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Effect of Crosslinking in Phosphorus-Nitrogen based Hydrogel on the Flammability of Cotton Fabric

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To improve the flame retardancy of cotton fabrics for applications requiring enhanced fire safety, a phosphorus- and nitrogen-containing crosslinked hydrogel, poly(N-hydroxymethyl acrylamide)-*bis*[2-(methacryloyloxy)ethyl]phosphate (PNBP), was synthesized and evaluated as a flame-retardant coating. The hydrogel was characterized by FTIR spectroscopy, mass spectrometry and thermogravimetric analysis. Cotton fabrics were coated using the admicellar polymerization technique, with cetylpyridinium chloride as the cationic surfactant and 2,2′-azobis(2-methylpropionamidine) dihydrochloride as the initiator. The concentration of *bis*[2-(methacryloyloxy)ethyl]phosphate crosslinker was varied to investigate its effect on flame-retardant performance. The fabric treated with the formulation containing 50% crosslinker did not ignite under the specified auto-flammability test conditions and exhibited a limiting oxygen index (LOI) of 36.0%. The results showed an improvement in flame-retardant performance with increasing crosslinker content under the conditions investigated. Field-emission scanning electron microscopy (FESEM) was used to examine the surface morphology of the treated fabrics.

Keywords: Cotton fabric, Flame retardant, Admicellar polymerization, Crosslinked hydrogel, Thermal stability.

INTRODUCTION

Cotton is widely used in apparel, bedding, curtains, carpets and other textile products due to its softness, comfort and versatility [1]. Its inherent flammability limits its use in applications where fire safety is a primary concern. Untreated cotton can ignite readily and sustain flame propagation, which increases the risk of fire-related damage and injury. The limiting oxygen index (LOI) of untreated cotton is commonly reported to be approximately 18%, although the value may vary with fabric structure and test conditions [2,3]. Developing effective flame-retardant treatments is important for improving the fire resistance of cotton textiles without compromising their practical utility.

Cellulose, the principal constituent of cotton, undergoes thermal decomposition through competing pathways involving dehydration, depolymerization, oxidation and the formation of volatile compounds and carbonaceous residue [4]. The relative contribution of these pathways depends on the heating conditions and surrounding atmosphere. Levoglucosan (1,6-anhydro-β-D-glucopyranose) is one of the volatile products formed during cellulose degradation and can contribute to the generation of combustible gases [5,6]. Flame-retardant treatments can alter the decomposition process by promoting char formation and limiting the release of combustible volatiles. The development of efficient treatments that provide adequate fire protection while maintaining acceptable material properties remains an important objective in textile research [7,8].

Phosphorus- and nitrogen-containing compounds are widely investigated for flame-retardant applications involving cellulosic materials [9-12]. Under thermal exposure, phosphorus containing groups can generate acidic species that promote cellulose dehydration and favour the formation of a carbonaceous layer. This layer may restrict heat transfer and reduce the release of combustible degradation products. Nitrogen-containing groups can contribute through the release of non-combustible gases or changes in condensed-phase decomposition, depending on their chemical structure and thermal behaviour [13-15]. Combining phosphorus and nitrogen within a single formulation may provide complementary flame-retardant effects, although the extent of their interaction must be established experimentally [16-19].

Several strategies have been explored to improve the flame resistance of cotton fabrics such as intumescent coatings [20],

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layer-by-layer assembly [21], chemical grafting [22] and sol-gel processing [23]. Admicellar polymerization offers an alternative approach to surface modification, using surfactant aggregates as localized reaction domains for polymer formation [24]. Previous studies [25-27] have applied this technique to polymer coatings on substrates such as alumina, aluminium and glass fibre. Its application to cotton fabrics presents an opportunity to investigate the formation of functional polymer networks at textile surfaces. A crosslinked hydrogel containing phosphorus and nitrogen may combine the structural contribution of a polymer network with the flame retardant functionality of these elements [28]. However, the influence of crosslinker concentration on coating characteristics and the resulting fire performance requires systematic evaluation.

The study aimed to assess the effect of crosslinker concentration on the flame-retardant performance of PNBP-coated cotton and examine its relationship with the thermal characteristics of the treated material. Hence, a phosphorus- and nitrogen containing crosslinked hydrogel, poly(N-hydroxymethyl acrylamide)-*bis*[2-(methacryloyloxy)ethyl]phosphate (PNBP), was synthesized and applied to cotton fabric by admicellar polymerization using a cationic surfactant. Different concentrations of the phosphorus-containing crosslinker were investigated to assess their influence on the thermal and flame retardant properties of the treated fabric. The hydrogel was characterized by FTIR spectroscopy, mass spectrometry and thermogravimetric analysis, while the surface morphology of the treated fabric was examined by FESEM. Flame resistance was evaluated using auto-flammability testing and limiting oxygen index measurements.

EXPERIMENTAL

The chemicals *viz.* N-hydroxymethyl acrylamide (NHMA) as monomer, 2,2′-azo*bis*(2-methyl propionamidine)dihydrochloride (AAPH) as initiator, cetylpyridinium chloride (CPC) as cationic surfactant and *bis*[2-(methacryloyloxy)ethyl]-phosphate (*Bis*MEP) as crosslinker were purchased from Sigma-Aldrich, India. Cotton fabric (165 g/m^2) was procured from Aditya Birla, Grasim Industries Ltd., India. The cotton fabric was washed with normal water and dried prior to use.

