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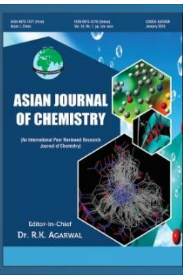

Synthesis of Phosphoric Acid Ester-based Flame Retardants and their Application *via* Sol-Gel Process to Enhance Flame Retardancy of Cotton Fabric

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In order to enhance the flame retardancy of cotton fabric and expand it use in regions with strict fire safety regulations, the phosphoric acid esters-based organic compounds such as diphosphoric acid ethyleneglycol ester (diPEG), triphosphoric acid glycerol ester (triPGE) and tetraphosphoric acid pentaerythritol ester (tetraPPE) were synthesized and characterized by ^{1}H NMR, ^{31}P NMR and HRMS method. The cotton fabric was treated with these compounds and aminopropyltriethoxysilane (APTES), a sol-gel precursor, by using sol-gel process. FTIR spectrum of treated fabric shows additional peaks at 1240 (P=O), 1044 (Si-O-Si) and 794 cm^{-1} (Si-O), which shows the presence of flame-retardant coating on fabric. Thermogravimetric analysis was employed to conduct the change in path of thermal degradation of treated cotton fabric. The behaviour of flame retardancy was assessed through auto-flammability and limiting oxygen index examinations. The treated cotton fabric exhibits outstanding flame-retardant properties during combustion and are extinguished promptly once the ignition is eliminated. The limiting oxygen index value of the cotton fabric after treatment with tetraPPE/APTES flame retardant reached 38.3%, which is sufficiently above the flame-retardant value of about 27%. The morphological structures of fabric were also investigated using FESEM.

Keywords: Cotton fabric, Sol-gel, Flame retardancy, Thermal studies, Morphology.

INTRODUCTION

The cotton fabric is the most easily available textile with a wide range of uses for clothing, furniture upholstery and industrial products [1,2]. But cotton is more likely to catch fire and burn with a high combustion rate being having a large surface area, therefore, it may cause loss of life and property due to fire incidents [3,4]. Therefore, improved flame retardant properties are needed for cotton fabric to lessen the risk of fire and expands their uses. Halogenated flame retardants particularly containing bromine have been used the successful flame retardants. But the halogenated flame retardants have been found to release harmful and carcinogenic compounds into the environment during combustion, endangering human health, therefore, several nations and regions have banned their use [5]. Presently phosphorus and silicon-based flame retardants are thought to be environmental responsive and these have shown a practical way to greatly increase the thermal stability and flame retardancy of materials, especially when paired with nitrogen [6-8].

Thermal degradation of cotton cellulose occurs when exposed to heat, resulting in the production of a highly flammable compound called levoglucosan [9], mainly responsible for spreading the fires. Nitrogen, phosphorus and silicon-based systems facilitates the development of a protective char layer, thus causing the combustion to be extinguished [10-13]. Several methods such as chemical modification, pad-dry cure, sol-gel treatment, graft polymerization and intumescent treatment have been documented to increase cotton textiles' fire resistance [14-18]. For a number of reasons, sol-gel process is a significant and influential technology in the textile sector to enhance effectiveness, reduce chemical consumption, functionality and durability of crucial properties. The key advantage of sol-gel technology is its minimal environmental impact [19] and provides a convenient synthetic route in which hydrolysis and condensation reactions generate organic-inorganic hybrid coatings at room temperature. Thus, through the creation of a physical barrier, these coatings can protect the polymer surface and improve certain desirable properties including flame retardancy [20].

In order to determine their effects on the thermal stability and flammability of cotton fabric, in the present study, phosphorus and nitrogen containing flame retardants were synthesized and applied on cotton fabric with alkoxysilane using the sol-gel method [21]. The structure and surface morphology of the untreated and treated cotton fabric were investigated by FTIR spectroscopy and SEM equipped with an energy dispersive X-ray spectroscopy (EDS). Thermogravimetric analysis (TGA) and flammability tests (LOI and auto-flammability) were used for evaluating the thermal degradation and stability and combustion behaviour of the treated cotton samples.

