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Fabrication and characterization of flame-retardant polyester fabric with a Polyurethane coating and Ammonium poly Phosphate and investigation of its mechanical and flame retardancy properties

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The advancement of fireproof fibers into diverse applications highlights the importance of developing effective flame-retardant textiles. These materials not only enhance safety by reducing flammability but also meet specific needs in challenging environments, such as High-performance operations. Understanding the mechanisms of fire retardancy and the methods of textile fabrication is essential for improving safety standards across various industries. This study is focused on fabrication and characterizations of a flame-retardant fabric based on Polyester and Polyurethane coating. Additionally, Ammonium Poly Phosphate (APP) was added to the Polyurethane coating as the flame-retardant component. The results showed that the introduction of Ammonium Poly Phosphate in the coating layer not only enhances the mechanical strength of final fabric but also has a significant influence on surface morphology which further affected the hydrophobicity and oil repellency of the final sample. Moreover, after inclusion of APP, the Limiting Oxygen Index reached 35%, char length was calculated around 16 cm and self-extinguish time was decreased to almost 0s, representing an enhancement in flame-retardancy properties. Our findings in this research prove that our final flame-retardant sample can be an ideal candidate for high-performance flame-retardant applications.

Keywords: Flame-retardant-Polyester-Polyurethane Coating-Ammonium poly Phosphate-Spraying

1. Introduction

In today's world, the application of fireproof fibers extends beyond traditional construction and industrial sectors. These fibers are now widely used in protective clothing, medical applications, specialized coatings, and even everyday garments[1]. When a fabric burns, they can release toxic substances and gases, posing severe health risks[2]. This has made the development of fireproof or flame-retardant textiles essential. Flame-retardant fabrics are either inherently flame-resistant or treated to resist ignition and self-extinguish once the flame source is removed[3]. Although they are not completely fireproof, they can char instead of igniting rapidly, forming a barrier that limits oxygen access and slows combustion. These textiles play a vital role in ensuring safety across various applications[1-3].

Key performance indicators of flame-retardant textiles include burning speed, Limiting Oxygen Index (LOI), and self-extinguishing behavior. Burning speed refers to how quickly a fabric burns when exposed to a direct flame. Shorter burn times and minimal damage indicate better flame-retardant performance[4]. LOI measures the minimum concentration of oxygen required to sustain combustion[5]. Higher LOI values signify better flame resistance. For instance, materials with LOI > 35% (e.g., carbon fibers) are considered non-flammable, while those with LOI < 20% (e.g., cotton) are easily ignitable. Self-extinguishment, the ability of a material to cease burning after the flame is removed, is critical for evaluating safety in textiles used in clothing and upholstery[4,5].

The fabrication of flame-retardant textiles involves chemical treatments, mechanical processes, and strict adherence to safety standards. It typically includes the use of high-strength, heat-resistant polymer fibers and methods that enhance the durability of the material. Flame-retardant clothing often combines natural fibers, which are skin-compatible and flexible, with synthetic fibers and fire-retardant additives to improve mechanical and thermal resistance.

The flame-retardant mechanism often relies on the formation of an anti-fire layer that lowers the

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material's temperature below its pyrolysis threshold[6]. This layer can release water vapor and form a protective coating that prevents flammability[7]. Based on their burning behavior, fibers are categorized as flammable, combustible, flame-resistant, or non-combustible. Flame-retardant fibers typically have an LOI above 25%[6].

Essential features of flame-retardant fibers include resistance to drying, cleaning, and repeated washing, as well as lightness, comfort, breathability, and high mechanical strength[8]. Depending on the application, they may also offer waterproof, antifungal, and antibacterial properties. Non-woven fabrics are particularly well-suited for high-performance garments due to their cost-effectiveness and material flexibility[8].

Polyester is among the most commonly used synthetic fibers in flame-retardant textiles[8]. It is produced by polymerizing ethylene glycol with terephthalic acid, resulting in a durable fabric with high resistance and easy processability[9]. However, polyester is inherently flammable, and when ignited, it tends to melt and drip. To counter this, structural modifications are necessary[9].

Creating flame-retardant polyester typically involves applying flame-retardant chemicals or incorporating these agents during fiber production[10]. The most effective approach is to treat polyester with flame-retardant additives through chemical processes such as dipping, spraying, or coating[10]. Dipping immerses the fabric in a flame-retardant solution; spraying distributes the solution evenly over the surface; coating forms a protective layer that restricts oxygen and enhances self-extinguishment[10,11]. These treatments may require reapplication after washing or wear[10,11].

Polyurethane (PU) is a widely used coating material for polyester fabrics due to its excellent flame-retardant capabilities. PU coatings form a barrier that limits ignition and slows flame spread[12]. When applied to polyester, PU enables the fabric to self-extinguish and reduces the release of toxic gases during combustion[13]. Toluene diisocyanate (TDI), a component of PU, plays a crucial role in forming cross-linked networks that improve mechanical properties and alter surface energy, further enhancing fire resistance[12,13].