Synthesis of poly(*N*-hydroxymethyl acrylamide)-*bis*-[2-(methacryloyloxy)ethyl]phosphate crosslinked hydrogel (PNBP): The PNBP crosslinked hydrogel containing 20% *Bis*MEP relative to NHMA monomer was synthesized *via* free-radical polymerization for its characterization as a flame retardant compound before its use *in situ* coating process on the fabric. The NHMA (1.0 g, 9.89 mmol), *Bis*MEP (0.63 g, 1.95 mmol) and AAPH (2.5 mg, 9.2 × 10^{-3} mmol) were dissolved in distilled water (3.0 mL) in a Schlenk flask. Prior to polymerization, the reaction mixture was bubbled with N_2 gas to avoid oxygen. The polymerization was then conducted at 65 ºC for 4 h under a continuous flow of nitrogen gas. Upon completion of the reaction, the prepared hydrogel was washed with alcohol to remove residual monomers and unused crosslinker if any. The purified material was subsequently filtered and dried in vacuum at 60 ºC for 2 h. The synthetic route for the PNBP crosslinked hydrogel is given in **Scheme-I**.

Coating of crosslinked hydrogel on cotton fabric by admicellar polymerization: A coated cotton fabric sample (TC_{PNBP20}) having 20% *Bis*MEP crosslinker relative to NHMA monomer in PNBP hydrogel was prepared *via* an *in situ* admicellar polymerization process as follow. A cotton fabric sample (15 cm × 5 cm, 1.06 g) was placed in a Schlenk flask, followed by the addition of NHMA (1.0 g, 9.89 mmol), *Bis*MEP (0.63 g, 1.95 mmol), AAPH (2.5 mg, 9.2 × 10^{-3} mmol) as the radical initiator, CPC surfactant (0.8 mM) and distilled water (10 mL). The reaction mixture was purged with nitrogen to remove dissolved oxygen and maintained at 30 ºC under a continuous nitrogen atmosphere for 8 h to facilitate admicelle formation and to achieve equilibrium monomer solubilization within the admicelles. Subsequently, the temperature was increased to 65 ºC to initiate polymerization, which was allowed to proceed for an additional 4 h. After completion of polymerizetion process, the coated fabric was taken out from the flask, washed thoroughly with water to eliminate residual reagents and then dried in vacuum oven at 60 ºC to constant weight. Using the same procedure, the TC_{PNBP30}, TC_{PNBP40} and TC_{PNBP50} samples were also prepared by having 30, 40 and 50% *Bis*MEP crosslinker concentration relative to NHMA monomer in PNBP hydrogel, respectively (Table-1).

TABLE-1
DESCRIPTION OF COTTON FABRIC SAMPLES

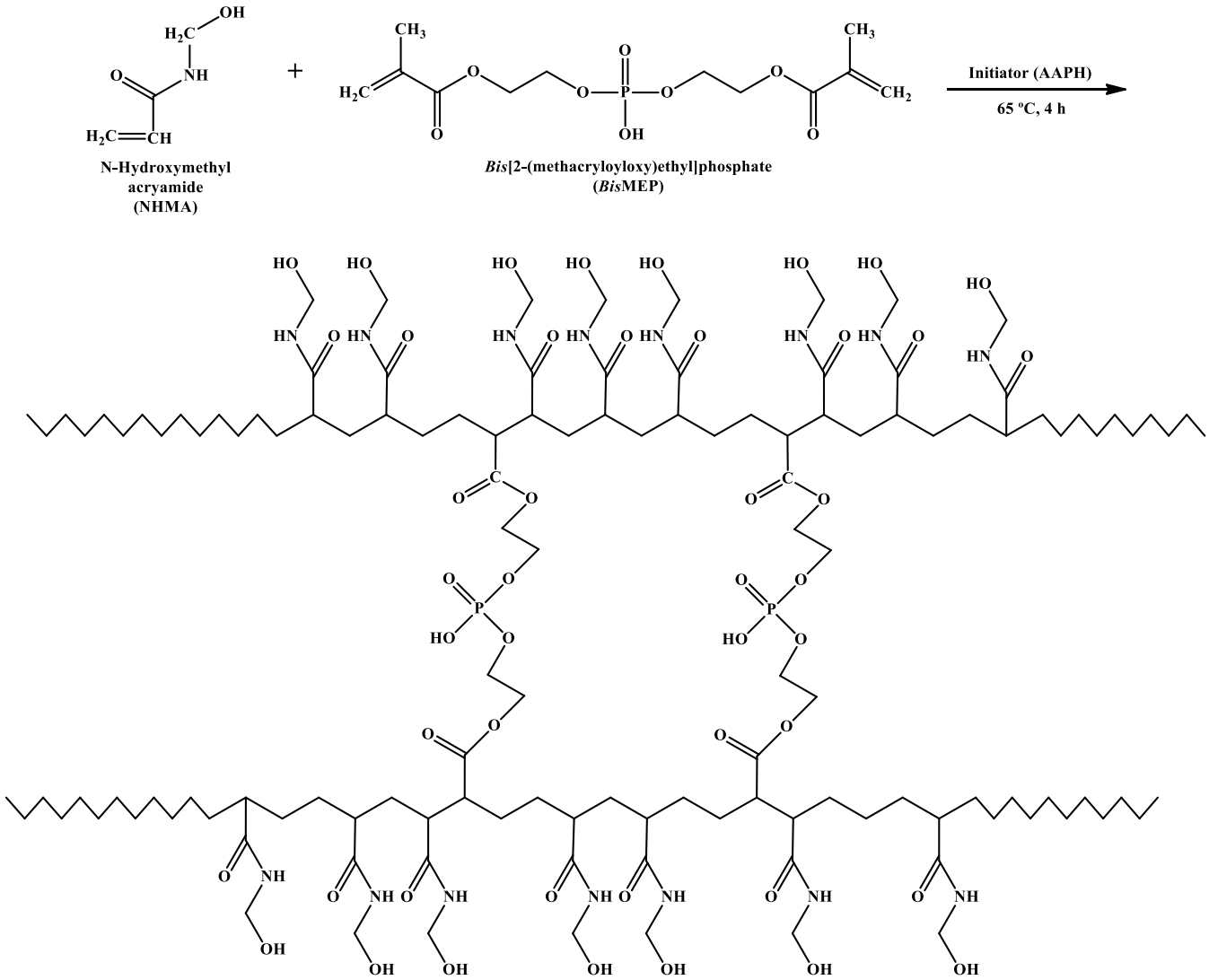
Sample	NHMA monomer (mM)	*Bis*MEP crosslinker (mM)	AAPH initiator (mg)	CPC surfactant (mM)
Pure cotton (PC)	–	–	–	–
TC_{PNBP20}	9.89	1.95	2.5	0.8
TC_{PNBP30}	9.89	2.94	2.5	0.8
TC_{PNBP40}	9.89	3.93	2.5	0.8
TC_{PNBP50}	9.89	4.92	2.5	0.8
$TC_{PNBP40L}$	Sample after home laundering of TC_{PNBP40}			
$TC_{PNBP50L}$	Sample after home laundering of TC_{PNBP50}			