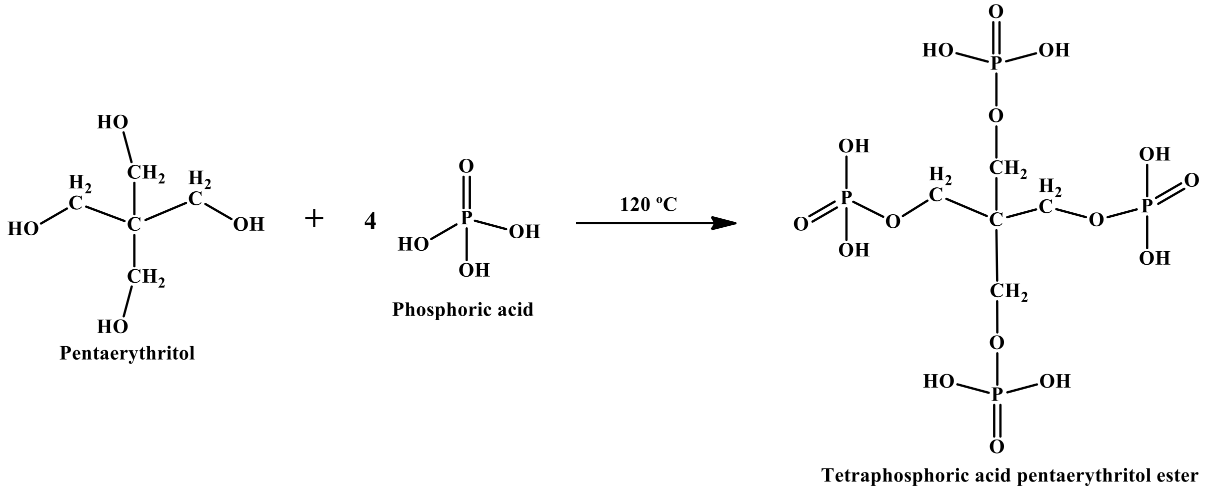
EXPERIMENTAL

Grasim Industries Limited (Aditya Birla Group) supplied the cotton fabric (165 g/m^2). The fabric was washed with water and then dried before use. The chemicals *viz.* aminopropyltriethoxysilane (APTES) and phosphoric acid (PA) from Sigma-Aldrich (India), while ethylene glycol, glycerol, pentaerythritol and ammonia were bought from Himedia Chemicals Co. (India). Without any additional purification, all of these chemicals were used as supplied.

Synthesis of phosphoric acid esters: In a 250 mL beaker, ethylene glycol (5.49 mL, 0.1 mol) and phosphoric acid (9.6 mL, 0.2 mol) were added and the mixture was heated to 120 ºC while being stirred magnetically. A viscous faint yellow liquid was collected after 4 h. The formation of diphosphoric acid ethyleneglycol ester (diPEG) was characterized by ^{1}H NMR, ^{31}P NMR and HRMS. In the same way, triphosphoric acid glycerol ester (triPGE) was synthesized using glycerol and phosphoric acid in 1:3 molar ratio, respectively. The tetraphosphoric acid pentaerythritol ester (tetraPPE) was synthesized in similar way using pentaerythritol and phosphoric acid in 1:4 molar ratio as shown in **Scheme-I** and characterized by ^{1}H NMR, ^{31}P NMR and HRMS.

Treatment of cotton fabric: After thoroughly mixing 40 g of ethanol and 50 g of distilled water in a flask, 5 g (0.02 mol) of diphosphoric acid ethylene glycol ester (diPEG) was added with continuous stirring for 15 min. Then 5 g (0.02 mol) sol-gel precursor (APTES) was added to the same flask with continuous stirring within 15 min. A piece of 30 cm × 8 cm size of washed and dried cotton fabric was soaked in the prepared solutions and kept soaked for 30 min. The pH value around 7 in neutral range was maintained by adding ammonia solution. The cotton fabric sample was then squeezed to get rid of any extra solution and the sample was vacuum-dried at 120 ºC until its weight remained constant. This sample is named as CF-diPEG. Similarly, samples of cotton fabric were treated with triPGE and tetraPPE flame retardants along with APTES a sol-gel precursor and abbreviated as CF-triPGE and CF-tetraPPE, respectively. The mass of each fabric sample was recorded before and after impregnation with the sol-gel solution to determine the weight gain (Table-1).

Characterization: Infrared spectroscopy was used to examine the bonding interaction between cotton cellulose and the applied formulations. FT-IR spectra were recorded using a Shimadzu IR affinity-I FTIR spectrometer at room temperature, in the range of 4000-400 cm^{-1}. A FESEM (7610F Plus/JEOL) with a 20 kV beam voltage and energy dispersive X-ray spectroscopy (EDS) was employed to analyze the surface morphology and elemental makeup of the treated samples. Pieces of cotton fabric measuring 0.5 cm × 0.5 cm each were cut, adhered to conductive tapes and then gold-metallized before analysis. Thermogravimetric analyzer (STA-TGA) was used to perform thermogravimetric analyses from 10 to 600 ºC at a heating rate of 10 ºC/min in nitrogen and air atmospheres



Scheme-I: Synthesis of tetraphosphoric acid pentaerythritol ester (tetraPPE)

TABLE-1
COTTON FABRIC SAMPLES DESCRIPTION AND WEIGHT GAIN

Sample	Phosphoric acid ester	Sol-gel precursor	Weight gain (%)
Cotton fabric (CF)			
CF-diPEG	Diphosphoric acid ethylene glycol ester (diPEG)	Aminopropyltriethoxy-silane (APTES)	06.9
CF-triPGE	Triphosphoric acid glycerol ester (triPGE)	APTES	07.2
CF-tetraPPE	Tetra phosphoric acid pentaerythritol ester (tetraPPE)	APTES	11.8

(100 mL/min). The samples were placed in open alumina pans (about 10 mg) to assess the thermal stability of the fabrics.

Burning behaviour of cotton fabric samples was assessed using a Paramount 45º Automatic flammability tester (ISO 9001:2008 group) in accordance with ASTM D1230-94, the Standard Test Method for Flammability of Apparel Textiles. Five seconds was chosen as the ignition time for this investigation. The studies were carried out at room temperature (30 ºC). The specimen's (15 cm × 5 cm) burning characteristics and duration were recorded after it was exposed to flame using a digital camera. Limiting Oxygen Indexer equipment ISO 4859-2 was used to analyze cotton fabric samples to measure Limiting Oxygen Index in accordance with ASTM D 2863. The cotton specimen of size 15 cm × 5 cm was ignited at the top. The oxygen level was adjusted until the sample could hardly continue to burn. Greater values in the LOI test signify a higher degree of flame resistance in cotton fabric.