The addition of flame-retardant additives like ammonium polyphosphate (APP) to PU formulations boosts thermal stability and flame performance[14]. APP promotes the formation of a char layer that insulates the underlying material from heat and flames[15]. Phosphorus-based compounds, including APP, are among the most effective flame-retardant additives. These compounds decompose to form phosphoric acid, which facilitates char formation, helping to limit flame spread[14]. They are also more environmentally friendly, and less toxic compared to halogenated flame retardants[16].

A major challenge in coating processes is maintaining fabric flexibility[16,17]. Therefore, selecting suitable coating materials and additives tailored to specific requirements, such as antistatic, antibacterial, or antifungal properties, is crucial[17]. Choosing the most efficient method and materials while considering economic and availability factors significantly impacts the quality and longevity of flame-retardant textiles[17].

In this research, woven polyester fibers are used for manufacturing flame-retardant clothing due to their favorable properties, cost-effectiveness, and availability. Phosphorus-based additives like APP are chosen over halogenated compounds for their lower toxicity and environmental impact. The spraying method is employed for applying the flame-retardant treatment, offering an efficient and practical approach to enhancing fabric safety Fig. 1.

2. Materials and Methods

To make flame retardant fabric from polyester. First, 100 parts per hundred (phr) of Polyurethane 691 (Interplast ANEX, Turkey)was dissolved in 15 phr of Iso propanol (Mojalali, Iran)and 15 phr of Toluene (Mojalali, Iran). Then,30% wt. (of polymer) Ammonium Poly Phosphate (149.08 g/mol) (Parsa

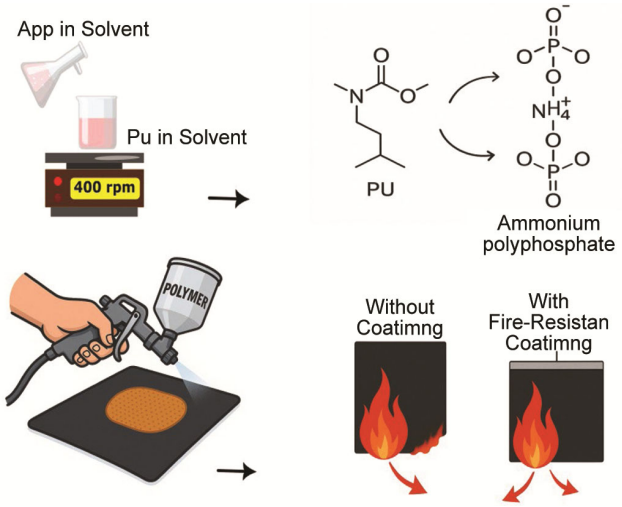

Fig. 1 — Graphical abstract of the fabrication process

Polymer, Iran) powder is dissolved and sonicated in the solvent. In this project the flame-retardant fabric was developed based on coating method to decrease the fabrication process cost and increase the variety of properties of the final product. In the next step, 1.5% wt. Toluene Diisocyanate (TDI)(808264, Merk) was dissolved in the solvent in order to increase the dispersion of APP in the PU solution[18]. Following this step, this solution was sprayed on the surface of the fabric so that its entire surface was rich in flame retardant material. Finally, the samples were cured at 60 °C in oven for 1 min. Before carrying out any characterizations, all the fabrics were weighed via ISO 3801 to ensure that mass per unit length measurements are accurate and standardized across various woven fabrics, facilitating quality control and compliance in textile manufacturing.

2.1. Breaking Strength

ISO 13934-1 is an international standard for testing the tensile properties of woven and knitted textile fabrics using the strip method to determine the maximum force and elongation at maximum force[19]. Each sample is cut with a width of 50 mm ± 0.5 mm and a length sufficient to achieve a gauge length of 200 mm. Then, they are clamped securely in the tensile test machine (Instron 5944), and one end remains fixed while the other is pulled at a constant speed. The maximum force and elongation at maximum force are recorded in the 1atm[19].

2.2. Tearing strength

ISO 13973-1 is the international standard for determining the tearing strength of textile fabrics, essential for evaluating their durability and performance across various applications[19]. The standard primarily focuses on two methods: the ballistic pendulum method (Elmendorf), which measures the force needed to propagate a tear initiated in a fabric, and the single-tear method, which assesses the force required to continue tearing a single tear in a specimen. The samples should be rectangular, with a width of 50 mm ± 0.5 mm which then were placed in SDL Atlas Elmendorf Tear Tester (M008) with a sufficient length for a gauge of 200 mm. They must be aligned with the warp or weft direction to ensure consistent tearing behavior. The test results provide insights into tear force and tear length, both critical for assessing fabric performance[19].

