

# A Novel Route for Preparation of PVC Sheets with High UV Irradiation Resistance

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**ABSTRACT:** A novel acrylonitrile acrylate copolymer coating was proposed to improve the UV irradiation resistance of poly(vinyl chloride) (PVC) sheets. Its effect on the photoaging properties of the sheets was evaluated by means of Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), yellowness index test, and the oxygen permeability coefficient and mechanical properties measurement. The experimental results show that the novel acrylonitrile acrylate copolymer coating significantly improves the UV irradiation resistance of PVC sheets. Compared with the PVC sheets without coating, the coated PVC sheets show better mechanical properties, lower yellow-

ness index, and smaller oxygen permeability coefficient after UV irradiation. The analysis on the SEM images of the PVC sheets indicates that the coating acts as a film to prevent the PVC sheets from UV energy and oxygen that can accelerate the photochemical reactions of PVC. The FTIR analysis confirms that the coating can slow down the microstructures development of the PVC sheets, also indicating that the coating impeded the photochemical reactions of PVC. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 117: 2899–2906, 2010

**Key words:** poly(vinyl chloride); acrylonitrile acrylate copolymer; coating; photoaging; stabilization

## INTRODUCTION

Poly (vinyl chloride) (PVC) is one of the most important and widely used thermoplastics in the world. PVC plastics are chosen for many outdoor applications because of their low costs, good processability, excellent chemical, and fire resistance.<sup>1–5</sup> However, the PVC resins are known to be low weathering-resistant. Indeed, under the combined interactions of sunlight, atmospheric oxygen, moisture, pollutants and heat, severe chemical changes occur in PVC resins, resulting in high brittleness, discoloration, and loss of gloss.<sup>6</sup> Among outdoor conditions, sunlight is the main factor that induces the photoaging and causes the poor properties throughout PVC service life.<sup>5</sup>

The photoaging mechanism of PVC is generally accepted that the aging proceeds primarily with a radical-type mechanism.<sup>7–13</sup> When a sample is exposed to sunlight, the high UV energy of sunlight

absorbed by PVC is able to initiate free radicals by the breakage of chemical bonds in PVC molecules, and then activate a series of chemical reactions, such as molecular chains scission and crosslinking, hydrogen chloride elimination to form alkene or polyene linkages.<sup>7,10,11</sup> As a result of that the bulk properties and microstructures of the sample change.

Much research has been conducted to improve the stability of PVC under UV irradiation.<sup>14–18</sup> Conventionally, UV-absorbers, which can convert the UV energy into heat before exciting any free radical, are used to improve the photo-stability of PVC. An interesting finding is that the protection efficiency increases about 20-fold when the same amount of UV-absorber is introduced in the surface layer instead of in the bulk of the polymeric substrate.<sup>6</sup> That reminds surface modification of polymer or applying protective coating should be a more effective way to improve the UV resistance of polymer.

The epoxy-acrylate compound is a main type of UV coating, but the coating has poor adhesion on PVC matrix. The grafting method could overcome this shortcoming in some degree,<sup>6,19</sup> but the complicated procedure blocked its application. The same disadvantage could be found in UV protecting layers made by plasma polymerization.<sup>20</sup> Some highly crosslinked thermosetting polymers can provide high UV resistance, but the curing temperature is above 100°C which is too high for heat sensitive polymers like PVC.<sup>21</sup> To date, no ideal technology

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**TABLE I**  
**The Retained Percentage of Elongation at Break and Impact Strength of Samples**

Irradiation time (day)	The retained percentage of elongation at break (%)		The retained percentage of impact strength (%)	
	Coated samples	Uncoated samples	Coated samples	Uncoated samples
0	100.0	100.0	100.0	100.0
3	75.3	68.5	94.4	87.1
5	60.5	49.1	88.2	82.5
8	57.2	42.2	87.0	78.0
10	53.7	38.4	84.3	75.7
15	53.9	32.3	82.1	67.9

has been found to effectively and efficiently protect PVC from the UV irradiation.

The aim of this article is to find a new route for the preparation of PVC sheets with high UV resistance. A novel acrylonitrile acrylate copolymer is synthesized and used as a coating on PVC surface to improve the UV irradiation resistance of PVC sheets. The photoaging properties of the coated PVC sheets are investigated.