The air permeability of both untreated and treated cotton fabric samples was examined using air permeability tester (IS: 11056-1984). The results reported were the average of three examinations. In compliance with ASTM D 1388 fabric testing protocols, the stiffness of cotton fabric samples was assessed using the Shirley stiffness tester. Each sample was tested four times and an average result was reported.

RESULTS AND DISCUSSION

NMR and HRMS studies: ^{1}H NMR, ^{31}P NMR and HRMS spectra of diphosphoric acid ethyleneglycol ester (diPEG), triphosphoric acid glycerol ester (triPGE) and tetraphosphoric acid pentaerythritol ester (tetraPPE) were recorded. The structure of diPEG compound was confirmed by ^{1}H NMR (DMSO, δ ppm): 3.51(m, 4H, CH_2), ^{31}P NMR: 0.04 ppm and HRMS (ESI): *m/z* calculated $[M+H]^+$ $C_2H_8O_8P_2$: 221.9694, found: 222.9767 (Fig. 1). In the same way, the structure of triPGE compound was confirmed by ^{1}H NMR (DMSO, δ ppm): 3.34 (s, 4H, CH_2), 3.25(m, 1H, CH), ^{31}P NMR: 0.21 ppm and HRMS (ESI): *m/z* calculated $[M+H]^+$ $C_3H_{11}O_{12}P_3$ *m/z* = 331.9463, found: 332.9594 (Fig. 2). Also, the tetraPPE compound was confirmed by characterizing with ^{1}H NMR (DMSO, δ ppm): 3.38(s, 8H, CH_2), ^{31}P NMR: 0.07ppm and HRMS (ESI): *m/z* calculated $[M+H]^+$ $C_5H_{16}O_{16}P_4$ *m/z* = 455.9389, found 457.2893 (Fig. 3).

FTIR spectral studies: Fig. 4 displays FTIR spectra of the cotton fabric samples. For the untreated cotton fabric, the characteristic bands at 3434 and 2890 cm^{-1} (*str*. vibration) are due to -OH and CH groups [22]. The spectrum of treated cotton fabric shows a few characteristic bands that are not present in untreated CF. The band at 1240 cm^{-1} (*str*. vibration) is due to the P=O structure. The stretching vibration band at 1044 cm^{-1} is due to the Si–O–Si bond. Absorption band at 794 cm^{-1} is due to stretching vibrations of the Si-C structure. The aforementioned analysis demonstrates that the surface of treated cotton contains phosphate and silane.

Morphology: The surface morphology and elemental content of the cotton fabric samples were examined using SEM in conjunction with EDS to gain a better understanding of the changes after treatment of cotton fabric. The SEM morphology of CF, treated fabric samples (CF-diPEG, CF-triPGE and CF-tetraPPE) and heated samples of CF and CF-tetraPPE are shown in Fig. 5. The micrographs show that all treated

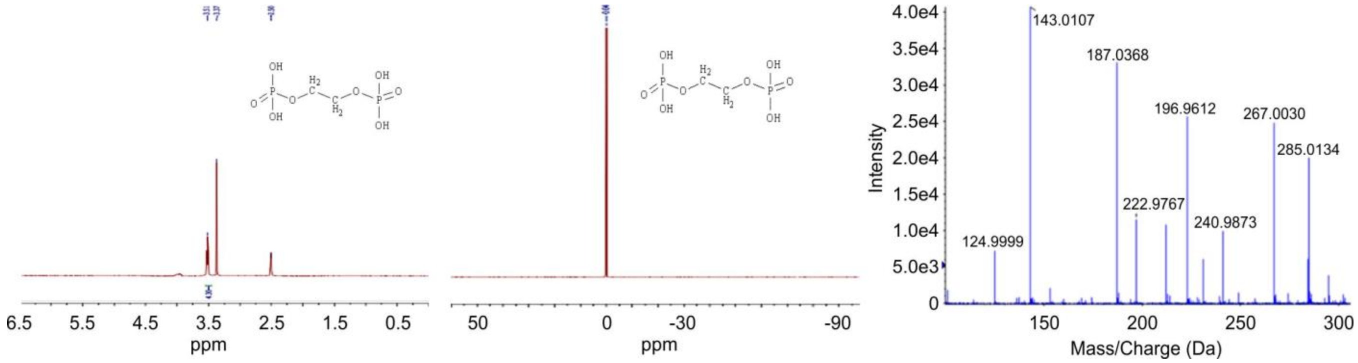


Fig. 1. ^{1}H NMR, ^{31}P NMR and HRMS of diphosphoric acid ethyleneglycol ester (diPEG)

