

New UV-Curable Cyclotriphosphazenes as Fire-Retardant Coating Materials for Wood

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ABSTRACT: A series of (2-hydroxyethylmethacrylate)(*n*-propoxy)cyclotriphosphazenes (HPCP) has been prepared by the reaction of $N_3P_3Cl_6$ with *n*-propanol and 2-hydroxyethylmethacrylate (HEMA) sequentially in the appropriate solvents. These phosphazene compounds are characterized by a combination of Fourier transform infrared, ultraviolet, and nuclear magnetic resonance spectroscopies. It was found that the compounds were soluble in most of the organic solvents, exhibited an absorption between 207–216 nm, and were curable by UV irradiation. After curing, the surfaces of the HPCP-coated woodblocks were transparent and had a fire-retardant property, which has been ascribed to the intumescent behavior during the burning process. © 1998 John Wiley & Sons, Inc. *J Appl Polym Sci* 69: 115–122, 1998

Key words: UV curing; cyclotriphosphazene; fire-retardant; 2-hydroxyethylmethacrylate (HEMA); coating materials

INTRODUCTION

As is known, ultraviolet (UV)-curable resins have been used extensively in surface treatment, electronics, pigmentation, and adhesion due to their characteristic properties, such as high cure speed, energy conservation, pollution reduction, and cost-effectiveness.¹ Nevertheless, most of the conventional commercially available monomers and oligomers used in the radiation-curable coatings are flammable and cannot fulfill certain specialized applications, which require significant levels of fire retardancy. Although this level of fire retardancy can normally be attained by the addition of inert fire-retardant additives in conventional plastics,² since the high concentration of additives

leads to difficulty in curing and severe degradation, such additives do not usually perform well with the radiation-curable materials.³ To eliminate these problems, some halogenated reactive-type fire-retardant monomers and oligomers have been prepared and used with some common acrylate resins to reduce their flammabilities.⁴ These monomers, like other halogenated fire-retardants, however, have been suspected to form highly toxic gases (brominated dibenzo-dioxines, dibenzofurans) and corrosive smoke upon burning.⁵ Therefore, searching for halogen-free flame retardants, such as phosphorus-containing materials, has drawn much attention.⁶

Alkoxyphosphazenes, which contain both phosphorus and nitrogen elements, have been developed as halogen-free flame-retardant additives.⁷ Recently, hexa(2-hydroxyethylmethacrylate)-cyclotriphosphazene, has been reported to be extremely sensitive to many forms of radiation, leading to rather efficient crosslinking.⁸ In order to combine UV curability and fire retardancy

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properties into a compound, a series of cyclotriphosphazenes containing various amounts of 2-hydroxyethylmethacrylate (HEMA) and propoxy groups has been synthesized by the substitution reactions in this study. These cyclotriphosphazenes were further coated on the selected woodblocks and cured with UV light for investigating their fire-retardant effect.

EXPERIMENTAL

Materials

The HEMA, benzoinethylether (BEE), used in this study were purchased from Aldrich Chemical Co. The HEMA was purified by extracting diacrylate with *n*-hexane and distilling in a high vacuum before use. The THF, *n*-propanol, benzene, sodium metal, ethyl ether, and pyridine were purchased from Merck Chemical Co. The THF and ethyl ether were dried with sodium metal and distilled prior to use.

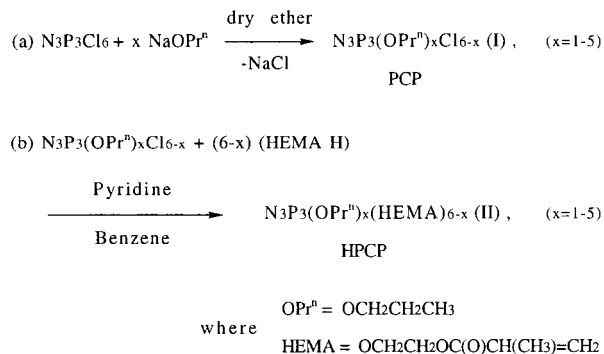
The woodblocks used were quercus ones bought from a local store at Chung-Li, Taiwan, Republic of China.

