;
- 5) Sodium silicate-based chemicals (treated fabric turned rough, harsh and slightly whitish);
- 6) Sulphur-based thiourea (treated jute fabric turned brownish and toxic odour of sulphur);
- 7) Ammonium sulfamate (release inert ammonia gas and sulphuric acid dehydration mechanism);
- 8) Diammonium phosphate and urea combination (phosphorylation of cellulose and liberation of ammonia restrict flammable gas formation) have been used successfully by researchers and jute industries to make it fire retardant. But, treated fabric exhibited afterglow and smoke (when add-on% is less) and poor physical properties and hand value (when add-on% is more) [12] [14]. This is a major drawback that needs attention.

In day-to-day life, vegetables and fruits are important consumable commodities. The consumption of such imperative commodities has expanded with the increase in world population [15]. Mass consumption has led to a higher generation of waste and by-products, creating disposal problem [16]. Vegetable and fruit by-products could be an important and profitable source of natural compounds [15]. Several research works have revealed that the generated compounds are potential sources of phenolic, natural acids, sugar, colours and minerals. Some of

these natural compounds exhibit bioactive functions such as anti-bacterial, anti-tumour, anti-fungal, anti-viral, anti-mutagenic and cardio-protective [16] [17]. Although some of the generated by-products can be considered unavoidable, others can be utilized in different domains, including pharmaceutical, food, textile and cosmetic industries [15] [18] [19]. These by-products may be a promising way to establish sustainable development and reduce environmental problems. Adoption of waste management procedures is necessary with the increasing valorisation of by-products and industries should develop innovative methods of recycling waste [19].

#### 2. Development of Fire Retardant Cotton Fabric

During the past 50 years research workers have attempted to evolve various flame retardant chemicals that could halt the combustion process of the cellulosic polymer and retard the burning rate of the finished cotton fabric. Phosphorus, nitrogen, sulphur-based and the halogen-based composition were effectively studied by various research workers in the past. Halogen-based chemicals have also been widely used considering their use as coating for the cellulosic textile material. However, due to environmental problems such as, halogen-based chemicals (release of dioxins and furanes) have been very much restricted in the market [20]. Considering this aspect, during the past five decades various flame retardant chemicals based on the composition of phosphorous, and nitrogen (particularly proban, pyrovatex-based chemicals with combination of melamine resin) came into existence in the market for imparting flame retardancy [3].

But, in this case also greater extent of add on percentage is necessary on the fabric surface and greater amounts of toxic chemicals have been used. Hence, achieving an eco-friendly, economical, and abundant intumescent-based flame retardant material from the wastage resource continues to pose a great problem and unmet needs. To this end, researchers have reported some of the protein and the plant-based natural biomolecules consist of different components (acid source, blowing agent and carbon source) process is much easier (can be apply in neutral pH) on the textile surface [13] [21] [22]. Research workers have effectively studied nature-based protein material such as DNA, casein, whey protein, sulphur-based hydrophobins (surface active proteins produced by filamentous fungi), and so on, for the cotton textile material.

According to studies conducted in 2014 [3] [22], DNA of natural protein indicates adequate flame retardancy by adding 10% - 15% higher concentrateson the fabric surface. Furthermore, the published document explicitly reports the flame retardancy mechanism [3]. Various plant-based biomolecules, such as extracts from spinach leaves, green coconut shells, and banana pseudostem sap, have previously been utilised to create cellulosic textile materials that are fire retardant [7] [13]. However, till date no published works have described in detail fire retardancy mechanism (by analysing compounds of volatile species, char, etc.)

of the cotton fabric imparted by plant-based biomolecules [7] [23].

Besides the flame retardancy, little study has been done on plant-based extracts in the multifunctional finishing (antimicrobial, naturally dyed) of the cotton textile materials. Hence, an incessant attempt was made to find a new plant-based natural biomolecules for imparting multifunctional finishing to the cotton fabric. Considering this aspect, Pomegranate Rind Extract (PRE) (original pH: 4.5), which is an abundantly available waste plant-based bio-molecule, offers a good option and has been well studied in producing combined multifunctional (fire retardant, antimicrobial, naturally dyed) cotton (cellulosic material) textile from a single treatment bath [10].

Pomegranate Rind Extract (PRE) as a fire retardant for jute (lingo-cellulosic material) fabric was studied. Researchers had applied the extract under various pH states and the detailed fire retardancy property, as well as the thermal kinetics confirmed. PRE-treated jute fabric exhibited satisfactory flame retardancy, despite that a great void was found regarding the mechanism relating to the fire retardancy action of PRE on the cellulosic substrate. Also, in detail forced combustion behaviour, volatile species analysis, and char analysis of the treated fabric have not been observed in this research [1] [6].