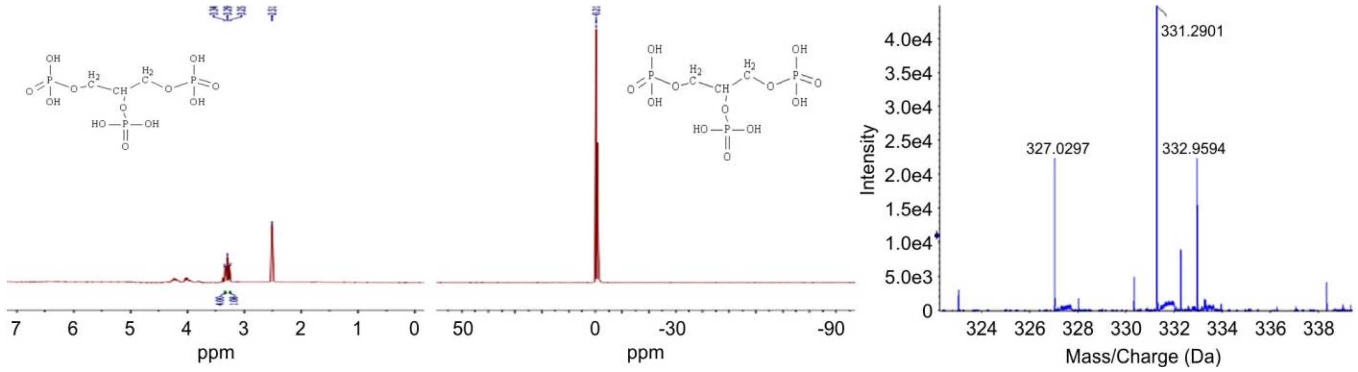


Fig. 2. ^{1}H NMR, ^{31}P NMR and HRMS of triphosphoric acid glycerol ester (triPGE)

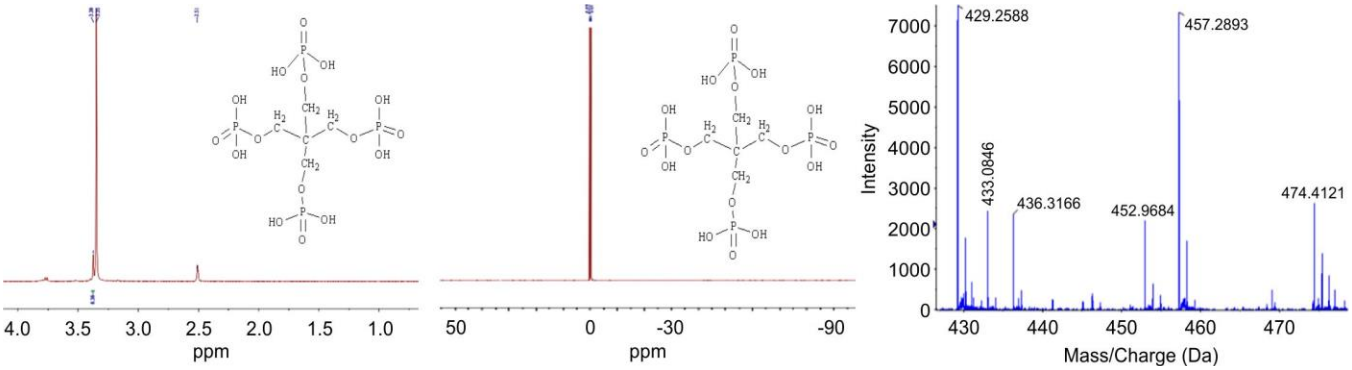


Fig. 3. ^{1}H NMR, ^{31}P NMR and HRMS of tetraphosphoric acid pentaerythritol ester (tetraPPE)

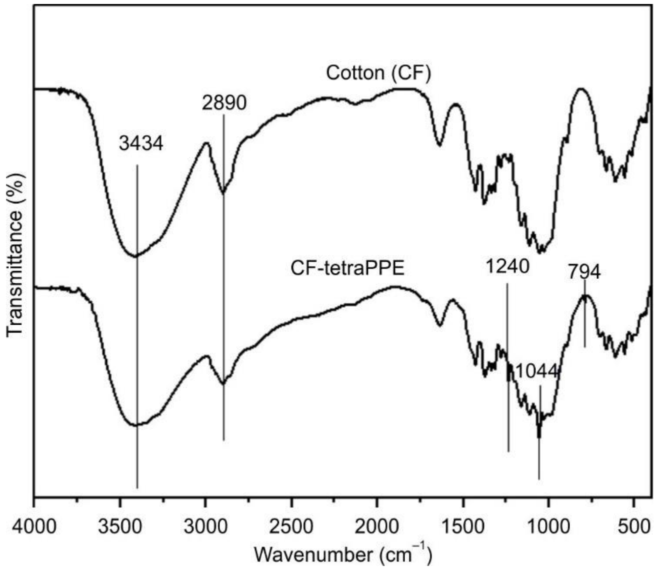


Fig. 4. FTIR spectra of cotton fabric (CF) and treated cotton fabric (CF-tetraPPE) samples

cotton samples share a like shape and structure to CF, while the surface of untreated fabric is comparatively smooth. The treated fibres exhibit an uneven film on their surface and appear to have a larger diameter, giving them some rough and uneven appearance. These variations indicated that flame retardant had been effectively applied to the cotton fabric surface. The phosphate group in flame retardant and OH groups in cotton cellulose absorb and immobilize phosphorous acid molecules on their surface through a covalent bonding. Meanwhile, since APTES has a -NH$_2$ group, which reacts chemically with phosphorus flame retardant. When water easily hydrolyzes APTES to make ethanol, a thin layer of silanol condensate is created [23-25]. The plausible mechanism of the reaction is shown in **Scheme-II**. When the treated fabric (CF-tetraPPE) is laundered, its surface seems smoother than that of treated cotton, but it is not as smooth as that of untreated cotton fabric, signifying that some flame retardant is still present on the surface of fabric.

