Egypt. J. Chem. **Vol. 68**, No. 1 pp. 573-582 (2025)

Egyptian Journal of Chemistry

http://ejchem.journals.ekb.eg/

Flame Retardant And Bioactive Polymer For The Finishing Of Cotton and Polyester: Synthesis, Characterization and Application

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Abstract

Novel environmental friendly flame retardant was synthesized for treating cotton and polyester textiles to impart fire resistant and antimicrobial properties. The synthesized flame retardant (polyvinyl phosphate urea (PVPU)) was based on a chemical modification of polyvinyl alcohol (PVA) without using the solvent that water only used. The chemical structures of the prepared PVPU polymer were investigated using FTIR. The ATR-FTIR spectra were examined to investigate the chemical composition of both untreated and treated cotton as well as polyester fabrics some properties such as fire resistance for untreated and treated fabrics were studied through carrying flammability test, limited oxygen index test (LOI) and tensile strength test. The surfaces of fabrics before and after treatment with PVPU were reported through Scanning electron microscopy (SEM) images. Thermal behaviors of untreated and treated fabrics were reported by thermo-gravimetric analysis (TGA). From all studies, the results showed a good fire resistance of the prepared PVPU when used for treating cotton and polyester fabrics. The antimicrobial resistance for cotton and polyester fabrics before and after treatment was investigated against some of the microorganisms. The results showed that the treated cotton and polyester textiles give the highest efficiency against fungi and bacteria compared to the untreated ones.

Keywords: PVA; flame retardant; cotton fabrics; LOI; tensile strength and microorganisms.

1. Introduction

The presence of polymer materials is becoming more convenient as they have more applications in many facets of our lives and economy [1,2]. However, some risks are found in polymer materials for example containing carbon, hydrogen, and other elements that control textiles' combustibility and flammability. Higher standards are expected of polymers in the modern environment of upgraded consumption and rapidly developing innovative industries. The most interesting application for polymer materials in the fields of textile products is improved by increasing their flame retardance for producing protective work clothes, firefighter uniforms, protective clothing for used in welding and related procedures, as well as the protection against heat and flames.

Another important application for polymer materials is improving the textiles products through increasing for antimicrobial efficiency. One of the most important fabrics is cotton fabrics that support the growth of microorganisms especially in moisture, nutrients and temperature. Since cotton fabrics contain carbohydrates that represent energy sources for the growth of microorganisms [3], this raised the risk of infection. So, it's necessary applying treatment for textiles to increase the microbial resistance. Wound infection is increased due to one of the most frequent bacteria (Staphylococcus aureus), which is referred to be the "transient flora" of the skin [4]. In order to prevent maceration of the surrounding tissue, regulate bacterial colonization, absorb exudate, and maintain the ideal moisture balance at the wound surface, skin wounds frequently require covering. As a result, picking the right outfit is crucial [5]. Cotton is a common dressing used in wound care because it has a higher gas permeability than occlusive dressings [6]. In hospitals and nursing homes, cotton gauze is still a successful long-term wound care option for extremely exudative wounds that require regular dressing changes [6].

Nowadays, halogen-free flame retardants that are environmentally friendly are developed in response to the growing environmental consciousness that is characterised by low toxicity and smoke with low corrosion [7–10]. According to statistics, the market was dominated by halogen-free flame-retardant goods in 2020, up from a 59% share [11]. Phosphorus-nitrogen FRs [16–19], metal-compound FRs [20–22], nitrogen FRs [14,15], and phosphorus FRs [12,13] have all been extensively researched. Polyvinyl alcohol (PVA) is a high-efficient FRs polymer, biocompatible, nontoxic artificial polymer and environmentally friendly. It widely used water-soluble polymer in FRs, PVA can quickly dissolve in water, forming a stable colloid, performance between plastic and rubber [23].

For controlling the flammability of textile products, there are three primary methods. The first one includes the incorporation during the production of the flame retardant. The second one is the wet treatment of the fabric after production and third one is

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Receive Date: 04 May 2024, Revise Date: 01 June 2024,Accept Date: 08 June 2024
DOI: 10.21608/EJCHEM.2024.287292.9676

back-coating the fabric. Cotton textiles are among the most widely used natural fibers because of its capacity to absorb sweat, renewable nature, and cozy feel [24, 25]. But, the flammability nature of cotton fabric and moreover the flame will spread rapidly causing fire [26,27]. Textiles can be treated with flame retardant by layer-by-layer (LBL) coating. The fabrics become flame-retardant due to the supramolecular coatings created by this approach (LBL), which creates attractive forces between the layers such as electrostatic attraction, hydrogen bonding, and coordination bonds [28, 29].

In this paper novel environmental friendly flame retardant was synthesized for treating cotton and polyester textiles to impart fire resistant and antimicrobial properties to develop new protective clothing with respect to industrial burn injuries with optimized ergonomic and improvised thermal management properties. The synthesized flame retardant (polyvinyl phosphate urea (PVPU)) was based on polyvinyl alcohol (PVA).

2. Experiments and characterization techniques

2.1. Materials

2.1.1. Fabrics

Unbleached 100 % cotton fabric (CF) (area density 430 g/m2) was supplied from MCI Egypt Company. In 3 % non-ionic detergent, the CFs were scoured at pH (6.5-7) for 15 min at 40 ^{o}C, then immersed in deionized water for several times, dried and kept into drier for storage [30].

2.1.2. Chemicals

Polyvinyl alcohol (PVA) was obtained from Sigma-Aldrich Co. Urea, absolute ethyl alcohol and phosphoric acid were supplied from AL Nasr Co.