Moreover, analysis of the phytochemicals, and multi-functionality behaviour (dyeing, antimicrobial efficacy) of the treated fabric was explored. The PRE comprises various alkaloids having nitrogen, high molecular weight phenolic compounds, carbon sources, and so on, which cause the fire retardant behaviour of the treated cotton fabric by condensed phase, aromatised mechanism. The fabric so treated soon exhibits dehydration, having less flammable gas formation and more carbonaceous char mass generation in thermal decomposition. Extract and the treated fabric also contain phenolic-based acids, tannin, protein precipitation capability the various positive metal ions, and so on, which cause the antimicrobial action against both the Gram-positive and the Gramnegative bacteria. PRE-treated cotton fabric is constituted of beta-cyanin, coumarin-based phytochemicals that cause the natural coloured effect on the treated cotton fabric. Hence, the present context reveals the multifunctional efficiency (fire retardancy, antimicrobial, mordanting and natural dyeing efficacy) of the waste Pomegranate Rind Extract (PRE) on the cotton textiles in details [10] [24].

#### **Technical Details**

In general, the materials used for the study purpose are mentioned below [10]:

Fabric	Natural Extra	ct Commercial Dye	Temp.	M:L Ratio	Rh & Time
Plain woven bleached cellulosic cotton fabric	Pomegranate rind	Acid Red dye (anionic dye) Methylene Blue (cationic dye)	90°C to 110°C	1:20	Rh: 65% Time: 24 hrs

Through this experiment, the parameters and characterisation that were tested [23]:

Parameters	Characterisation
	Thermo-gravimetric analysis,
	Fourier Transform Infrared spectroscopy,
	Antimicrobial efficacy of treated fabric,
Limiting oxygen index	Colour strength of the treated cotton fabric,
	Phytochemical analysis of the pomegranate
	rind extracts, Tensile strength test of the treated
	fabric.
Total heat release	Fourier Transform Infrared spectroscopy.
Heat release rate	Scanning electron microscopy.
Time to ignition	Gas chromatography-mass spectroscopy.
Time to flame in and flame out	Antimicrobial efficacy of treated fabric.
Maximum average rate of heat emission	Colour strength of the treated cotton fabric.
Peak heat release rate	Phytochemical analysis of the pomegranate
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	Tensile strength test of the treated fabric.

# 3. PRE Extracts and Its Composition

Pomegranate Rind Extract (PRE) has been observed in order to determine its composition and the relevant spectra that were obtained. The chemical substance included in the PRE extract can be identified by examining its complete mass spectrum. Chromatographic peaks have been found at three distinct retention times during the experiment. The molecule 1,3-amino guanidine, 1,4-dioxane-2,5-dione, 3-amino-1,2-propanol, 1-propanol, and 2-amino are yielded by the chromatogram's peaks corresponding to the various molecular weight components included in the PRE peak observed at 10.69 s. Compounds such as tetrahydro-4H-pyran-4-ol, propanoic acid, 2-hydroxy, aziridin, and 1-(methoxy methyl) are responsible for the peak that was seen at 11.35 s. Due to the presence of 3-methyloxazolidine, 1,4-dioxane-2,5-dione, 3,6-dimethyl, and 1,4-dioxane-2,5dione, 3,6-dimethyl-(3S CIS), a large peak was seen at 12.46 s. The majority of the constituents are alkaloids that include nitrogen and have an aromatic basis. The PRE extract contains several high molecular weight pro-carcinogenic chemicals. However, the quantity of the material is lower. The mass spectra curves unequivocally demonstrate that the majority of the material contained in the PRE has a molecular weight in the region of 17 to 100. The PRE extract has higher concentrations of the following materials: hydroxyl amine, hydrazine methyl, 1,3-amino guanidine, glycidol, propanoic acid, alanine, carbamic acid, 5-methyloxadolizine, aziridine, etc. Most of these components are composed of nitrogen-containing alkaloids, amino acids, and other substances. However, a few major molecules have been identified in the extract, albeit in smaller concentrations [23].

# 4. Thermo-Oxidative Decomposition of PRE and the Treated Cotton Fabric

Study of thermo-oxidative decomposition of the dried Pomegranate Rind Extract (PRE), control and the specified concentration of "PRE" treated cotton fabric has been determined. PRE extract reveals major mass loss at a particular temperature to the degradation of the nitrogen-containing blowing agent (amino guanidine, 1,3-di-amino guanidine, piperidine, etc., as found in the GC-MS analysis). There has been the liberation of non-combustible gases in the event of the blowing agent present in the PRE extract. This gas liberation phenomenon is an endothermic (heat absorption) process and has been found in the Differential Scanning Calorimetric (DSC) analysis of the PRE extract at a particular temperature. The next weight loss of the PRE extract happened at a particular temperature, due to the oxidation of the carbonaceous char mass to carbon monoxide, carbon dioxide, and so on [6] [25].

