Supporting Information

Phosphorous grafted chitosan functionalized graphene oxide based nanocomposite as novel flame retardant materials for textile and wood

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Abbreviation:

PCG (phosphorus chitosan GO nanocomposite)

GC (GO coated cloth),

CC (chitosan-coated cloth)

PCGC (phosphorus chitosan GO -coated cloth)

Characterization details:

FTIR analyses were carried out on FTIR spectrophotometer (Bruker) Vertex 70 in the range of 400–4000 cm^{-1}. XRD patterns were generated using Cu-Kα (1.54 Å) radiation in a Bruker AXS diffractometer. SEM and EDXA studies of the material were performed on a JEOL – JSM 6380 LA instrument. Thermogravimetric analysis (TGA) of the catalysts was carried out with (Shimadzu DTG-60 H, Japan) in an aluminium pan. XPS studies of the material were performed on PHI 5000 Versa Prob II,FEI Inc. instrument.

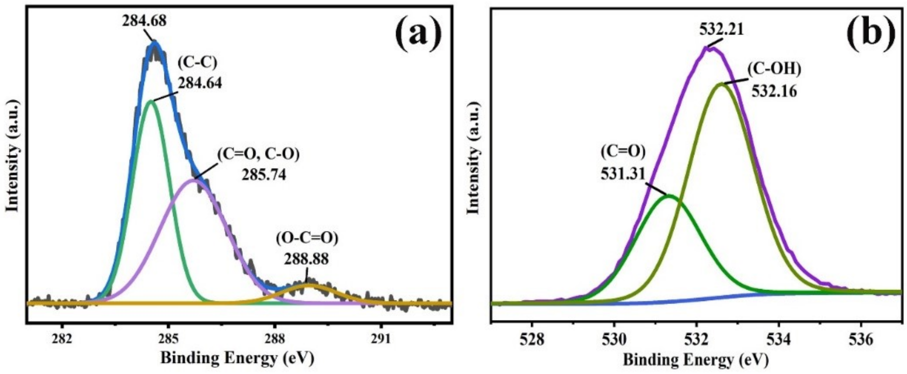

Figure S1: XPS spectra of GO (a) C 1s (b) O1s

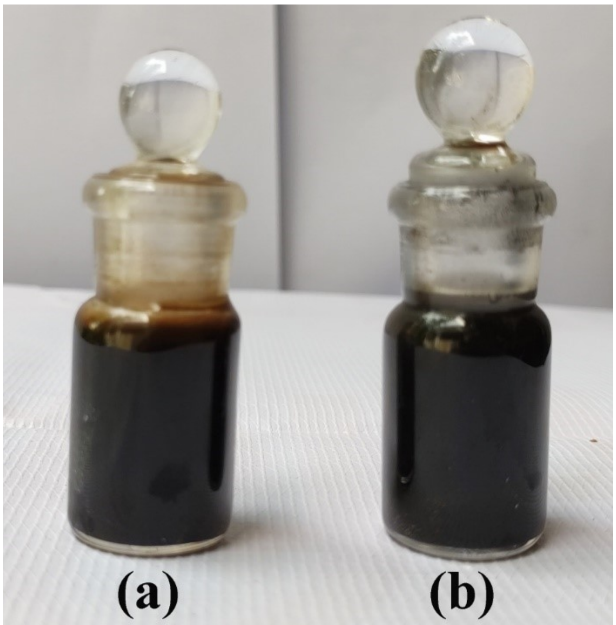

Figure S2: Images of GO and PCG dispersed in DI water 1 mg/mL

The distribution of carbon, phosphorous, oxygen and nitrogen on the surface of the cloth confirmed by SEM. The possible mechanism of PCG surface is shown in Figure S3. When the PCGC comes in contact with fire, the carbon skeleton of GO and chitosan decomposes and it can act as template to form char and prevent heat transfer and delay in fire. Phosphorous help in char formation.