2.2. Method

2.2 .1 Synthesis of polyvinyl phosphate urea (PVPU)

0.22 g (0.005 moles) of PVA was dissolved in 7.8 g (0.08 moles) of H3PO4. Then the mixture was heated at 130 °C for 3 hrs until the appearing of a viscous transparent liquid. After this, add 4.8 g urea (0.08 moles) and then decrease the temperature to 100 °C with stirring for 1 hr to appear off-white viscous liquid (PVPU) [31]. The FR powder was obtained by precipitation in diethyl ether and followed by filtration then dried at 40 ºC.

2.2.2. Fabric treatment

A Pad-dry-cure method was used in the treatment, where the CF and PE fabrics were first rinsed in a padder containing a solution of PVPU with different concentrations (25, 37.5, 50, and 62.5 %) for CF and with concentrations of 50 and 62.5% for PE for 3 min. Then the treated fabrics were dried at 90 ºC for another 3 min and finally, the curing is carried out at a temperature between 120-140 ºC for 2.5 min Following the curing process, the treated CF and PE samples were laundered for a predetermined number of times in accordance with AATCC Test Method 124-1996 [32].

2. 3. Characterization techniques

Using a Nicolet 380 Spectrometer, the ATR-FTIR spectra of selected treated and untreated fabric samples as well as synthetic PVPU were acquired within the range of 650–4000 cm^{-1}. Utilizing a TGA-50 Shimadzu apparatus, the thermal analyses of both treated and untreated textiles were recorded at a flow rate of 10 ml/min and a heating rate of 10 ^{o}C/min in a nitrogen atmosphere, with temperatures ranging from ambient temperature to 650 ºC. Utilizing a SEM Model Philips XL 30 with an accelerating voltage of 10 KV, magnification ranging from 10 x to 400.000x, and resolution for W (3.5 nm), the morphology of both treated and untreated fabrics was examined.

2.4. Tests

2.4.1. Flammability study

By Horizontal burning test according to ISO 3795, the flammability of treated and untreated fabrics was investigated. In this test, the fabrics samples stand horizontally and from end of sample, it was supplied by flame [32]. It was determined how long it took the flame to go from the first reference mark, which is located 25 mm from the end, to the second reference mark, which is located 100 mm from the end. Sample dimensions were 7x20 mm, maintained at 23 ºC and 50 % humidity during 48 hr prior to analysis.

2.4.2. Tensile strength test

According to BS EN ISO 13934–1: 1999 [33], the tensile strength of both treated and untreated fabric samples was assessed using six specimens (three for the warp and three for the weft) and a gauge length of 200 mm with a strain rate of 30 mm/min. The specimens were 50 mm wide.

2.4.3. Limiting oxygen index LOI

According to ASTM D2863- 97 standard methods [34], Limiting oxygen index (LOI) values of both treated and untreated fabrics were detected.

2.4.4. Washing Process

In order to investigate the effects of multiple washings on the treated cotton and PE fabrics by PVPU, the durability of PVPU on fabrics was evaluated by washing the samples five times following the finishing process in accordance with the AATCC M7 standard. It is a laboratory procedure used to wash fabrics at home before flammability test to distinguish between finishes that are durable and those that are not [35].

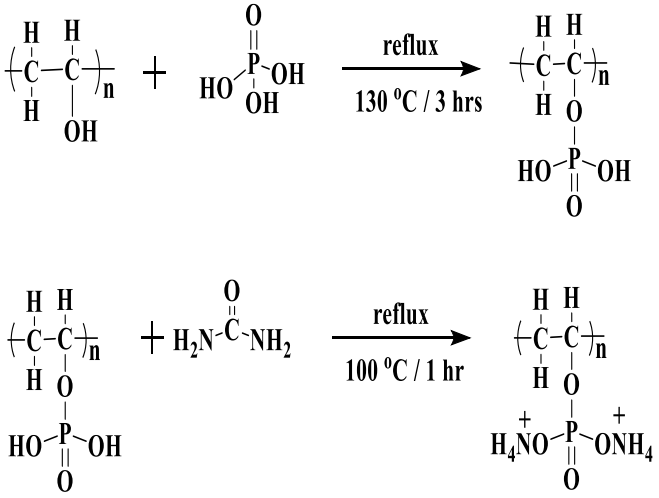
2.4.5. Antimicrobial studies

The antimicrobial efficiency of both treated and untreated CF as well as PE fabrics were investigated on unicellular fungi (Candida albicans ATCC 10231), two types of bacteria (Listeria monocytogenes ATCC 7646 and Staphylococcus aureus ATCC 6538) as Gram-positive and on another two types of bacteria (Salmonella sp. ATCC 14028 and Escherichia coli ATCC 25922) as Gram-negative.The tests were applied through disc diffusion method, in which samples of treated and untreated CF and PE fabrics were poured into wells in agar plates containing fungi and bacteria in the form of squares. After that, the bacterial and fungal plates were incubated for 24 h at 37 °C and for 48 hrs at 30 °C, respectively. The inhibition zone diameter was determined in mm.