Connected to the textile material, control cotton textile showed one major mass loss stage at a particular temperature, where more than 70% mass loss was observed. The temperature is generally termed as pyrolysis temperature where cellulosic cotton polymer has been depolymerized by the generation of aliphatic char and flammable gases such as levoglucosan, carbon monoxide, pyroglucosan, and so on. Due to the depolymerisation of the cellulose polymer at higher temperature only 5% char mass remains and the aliphatic char has been converted into the aromatic char mass [26] [27] [28]. On the other hand, PRE treated cotton fabric exhibits mass loss in three phases. The initial loss of mass (5%) happened at a particular temperature, possibly owing to the evaporation of the attached water molecule in the PRE treated fabric. 2nd mass loss peak has been observed at a particular temperature (50°C lower than the control cotton cellulose) where only 25% mass loss has been noticed. It provides clarification that the PRE treatment reduced the decomposition temperature and enabled the dehydration of cellulose (more water liberation which has been proved by the GC-MS analysis of the pyrolysate of the treated fabric) at a particular temperature and limits the flammable gas formation (also has been proven from the GC-MS analysis of the pyrolysate of PRE treated fabric) and raise aliphatic char mass generation. Indeed PRE treatment helps to catalyse the dehydration of the cellulosic polymer by blocking the reactive primary-OH group of cellulose (autocrosslinking phenomena) [29].

The primary hydroxyl group of the gluco pyranose structure of cellulose has mainly caused the formation of flammable gas such as levoglucosan. At a certain temperature, *i.e.* at pyrolysis temperature, the control fabric reveals 22% remaining mass; while the PRE treated fabric retains 50% mass. The process has been strengthened with the dehydration tendency of the PRE treated cellulose polymer. At 450°C, the treated fabric exhibits strong peak that implies the aromatisation of the char mass [8].

The concerned peak for the control fabric is significantly lower. At an elevated

temperature of 450 degrees Celsius, treated cotton cloth lost just 60% of its mass in the air atmosphere compared to 80% for the control cotton fabric. Yet, as char products have been oxidised at higher temperatures in an environment of air, there has not been much of a difference in residual mass at 500 degrees Celsius. The control and PRE treated cotton fabrics went through isothermal TG analysis at 350 degrees Celsius (pyrolysis temperature), for 20 minutes in an environment of air. It confirmed that after 15 minutes of thermo-oxidative breakdown, 350 degrees Celsius had been attained from 50 degrees Celsius (which is due to temperature gradient of 20 C/min) [29].

A steady temperature of 350 C was subsequently maintained for 20 minutes. The reduction in weight of PRE treated cotton is slower once the temperature gets to 350 C than it is for control cotton fabric, according to the results. Ultimately, after reaching 350 C, control cotton lost 50% (90% to 40%) of its weight within 3 minutes, whereas treated cotton cloth only lost 25% (75% to 50%) at the same time. It strongly supports the PRE-Treated cellulosic material's weight and temperature stabilization phenomenon.

In the nitrogen atmosphere, thermo-gravimetry of the control and PRE-Treated (400 g/L) cotton fabric was also completed. It primarily denotes the behaviour of pyrolysis and the amount of char material [8].

The cotton cloth under control degrades in two steps, according to the TG curve. The cotton fabric which constitutes of theglucosyl units were broken down to char in the first phase at a lower temperature and thereafter depolymerized into volatile combustible compounds a higher temperature range in the second step [27] [29]. Conversely, treated cotton fabric indicated two minor deterioration phases at lower temperatures (under 300 C), which may be related to the degradation caused by particular PRE coating components. According to the data shown in the curve, the major mass loss peak of the treated cotton fabric (D) is almost 60 C lower than that of the control cotton fabric [29].

It indicates a speeding up of the pyrolysis process and a decrease in the production of combustible volatiles for the treated fabric, which causes the amount of char mass remaining at 450 C to be around 20% more than that of the control cotton fabric. Additionally, a larger portion of the char mass generation can also be understood by both TG curves. The PRE treated cotton fabrics' flammability property. The findings of vertical flammability test and the Limiting Oxygen Index (LOI) value examinations of control and PRE treated cotton fabrics have been determined [29].