2.3. Oil Repellent Degree

ISO 14419:1997 is the international standard for assessing the oil-repellent degree of textile fabrics. It is crucial for evaluating their resistance to oil-based substances in various industries, including apparel and technical textiles[20]. The procedure involves applying a series of hydrocarbon liquids with varying surface tensions to the fabric and observing how the oil behaves on the surface. Specimen Preparation: Fabrics are cut to specified dimensions: 50 mm ± 1 mm in width and approximately 200 mm ± 2 mm in length. Five drops of standard test liquids (eight standardized hydrocarbon liquids with increasing surface tensions, starting from 100% n-hexadecane (liquid 1) to mixtures with higher proportions of hydrocarbons and surface tensions up to about 50 mN/m (liquid 8)) are placed on the fabric. After 30 seconds, the drops are observed for absorption and contact angle. The highest numbered liquid that is not absorbed determines the oil repellency grade[20]. As a result of this test, Fabrics are graded based on their ability to repel oil, with higher grades indicating better resistance.

2.4. Contact angle degree

The most regular and widely used contact angle test method for flame retardant textiles is the sessile drop method performed with a standard contact angle goniometer. AKrüss DSA contact angle goniometer was used to perform this test. For measuring the contact angle in fabrics, a small droplet of water (usually around 2-5 μL) was first poured onto the surface of the clean and dry fabric. Then, a high-quality camera was positioned at a suitable angle to capture the droplet profile, ensuring accurate results. The final images were processed and analyzed via the Image J application to report the results.

2.5. Limiting Oxygen Index (LOI) test

The LOI test following established standards such as ISO 4589, is a key determinant in fire safety, reveals the minimum oxygen concentration required to sustain a material's combustion[21]. A higher LOI value, a significant indicator of fire resistance, is crucial in evaluating materials used in applications where fire safety is paramount. The LOI value is expressed as the percentage of oxygen in the gas mixture at which the sample maintains combustion. For example, if a material continues to burn in a 21% oxygen atmosphere but extinguishes at 20%, its LOI would be recorded as 21%. Samples, typically in strips or small pieces with dimensions around 3 inches or 50 mm long. You will need a vertical glass chimney or combustion chamber designed for

LOI testing and a controlled gas supply system capable of mixing oxygen and nitrogen.

2.6. Bending Length

ISIRI 4824 focuses on evaluating the bending length of textile materials, which is a measure of the fabric's stiffness and flexibility[19]. Bending length is defined as the distance over which a fabric can bend under a specified load or force. It indicates how flexible or stiff a fabric is, which is crucial for its application in various products. Before testing, it is crucial that specimens are cut from the fabric to a specified size, typically a rectangular shape with a width of 50 mm ± 0.5 mm. . Equally important is the conditioning of these specimens under standard temperature and humidity conditions. This step is vital as it ensures the accuracy of the results.

The test involves a crucial component-the bending length apparatus. This apparatus, which may consist of a horizontal support with a known weight applied at a specific point on the fabric, plays a significant role in the testing process. The bending length is determined by measuring the distance from the support point to where the fabric begins to bend under the applied Load. This measurement is typically taken when the fabric is subjected to a defined force or weight, which causes it to flex.

The bending length can be calculated based on the formula:

$$\text{Bending Length} = \frac{W \times L^3}{E \times I}$$

Where:

- W = Load applied (weight) (N)
- L = Length of the specimen (Cm)
- E = Modulus of elasticity of the fabric (Pa)
- I = Moment of inertia of the cross-section of the fabric (Cm^4)

A shorter bending length indicates greater flexibility, while a longer one suggests increased stiffness. The results of the bending length test are not just numbers. They are practical tools that help assess a fabric's suitability for specific applications, influencing design choices in clothing and other textile products[18].

2.7. Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is a powerful technique used for high-resolution imaging and analysis of the surface morphology and composition of materials. The SEM test method is an invaluable tool in material science, providing detailed insights into the structure and composition of various materials. Its ability to combine high-resolution imaging with elemental analysis makes it essential for research, quality control, and failure analysis across multiple industries.

Table 1 — Name and Abbreviated name of samples

Samples	Abbreviated name
Polyester Fabric	PS
Polyester/Polyurethane coating	PPU
Polyester/Polyurethane coating/Ammonium poly Phosphate	PPA

2.8. Energy Dispersive X-ray Spectroscopy

The EDAX test method refers to the use of Energy Dispersive X-ray Spectroscopy (EDX) or Energy Dispersive X-ray Analysis (EDAX), typically in conjunction with Scanning Electron Microscopy (SEM). This technique is widely utilized for elemental analysis and chemical characterization of materials.

3. Results and Discussion

3.1. Sample preparation

In this research, three samples were investigated. The properties of samples are shown in Table 1.

The final sample (PPA) microscopic image is shown in Figure 2. The fabric and coating structure is clear in Figures 2a) and b).