## EXPERIMENTAL

### Materials

PVC, SG-8 with a number-average molecular weight of  $5.0 \times 10^4$ , was purchased from Jinlu Group, Sichuan, China. Tribasic lead sulfate and dibasic lead phosphate were produced by Tianshen Plastic Auxiliary Co., Zhejiang, China. Stearic acid was from Kelong Chemical Reagent Plant, Sichuan, China. Acrylonitrile acrylate copolymer latex was prepared in our Lab.

### Sample preparation

One hundred parts of PVC, tribasic lead sulfate (powder, 2 phr), dibasic lead phosphite (powder, 1 phr), and stearic acid (powder, 0.5 phr) were mixed. The mixture was blended using an open twin-roller at 170°C for 5 min. The mixture was then molded into about 1.0 mm and 4.0 mm thickness sheets at 180–190°C. Dumbbell-shaped specimens according to the type IA in GB/T1040 for tensile strength test were cut from the 1.0 mm sheets. The impact test specimens (60 mm × 12.5 mm × 4 mm with 2.5 mm notch) were cut from 4.0 mm thickness sheets.

Acrylonitrile acrylate copolymer (AA copolymer) emulsion polymerization was carried out in a 500 mL four-necked flask equipped with a condenser, required amount of surfactant and sodium bicarbonate were placed into the flask under stirring for 0.5 h in a nitrogen atmosphere. Quantitative monomer (acrylonitrile and acrylate) and potassium persulfate solution were dropped into the reactor at 82°C, and

dropped off in 1 h. The reaction went on for 2 h at 84°C, completing the emulsion polymerization reaction.

Acrylonitrile acrylate copolymer latex was sprayed on the sheets. Then, the coated sheets were dried at 70°C for 5 min. The thickness of the film is about 0.05 mm. The dried coated sheets were ready for property test and structure characterization.

### Accelerated ultraviolet-weathering tests

Accelerated artificial UV-weathering tests were conducted in a chamber equipped with 500 W high-pressure mercury lamps, ventilation, and heating equipment. The samples were subject to continuous exposure at  $40 \pm 5^\circ\text{C}$ , and the average irradiation intensity was  $5.5 \text{ W/m}^2$  at 340 nm.

### Measurements and characterization

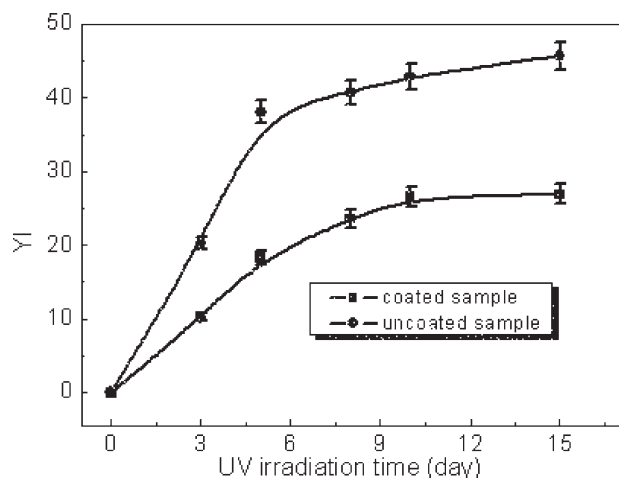
The tensile testing was carried out according to Standard GB/T1040 using a SANS CMT4104 testing machine at an extension rate of 20 mm/min. The impact testing was conducted according to GB/T1843 using a XJ-40A impact tester.

The yellowness index (YI) was measured using the Tristimulus Colorimeter SC-80A according to Standard GB/T2409.

The gas-permeability was measured in the pressure-difference gas permeation instrument (VAC-V1). The testing temperature was 40°C and oxygen was used as the testing gas. Permeability coefficient dependent on time was obtained in accordance with GB/T1038.

The surface morphologic analysis was characterized using a SEM (JSM-5900) with the pretreatment of Au deposition. The accelerating voltage of the SEM was 20 kV.