The vertical flammability test proved that the control cotton fabric (250 mm  $\times$  40 mm) burned in one minute (burning rate: 166 mm/min) with flame (60 s) and afterglow (30 s) while PRE treated cotton fabric (treated with various concentrations, 100, 200, and 400 g/L) of the same dimension burned in 7 12 and 17 minutes (burning rate: 31, 21, and 14 mm/min), respectively. The vertical burning behaviour of the control and the 400 g/L PRE treated fabric have been obtained in order to achieve this. In fact, regardless of the concentrations utilised in the experiment. The PRE-treatment demonstrated possible flame-stopping behav-

iour because a significant portion of the treated samples burned slowly and only produced afterglow. As the PRE treatment's concentration was increased, the afterglow's intensity and pace of propagation reduced. It might be related to the PRE's chemical makeup and the steady improvement of the add-on% (18%, 24%, and 35%) of the treated cotton fabric with increasing PRE treatment concentration [3] [30].

In relation to the analysis of the oxygen consumed, control cotton fabrics had a Limiting Oxygen Index (LOI) value of 18.5% while PRE treated fabrics (100, 200, and 400 g/L) had LOI values of 24%, 28%, and 32%, respectively (Note: LOI is the amount of oxygen used in the combination of oxygen and nitrogen for only burning the sample). The LOI value is greater than or equivalent to 26% for a common fire retardant textile material [28].

According to the study, it has been found that the treated sample's oxygen index value is 32, which is greater than the LOI index of any cotton fabric compared to the control sample's oxygen index of 18, which is cotton fabric [31]. In fact, it depicts how the treated and control cotton fabrics decompose by thermo-oxidation. The treated sample failed the vertical flammability test (after flame: nil). Only the afterglow has been seen, and it spreads very slowly. The flammability assays indicated above confirmed that the chemicals found in the PRE extracts are the only ones responsible for the treated material's observed flame retardancy effect. The PRE extract (pH 4.5) contains diverse blowing agents like aminoguanidine, nitrogen-based carbamic acid, ammonium salt, and hexacontanoic acid, asparagines, hydrazine, ethanamine, piperidine, aminoguanidine, 1,3-di-ammino guanidine, and other blowing agents are examples [30].

These nitrogen-containing alkaloid-based bio-molecules that are ejected (rise in volume) upon heating might be helping to improve the flame retardancy of the treated cotton fabric. According to research, the salt of guanidine, such as guanidine sulphate and guanidine phosphate, has an intumescent flame retardant effect on polymeric materials. Additionally, PRE extract includes a small number of carbon-containing substances such carbonic dihydrazide, nonhexacontanoic acid (an aromatic polyphenolic structure), other aromatic compounds with high molecular weights, and sugar-based materials. These biomolecules with carbon-based structures aid in the production of greater char mass at higher temperatures [20] [30].

These aforementioned bio-molecules are the sources of nitrogen found in both the PRE extract and the fabric surface, as seen from the EDX analysis. It's possible that not all of the nitrogen-based biomolecules found in the PRE extract are also found on the treated fabric surface. PRE treated cloth has been washed in methanol solution for further confirmation and the extracted washed solution has been analysed by GC-MS (Gas Chromatography-Mass Spectrometry). It was discovered that the solution contained carbamic acid, amminoguanidine, asparagine-like nitrogenous protein material, monoammonium salt (perhaps ammonium nitrate), and sulphur-based morpholinothiophenol and thiodiazole. Based on the surface morphology analysis, these substances (the acid producing agent

and the blowing agent) created a consistent thick coated layer on the cotton fabric surface [23] [30].

# 5. Kjeldahl Analysis of the PRE Treated Fabric

As part of on-going research efforts in this regard, nitrogen analysis using the Kjeldahl method has been completed for both the control and treated cotton fibres. While the high concentrated (400 g/L PRE extract) PRE treated fabric showed 10.2% moisture content and the presence of 1.5% nitrogen with protein content of 9.4% (possibly from the nitrogen containing blowing agent like oxy-bisalanine, asparagines, aminoguanidine, etc. observed from the GC-MS analysis), control cotton fabric had a moisture content of 8.2%, registered 0.35% nitrogen, and 2.5% protein. The findings of the EDX study likewise demonstrated that the treated fabric surface had increased nitrogen content (3.33 weight%) compared to the control cotton fabric (0.756 weight%) analysis of volatile species and char morphology. A GC-MS analysis of the volatile species (by-products left after burning or pyrolysis) of the control and treated cotton fabrics was conducted to better understand the mechanism underlying the fire retardancy imparted by the PRE biomolecule [32] [33]. It was discovered that the control cotton fabric's volatile species primarily contained phenolic compounds, various aliphatic and benzoid aromatic-based saturated and unsaturated hydrocarbons (m/z = 41, 50, 51, 53, 55, 57, 58, 64, 65, 67, 69, 70, 71, 80, 81, 91), terpenic compounds (m/z = 105, 109, 107, 108, 177, 193, m/z = 73, 74) [32].