Equipment

The ^1H -, ^{13}C -, and ^{31}P -nuclear magnetic resonance (NMR) spectra were obtained with a Bruker AC 200 spectrometer. An 85% H_3PO_4 aqueous solution assigned to 0 ppm was used as an external reference for the ^{31}P -NMR spectra. As convention, the ^1H - and the ^{13}C -NMR spectra were referenced to tetramethylsilane. UV-visible spectra of all the samples dissolved in spectroscopic-grade methanol (3 mg HPCP in 20 cc methanol) in a quartz cell were obtained with a Shimadzu UV-160 spectrometer.

For ease in monitoring the UV irradiation reaction, a C Sum Model UC-600W Lot #1628 with a low-efficiency medium-pressure mercury lamp, Wesernoquartz CS99, was used. The emission spectrum from this light source extends from 200 to 400 nm with a high emission at 365 nm.

On the other hand, the light source used in the UV irradiation for the coated woodblocks was a 1000-W/cm² medium-pressure mercury lamp of C SUM UVC-202. The emission spectrum from this light source also extends from 200 to 400 nm with a high emission at 365 nm.



Scheme 1

Preparation of (2-Hydroxyethylmethacrylate)-(*n*-propoxy) cyclotriphosphazenes (HPCP)

The HPCP mixtures with various amounts of HEMA were synthesized according to Scheme 1. The preparation procedures were as follows.

Synthesis of (*n*-Propoxy)-chlorocyclotriphosphazenes (PCP)

An *m*-equivalent of sodium (9.93 g, 0.432 mole, for $m = 3$) and 300 mL of dry ethyl ether was placed in a three-necked flask provided with a stirrer, reflux condenser, and a nitrogen inlet. The flask was placed in a cold water bath and an *m*-equivalent of *n*-propanol (25.93 g, 0.432 mole, for $m = 3$) was added dropwise with continuous stirring until the sodium had completely reacted with the *n*-propanol to form the sodium salt. A 200-mL dry ethyl ether solution of hexachlorocyclotriphosphazene, $\text{N}_3\text{P}_3\text{Cl}_6$, (50 g, 0.144 mol) was then added dropwise into the sodium salt solution with continuous stirring. After reaction at 40°C for 48 h under nitrogen, the sodium salt produced was separated by filtration, and the solvent was removed under distillation. The residue obtained was then dissolved in dichloromethane, washed several times with water, and dried over magnesium sulfate. This solution was finally vacuum-distilled, and a light-yellow liquid PCP mixture was obtained (45 g for $m = 3$).

Synthesis of (*n*-Propoxy)(2-hydroxyethylmethacrylate) cyclotriphosphazene (HPCP)

A solution of 50 g of PCP in a 500-mL benzene-pyridine (2.1 : 1) cosolvent was prepared first. An appropriate amount of 2-hydroxyethylmethacrylate was added dropwise to the solution and stirred at 55°C for 48 h under nitrogen. The pyridine salt was separated, and the solvent was re-

moved under a high-vacuum distillation. The residue obtained was then dissolved in dichloromethane, washed with water, and dried over magnesium sulfate. The solution was finally concentrated to produce a pale yellow viscous liquid (40 g for $m = 3$).

The PCP and HPCP mixtures obtained were characterized by Fourier transform infrared (FTIR), ^1H -, ^{13}C -, and ^{31}P -NMR, and UV spectroscopies and used without separation throughout the study for economic considerations.

UV Curing of HPCP Film

In order to investigate the UV curing of an HPCP film, a polished potassium bromide plate was coated with an approximately 10- μm -thick layer of HPCP. The plate was then exposed to radiation from a distance of 12 cm in an atmosphere of air several times until the amount of the residual double bonds reached a constant level, which was monitored by the vinyl peak of the FTIR spectrum.

Preparation of HPCP-Coated Woodblocks

The phosphazenes (HPCP) prepared were coated onto the quercus woodblocks ($30 \times 15 \times 1 \text{ cm}^3$) and cured with UV irradiation. The tested woodblocks were kept in a controlled atmosphere of $50 \pm 5\%$ relative humidity and $23 \pm 2^\circ\text{C}$ for 14 days

prior to coating. A coated sample was exposed to the UV source at a belt speed of 5 cm/min from a distance of 12 cm in an atmosphere of air several times until the surface was completely dry, and the surface was washed with acetone to remove the incompletely cured HPCP. After curing, the specimens were kept under the controlled conditions described above. Prior to the burning test, the specimens were kept in the oven at $50 \pm 0.2^\circ\text{C}$ for a period of 40 h.