RESULTS AND DISCUSSION

Characterization of poly(N-hydroxymethyl acrylamide)-*bis*[2-(methacryloyloxy)ethyl]phosphate (PNBP) crosslinked hydrogel

FTIR analysis: Fig. 1 depicts spectra of NHMA monomer, *Bis*MEP crosslinker and PNBP crosslinked hydrogel. The spectrum of *Bis*MEP gives bands at 1721 (C=O *str.* vibration of ester group), 1627 (C=C in methacrylate), 1383 (CH_3 symmetric bending vibration) and 1165 cm^{-1} (P=O *str.* vibration of phosphate). FTIR spectrum of NHMA gives bands at 1660 (C=O *str.* vibration, amide-I), 1540 (N-H bending coupled with C-N *str.* vibration, amide-II) [29], 1627 (vinyl C=C *str.* vibration) and 1024 cm^{-1} (C-O-H *str.* vibrations of hydroxymethyl group). Bands present in the spectrum of PNBP at 1739, 1648, 1552, 1383, 1190 and 1027 cm^{-1} indicated the successful incorporation of *Bis*MEP crosslinker in the polymeric chain of NHMA. The disappearance of the band at 1627 cm^{-1} (C=C *str.* vibration) in backbone of polymeric chain also confirms the crosslinked polymerization.

HRMS analysis: Fig. 2 represents the mass spectra of monomer NHMA monomer, *Bis*MEP crosslinker and PNBP



Scheme-I: Synthesis of poly(*N*-hydroxymethyl acrylamide)-*bis*[2-(methacryloyloxy)ethyl]phosphate (PNBP) crosslinked hydrogel

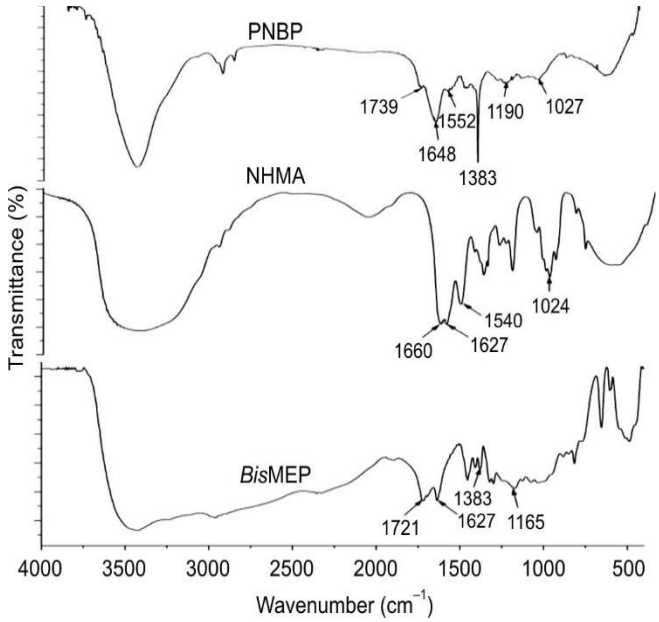


Fig. 1. FTIR spectra of *Bis*MEP, NHMA and PNBP hydrogel

hydrogel. In mass spectrum of NHMA monomer, the experimental value ($M+H^+$) was observed to be the same as the calculated value of the monomer (102.0540). The experimental value ($M+H^+$) for *Bis*MEP crosslinker was observed to be the same as the calculated value of the crosslinker (323.1493). Similarly, the experimental value ($M+H^+$ = 424.2249) of PNBP hydrogel was observed to be ($M+H^+$ = 423.2033) the same as the calculated value for the smallest unit of hydrogel, which confirmed the successful formation of PNBP crosslinked hydrogel.