# 6. Dyeing with the Anionic and Cationic Dye

The dye medium used in the acid dye (1% depth) and basic dye (0.1% depth) and PRE dye was used in the study. The fabric dyed with anionic acid dye from the PRE medium showed a darker tint than the fabric dyed with water, according to the results. This is a result of PRE's ability to effectively mordant anionic dyes. Anionic acid dye molecules were captured by the zeta potential of cellulose in solution, which promoted the deposition of positively charged metal ions from the PRE onto the fabric's surface. As a result, compared to cotton dyed with water, the treated cloth had a deeper, redder, and more yellow appearance. Increased yellowness (from natural coumarines and betacyanines), redness (from more attached anionic dye), and overall shade depth were all accentuated by this impact, which was more noticeable with the concentrated 200 g/L PRC medium. The reason for tonal changes could be that at higher dyeing temperatures, colour groups in the PRC extract connect to the fabric surface through van der Waals forces, changing the shade. The plant extract was also subjected to a thorough phytochemical study by the researchers [34].

Phytochemical analysis of PRC extract reveals natural colorants. Basic-dyed cotton from PRC appears lighter and differently shaded than water-dyed cotton due to positively charged metal ions in the treatment liquid depositing on the negatively charged fabric surface. This results in a greenish tone, contrasting with

the bluish tone of water-dyed fabric. The yellow hue is attributed to light positive ions deposition on the negatively charged fabric. Interaction between negatively charged cotton surface and positively charged basic dye molecules produces green colour in PRC-treated fabric, unlike the blue shade in water-dyed fabric. Researchers used Methylene Blue and Acid Red dyes on paper, finding no negative effects of the popular and attractive Methylene Blue colour [35].

The skin application of the dye at 2000 mg/kg showed no observed toxic effects. However, further research is needed for the acid red skin irritation test. Regarding fire resistance, cotton fabric painted with the PRC substrate exhibited lower thermal stability, burning and glowing on contact with honey for 6-7 seconds, compared to pre-treated fabric. The anionic and cationic stains on the fabric surface did not improve flame performance, and the Bepaint patch interfered with the flame retardant effect of PRC alone. Despite lower thermal resistance, the PRC-dyed fabric demonstrated greater colour depth and improved flame retardancy compared to water-based cotton. Bacterial analysis revealed significant antibacterial activity against both gram-positive and gram-negative bacteria, indicating potential antimicrobial efficacy in PRE-Treatedtissues [5] [36] [37].

The fabric treated with PRC at pH 7.0 exhibited significantly lower bacterial growth against both gram-positive (S. aureus) and gram-negative (E. coli) bacteria compared to untreated cotton fabric. The treated fabric independently demonstrated a bacterial reduction of 97.5% and 97.7% against S. aureus and E. coli, respectively, while the control fabric showed no bacterial reduction (0%) in either case. Positive metal ions, guanidium ion, tannin, and phenolic-based acids found in PRC extract contribute to its antibacterial properties by forming complexes that harm the negatively charged bacterial cell wall. The presence of ellagitannins and secondary metabolites, particularly punicalagin, in the extract plays a crucial role in its antibacterial activity. Tannin aids in protein precipitation and leakage formation in bacterial cell walls, leading to cell death. Alcoholic pomegranate rind extract, after 28 hours of incubation, significantly reduces bacterial cell growth and protein content in cell walls. The polyphenols in PRC contribute to protein precipitation and microbial enzyme inhibition, with tannic acid destroying bacterial cell walls. Coumarins in the plant extract negatively impact Gram-positive bacteria, while the phytochemical study indicates that PRC contains various compounds contributing to its antibacterial properties, including alkaloids revealed by GC-MS analysis [5] [36].

#### 7. Technical Characteristics

The tensile strength of the untreated cotton fabric and the treated cotton fabric has been determined. Tensile strength of the control cotton textiles utilised in the experiment was 574 N with an extension of 11.5%. Contrarily, cotton fabric treated with PRE extract exhibits an average tensile strength of 693 N (9.2% higher than the control fabric; 8.7% higher than the control fabric). The increased add-on% of the PRE after the treatment may be the cause of the higher tensile

strength of the PRE treated cotton fabric. The addition of softeners to the PRE formulations can reduce the stiffness of the treated fabric. The treatment may enhance the fabric's hand quality by making it softer. In our research lab, additional study is being conducted in this approach [5] [36].

# 8. Assessment of Flame Retardant Property in Jute

For imparting flame retardant properties to cellulosic material, halogen-based back coating has been widely used. However, treatment is toxic because of the liberation of dioxins, furanes, etc. and banned in European countries [37]. Under such a situation, continued efforts have been taken by research workers over the past centuryin order to render the jute textile material fire retardant. Among different chemicals used, phosphorous-based flame retardants along with nitrogenous compounds are the most effectiveformulations reported so far due to their synergistic flame retardant action and condensed phase (change in pyrolysis path of cellulose and char formation) mechanism of fire retardancy [38].