3.2. Mechanical strength

3.2.1 Breaking strength

Fig. 3 Mechanical Strength of flame-retardant Fabrics: a) Breaking Strength, b) Bending Length of Flame-retardant Fabrics

3.2.2 Tearing strength

As demonstrated in Tables 2 and 3 and Figures 3 and 4, the incorporation of polyurethane (PU) coating onto polyester fabric leads to a substantial enhancement in the mechanical strength of the final textile. This improvement is attributed to the PU layer acting as a protective barrier that distributes tensile stress more evenly across the fabric, thereby minimizing localized failures and increasing overall durability, including resistance to tearing.

A key contributor to this mechanical enhancement is toluene diisocyanate (TDI), a diisocyanate used in the synthesis of PU. TDI reacts with polyols through a nucleophilic attack of hydroxyl groups, forming a

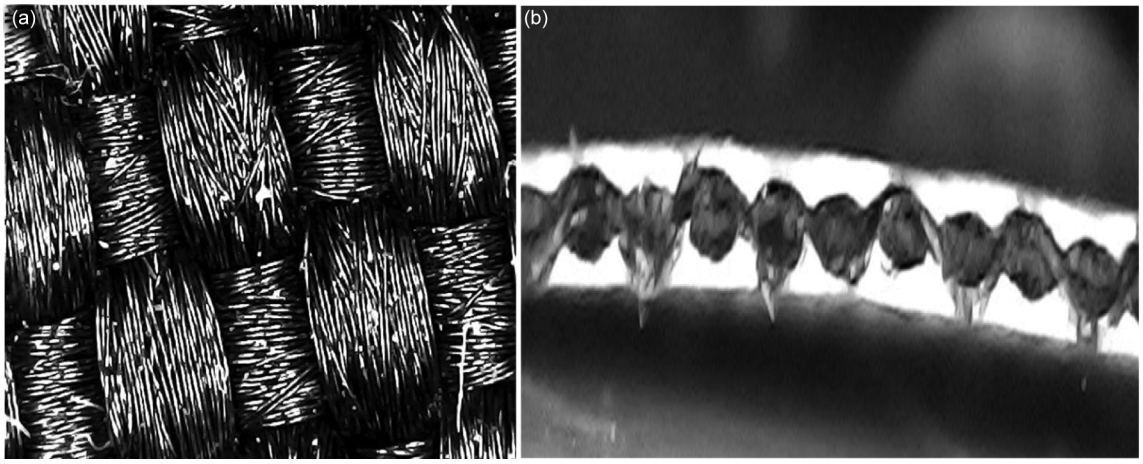

Fig. 2 — The microscopic images of PPA fabric. a) a microscopic image from plan view, b)a microscopic image from cross-section view

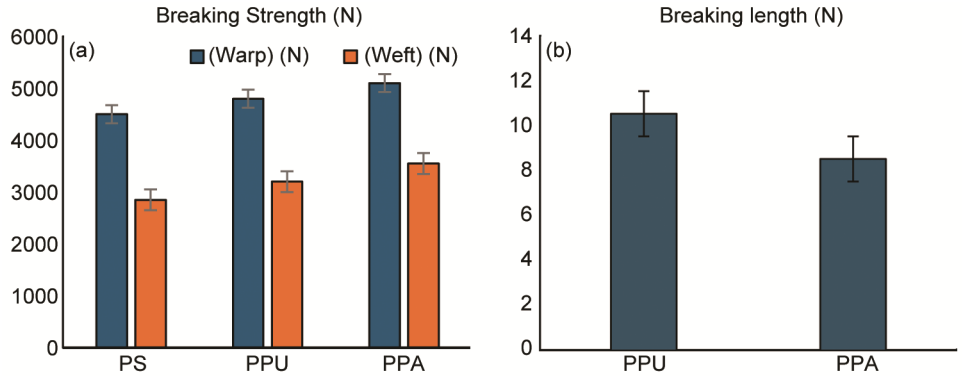

Fig. 3 — Mechanical Strength of flame-retardant Fabrics: a) Breaking Strength, b) Bending Length of Flame-retardant Fabrics

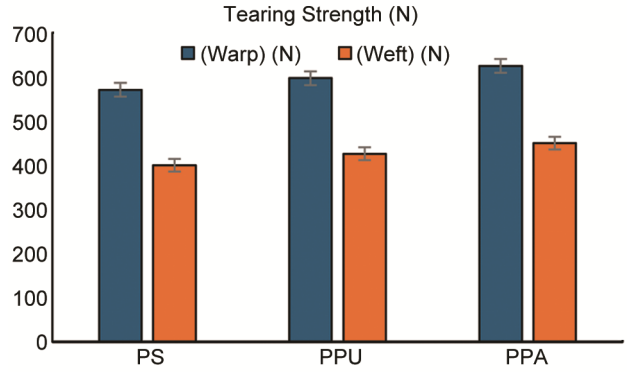

Fig. 4 — Tearing strength of flame-retardant textiles

Table 2 — Mechanical properties of Final flame-retardant fabrics

Sample	Breaking strength (warp) (N)	Breaking strength (weft) (N)	Bending length (Cm)
PS	4500	2851	∞
PPU	4800	3200	10.5
PPA	5100	3550	8.5

Table 3 — Mechanical properties of Final flame-retardant fabrics

Sample	Tearing strength (warp) (N)	Tearing strength (weft) (N)	Weight ($\frac{g}{m^2}$)
PS	571	400	341
PPU	597	426	471
PPA	625	450	510

highly cross-linked polyurethane network[22,23]. This increased cross-link density results in better adhesion and a more interconnected structure within the PU matrix, significantly improving its bonding to the polyester substrate and thereby strengthening the composite material[22-24].