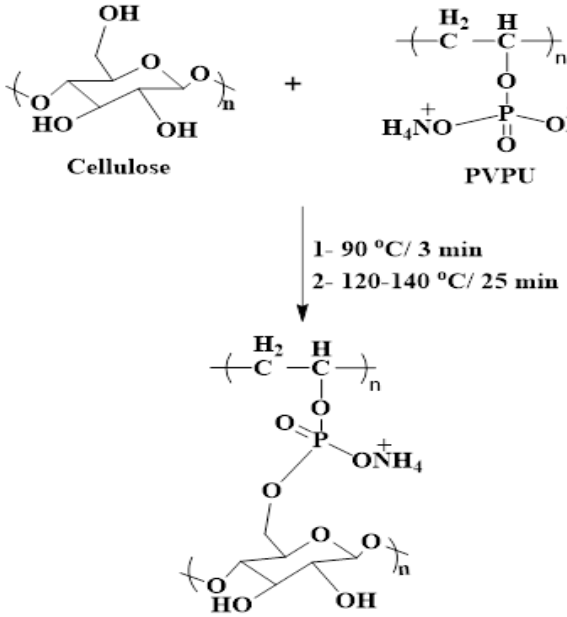
3. Results and discussion

3.1. Synthesis of PVPU FR

Novel PVPU polymer was synthesized for treating cotton and PE fabrics to impart fire resistance and antimicrobial properties. The FR was synthesized by mixing PVA, phosphoric acid and urea to obtain PVPU as a flame retardant as observed in Scheme 1. Whereas Scheme 2 showed the reaction between PVPU and cotton fabrics



Scheme 1: The mechanism of the preparation of flame retardant of PVPU.



Scheme 2: Reaction between PVPU and cotton fabrics.

3.2. FT-IR

Fig.1. showed FT-IR spectra for PVA and flame retardant (PVPU). For PVA in spectrum (Fig.1$_a$), peaks at 3424 cm^{-1}, 2936 cm^{-1} and 2906 cm^{-1} are corresponding to OH stretching, asymmetric and symmetric stretching of CH_2, respectively. A band at 1634 cm^{-1} was showed that respect to water absorption. The spectrum of PVPU (Fig.1$_b$) exhibited new peaks at 1205 cm^{-1} for the stretching of P=O from phosphate, 831 cm^{-1} for the stretching of P-O and the stretching of C-O-P observed at 1064 cm^{-1}. Fig.2(a-e) showed ATR-FTIR for treated and untreated CF with different concentration of flame retardant (PVPU). In Fig.2(a), peak at 3418 cm^{-1} was observed due to the stretching of OH that is present in the structure of cellulose. At 2920 and 2853 cm^{-1}, asymmetric and symmetric stretching of C-H vibrations appeared, respectively. On treating CF with flame retardant of different concentrations (Fig.2(b-e)), new bands at 1204 for P=O, 1196 for P-O, and 849 cm^{-1} corresponding to P-O-C were recorded. New appeared peaks in samples of treated CF confirmed the grafting of flame retardant (PVPU) onto CF. Fig.3(a-c) showed ATR-FTIR for untreated and treated PE with different concentrations of flame retardant (PVPU). Fig.3(a) showed the spectrum of untreated PE that exhibited peaks at 2963, 725 and 874 cm^{-1} corresponding to stretching, bending of C-H and vibration of C–C out-of-plane bending, respectively. Peaks at 3434 and 1014 cm^{-1} were observed due to the intermolecular O–H bonded to C=O groups and O–H out-of-plane bending in the terminal carboxylic groups in the polyester chains. The peak of C=O of ester group was observed at 1721-1735 cm^{-1}. In Fig. 3(b-c), peaks for P=O, P-O and P-O-C were

observed at 1206, 1194 and 838 cm^{-1} , respectively. New appeared peaks in samples of treated PE confirmed the grafting of flame retardant (PVPU) onto PE.

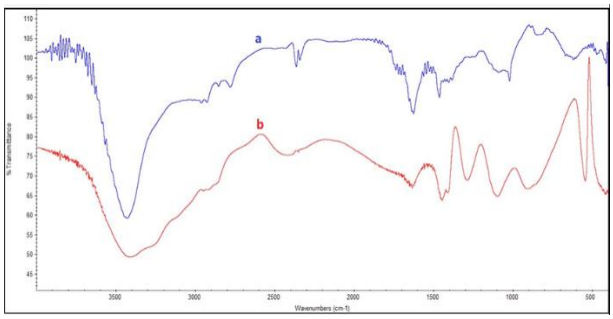


Figure.1. FT-IR for (a) PVA and (b) PVPU flame retardant.

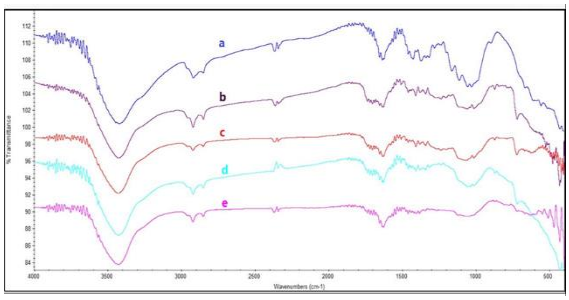


Figure 2. FTIR-ATR for (a) untreated CF and (b,c,d and e) treated CF with (b) 25 %, (c) 37.5 %, (d) 50 % and (e) 62.5 % of PVPU flame retardant.

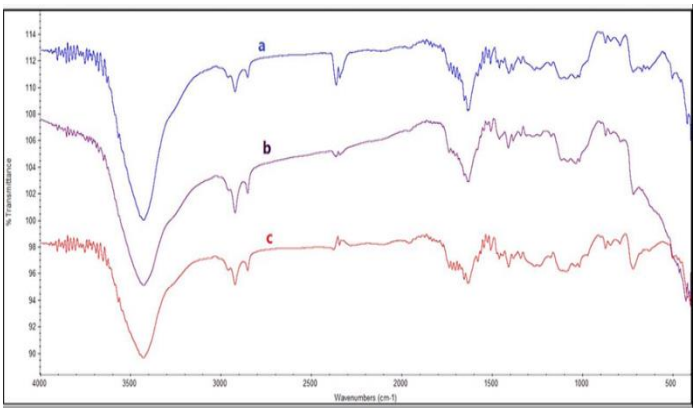


Figure.3. FTIR-ATR for (a) untreated PE and (b and c) treated PE with (b) 50 % and (c) 62.5 % of PVPU flame retardant.