The market has seen the emergence of several flame-retardant chemicals based on the composition of phosphorous and nitrogen during the past fifty years, particularly those based on tetrakids-hydroxymethyl salt and N-alkyl phospopropionamide. Although the textile fabric loses its tensile strength and stiffens, these commercially acceptable fire retardant formulations must be placed in an unfavourable acidic environment for cross-linking dehydration and more add-on% (usually more than 15%) is needed for flame retardancy. In addition, this type of treatment is costly because it requires using more chemicals and using a hightemperature curing method [39]. Consequently, ongoing research has been done to find a less expensive, environmentally friendly alternative to add fire retardancy to textile materials in order to overcome the aforementioned problems in this field. In order to do this, researchers have documented the usage of many bio-macromolecules in the very recent past, including plant-based casein, whey protein, hydrophobin, DNA, green coconut shell extract, and banana pseudo stem sap. Pomegranate Rind Extract (PRE) has been discovered to be a respectable substitute for the traditional flame retardant chemicals as a result of on-going research into bio-molecules for fire retardancy [1] [7] [8] [13] [15] [21].

This was the first attempt to investigate pomegranate rind extract as a fire retardant for any material. It appears that PRE comprises a variety of aromatic phenolic groups that are deposited on the surface of the fabric and operate as an intumescent heat barrier and flame propagation barrier (expand in volume upon heating, foamy char mass generation). Additionally, PRE contains nitrogen-containing groups that could create azo-compounds and aid in the development of char [10] [40].

Additionally, it might aid in the release of non-combustible gases such as nitrogen, carbon dioxide, and ammonia. A variety of inorganic metallic salts and metallic oxides are present in PRE and help to catalyse the dehydration phenomenon with increased char production. Ligno-cellulosic material that had under-

gone PRE treatment collectively had shown condensed phase mode fire retardancy. Hence, the article describes a novel technique for enhancing the fire resistance of lingo-cellulosic materials using PRE (waste bio-macromolecules). The systematic impact of varying application pH on the flammability behaviour of treated materials—which was evaluated by the limiting oxygen index, the vertical flammability test, etc.—has been shown. By examining the PRE's elemental composition, degradation behaviour, char form, and heat release phenomena, the study also explains in detail the mechanisms underlying fire retardancy [34] [38].

# 9. Studies on Flammability

The study involved treating lignocellulosic jute fabrics with Pomegranate Rind Extract (PRE) at different pH levels. Flammability testing, including Limiting Oxygen Index (LOI) and vertical flammability, revealed enhanced thermal stability in all treated fabrics compared to the control, which burned entirely within 180 seconds. Regardless of pH, PRE-treated fabrics showed no ignition during testing, with afterglow limited to the middle of the fabric. Interestingly, increasing the application pH of PRE reduced afterglow intensity and propagation rate, along with a significant decrease in smoke generation. The sample treated at pH 10 displayed self-extinguishing behavior and a lower burning rate than those treated at pH 4.5 and 7. The afterglow in pH 10 treatment was minimal, and the fabric demonstrated complete self-extinguishment. pH 4.5 treatment showed a more scattered, bright, and prolonged afterglow compared to pH 7, with a higher burning rate. pH 7 treatment exhibited a 92.7% lower burning rate than the control, with a regular and shorter afterglow [29] [38].

The proposed model suggested that the control fabric's afterglow followed a distributed path at high speed, while pH 4.5 treatment showed a slower, more predictable afterglow with increased char mass. The study concluded that PRE treatment, especially at higher pH, significantly improved jute fabric's flame resistance, reducing afterglow, burning rate, and smoke generation [8].

#### 10. Elements and Surface Morphology

SEM studies were conducted to examine the surface morphology of dried Pomegranate Rind Extract (PRE)-treated, control, and untreated jute materials. The burnt PRE extract exhibited a rough, foamy structure with vacuum bubbles, attributed to phosphorus and nitrogen content. EDX analysis (Energy-Dispersive X-ray) confirmed the presence of nitrogen, phosphorous, and other elements on the extract's surface, supporting its intumescent behaviour. Control jute fibers had a smooth surface, while PRE-Treated samples at pH 4.5 (Sample B) displayed a thick, smooth coating. Sample C (PRE-treated fabric at pH 10) showed a thicker and rougher surface, indicating increased PRE add-on with alkali. Char morphology after burning revealed well-preserved structural integrity in PRE-Treated samples, contrasting with deformed structures in control burnt jute. The char

morphology suggested an intumescent category of fire retardant action [20] [41].