Characterization of coated cotton fabric samples with phosphorus-nitrogen based crosslinked hydrogel

FTIR analysis: Fig. 3 depicts the spectra of FTIR of pure cotton fabric (PC) and treated fabric (TC_{PNBP50}) samples showing characteristic absorption bands at 3412 (O–H *str.*), 2902 (C–H *str.*), 1632 (C=C *str.*), 1426 (CH_2 sym. *bend.*), 1338 (C–H *bend.*), 1164 (C–O–C asym. *str.*) and 1049 cm^{-1} (C–O *str.*). In the hydrogel-coated cotton fabric, additional absorption bands were observed in the range of 3500-3350 cm^{-1} due to the appearance of the O–H and N–H stretching vibrations. New bands at 1723 (C=O *str*. of *Bis*MEP), 1159 (P=O *str*. of

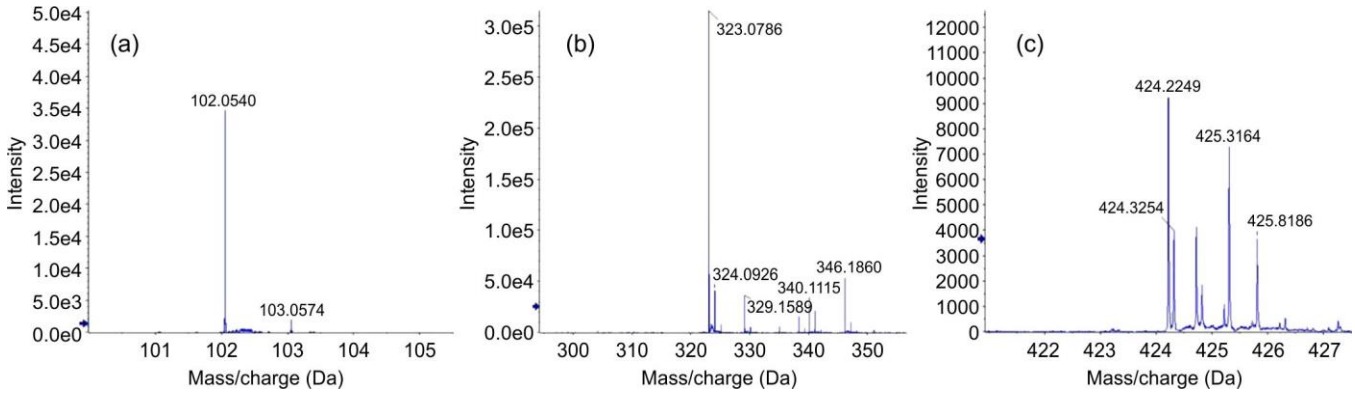


Fig. 2. Mass spectra of (a) NHMA, (b) *Bis*MEP and (c) PNBP hydrogel

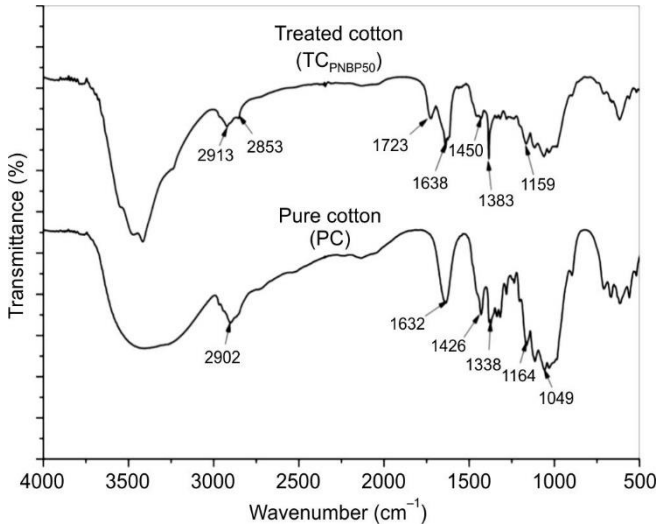


Fig. 3. FTIR spectra of pure cotton (PC) and treated cotton fabric (TC$_{PNBP50}$)

the phosphate group in *Bis*MEP), 1638 (amide C=O *str*. of NHMA), 1383 (amide C–N *str*. of NHMA) and 1450 cm^{-1} (CH_2 *bend*.) further confirm the hydrogel formation. The presence of these additional characteristic bands indicates the successful deposition of a phosphorus and nitrogen-based polymeric layer on the cotton fabric.