3.3. Thermal analysis

The thermo-gravimetric analysis for treated and untreated cotton with PVPU concentrations of 25, 37.5, 50, and 62.5 % was studied under nitrogen and a heating rate of 10 ^{o}C/min and were shown in Fig. 4 (a-e). The data revealed that treated and untreated CF with PVPU of a concentration 25, 37.5, 50, and 62.5 % started decomposition at temperatures 293, 287, 278 and 275 ^{o}C, respectively. This proves that introducing PVPU within the structure of CF lowers the temperature of decomposition of CF. Moreover; this influence is more observed at higher concentration of PVPU flame retardant. At higher temperatures, untreated CF decomposed at 392 ^{o}C with residue 0.92 %, whereas treated CF with PVPU of a concentration 25, 37.5, 50, and 62.5 % decomposed at 480, 510,515 and 521 ^{o}C with residues 16.19, 40.93, 39.49 and 47.84 %, respectively. The results showed that PVPU is chemically bonded onto CFs which reduces the output of volatile components in treated fabrics than untreated ones decomposition at higher temperatures. So, the higher residue contents of the treated CFs were observed from data obtained from TG and DTG (Fig. 4 (a-e)). Also, the TG and DTG for treated and untreated PE fabrics with PVPU of concentrations 50, and 62.5 % (Fig. 5(a-c) were studied. The results showed that untreated PE started to decompose at temperature 371 ^{o}C, while treated PE with PVPU concentrations of 50, and 62.5 % decomposed at 349 and 350 ^{o}C, respectively. This proves that introducing PVPU within structure of PE lowers the temperature of decomposition. At higher temperatures, the residues for untreated PE are 9.41, 24.29 and 22.14 %, respectively. The results showed that PVPU is chemically bonded onto PE that reduces the output of volatile components in treated fabrics than untreated one's decomposition at higher temperatures. So, the higher residue contents of the treated CFs were observed from the data obtained

from TG and DTG (Fig. 5(a-c)). Finally, all data showed that the treated CF and PE fabrics have better thermal stability than untreated.ones.

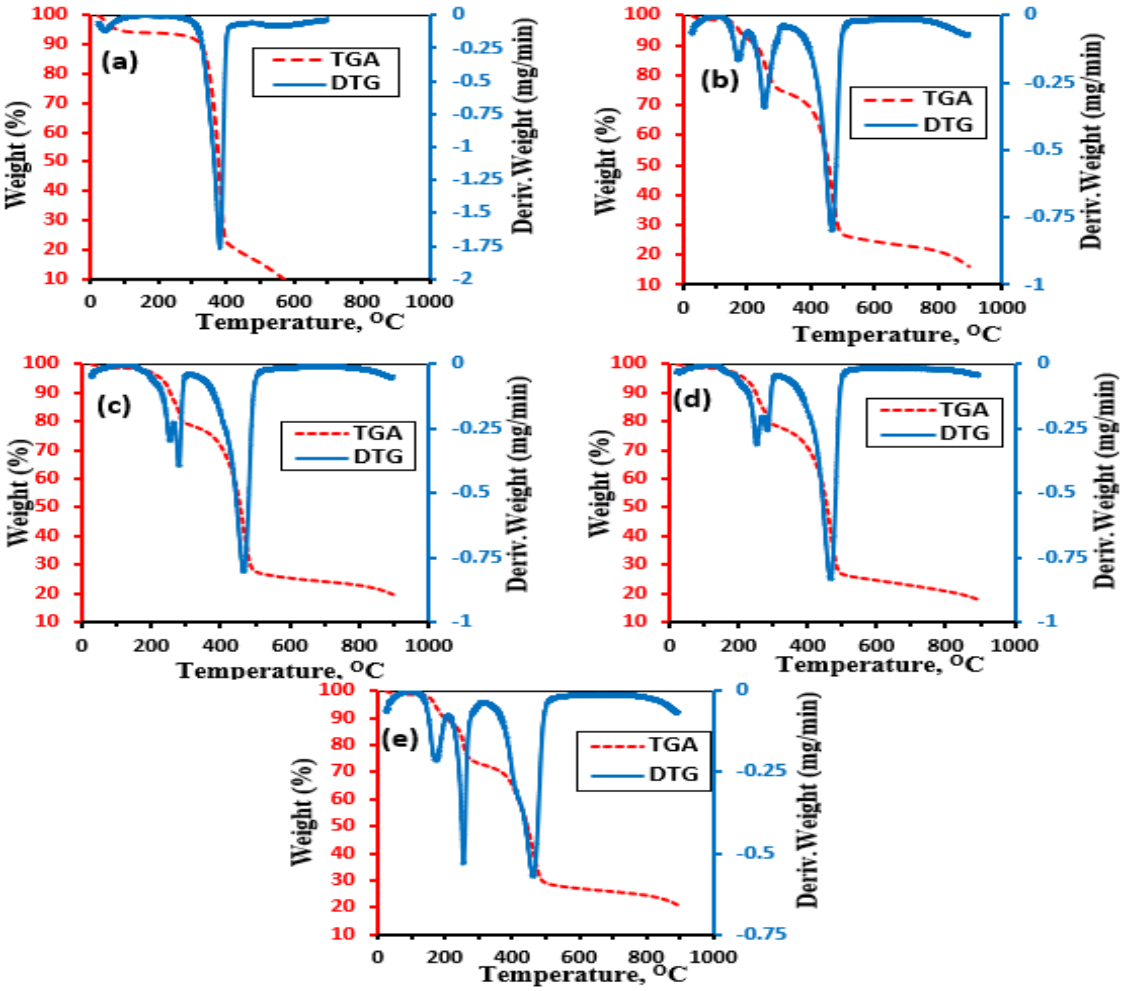
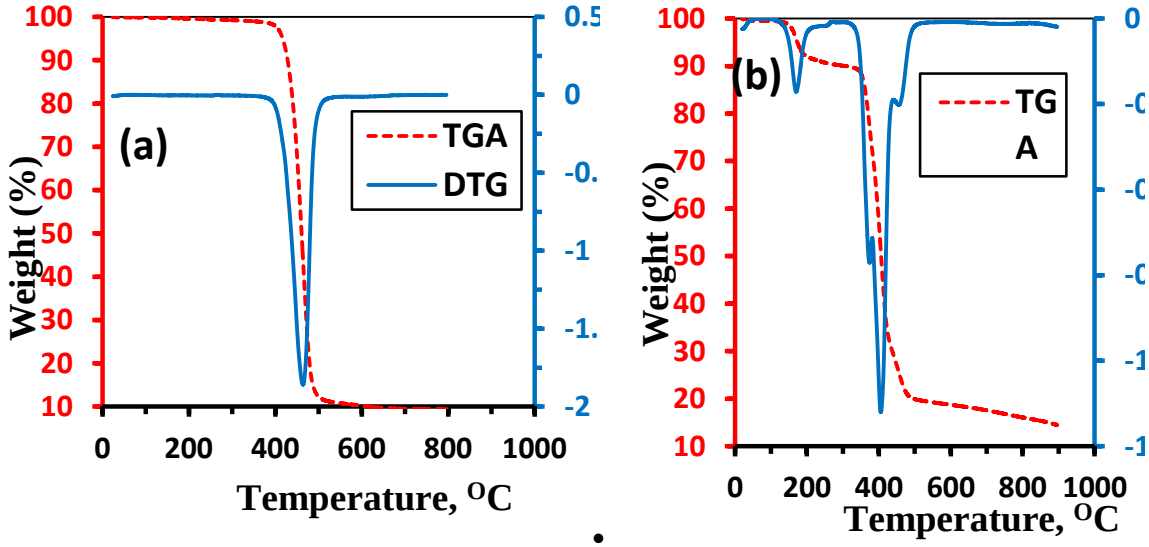