The biosynthesis of silver nano-conjugates using agro-waste has drawn the attention of researchers in recent years due to eco-friendly and low-cost methods. A comparative study on various techniques (sunlight, probe ultra-sonication, and microwave irradiation) of silver-bio-nano-conjugate production was carried out using lyophilized pomegranate peel polyphenols. Probe ultrasonic-assisted-silver nano-conjugates showed good antibacterial activity against *S. aureus* and *E. coli* as compared to microwave-assisted silver nano-conjugates [41].

# 11. Thermal Stability

The dried Pomegranate Rind Extract (PRE) exhibited an initial weight loss of 15% - 20% below 100°C due to moisture evaporation, with a subsequent char mass of 55% at 350°C. The presence of nitrogen, chlorine, potassium, and aromatic phenolic rings contributed to thermal stability and flame inhibition. A second peak at 500°C indicated the transition from aromatic to aliphatic char. The thermal degradation of jute materials (control, PRE-treated, untreated) revealed initial moisture removal, hemicellulose decomposition, cellulose pyrolysis, and lignin transformation into phenols. PRE-treated jute fabrics showed a slower weight loss rate and higher char mass production. Higher application pH increased char mass and shifted the cellulose degradation peak to lower temperatures. Notably, PRE treatment enhanced dehydration, altered pyrolysis paths, and improved char mass generation, suggesting condensed phase fire retardancy [23].

Isothermal TG at 350°C for 20 minutes revealed that the dried pomegranate rind extract (D) had a lower rate of weight loss (4.9%) compared to the control jute polymer (37.2%). The PRE-treated jute fabric at pH 7 (B) lost 11.25% weight, while pH 10 treatment (C) showed only 5.25% mass loss after 20 minutes. The TG curves in air atmosphere demonstrated four peaks in the control fabric, with hemicellulose degradation at 180°C, cellulose structure breakdown at 260°C and 330°C, and lignin decomposition at 420°C. pH 4.5 treatment had three peaks, lacking the 180°C peak due to PRE's reactivity with amorphous hemicellulose. Alkaline pH treatment displayed one peak at 300°C, eliminating hemicellulose peaks, retaining over 30% char mass at 500°C, and exhibiting a self-extinguishing effect. Overall, PRE treatment demonstrated effective fire retardancy with altered weight loss patterns and enhanced char formation [42].

The DSC (Differential Scanning Calorimetry) curves for untreated and treated jute samples up to 300°C indicate initial endotherm below 100°C, attributed to water evaporation. Increasing treatment pH correlates with higher heat absorption, suggesting elevated moisture content in the treated material. Phenolic groups, inorganic salts, and metal oxides in the PRE extract contribute to moisture absorption and fire retardancy. Higher PRE add-on% increases moisture absorption intensity. Control jute exhibits abrupt endothermic trends (150°C - 300°C) indicating pyrolysis, while treated fabric shows decreased endothermic intensity

and initiates an exothermic reaction around 250°C. Higher pH treatments exhibit intensified exotherms, signifying enhanced dehydration processes and increased char mass production [23] [42].

#### 12. FTIR Evaluation

The dried PRE extract's FTIR (Fourier Transform Infrared Spectroscopy) measurement displayed a significant peak at about 3271 cm<sup>-1</sup>, which could be attributed to the phenolic -OH group's stretching vibration. The region beneath this peak indicates the hydrogen bonds that exist within individual polyhydroxy aromatic compounds. Peak at 1608 cm<sup>-1</sup> was noticed and is connected to the aromatic rings in PRE (Al-Rawahi 2014). Another prominent peak can be attributed to the -O-C- aliphatic stretching vibration at 1045 cm<sup>-1</sup>. A small peak at 1701 cm<sup>-1</sup> may be attributed to the N-containing groups' C=O vibration. The C=C and C=N stretching modes may be used to explain the weak peak found at 1328 cm<sup>-1</sup>. Mohammed et al. most recently reported that this specific peak is the extract's nitro group's distinctive characteristic. Additionally, a few minor peaks have been seen below 1000 cm<sup>-1</sup>, which could indicate the existence of certain alkenes. The P-O-C stretching vibration and the phosphate motion, respectively, may be represented by the peaks that are also present at 1118 and 963 cm<sup>-1</sup> (small peak). In contrast to the control jute, the FTIR curves of the PRE treated samples do not exhibit any appreciable change in the peak intensity. Additionally, the treated jute sample's FTIR curves show no discernible additional peak. It can be as a result of the lower PRE addition percentage on the treated sample surface. The similar result was seen by a research team after applying banana pseudostem sap to jute fabric at various pH levels [40] [42].