In the PU/APP system, TDI plays a dual role by not only contributing to cross-linking but also enhancing the coating's mechanical and thermal performance[22-24]. The interaction between TDI and other components, such as ammonium polyphosphate (APP), further reinforces the network. APP, when used as a filler in the PU matrix, reacts with isocyanate groups to form additional urethane linkages, increasing the cross-link density. This structural reinforcement translates into improved tensile strength, abrasion resistance, and thermal stability of the coated fabric[7]. Overall, the synergy

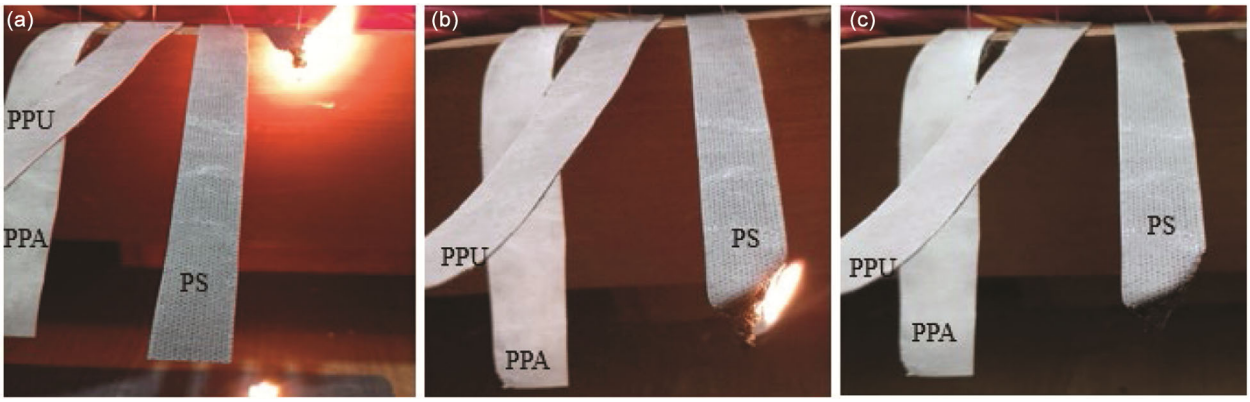

Fig. 5 — Self-Extinguishing test process of Fabric samples during 10s a) at the beginning of the test (t=0s), b) after 5s(t=5s) and c) after 10s (t=10s)

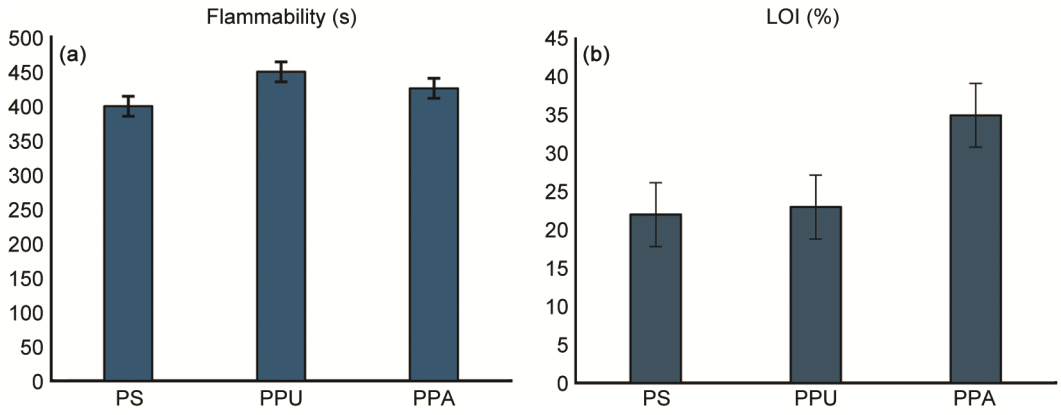

Fig. 6 — The flame retardancy properties of textiles. a) flammability and self-extinguishing time, b) Limiting Oxygen Index

Table 4 — The fire-retardancy of fabrics

Sample	LOI (%)	Flammability (s)	Self-extinguishing (s)	Char length (cm)
PS	22	400	∞	∞
PPU	23	450	5	5
PPA	35	426	0	16

between PU, TDI, and APP is instrumental in tailoring the mechanical properties of flame-retardant polyester textiles[25].