Figure 4: TG and DTA for (a) untreated CF and (b,c,d and e) treated CF with PVPU of concentration (b) 25 %, (c) 37.5 %, (d) 50 % and (e) 62.5 %.



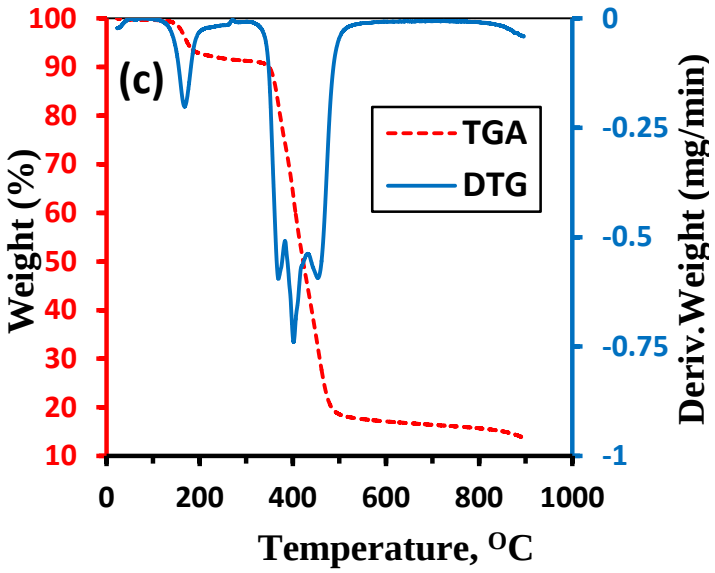


Figure 5: TG and DTA for (a) untreated PE and (b and c) treated PE with PVPU of concentration (b) 50 % and (c) 62.5 %.

3.4. Surface morphology

SEM technique (Figs. 6,7) is used to observe the surface of both treated and untreated fabrics. The untreated CF and PE fabrics were flat in comparison with treated ones which are fuller. The surface of treated fabrics becomes more smooth than that permeated PVPU fabrics. These was no observed change in fiber morphology of treated samples at different concentrations, but by increasing PVPU concentration, the fiber fracture of treated samples decreased. Moreover, there is a slight increase in the fiber diameter [36] by increasing the PVPU %with higher fiber stability. A clear bubble was generated on the treated fiber surface indicating the formation of a layer of PVPU on the treated fabrics [37].