The residues obtained after heating the sample at 300 ºC for 5 min were gathered and subjected to SEM analysis in

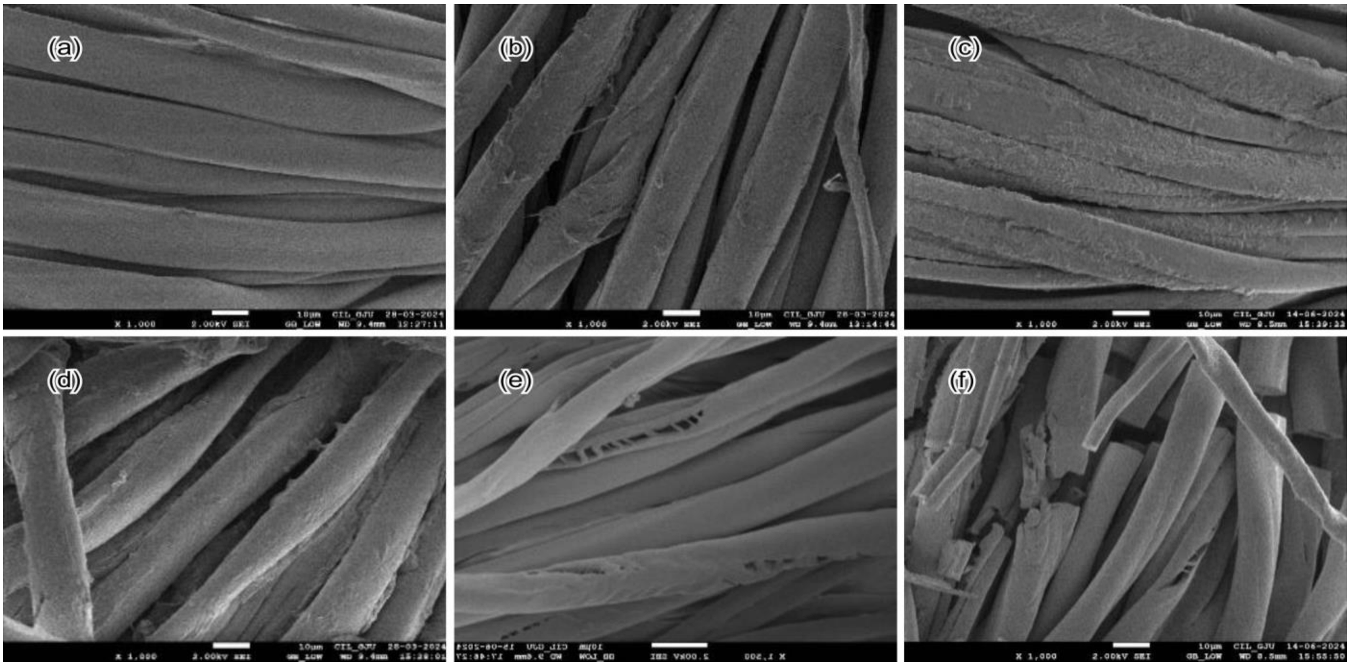
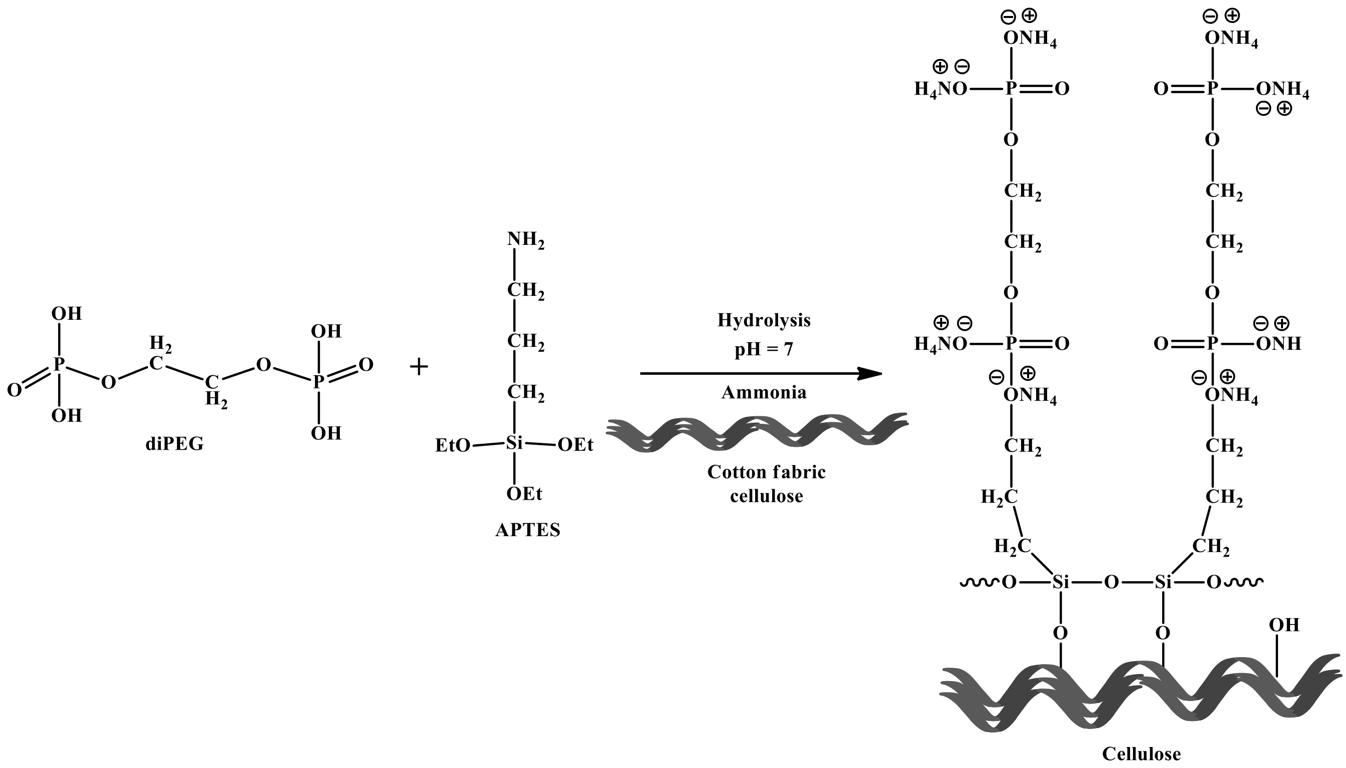