Flame Retardancy Test

The flame retardancies of the HPCP-coated woodblocks were evaluated by char indexes measured according to the ASTM Method D1360-79.

RESULTS AND DISCUSSION

Synthesis and Characterization of HPCP

The HEMA and *n*-propoxy containing phosphazenes (HPCP) were synthesized according to scheme 1.

The (*n*-propoxy)chlorocyclotriphosphazenes (PCP) were synthesized as step (a) in Scheme I and characterized by the combination of FTIR, ^1H -, ^{13}C -, and ^{31}P -NMR spectra.

The FTIR spectra of the PCP mixtures with

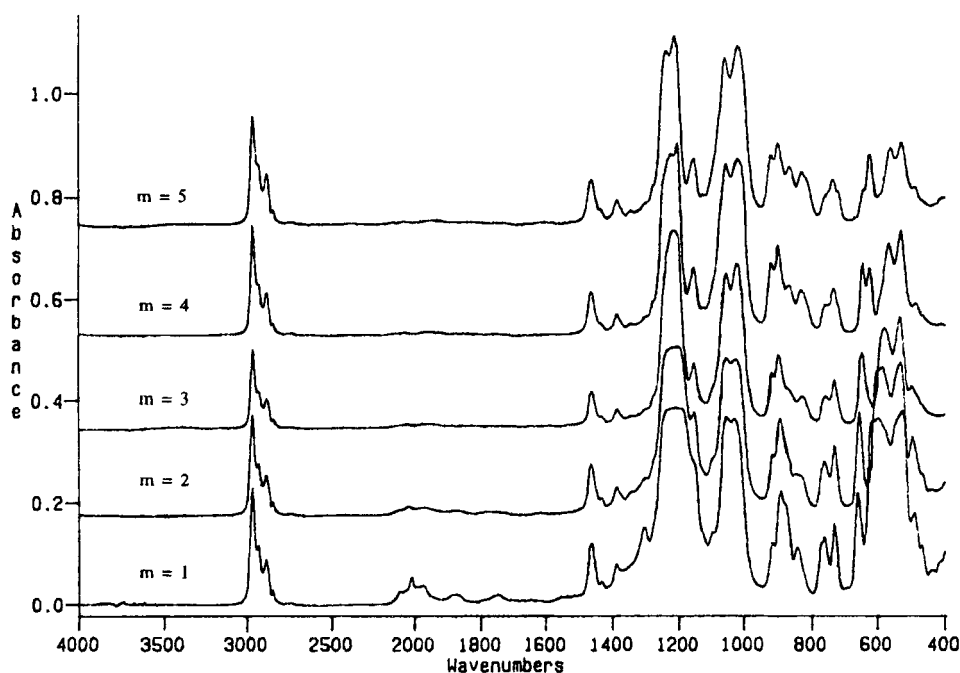


Figure 1 FTIR spectra of PCP ($m = 1-5$).

Table I Physical Characteristics of PCP

FTIR (KBr) (cm ⁻¹)	¹ H-NMR (ppm) (CDCl ₃)	¹³ C-NMR (ppm) (CDCl ₃)
2850–2975 (CH stretched)	0.9–1.0 (<i>t</i> , CH ₃)	9.9 (CH ₃)
1465 (CH asym)	1.7–1.9 (<i>m</i> , CH ₂)	22.9, 23.1, 23.1
732–736 (CH ₂ stretched)	4.1–4.2 (<i>m</i> , P—O—CH ₂)	(CH ₂ , <i>J</i> = 5.5 Hz)
1225–1240 (P=N stretched)	³ <i>J</i> _{PH} = 20 Hz)	68.3–71.4
1010–1030 (P—O—C aliph)		(P—O—CH ₂)
560–616, 526–530 (P—C1)		

various *x* values were similar. As shown in Figure 1, the strong absorption peaking at 1217 cm⁻¹ corresponding to the P=N stretching is a characteristic of cyclotriphosphazene compounds; the strong absorptions peaking at 1020 and 1050 cm⁻¹ correspond to the P—O—C (aliphatic) stretching, and the other absorptions are characteristic of the aliphatic and the functional groups attached to the phosphazene ring. These absorptions, summarized in Table I, support the structure expected for the PCP mixtures.

As expected, the ¹H-NMR spectra of the PCP showed a triplet at 1.0 ppm (CH₃), a multiplet at 1.8 ppm (CH₂) for the propoxy protons, and a multiplet at 4.2 ppm (³*J*_{PH} = 20 Hz) for the P—O—CH₂ group.