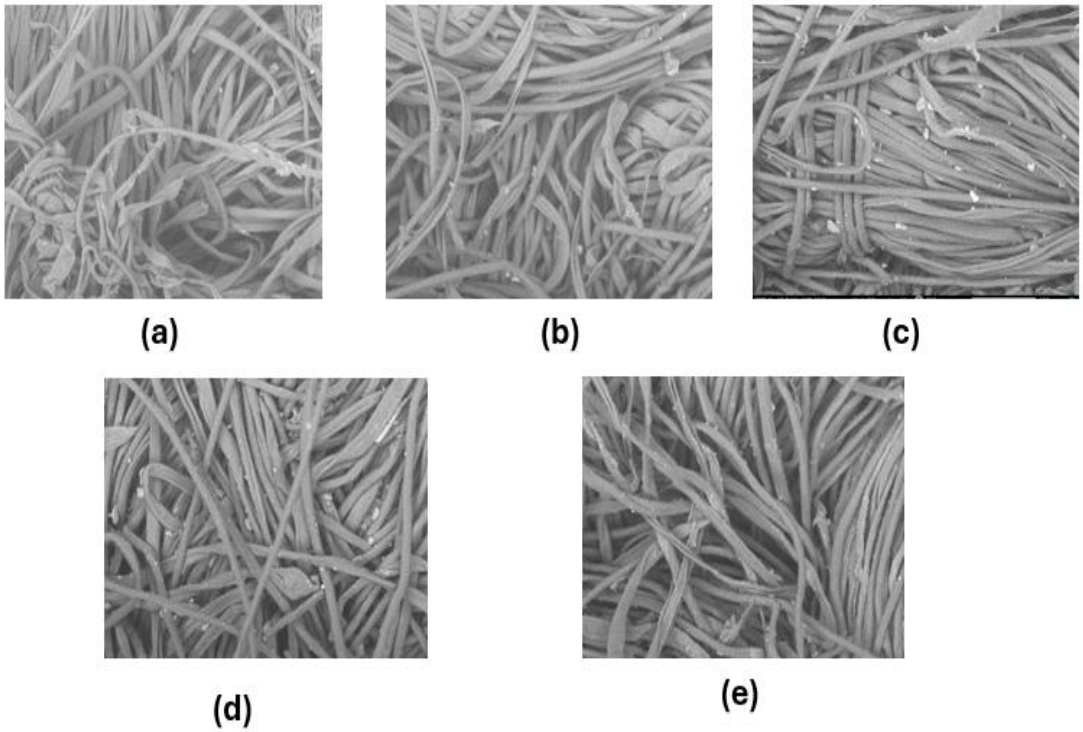


Figure 6: SEM images for (a) untreated CF and (b,c,d and e) treated CF with PVPU of concentration (b) 25 %, (c) 37.5 %, (d) 50 % and (e) 62.5 %.

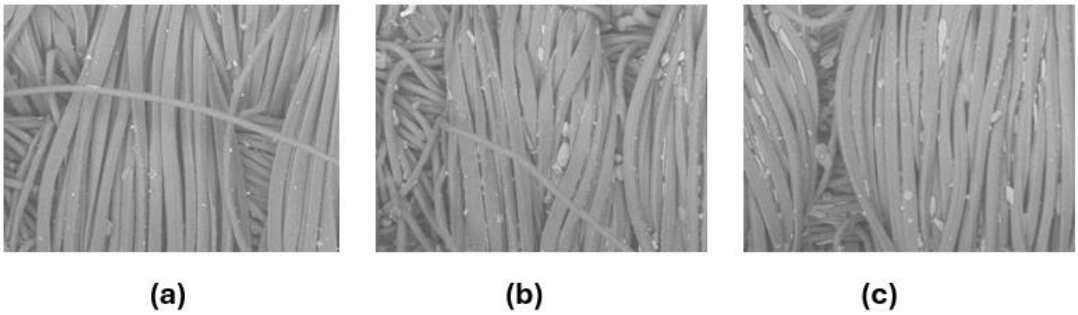


Figure 7: SEM images for (a) untreated PE and (b and c)treated PE with PVPU of concentration(b)50 % and (c) 62.5 %.

3.5. Flammability test

The examination of fire resistance of treated and untreated fabrics at different concentration of PVPU (62.5, 50, 37.5 and 25%) were investigated by vertical flammability test (Figs.8,9). The results can show that there is a serious damage in the untreated sample 22.0 cm of damaged length. In contrast, a slight damage appeared in the treated samples from 0.1 to 1.1cm of damaged length. All treated samples showed lower chare length than that of the untreated ones in both cotton and PE. Also, it was also clear that, increasing PVPU % gave excellent flame retardant fabrics. Where, the majority of the treated PVPU 62.5% and 50% samples showed the remarkable flame resistance comparing to the other treated ones suggesting that the cross linking between PVPU fire retardant and fabric has increased too [36].

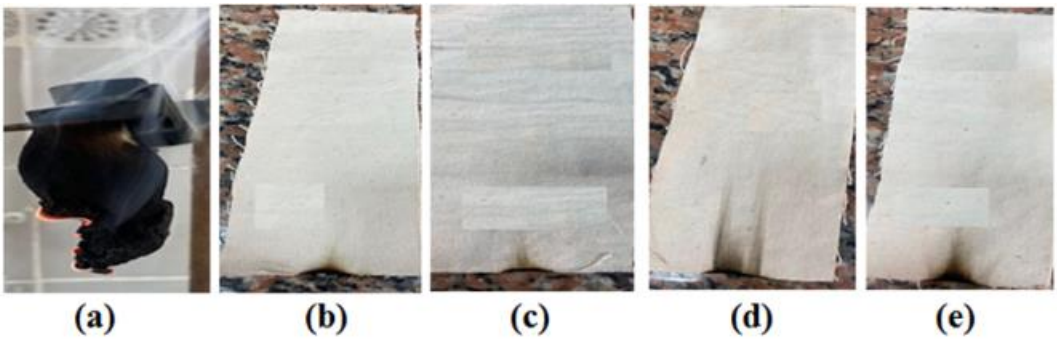


Figure 8: Flammability images for (a) untreated CF and (b,c,d and e)treated CF with PVPU of concentration (b) 25 %, (c) 37.5 %, (d) 50 % and (e) 62.5 %.