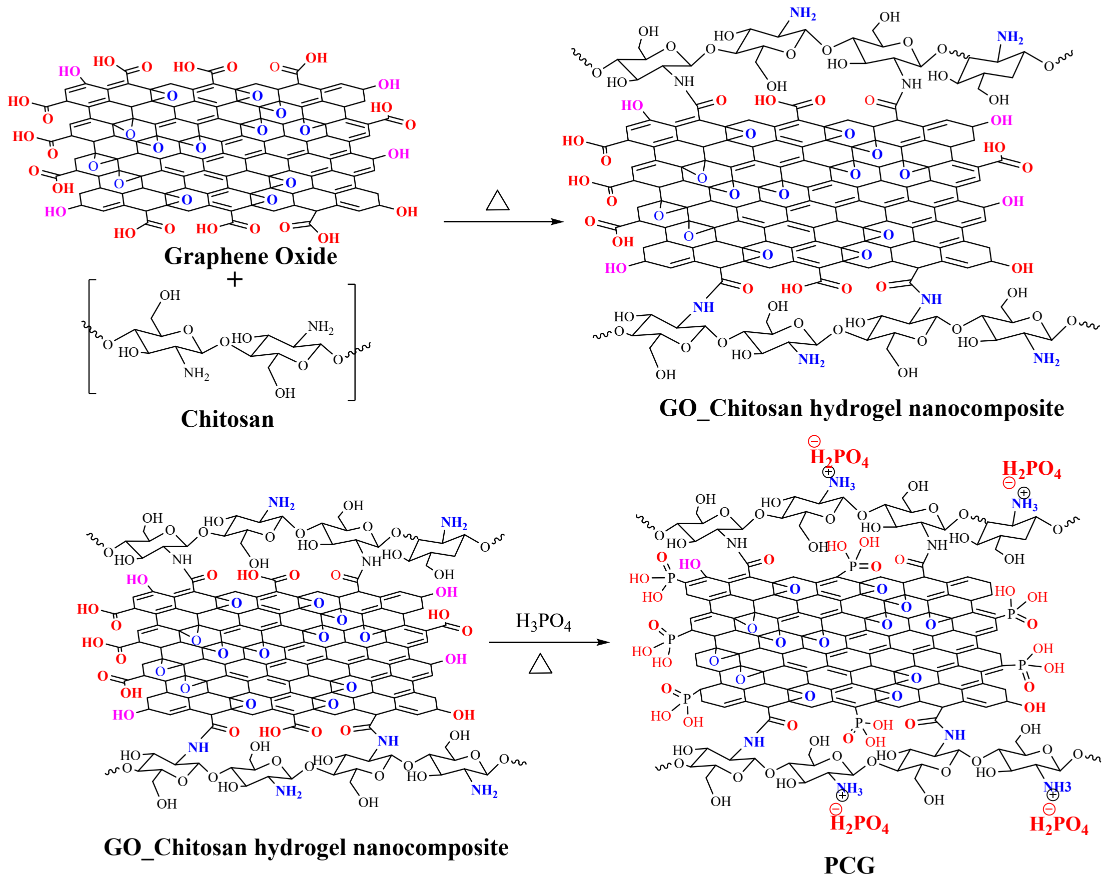

Figure S3. Probable mechanism of synthesis of PCG

Schematic an illustration of the dehydration of PCG that is suggested, which results in the formation of phosphoesters, phosphodiesters, and phosphoanhydrides through intramolecular and intermolecular reactions. While dehydration releases water vapour that reduces the concentration of gas fuel and acts as a physical flame retardant, burning is a gas phase reaction. After dehydration, the phosphoester, phosphodiester, and phosphoanhydride that develop at the margins of PCG serve as a physical insulation layer that blocks the flow of heat.

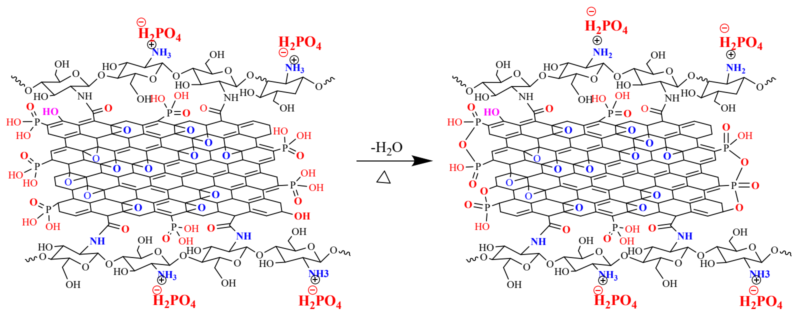

Figure S4: Schematic an illustration of the dehydration of PCG

Table S1. EDX, EA and XPS data of GO and PCG of (at. %).

Sample	Element	EDX (at. %)	XPS (at. %)	Elemental Analysis (wt. %)
GO	C	63.0	63.8	64
	N			
	O	31.0	32.1	32.3
	P			
PCG	C	31.0	28.7	29.9
	N	12.2	11.8	12.3
	O	28.9	30.6	30.8
	P	23.4	25.4	26.3

Table S2. Dip coating process (time, volume and concentration).

Flame retardant Material	Time (s)	Volume (mL)	Conc. (mg/mL)
GC	25	100	1
CC	25	100	1
PCGC	25	100	1

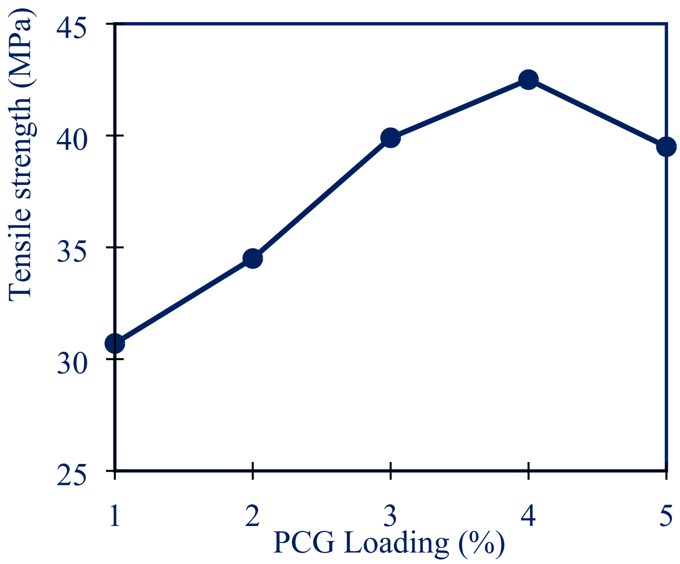

Figure S5. Tensile strength of PCGC.