The ¹³C-NMR spectra data listed in Table I indicate that the peak for the carbon of the P—O—CH₂ group was shifted to a lower field, due to the electron-withdrawing effect of the phosphazene ring and that the carbon of the propoxy end group (CH₃) was shifted to a higher field as compared to that for the corresponding carbons of propanol (64 ppm HOCH₂CH₂CH₃).

As indicated in Table II, the chemical shifts and the coupling constants show that the ³¹P-NMR spectra of all the PCP compounds were second-order with *J*/Δ*ν* values in the range from 0.06 to

0.12, and those partially substituted compounds were predominantly nongeminal. As listed in Table III, the composition of the PCP mixtures, estimated from the ³¹P-NMR spectra, was varied according to the amount of the propanol-fed *m* value.

The (*n*-propoxy)(2-hydroxyethylmethacrylate)-cyclotriphosphazenes (HPCP) were synthesized as in step (b) in Scheme 1. The FTIR spectra of the HPCP were similar, and a typical spectrum is shown in Figure 2. The strong absorption peaking at 1727 cm⁻¹ corresponds to C=O stretching. The absorptions at around 1635 and 811 cm⁻¹ correspond to C=C and C=C—H stretching of the vinyl groups. The other absorptions listed in Table IV are characteristics of the aliphatic and the functional groups present in HPCP. The absorptions support the structures expected.

The ¹H-NMR chemical shifts of HPCP summarized in Table IV indicate a triplet at 0.9 ppm of CH₃, a multiplet at 3.9 ppm of CH₂, and a multiplet at 4.3 ppm of P—O—CH₂ for the propoxy group. For the methacrylate group, the protons of the double bond are indicated by two doublets at around 5.6 and 6.1 ppm due to the electron-withdrawing effect of the carbonyl group, and the pendent methyl protons are shown by a triplet at 1.9 ppm. The chemical shifts of the propoxy group in the HPCP were similar to those in the PCP.

Table II ³¹P-NMR Chemical Shifts and Coupling Constants of PCP

PCP	Spectrum Type	Position (ppm)			<i>J</i> _{PA₂B} (Hz)	<i>J</i> /Δ <i>ν</i>
		A	B	C		
N ₃ P ₃ (OPr ⁿ) ₁ Cl ₅	AB ₂	15.2	22.9		61.6	0.1
N ₃ P ₃ (OPr ⁿ) ₂ Cl ₄ ^a	AB ₂	25.3	18.7		63.2	0.12
N ₃ P ₃ (OPr ⁿ) ₃ Cl ₃	A	22.0				
N ₃ P ₃ (OPr ⁿ) ₄ Cl ₂ ^a	AB ₂	12.3	24.7		73.7	0.07
N ₃ P ₃ (OPr ⁿ) ₅ Cl ₁	AB ₂	27.5	15.3		76.2	0.08

^a N₃P₃(OPrⁿ)₂Cl₄ and N₃P₃(OPrⁿ)₄Cl₂ are nongeminal.

Table III Composition of PCP

Component/PCP	$m = 1^a$	$m = 2$	$m = 3$	$m = 4$	$m = 5$
$N_3P_3Cl_6$	18.9				
$N_3P_3(OPr^n)_1Cl_5$	58.9	33.9			
$N_3P_3(OPr^n)_2Cl_4$	22.1	60.0	40.4		
$N_3P_3(OPr^n)_3Cl_3$		1.2	45.4	36.4	13.5
$N_3P_3(OPr^n)_4Cl_2$			14.2	62.5	65.3
$N_3P_3(OPr^n)_5Cl_1$					21.1
Other		4.8		1.1	

^a m = input mole ratio of *n*-propanol/ $N_3P_3Cl_6$. Other: impurity.

The chemical shifts of the ¹³C-NMR spectra of the HPCP are given in Table IV. The assignments were implemented using the normal additivity parameters of the substituents in the methacrylate groups and the consideration of the off-resonance spectra in conjunction with the difference in intensities among the methylene and methyl carbons.⁹ It can be seen that the data for the propoxy group in the HPCP are similar to that of the PCP.