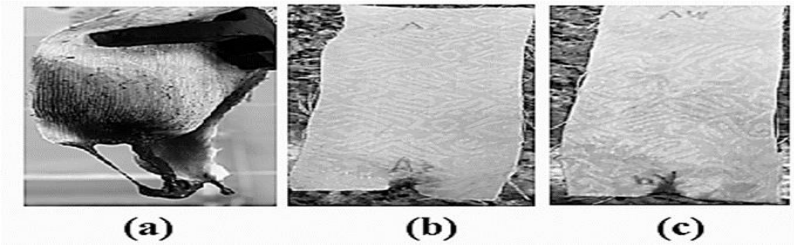


Figure 9: Flammability images for (a) untreated PE and (b and c) treated PE with PVPU of concentration (b) 50 % and (c) 62.5 %.

3.6. LOI test

One of the important tests that are used to examine the fire resistance of treated and untreated fabrics is LOI. Fig.10 shows the values of LOI for treated fabrics before and after the cycles of washing. From the obtained data untreated CF has a higher LOI value (17.1 %) than untreated PE fabrics. Also, the LOI values of treated samples were increased due to the cross-linking between fibers and an effective PVPU. For the cotton and PE samples treated with 62.5% of PVPU the LOI value raises to 28.2 and 24.2, respectively showing the permeability of PVPU on CF and PE fabrics even after 5 washing cycles with a little decrease in LOI values with increasing the number of washing cycles [38]. This permeability of PVPU with fabrics is due to covalent bond (C-O-P) between PVA and phosphorus-containing groups, which is confirmed by ATR-FTIR.

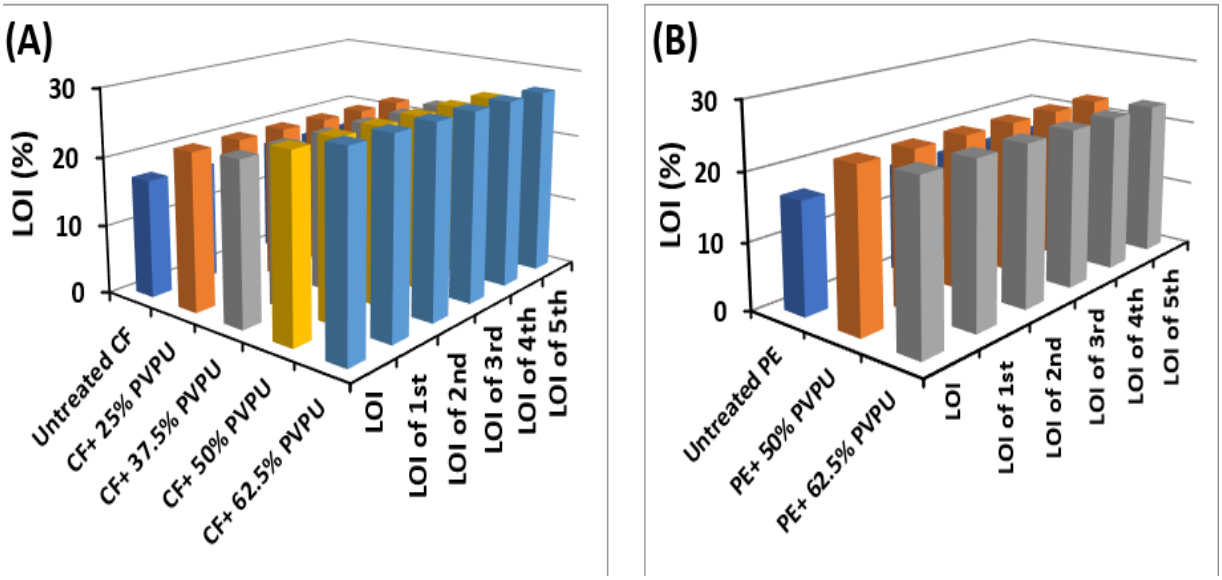


Figure 10: LOI for (A) untreated CF and treated CF with PVPU of concentration 25 %, 37.5 %, 50 % and 62.5 %, and (B) untreated PE and treated PE with PVPU of concentration 50 % and (c) 62.5 % with number of laundering cycles 1^{st},2^{nd},3^{rd},4^{th} and 5^{th}.

3.7. The Laundering Durability of the Treated Fabrics

Different laundering cycles for treated fabrics were measured at different concentrations of PVPU (62.5, 50, 37.5, and 25%). It was revealed that all the tested samples of cotton and PE indicate higher flammability resistance after as many laundering cycles as made. These data indicate that the presence of PVPU fire retardant polymer makes a cross-link with fabrics. Consequently, this is another confirmation that using flame retardant finishing (PVPU) (62.5 and 50%) increases the resistance of the fabrics even after multiple laundering cycles.

3.8. Tensile strength test

The application of PVPU polymer may affect fabric properties, like strength and bending rigidity. The tensile strength for treated and untreated fibers is collected in Table 1. We concluded that, the breaking strength of treated CF samples decreased compared with untreated cotton but remain nearly the same with untreated one and the strength of the fiber improved in PVPU (50%). From all results, the strength of treated PE samples increased compared to the untreated one, this proved the strong chemical bond connection between PVPU and PE fibers. In treated CF samples, there is a slight damage by PVPU but in polyester the tensile improved.