Fig. 5. SEM images of (a) CF, (b) CF-diPEG, (c) CF-tetraPPE, (d) after laundring: CF-tetraPPE$_L$, (e) heated CF$_B$ and (f) heated CF-tetraPPE$_B$



Scheme-II: Interaction of diPEG and aminopropyltriethoxysilane (APTES) with cotton fabric cellulose

order to assess the morphological and compositional changes following heating. After heating, the structure of treated cotton fabric appears to be similar to that of the original cotton with threads break down with no strength on touching indicating that the material might exhibit predominantly condensation flame retardancy.

The compound makeup of the fabric surface examined by EDS is presented in Fig. 6 and elemental data is given in Table-2. Following fabric treatment, the amount of C and O decreased slightly as compared to untreated cotton and lesser amounts of Si, P and N were observed in coated fabric samples, further demonstrating the effectiveness of flame retardant on the fiber's surface. After heating the CF-tetraPPE sample, an increase amount of Si and N elements is observed in the char residue, demonstrating the exceptional carbonization ability of flame retardant [26].

TABLE-2
EDX DATA OF THE COTTON FABRIC (CF) SAMPLES

Sample	C (%)	N (%)	O (%)	Si (%)	P (%)
CF	58.3	–	41.7	–	–
CF-diPEG	46.3	4.5	46.5	0.7	1.7
CF-triPGE	48.7	5.8	40.5	2.3	2.6
CF-tetraPPE	38.1	8.5	48.6	0.3	4.6
CF-tetraPPE$_L$	48.1	3.5	44.5	0.9	3.0
CF-tetraPPE$_B$	54.4	8.7	32.9	0.9	3.1

Flammability behaviour: The 45° Auto-flammability test and the limiting oxygen index (LOI) are the two popular quantitative techniques for assessing the flammability of the material. The flame resistance of textiles is positively correlated with both LOI and vertical flammability tests. The LOI values of cotton fabric samples treated using the sol-gel