Since the HEMA and propoxy groups have similar shielding effects on the attached phosphorus atoms, and the PCP compounds used were mixtures, the ³¹P-NMR spectra of the HPCP produced displayed broad multiplets between 4 and 27 ppm and were difficult to resolve. Nevertheless, the average composition of the HPCP could be esti-

mated from the ratio of the integration of the peak at 1.94 ppm (CH_3 protons in HEMA) to that at 0.95 ppm (CH_3 protons in *n*-propoxy) and are listed in Table V.

UV Irradiation Polymerization

The HPCP products are soluble in a wide range of organic solvents, such as THF, benzene, acetone, and chloroform.

As shown in Figure 3, the absorption spectra of the UV measurements for HPCP in methanol show an absorption peak between 207 and 216 nm for each sample. As listed in Table VI, the peak position, λ_{max} , and the molar absorptivity, ϵ_{max} , are decreased with an increasing x value.

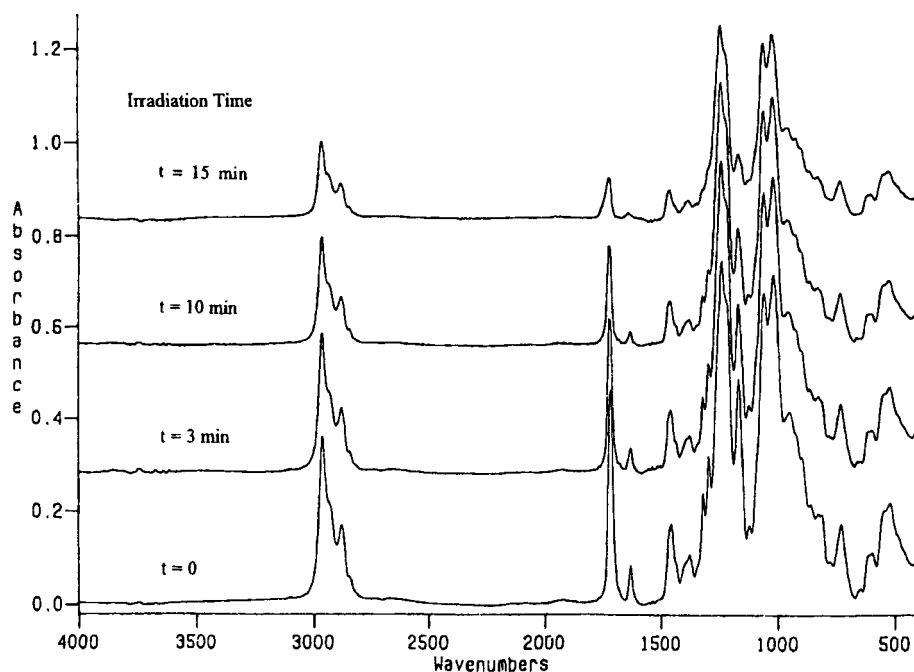


Figure 2 FTIR spectra of HPCP (II-4) before and after UV irradiation.

Table IV Physical Characteristics of HPCP

FTIR (KBr) (cm ⁻¹)	¹ H-NMR (ppm) (CDCl ₃)	¹³ C-NMR (ppm) (CDCl ₃)
2850–2975 (CH stretched)	Propoxy	Propoxy
1465 (CH asym)	0.9 (<i>t</i> , CH ₃)	9.9 (CH ₃)
732–736 (CH ₂ stretched)	1.7 (<i>m</i> , CH ₂)	23.2 (CH ₂)
1240–1247 (P=N)	4.0 (<i>m</i> , P—O—CH ₂)	67.9–69.6 (P—O—CH ₂)
1010–1030 (P—O—C aliph)	HEMA	HEMA
1727 (C=O)	1.9 (<i>t</i> , CH ₃)	18.2 (CH ₃)
1635 (C=C)	3.9 (<i>m</i> , CH ₂)	62.3–64 (P—O—CH ₂)
811 (C=CH)	4.3 (<i>m</i> , P—O—CH ₂)	66.3 (CH ₂)
	5.6 (<i>d</i> , C=CH _a)	166.8, 167.6 (C=O)
	6.2 (<i>d</i> , C=CH _b)	126.0 (C(CH ₃)C=)
		135.8 (C(CH ₃)=CH ₂)

This hypsochromic shift of the λ_{\max} and the decrease in absorption are attributed to the decrease in HEMA content. The HPCP can be cured by UV irradiation.