Thermal studies: TG curves of the PNBP hydrogel with 20% crosslinker, pure cotton fabric (PC) and treated fabric samples in air and nitrogen atmospheres are displayed in Fig. 4. The PNBP flame retardant in air atmosphere degrades gradually upto 250 ºC with about 15% weight loss including loss of absorbed moisture and bounded water alongwith decomposition of hydroxymethyl group due to interactions in oxidative environment. Whereas, no weight loss occurs upto 250 ºC in inert environment except some loss of absorbed moisture up to 100 ºC. Further, the amide and phosphate bonds cleavage takes place majorly up to 450 ºC with the evolution of volatile gases like NH_3, CO and CO_2 [30] leaving 42% residue in presence of air. In case of N_2 atmosphere, the PNBP leaves less residue (34%) at 550 ºC due to degradation of polyacrylamide chain and phosphate group.

The corresponding thermal degradation data of PC and treated cotton fabric samples in air and nitrogen atmospheres are shown in Table-2. The thermal degradation of pure cotton fabric in an oxygen atmosphere resulted in almost complete decomposition leaving only 1.5% residual char at 600 ºC. The observed weight loss is associated with dehydration, random chain scission, decomposition with the evolution of volatile products predominantly levoglucosan responsible for its high flammability [31]. In N_2 atmosphere, pure cotton fabric degraded in one step up to 400 ºC with less release of

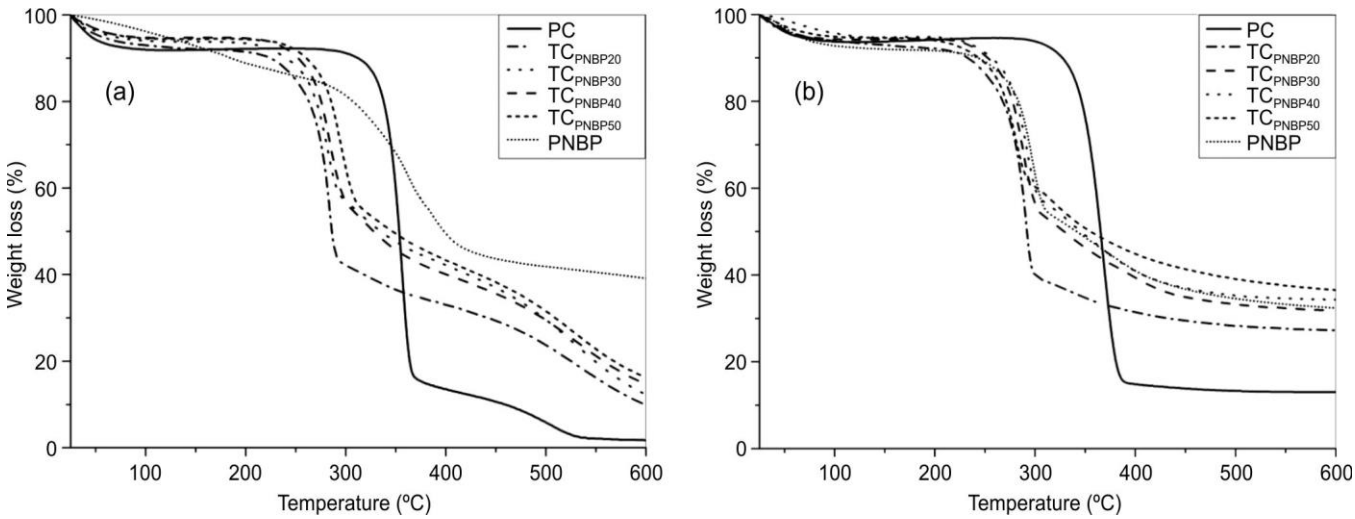


Fig. 4. TG curves of PNBP hydrogel, pure cotton (PC) and TC$_{PNBP20}$ to TC$_{PNBP50}$ in (a) air and (b) nitrogen atmospheres

TABLE-2
TG DATA OF PURE AND TREATED COTTON FABRIC SAMPLES IN AIR AND NITROGEN ATMOSPHERES

Sample	Stage	Temp. range (ºC)		Weight loss (%)		DTG (ºC)		$T_{10wt\%}$ (ºC)		$T_{50wt\%}$ (ºC)		Char at 600 ºC (%)	
		Air	N_2	Air	N_2	Air	N_2	Air	N_2	Air	N_2	Air	N_2
PC	1st	100-600	100-600	96.7	91.0	355, 509	368	312	334	353	362	1.5	7.5
TC_{PNBP20}	1st	100-380	100-315	56.9	55.8	295	298	231	232	326	290	9.7	27.2
	2nd	380-600	315-600	30.2	10.7	525	384						
TC_{PNBP30}	1st	100-375	100-320	55.2	40.8	289	292	257	254	325	339	10.1	31.8
	2nd	375-600	320-600	26.3	19.4	539	345						
TC_{PNBP40}	1st	100-400	100-315	53.5	39.2	284	286	225	257	286	350	14.6	34.3
	2nd	400-600	315-600	30.8	21.2	547	337						
TC_{PNBP50}	1st	100-385	100-315	49.8	37.8	283	281	250	243	319	352	16.2	36.5
	2nd	385-600	315-600	29.6	21.1	538	320						

volatiles in comparison to that of case in air atmosphere and after that almost no degradation occurred giving char yield of 7.5% at 600 ºC.