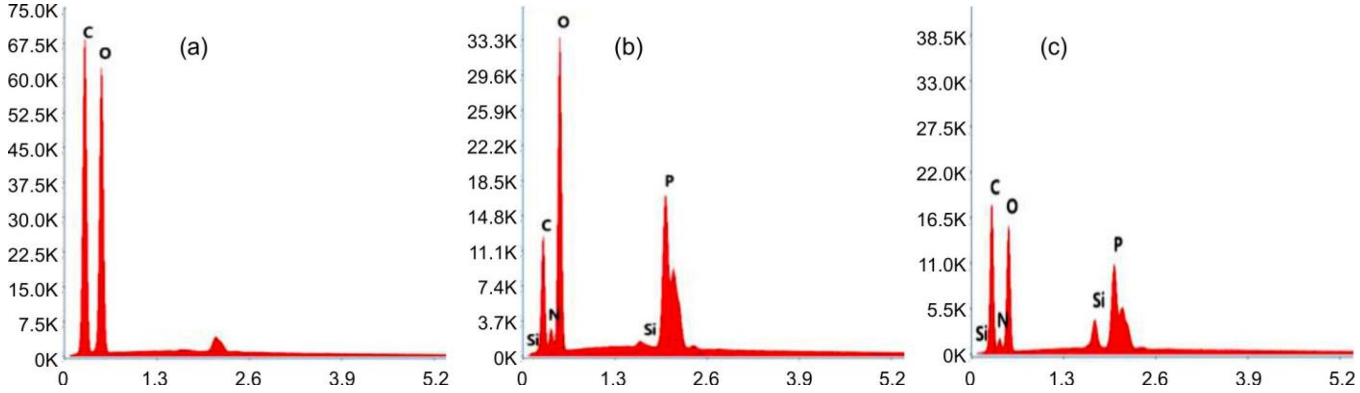


Fig. 6. EDS spectra of (a) CF, (b) CF-tetraPPE and (c) heated CF-tetraPPE$_B$

TABLE-3
FLAMMABILITY, AIR PERMEABILITY AND STIFFNESS VALUES FOR COTTON FABRIC SAMPLES

Sample	Flame spread time (s)	Char length (cm)	Auto flammability test (Pass/Fail)	LOI (%)	Air permeability ($m^3/m^2/min$)	Stiffness (cm)
CF	14	BL#	Fail	19.0	150.4	2.1
CF-diPEG	SE*	1.9	Pass	33.0	151.0	2.5
CF-triPGE	SE*	1.7	Pass	34.8	153.4	2.4
CF-tetraPPE	SE*	1.4	Pass	38.3	154.8	3.1

*SE = Self-extinguished, #BL = Burn entire length

technique are displayed in Table-3. The treated cotton fabric had a LOI value of up to 38.3%, while the control cotton fabric had a value of 19.0%. Also cotton fabric that has not been treated is easily ignited, burns completely and leaves no trace. The untreated cotton fabric of length 15 cm burned completely and intensely in 45ºAuto-flammability test within 14 s. When the flame source is removed after 5 sec, samples of cotton fabric treated with sol-gel self-extinguish char length only 1.4 to 2.2 cm. These results suggest phosphorus doped sol-gel precursor have a synergistic effect on the flame retardancy of cotton fabric.

Air permeability and stiffness measurement: The permeability of fabric is directly related to its structure, which is normally defined by its porosity. Table-3 shows that air permeability of untreated fabric is 150.4 $m^3/m^2/min$, whereas for treated fabric samples it ranges from 151.0 to 154.8 $m^3/m^2/min$. There is slight increase in air permeability after treatment of fabric, hence porosity of fabric is maintained after treatment and there is no process of shrinkage of fabric. The resistance of a fabric to bending or flexing is measured by its stiffness. The material, yarn, weave, finish and thickness of the fabric are some of the variables that affect it. Untreated cotton fabric has a stiffness of 2.1 cm, while treated fabric samples have stiffness values between 2.5 and 3.1 cm. Consequently, it is observed that following treatment, the treated flexibility of fabric declines while its air permeability increases slightly.

Thermal studies: TG and DTG of treated and untreated cotton fabric in N_2 and air are displayed in Fig. 7 and the corresponding data are displayed in Tables 4 and 5. There were basically three stages to this heat degradation process. Water evaporation was the primary cause of the slight weight loss that happened at temperatures lower than 200 ºC. As the temperature increased, two competing reactions occurred: at lower temperatures, glycosyl units underwent dehydration to form carbon, whereas at higher temperatures, they deploymerized to produce flammable volatile compounds [27]. According to data, initial decomposition temperature ($T_{5\%}$) and maximum first weight loss temperature (T_{max1}) for samples in both nitrogen and air atmospheres are close. For untreated fabric sample initial weight loss occurs near at 290 ºC while for all treated samples initial decomposition start at lower temperature, hence presence of flame retardant coating decrease $T_{5\%}$ but it reduces maximum degradation temperature T_{max1}. This may be due to the breakdown of flame retardant earlier, while phosphoric acid molecules are produced to encourage the dehydration and carbonization of cotton textiles at a comparatively lower temperature. The char layer that acts as a barrier to stop the underlying material from decomposition [28]. Finally leading to enrichment in residual mass for untreated fabric up to 46.2% for treated sample (CF-tetraPPE) in N_2 atmosphere. All samples had relatively fewer residues in air environments due to further oxidation, which are maximum up to 27.2% for CF-tetraPPE sample. Only in air atmosphere, second weight loss of untreated and treated fabric samples occur at higher temperature due to further oxidation referred to as $T_{max2.}$ According to TGA data, the flame-retardant samples exhibit enhanced carbonization and thermal oxidative stability, which helps to increase the flame retardancy of cotton textiles.

TABLE-4
TG-DTG DATA OF COTTON FABRIC SAMPLES IN NITROGEN ATMOSPHERE

Sample	$T_{5\%}$ (ºC)	DTG T_{max1} (ºC)	Residue at T_{max1} (%)	Residue at 600 ºC (%)
CF	290	349	66.0	00.0
CF-diPEG	213	268	81.8	41.6
CF-triPGE	204	262	82.8	43.5
CF-tetraPPE	201	248	81.7	46.2

Conclusion

The phosphoric acid esters-based organic compounds were synthesized and applied on cotton fabric with sol-gel precursor APTES using sol-gel approach. This research illustrates the notable flame-retardant effectiveness of APTES and phosphorus acid derivatives on cotton fabric. All treated samples passed the auto-flammability test. The significant rise in LOI from 19.0 to 38.3% in the treated samples underscores the considerable enhancement in flame retardancy.