#### 13. PRE-Based Finish

Energy Dispersive X-ray (EDX) analysis of the Pomegranate Rind Extract (PRE) indicates predominant elements such as oxygen, carbon, nitrogen, potassium, and chlorine, with traces of copper, zinc, phosphorous, sodium, aluminium, and silicon. Nitrogen, a recognized flame retardant, is present in the PRE-Treated material (pH 10) but in reduced concentrations. GC-MS analysis reveals nitrogen-based compounds in the PRE extract, contributing to flame retardancy in the treated cotton fabric. Carbon-containing substances in the PRE aid in aromatization, increasing insulating char mass at higher temperatures. Mass spectroscopy analysis confirms nitrogen-containing compounds in the PRE, supporting its reported thermal stability. Nitrogen and phosphorous in the treated fabric may limit combustible gas generation. Positive metal ions from the EDX analysis enhance the fabric's heat resistance by altering the pyrolysis path. The presence of aromatic phenolic groups in the PRE contributes to the treated fabric's thermal stability, as confirmed by Fourier transform infrared analysis (FTIR) and Gas Chromatography-Mass Spectrometry (GC-MS) [24] [31] [43].

Chemicals in the Pomegranate Rind Extract (PRE), including phosphorous,

nitrogen, metallic salts, oxides, and aromatic phenolic groups, create an intumescent coating on the treated fabric's surface, acting as a heat barrier. This coating, formed by high molecular weight phenolic aromatic rings, enhances charring and smoulderingbehaviours. In the treated fabric, the elements that were found are Nitrogen and Phosphorous. This helps in phosphorylating cellulose and limiting combustible gas generation. Positive metal ions, determined by EDX analysis, may be present as salts or oxides, altering the pyrolysis path to improve heat resistance. DSC curves indicate increased moisture content due to salt molecules in the treated sample. Aromatic phenolic groups contribute to the thermal stability of the treated fabric. The intumescent coating formed by the PRE's chemicals serves as a heat insulator, suggesting a role in flame retardancy and char promotion. Studies on similar phenolic monomers support the idea that phenolic compounds contribute to flame retardancy by promoting char formation. Overall, the multifaceted composition of the PRE induces intumescent behaviour in the treated fabric, providing enhanced fire resistance [40] [44].

Nitrogen and phosphorus in the Pomegranate Rind Extract (PRE) treated fabric potentially aid in phosphorylating cellulose, limiting combustible gas generation, and altering the pyrolysis path. Positive metal ions, identified by EDX analysis, may be present as salts or oxides, enhancing heat resistance. Aromatic phenolic groups contribute to thermal stability by forming a high molecular weight phenolic aromatic ring coating on the fabric surface. These chemicals collectively create an intumescent coating, acting as a heat barrier. The intumescent phenomenon involves volume expansion, foamy character, and altered physico-chemical properties. The application of PRE in an alkaline environment (pH 10) increases thermal stability, targeting the amorphous hemicellulose portion, enhancing PRE penetration, and encouraging char production. Alkali-treated jute materials exhibit increased thermal stability, supported by LOI values and vertical flammability tests. Overall, the combination of chemical elements in PRE and alkali treatment enhances fire resistance in the treated fabric [1] [42].

To produce fire-resistant cotton textiles, a combination of Sodium Lignin Sulfonate (SLS) and waste Pomegranate Rind Extract (PRE) has been investigated. The experimental results showed that the cotton fabric sample treated with only PRE and one treated with SLS was completely burned with flame and afterglow, while the cotton fabric sample treated with PRE combined with three different concentrations of SLS (5%, 7%, and 10%) demonstrated a proper self-extinguishing effect with measurable char length. The combination of nitrogen, sulphur (which forms nonflammable sulphur dioxide when burning), and the aromatization properties of the polyphenolic groups in the PRE and SLS fabrics results in fire retardancy. Commercial applications for treated fabric include the creation of non-permanent constructions, home textile items, curtains, sofa covers, and other items [6] [40].

#### 14. Conclusion

Pomegranate Rind Extract (PRE) emerges as a sustainable and effective flame sup-

pressant for cotton substrates, containing acid sources, protein-based blowing agents, carbon sources, and nitrogen-containing bases. The treated fabric finds applications in non-permanent outdoor structures, such as tents and stalls, and home textiles like sofa covers and curtains. The technology is adaptable for small-scale handloom sectors, reducing the need for frequent washing. PRE exhibits strong potential as a green fire retardant alternative for lingo-cellulosic textiles. While studies on polyamide materials demonstrate enhanced dye-ability and fastness properties with mordant use, further research is needed to evaluate the durability of antibacterial activity post-washing and light exposure.

#### **Conflicts of Interest**

The authors declare no conflicts of interest regarding the publication of this paper.

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