3.3. Flame retardancy properties

As illustrated in Figure 5, the PPU and PPA samples could not be ignited, and no flammability was observed. Further evidence from Figure 6 and Table 4 shows that incorporating a PU coating containing ammonium polyphosphate (APP) into polyester fabric, which is not inherently flame-resistant, significantly reduces its ignitability. The coated polyester exhibited a Limiting Oxygen Index (LOI) of 20–22%, indicating low flammability, therefore, in our research we obtained the LOI of 22% for polyester fabric. While untreated polyester tends to burn slowly and continue combustion until the flame source is removed—often melting and dripping—flame-retardant textiles form a protective char layer upon exposure to heat.

This char acts as a thermal and oxygen barrier, slowing down the combustion process and reducing heat transfer to the underlying material[26]. A thicker char layer offers better insulation and contributes to improved flame retardancy. This mechanism explains the enhanced self-extinguishing behavior and reduced flammability observed in PPA samples compared to untreated polyester (PS) and PU-coated polyester (PPU)[26].

APP plays a critical role in this flame-retardant behavior by chemically reacting with the isocyanate groups in the PU backbone, increasing the formation of urethane linkages and thereby enhancing the cross-linking density of the polymer matrix[15,26,27]. Additionally, the phosphate groups in APP interact with isocyanate groups in TDI-based PU systems, influencing the curing process and enabling APP to

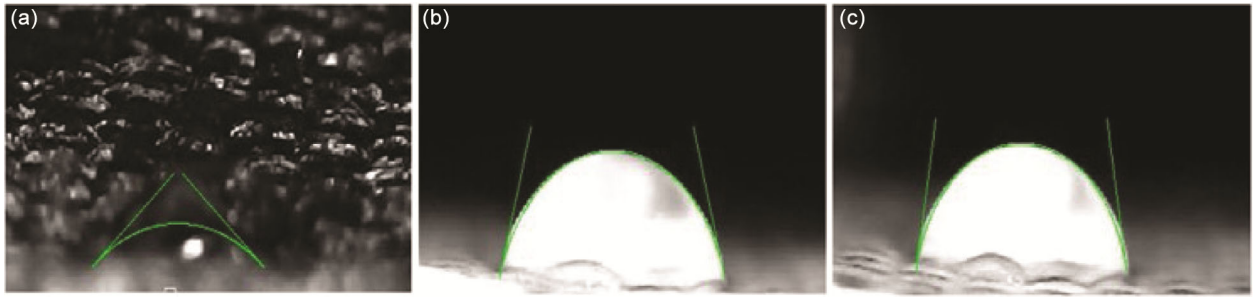

Fig. 7 — The contact angle degree for three fabric samples. a) Pure PS, b) PPU and c) PPA1 The EDAX analysis of Polyester/PU fabric a) Components contents in PPU matrix, b) Carbon content in PPU matrix, c) Oxygen content in PPU matrix and d) Nitrogen content in PPU matrix

Table 5 — The Air permeability, Oil repellency and Water contact angle for samples

Sample	Air permeability ($\frac{L}{m^2 s}$)	Oil repellent degree	Contact angle degree
PS	571	48	36
PPU	623	26	72
PPA	597	6	85

disperse evenly within the PU matrix[27,28]. This uniform dispersion enhances the coating's morphology, contributing to improved flexibility, toughness, and structural reinforcement[27,28]. During combustion, these interactions promote char formation, further enhance thermal stability and flame resistance. Beyond flammability, APP also significantly boosts the heat resistance of the PU coating, allowing it to maintain mechanical integrity under high temperature conditions[29,30]. This enhanced thermal durability makes the PU/APP system highly suitable for advanced protective textile applications[29,30].

3.4. Oil Repellent and contact angle degree

As presented in Figure 7 and Table 5, the surface properties of polyester fabrics were significantly altered following PU coating and the incorporation of APP filler. The PS sample (untreated polyester) exhibited the lowest contact angle (36°), indicating a highly hydrophilic surface and poor water repellency. In contrast, the PPU sample, which includes a pure PU coating, showed an increased contact angle of 72°, reflecting the moderate hydrophobic nature of polyurethane that contributes to water resistance by forming a barrier layer. Interestingly, the PPA sample, coated with a PU/APP composite, demonstrated a further increase in contact angle to 85°, indicating an enhanced hydrophobic surface, likely due to microstructural changes introduced by APP.

PU coatings reduce fabric wettability by lowering surface energy and forming a continuous film that limits water penetration[31]. The role of TDI in this system is critical; it enhances cross-linking within the PU matrix, which not only improves mechanical strength but also alters surface energy, thereby influencing wettability and contact angle behavior[31,32]. The interaction between TDI and APP during the curing process further contributes to increased cross-linking density and changes in surface morphology[31,32]. APP's inclusion results in a rougher surface texture, which promotes the Cassie-Baxter effect—a phenomenon where air pockets are trapped beneath water droplets, increasing the contact angle and enhancing hydrophobicity[32].