The tensile strength of PCGC and blank cloth were measured by the reported method for fibers.[1] We have observed that the tensile strength of the blank cloth is 30.524 MPa (Figure S5). Whereas, PCGC samples have shown the increasing trend of tensile strength in comparison to blank cloth, with PCG material loading (1-5 wt. %). The maximum tensile strength 42.75 MPa was observed by the incorporation of 4.0 wt. % PCG. After the loading of 4-5% PCG material gradually decreased the tensile strength up to 39.5 MPa.

Table S3: Thickness of uncoated cloth, PCGC and PCGC char

Sr.No.	Thickness of the uncoated cloth (mm)			Thickness of the PCGC (mm)			Thickness of the PCGC Char (mm)		
	M.S.R.	C.S.D.	T.S.R.	M.S.R.	C.S.D.	T.S.R.	M.S.R.	C.S.D.	T.S.R.
1	0	18	0.18	0	32	0.32	0	52	0.52
2	0	16	0.16	0	33	0.33	0	48	0.49
3	0	17	0.17	0	35	0.35	0	49	0.49
4	0	16	0.16	0	32	0.32	0	53	0.53
5	0	17	0.17	0	35	0.35	0	50	0.51
Average			0.17			0.33			0.51

*(M.S.R- main scale reading, C.S.D- circular scale division, T.S.R- total scale reading)
LC: 0.01 mm
Thickness of the uncoated cloth : 0.17 mm
Thickness of the coated cloth (PCGC) : 0.33 mm
Thickness of the Char cloth : 0.51 mm

Table S4: Thickness of uncoated wood, PCG@wood and burned coated PCG@wood char

Sr.No.	Thickness of the uncoated wood (mm)			Thickness of the PCG Coated wood (mm)			Thickness of the PCG@wood Char (mm)		
	M.S.R.	C.S.D.	T.S.R.	M.S.R.	C.S.D.	T.S.R.	M.S.R.	C.S.D.	T.S.R.
1	2	4	2.04	2	29	2.29	2	9	2.09
2	1	96	1.96	2	24	2.24	2	5	2.05
3	1	98	1.98	2	21	2.21	2	6	2.06
4	2	3	2.03	2	26	2.26	2	3	2.03
5	1	99	1.99	2	28	2.28	2	8	2.08
Average			2.00			2.26			2.06

*(M.S.R- main scale reading, V.S.D- circular scale division, T.S.R- total scale reading)
LC: 0.01 mm
Thickness of the uncoated wood : 2.00 mm
Thickness of the coated wood (PCG) : 2.26 mm
Thickness of the Char wood : 2.06 mm

Table S5: Different loading of PCG coating on flame retardant property

No. Of Dips	%PCG loading	LOI	Results after exposure to similar flame
1	2.1	31.34	Not an efficient flame retardant
2	3.4	43.65	Improved flame retardancy but not an efficient flame retardant
3	4.3	51.53	an efficient flame retardant

4	4.6	52.12	an efficient flame retardant
5	5.2	53.45	an efficient flame retardant

Video link:
Video S1 showing PCG coated cloth vs blank cloth flame retardancy test
https://drive.google.com/file/d/1nUGrzkpQDycVoiqoNpuPi_tRAm9dt5-q/view?usp=sharing
Video S2 showing CC vs blank cloth flame retardancy test
https://drive.google.com/file/d/1I04XjlFkPvJ95tp6TVXYeNeBHdpZEBEF/view?usp=sharing
Video S3 showing GC vs blank cloth flame retardancy test
https://drive.google.com/file/d/1QPGYySnr6VkjvpHDMLDV8f-woF-zB6PI/view?usp=sharing
Video S4 showing PCG coated wood vs blank wood flame retardancy test
https://drive.google.com/file/d/1ZTbFrw1kzbY-OQRsTaukYEI8Rrjj9gMr/view?usp=sharing
Video S5 showing PCGC vs blank cloth Bunsen burner flame retardancy test
https://drive.google.com/file/d/1lGfDaU3LshCmvwGty07ioMPGLtpwd6xE/view?usp=sharing

References:

[1] D.A. Pethsangave, R. V. Khose, P.H. Wadekar, S. Some, Deep Eutectic Solvent Functionalized Graphene Composite as an Extremely High Potency Flame Retardant, ACS Appl. Mater. Interfaces. 9 (2017) 35319–35324. https://doi.org/10.1021/acsami.7b09587.