In the UV-induced polymerization, BEE was chosen as the photoinitiator, which was used to induce photopolymerization in an atmosphere of air via homolytic cleavage. In order to monitor the reaction by FTIR, the sample was prepared by coating the HPCP on a KBr plate and irradiating by a medium-pressure Hg lamp for 15 min. As shown in Figure 1, the absorption corresponding to the carbon-carbon double bond at 1630 cm⁻¹ is decreased in intensity with an increasing irradiation time, implying that the double bond is the curing site. In addition, this region has a minimum interference with other functional groups; therefore, it was selected to monitor the extent of the polymerization. The residual unsaturation, calculated from the absorption of the peak at 1630 cm⁻¹ is plotted versus the irradiation time in Figure 3.

The expression used to estimate the initial polymerization rate R_p , is

$$R_p = (\% \text{ Converted}/100) * (A_0/T)$$

where A_0 is the initial methacrylate concentration (mol/L), and T is the total irradiation time (s).

As illustrated in Figure 4, at an early stage of the photoreactive irradiation, the sample with more methacrylate substituents was cured faster due to a higher concentration of the methacrylate groups. On the contrary, after about 3 min of irradiation, the residual unsaturation of the sample became higher, and with 15 min of irradiation, about 20% of the unsaturated groups still remained in the sample II-1. This result is ascribed to the rigidity of the crosslinked polymer formed in the early stage of irradiation, which inhibited the diffusion of the residual effective methacrylate groups toward the chain-propagation free

Table V Average Composition of HPCP^a

Sample	OPr ⁿ (<i>x</i>)	HEMA (6 - <i>x</i>)
II-1	0.7	5.3
II-2	1.5	4.5
II-3	2.3	3.7
II-4	3.7	2.3
II-5	4.8	1.2

^a HPCP is the abbreviation for N₃P₃(OPrⁿ)_{*x*}(HEMA)_{6-*x*}.

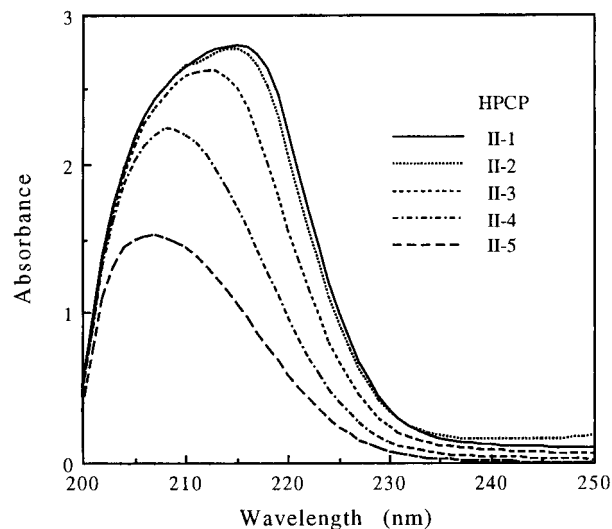
**Figure 3** UV absorption spectra of HPCP.

Table VI UV Absorption of HPCP with the Respective λ_{\max} and ϵ_{\max}

Sample ^a	λ_{\max} (nm)	ϵ_{\max}
II-1	215.7	20000
II-2	214.1	18000
II-3	213.4	16000
II-4	208.5	12000
II-5	207.3	7300

^a 3 mg HPCP in 20 cc of methanol.

radical sites. The data listed in Table VII confirm that both the A_0 value and the R_p value are decreased, while the residual unsaturation with 15 min of irradiation increases with a decreasing HEMA content, as discussed. This reveals that the curing polymerization was kinetically controlled in the beginning and became diffusion-controlled after about 3 min.

Flammability Studies of HPCP

To study the fire retardancy, each HPCP prepared was coated onto a quercus woodblock ($30 \times 15 \times 1 \text{ cm}^3$) and cured with UV irradiation. The light source used for the irradiation in this part of the study was a 1000-W/cm^2 medium-pressure mercury lamp. The emission spectrum from this light source extends from 200 to 400 nm with a high emission at 365 nm wavelength. The woodblock coated with about a 0.16 mm thickness of HPCP was exposed to the UV source at a belt speed of 5 cm/min from a distance of 12 cm in an atmosphere of air. The procedure was repeated until the surface was dry, and the surface was washed with acetone to remove the incompletely cured resin. It was found that the cured coating was all removed by acetone when the sample II-5 was used. This phenomenon is ascribed to the low crosslinking density.