Additionally, this rough surface contributes to improved oil repellency. While the PS sample had an oil repellency rating of 48, this value dropped to 26 in the PPU sample due to the low surface energy of the PU chains[33]. However, in the PPA sample, the oil repellency rating dropped even further to 6, indicating a substantial loss in oil resistance, which may be attributed to the increased surface roughness and altered chemical composition due to APP addition[33,34]. Although hydrophobicity was enhanced, the compatibility of the APP particles with oil-based liquids may have reduced oil repellency, highlighting the trade-off between water resistance and oleophobicity in these functional coatings[34].

3.5. SEM and EDAX analysis

Figure 8 represents the SEM analysis of PU coatings. As demonstrated in Figure 8a) a phase separation can be seen in pure PU morphology. The microstructures are completely observable among the PU morphology. In Figure 8b) the dispersion of APP nanoparticles isclear; however, phase separation has been reduced due to incorporation of APP and TDI in

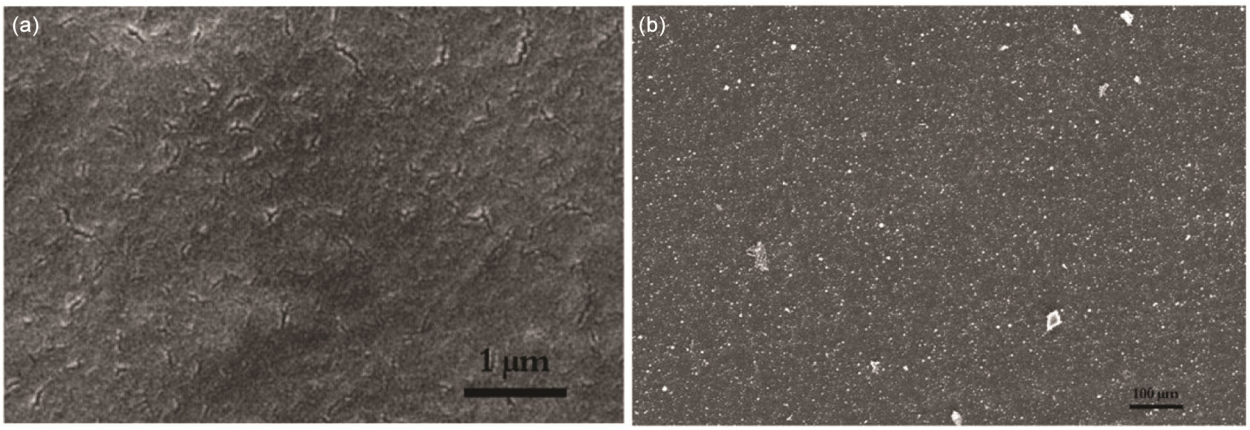

Fig. 8 — The SEM images of Polyester fabrics with a) pure PU coating and b) PU/APP coating

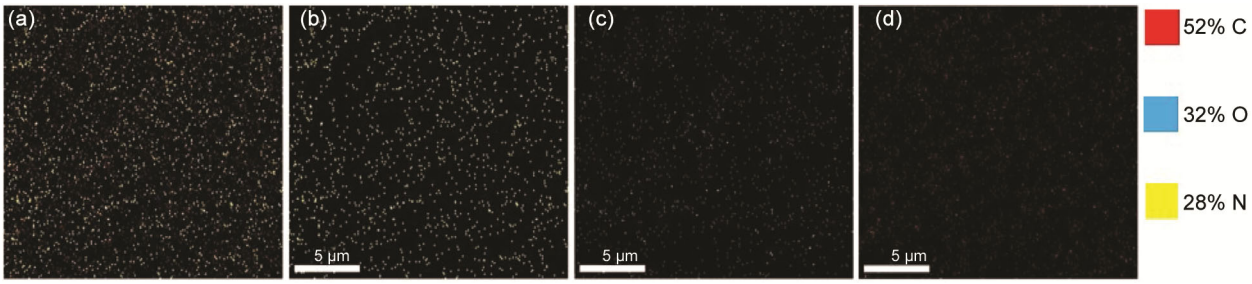

Fig. 9 — The contact angle degree for three fabric samples. a) Pure PS, b) PPU and c) PPA2 The EDAX analysis of Polyester/PU fabric a) Components contents in PPU matrix, b) Carbon content in PPU matrix, c) Oxygen content in PPU matrix and d) Nitrogen content in PPU matrix

PU matrix. The dispersion of APP within the PU matrix is significant, so poor component dispersion will lead to phase separation and generate weak points within the media, significantly impacting its overall performance. As shown in Figures 9 and 10, no phase separation was observed, indicating a proper APP distribution in the PU matrix.