In case of the treated cotton fabric samples, the hydrogel coating makes fabric thermally stable up to about 225 ºC, exhibiting only minor changes accompanied by slight weight loss attributed to the evaporation of absorbed moisture and other volatile impurities present in the material. The presence of the polymeric film on the fabric surface retards the rate of weight loss within the temperature range of 225-370 ºC due to condensed phase action of released phosphorus containing moiety on heating and develops a protective barrier effect. Furthermore, the thermal stability of the coated fabric significantly improves after 370 ºC temperature with increasing *Bis*MEP crosslinker content in the hydrogel network due to formation of more char as a barrier to retard the release of volatiles to vapour phase by blocking heat and oxygen transfer with the increased crosslinking in hydrogel. The enhanced crosslinked structure promotes carbonization during thermal degradation, resulting in higher char formation rather than complete combustion in both air and nitrogen atmospheres.

Morphology and elemental analysis: The surface morphology and elemental analysis of pure and treated cotton fabric samples with and without home laundering were studied using SEM and EDX techniques. The SEM images and weight percentages of detected elements of various regions of pure cotton (PC), treated cotton fabric (TC_{PNBP20} and TC_{PNBP50}) and $TC_{PNBP50L}$ (after first home laundering) samples are shown in Fig. 5. The SEM image of pure cotton exhibits a clean and smooth surface whereas the SEM images of treated cotton shows a uniform thin polymeric film coating on the surface of cotton fibres.

The EDX spectra of TC_{PNBP20} and TC_{PNBP50} exhibited phosphorus and nitrogen peaks, supporting the presence of the PNBP coating on the cotton surface. These signals are consistent with the deposition of the phosphorus- and nitrogen-containing hydrogel through admicellar polymerization. After one home-washing cycle, the $TC_{PNBP50L}$ sample showed a

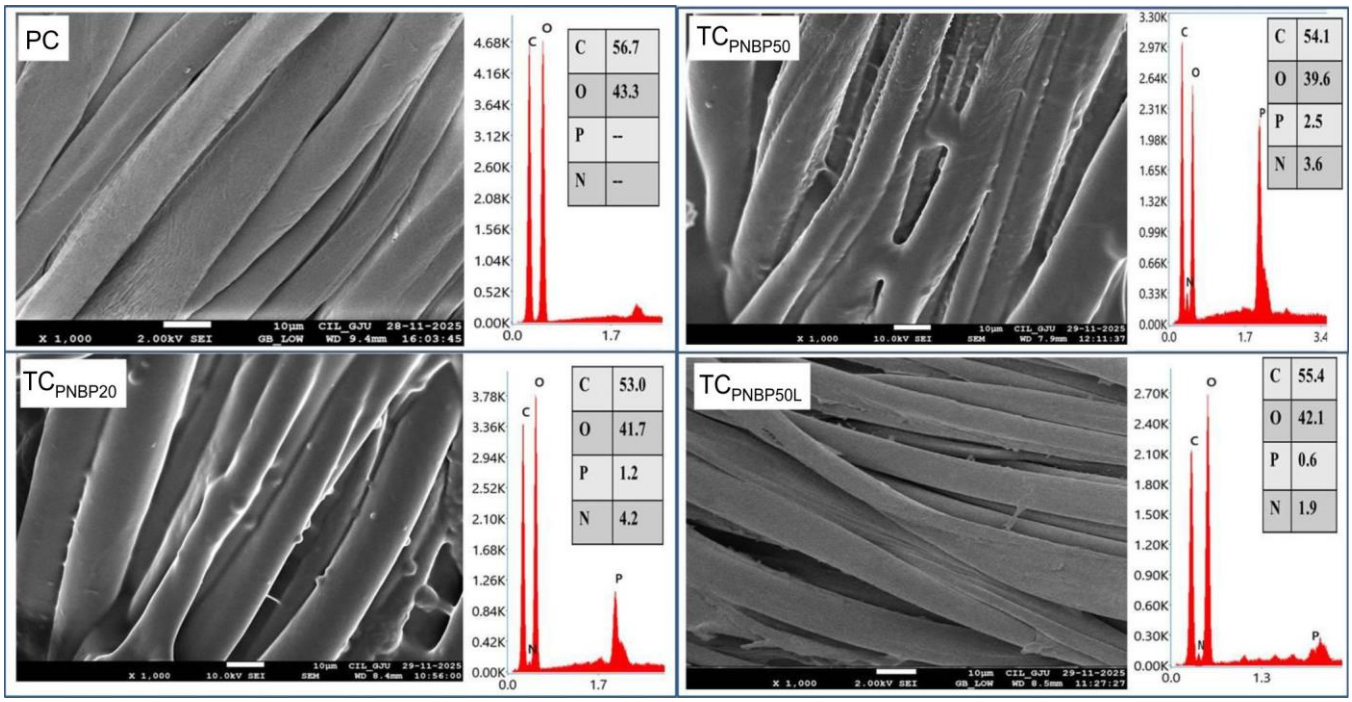


Fig. 5. SEM pictures and EDX patterns of the corresponding elemental contents of PC, TC_{PNBP20}, TC_{PNBP50} and $TC_{PNBP50L}$ fabric samples

lower relative contribution from these elements, which may be associated with partial removal of the coating during laundering. EDX analysis alone cannot confirm uniform coating coverage or quantify the extent of coating loss.