The surfaces of all the specimens were trans-

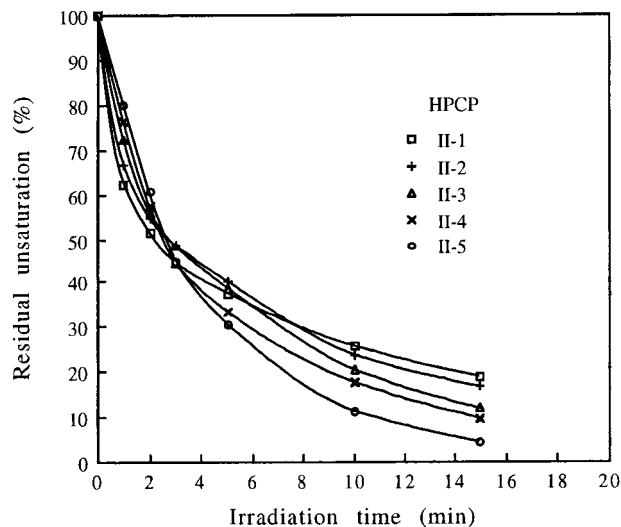


Figure 4 Photoreactivity profiles of HPCP (containing 5% BEE).

parent and chemically stable in the common organic solvents. The hardness of the coated surfaces was measured by the pencil hardness technique. As indicated in Table VIII, the hardness of the surface is increased with an increase in HEMA content due to the higher crosslinking density and can be adjusted as required by changing the HEMA content.

The fire retardancies of the woodblocks were evaluated by the char index according to the ASTM Method D1360-79. As shown in Table VIII, the char index is decreased with an increasing propoxy substituent and is less than 25 when the content of the propoxy substituent is increased ($x > 0.7$). It is also interesting to note that swollen chars were formed on the samples with a higher propoxy content during the burning process. This implies that the fire retardancy can be ascribed to an intumescent mechanism in which propylene, a gaseous product evolved from the propoxy group on burning, acts as a spumific agent, and the cellulose evolved from the woodblock acts as a source

Table VII Photoreactivity Evaluation of HPCP

Sample	A_0 (mol/L)	$R_p \cdot 10^4$ (mol/L s ⁻¹)	Residual Unsaturation with 15 min of Irradiation (%)
II-1	6.8	14.3	18.7
II-2	5.6	10.4	17.5
II-3	4.7	6.2	12.4
II-4	3.1	3.4	9.3
II-5	1.8	0.9	4.8

Table VIII Flammability Test of UV-Cured Polymer HPCP^a Coated Onto a Quercus Woodblock

Sample	x	Wt. Loss ^b (g)	Char Index ^c (cm ³)	Pencil Hardness (H)
Blank ^d	0	11.8 ± 0.9	33.9 ± 2.4	—
II-1	0.7	8.5 ± 0.7	26.1 ± 1.3	6H
II-2	1.5	7.6 ± 0.3	21.8 ± 1.5	5H
II-3	2.3	6.0 ± 0.5	16.7 ± 0.9	4H
II-4	3.7	5.1 ± 0.6	12.5 ± 0.8	3H

^a N₃P₃(OPrⁿ)_x(HEMA)_{6-x}.

^b Mean average weight loss of 5 unleached specimens.

^c Mean average char index of 5 unleached specimens.

^d Mean uncoated HPCP polymer on woodblock.

of carbon for the char. This result indicates that the high crosslinking density of the samples with a high HEMA content hinders expansion during foaming, as found in other thermosetting resins.^{10,11} Since a char index of about 25 or lower is considered generally acceptable for wood-coating applications, the woodblocks coated with the HPCP containing sufficient propoxy groups are considered to be fire-retardant.

CONCLUSION

The UV-curable fire-retardant phosphazene compounds (HPCP) were prepared by the reaction of N₃P₃Cl₆ with *n*-propanol and HEMA, sequentially, in the appropriate solvents. These HEMA-containing phosphazene compounds are curable by UV irradiation. It was found that the HPCP with a higher concentration of methacrylate had a higher photoreactivity to produce a higher crosslinking density. The hardness of the cured surface on the woodblocks can be adjusted by the ratio of HEMA and propoxy substituents. The woodblocks coated by the HPCP products with an appropriate propoxy content exhibited fire retardancy after UV curing. The fire retardancy was ascribed to the intumescent behavior during the burning process.

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