According to Figures 9 and 10, the EDAX elemental mapping images reveal the spatial distribution of elements across the coated textile samples at the microscale (5 µm scale). The varied color intensities and dot patterns suggest differences in elemental composition and dispersion between the samples. In images with denser and more uniformly distributed dots, particularly in red, yellow, or blue, the presence of elements like phosphorus and nitrogen—likely from the ammonium polyphosphate (APP) filler—is evident, indicating effective integration into the polyurethane (PU) matrix[35]. Such uniform dispersion reflects a well-applied coating that is critical for achieving consistent flame-retardant properties. In contrast, images with sparse or uneven dot patterns suggest less homogenous element distribution, potentially corresponding to uncoated polyester (PS) or samples with poor additive dispersion[35,36]. The elemental homogeneity in PU/APP-coated samples (PPA) supports improved coating morphology and functionality, while limited elemental signals in the PS sample affirm the absence of flame-retardant treatment[36,37].

As mentioned earlier, polyurethane (PU) coatings can undergo phase separation due to the presence of distinct hard and soft segments in their polymer structure. Achieving a strong and durable coating requires a homogeneous microstructure with a well-balanced distribution of these segments[38]. To form such an integrated structure, a compatibilizer is often needed. Toluene diisocyanate (TDI) serves this role by promoting uniform mixing and enhancing compatibility within the PU matrix, especially when additives like flame retardants are introduced[38,39]. TDI also increases cross-linking density, strengthening the polymer network[39]. Additionally, the incorporation of ammonium polyphosphate (APP) can influence phase separation by interacting with TDI during curing, further modifying the cross-linking behavior and overall morphology of the PU coating[38-40].

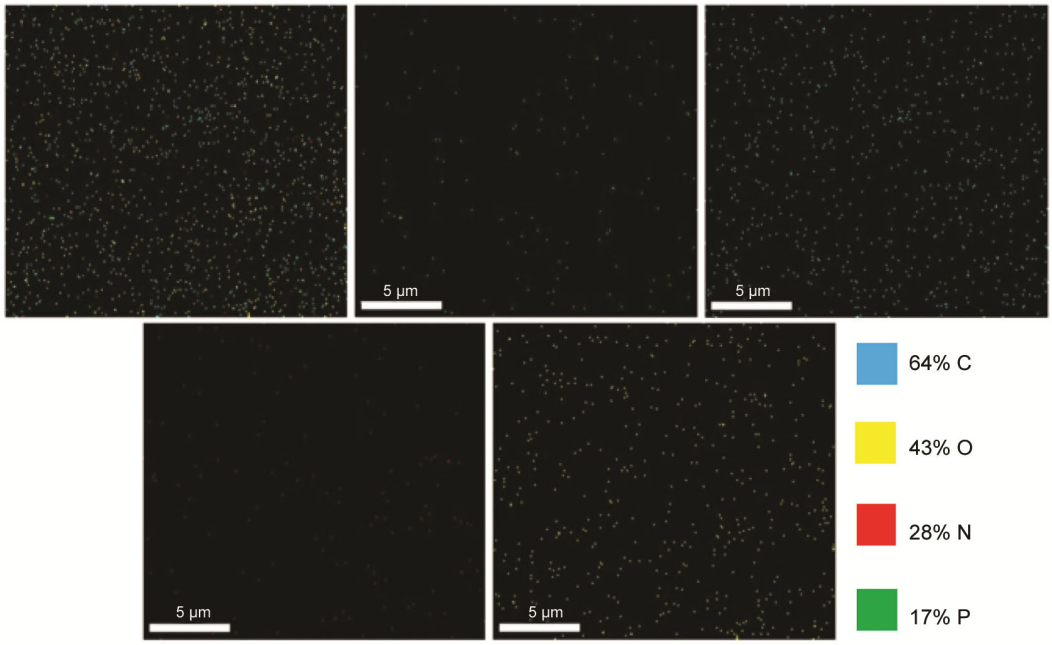

Fig. 10 — The EDAX analysis of Polyester/PU/APP sample a) Components contents in PPA matrix, b) Phosphorus content in PPA matrix, c) Carbon content in PPA matrix, d) Nitrogen content in PPA matrix and e) Oxygen content in PPA matrix

4. Conclusion

In this study we focused on preparation of a flame-retardant Polyester fabric with Polyurethane based coating and Ammonium poly phosphate as a flame-retardant component. The coating was applied on polyester via spraying method and cured at 60 °C for 1 min. The outcomes revealed that toluene diisocyanate (TDI) and ammonium polyphosphate (APP) have a significant role in enhancing the properties of polyurethane (PU) coatings. TDI is essential for forming strong cross-linked networks in PU, which improves mechanical strength, durability, and adhesion to various substrates. This results in coatings that can withstand physical stress, impact, abrasion, and environmental exposure without cracking or peeling. On the other hand, APP enhances the flame retardancy of PU foams by promoting an intumescent effect that forms a protective char layer during combustion. This char layer not only protects the underlying material from flames and heat which influentially reduces heat release rates during combustion. Additionally, APP can enhance the mechanical properties of PU, such as tensile strength and flexibility, particularly when used in combination with other flame retardants. Overall, both TDI and APP play crucial roles in optimizing the performance characteristics of PU coatings for various applications particularly fabrication of high- strength and durable flame- retardant products.

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