SEM and EDX analysis of char: Fig. 6a displays that the morphology is retained on heating PC at 250 ºC for 10 min. The image of PC after heating at 300 ºC (Fig. 6b) displays the compression and major fractures of the fibres. However, the fibrous structure of residue of TC_{PNBP50} (obtained at 250 ºC) develops swallowed, twisted with some minor cracks (Fig. 6c). The residue of TC_{PNBP50} (obtained at 300 ºC) becomes squeezed and cracked surface structure (Fig. 6d). The morphology of pure fibres does not change much on heating upto the temperature of 300 ºC. Fig. 6e-f reveals the EDX pattern

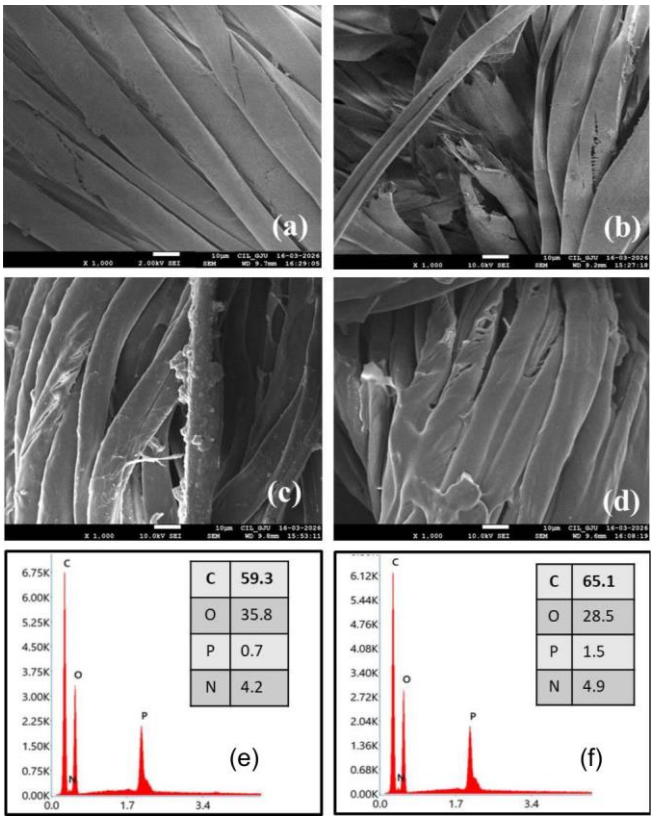


Fig. 6. SEM images of residues of pure cotton obtained at (a) 250 ºC, (b) 300 ºC, residues of TC_{PNBP50} obtained at (c) 250 ºC, (d) 300 ºC and EDX pattern of residues of TC_{PNBP50} obtained at (e) 250 ºC and (f) 300 ºC

of residues of treated cotton sample (TC_{PNBP50}) obtained by heating at 250 and 300 ºC, which shows the increase of wt.% of P and N elements with increase in temperature due to condensed phase action of hydrogel flame retardant.

Flammability behaviour: The flammability behaviour of untreated and treated cotton fabric samples was evaluated using a "Paramount 45º Automatic-flammability tester" with a constant ignition time of 2 sec and limiting oxygen index (LOI) tester. To assess durability, selected treated samples were also tested after normal water washing. The untreated cotton fabric exhibited rapid ignition and intense flame propagation. After removal of the flame source at 1 sec, the un treated cotton fabric burned completely within 11.5 sec, leaving no char residue. The treated samples exhibited improved flame resistance, with TC_{PNBP20}, TC_{PNBP30} and TC_{PNBP40} showing slower flame propagation and char formation of varying lengths (Fig. 7 and Table-3). The LOI values increased from 28.0 ± 0.1% for TC_{PNBP20} to 36.0 ± 0.2% for TC_{PNBP50} as the crosslinker concentration increased from 20 to 50%. TC_{PNBP50} did not sustain combustion after flame exposure and left only a small charred area. After one washing cycle, the flame-retardant performance declined. $TC_{PNBP40L}$ burned completely within 29.2 sec, whereas $TC_{PNBP50L}$ exhibited slower flame propagation and self-extinguished after 23 sec, leaving char residue. These findings indicate that higher crosslinker concentrations were associated with improved flame resistance under the test conditions, although the reduction in performance after washing raises concerns about coating durability. The improved behaviour of the treated fabrics may be related to the formation of a protective char layer that limits heat transfer and the release of combustible degradation products. Further washing-cycle studies are needed to assess the long-term durability of the coating.

Air permeability and stiffness: The air permeability performance of the cotton fabrics was determined using a standard air permeability testing instrument, with results expressed in $m^3/m^2/min$ as the rate of airflow through the fabric surface. The stiffness properties of both untreated and flame-retardant-treated samples were measured using a Shirley Stiffness Tester. All measurements were conducted in triplicate and the mean values were used for analysis. According to Table-3, treatment of fabric with the flame-retardant led to a moderate drop in air permeability in comparison to the untreated fabric, which recorded 179.7 $m^3/m^2/min$. The treated