Table 1: Tensile strength for untreated, treated CF and PE with different concentrations of PVPU

Samples	Tensile strength in warp Direction (N)	Tensile strength in weft direction (N)
Untreated CF	120	105
CF+ 25 % (PVPU)	100	95
CF+ 37.5 % (PVPU)	105	97
CF+ 50 % (PVPU)	110	100
CF+ 62.5 % (PVPU)	100	103
Untreated PE	100	95
PE+ 50 % (PVPU)	130	120
PE+ 50 % (PVPU)	120	110

3.9. Antimicrobial studies

The antimicrobial efficiency of the untreated, treated CF and PE were investigated against some microorganisms (Figs 11,12 and 13). The results showed a good efficiency of treated CF samples with most microorganisms than untreated CF. Also, the results obtained from treated CF samples showed that the highest inhibition zone for treated CF with 62.5% of FR was recorded against C. albicans and S. aureus. In another side, the treated PE samples showed an interesting result than untreated ones. All treated PE samples recorded the highest efficiency against all microorganisms. Last but not least, the use of PVPU as a cutting-edge technique to give cotton and polyester fibers antibacterial qualities is a creative and successful tactic that made it possible to first report the use of antimicrobial for cellulosic fibers like cotton gauzes, an emerging class of antibiotics that is particularly effective against resistant bacteria. Furthermore, the newly developed procedures have demonstrated efficacy without causing cytotoxicity, a significant issue in the development of novel antimicrobial finishing techniques for textiles and wound dressings.

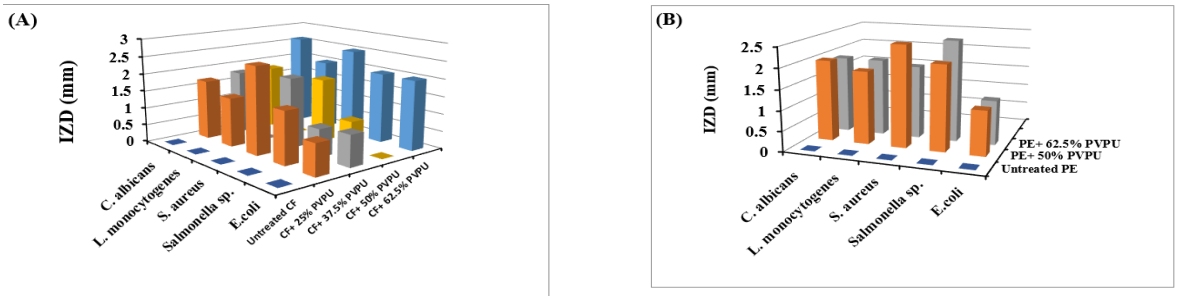


Figure 11: Inhibition zone for (A) untreated and treated CF with PVPU of concentration 25, 37.5, 50 and (c) 62.5 % and (B) untreated and treated PE with PVPU of concentration 50 and (c) 62.5 %.

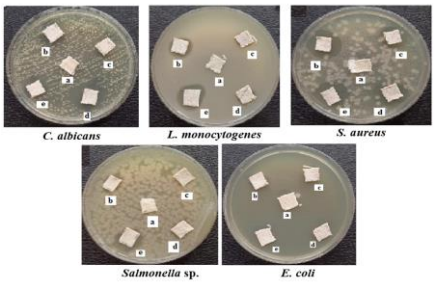


Figure 12: Antimicrobial images (a) untreated CF, (b,c,d and e) treated CF with PVPU of concentration (b) 25%, (c) 37.5 %, (d) 50 % and (e) 62.5 %.

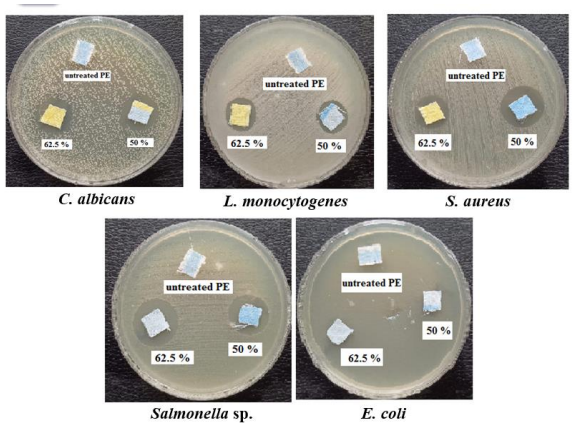


Figure 13: Antimicrobial images for untreated PE and treated PE with PVPU of concentration 50 and 62.5 %.

4. Conclusion

Novel environmental friendly flame retardant was synthesized for treating cotton and polyester textiles to impart fire resistance and antimicrobial properties. The synthesized flame retardant (polyvinyl phosphate urea (PVPU)) was based on a chemical modification on polyvinyl alcohol (PVA) without using solvent that water only used. PVPU polymer structure was investigated using FTIR. The composition of untreated and treated cotton and polyester fabrics were reported ATR-FTIR. Some properties such fire resistance for treated and untreated fabrics were studied through carrying flammability test, limited oxygen index test (LOI), tensile strength test. The surface images of untreated and treated fabrics were reported by SEM. The thermal behaviour of both treated and untreated fabrics were reported by TGA. From all studies, the results showed a good fire resistance of the prepared PVPU when used for treating cotton and polyester fabrics with more durability. Last but not least, the use of PVPU as a cutting-edge technique to give cotton and polyester fibers antibacterial qualities is a creative and successful tactic that made it possible to first report the use of antimicrobial for cellulosic fibers like cotton gauzes, an emerging class of antibiotics that is particularly effective against resistant bacteria.

Ethics approval
Not applicable.
* **Consent to participate**
The authors agree to the journal's policy
* **Consent for publication**
The authors agree to the journal's policy
* **Availability of data and materials**

Data will be available from the corresponding author upon reasonable request.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

The authors confirm contribution to the paper as follows: study conception and design: Laila M. Reda, Amal M. Metwally; data collection: Mohamed M. Thabet; analysis and interpretation of results: Mohamed M. Azab, Laila M.Reda, Amal M. Metwally. Author; draft manuscript preparation: : Mohamed M. Azab, Laila M.Reda, Amal M. Metwally.Hanna A. Elkhawaga wrote the references. All authors reviewed the results and approved the final version of the manuscript.

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