TABLE-5
TG-DTG DATA OF COTTON FABRIC SAMPLES IN OXYGEN ATMOSPHERE

Sample	$T_{5\%}$ (ºC)	DTG T_{max1} (ºC)	Residue at T_{max1} (%)	DTG T_{max2} (ºC)	Residue at T_{max2} (%)	Residue at 600 ºC (%)
CF	288	340	57.6	485	02.8	00.0
CF-diPEG	210	272	73.1	505	27.0	14.7
CF-triPGE	183	246	73.8	503	31.5	16.9
CF-tetraPPE	195	260	74.5	517	36.7	27.2

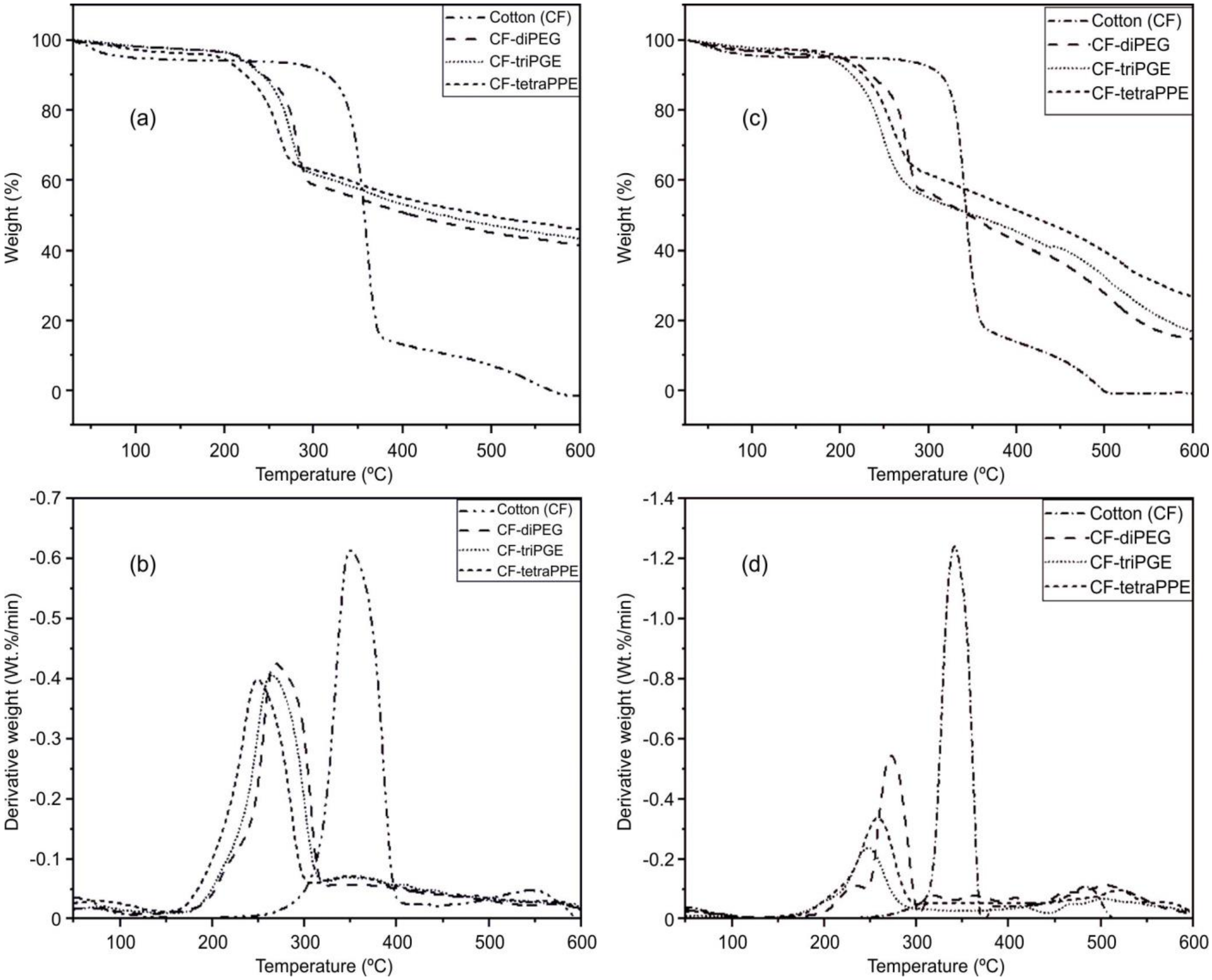


Fig. 7. TG and DTG thermograms of cotton fabric samples in (a, b) nitrogen and (c, d) air atmospheres

The thermal stability of the treated cotton fabric samples is significantly enhanced when compared to that of virgin cotton fabric at elevated temperatures. The findings from the TGA demonstrated that the ability to form char can be significantly enhanced following treatment, with 27.2% and 38.3% of the residue for the CF-tetraPP sample at 600 °C in air and N_2 atmospheres, respectively. The created char film on the surface obstructs the transfer of heat and oxygen, thereby inhibiting the further degradation of fabric. This research presents an easy approach to create eco-friendly cotton fabrics that exhibit outstanding flame resistance properties.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

The authors declare that no AI tools were used in the preparation or writing of this research/review article.

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