TABLE-3
AUTO FLAMMABILITY, LIMITING OXYGEN INDEX (LOI), AIR PERMEABILITY AND STIFFNESS DATA OF COTTON FABRIC SAMPLES

Sample	Auto flammability		LOI (%)	Air permeability ($m^3/m^2/min$)	Stiffness (cm)
	Flame spread time (s)	Char length (cm)			
Pure cotton (PC)	11.5 (BEL)*	BEL	18 ± 0.2	179.6 ± 0.3	1.71 ± 0.1
TC_{PNBP20}	15.0 (SE)	3.5×2.4	28 ± 0.1	171.2 ± 0.2	2.30 ± 0.2
TC_{PNBP30}	5.0 (SE)	3.5×1.2	32 ± 0.2	166.3 ± 0.2	2.63 ± 0.2
TC_{PNBP40}	4.0 (SE)	2.0×1.1	33 ± 0.2	154.4 ± 0.3	2.90 ± 0.1
TC_{PNBP50}	0.0 (DCF)	1.7×0.9	36 ± 0.2	140.8 ± 0.4	3.33 ± 0.1
$TC_{PNBP40L}$	29.2 (BEL)	BEL	–	–	–
$TC_{PNBP50L}$	23.0 (SE)	7.0×1.2	26 ± 0.2	–	–

*BEL: burn entire length, SE: self-extinguished DCF: doesn't catch fire

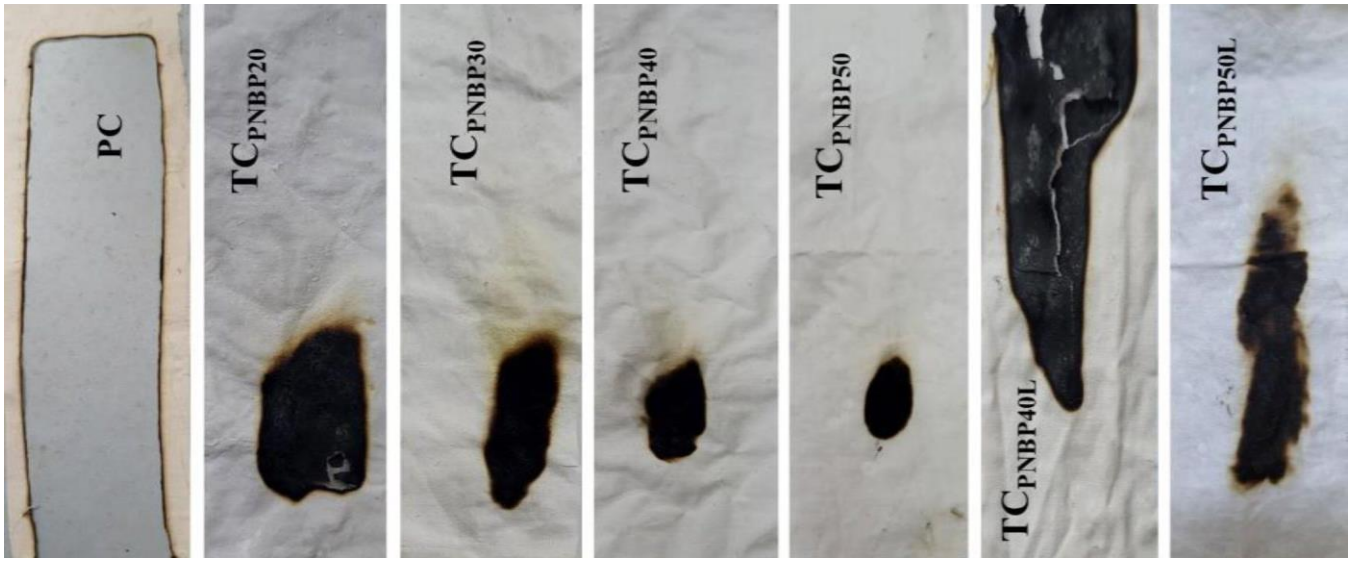


Fig. 7. Auto-flammability test results of pure and treated cotton fabric samples

samples showed values ranging from 132.4 to 171.2 $m^3/m^2/$ min. In terms of stiffness, the untreated fabric exhibited stiffness of 1.71 cm, whereas the treated fabrics (TC_{PNBP20} – TC_{PNBP50}) displayed slightly higher values between 2.30 and 3.33 cm. Despite these changes, the treated fabrics retained adequate breathability and acceptable flexibility, indicating that the finishing process had only a limited impact on overall comfort-related properties.

Conclusion

A phosphorus and nitrogen-based crosslinked hydrogel flame retardant namely poly (N-hydroxymethyl acrylamide)-*bis*[2-(methacryloyloxy)ethyl]phosphate (PNBP) was synthesized and coated onto cotton fabric *via* admicellar polymerization. The treated cotton fabrics exhibited remarkable flame retardant performance with the limiting oxygen index (LOI) increasing significantly from 18.0% of pure cotton fabric to 36.0% as well as passed the auto-flammability test with the increase of crosslinking in hydrogel of *Bis*MEP crosslinker concentration from 20 to 50%. The incorporation of varying amounts of *Bis*MEP crosslinker in the hydrogel further improved thermal stability, char yield and flame retardancy. This study demonstrates a simple and eco-friendly approach for developing durable flame-retardant cotton fabrics.

CONFLICT OF INTEREST

The authors declare that there is no conflicts of interest regarding the publication of this article.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this manuscript, the authors used an AI-assisted tool(s) to improve the language and no data, results or scientific conclusions were generated by AI. All analyses, interpretations and conclusions are the authors' own. The authors reviewed and edited the content and take full responsibility for the published work.

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