The infrared spectra were obtained using a Nicolet 560 Fourier transform infrared (FTIR) spectrometer at a resolution of  $2 \text{ cm}^{-1}$  with the reflection mode. For the coated PVC sheets, the coating was removed in toluene or ethyl acetate in dissolving method



**Figure 1** Changes in the yellowness index of the samples with the irradiation time.

before detected to determine the characteristic group of PVC matrix accurately. The  $1430\text{ cm}^{-1}$  peak, which is attributed to the stretching vibration of  $\text{CH}_2$ , was selected as an internal reference.<sup>13,22,23</sup> The carbonyl index (CI) was defined to show the photo-

oxidation degree of the material and the formation of the vinyl group was characterized by the vinyl index (VI) as follows:

$$\text{CI} = S_{\text{C=O}}/S_{1430} \quad (1)$$

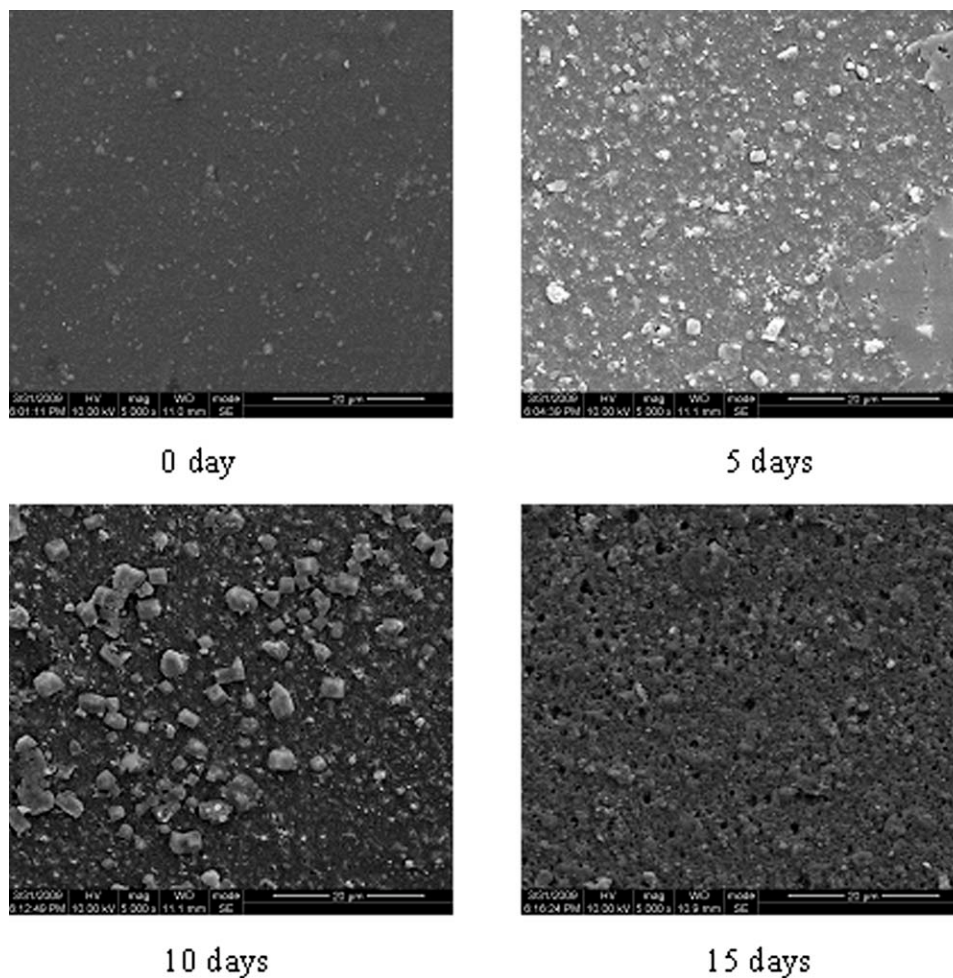
$$\text{VI} = S_{\text{C=C}}/S_{1430} \quad (2)$$

Here, the peak areas of carbonyl group and vinyl group were determined in the range of  $1800\text{--}1670\text{ cm}^{-1}$  and  $1660\text{--}1600\text{ cm}^{-1}$ , respectively.

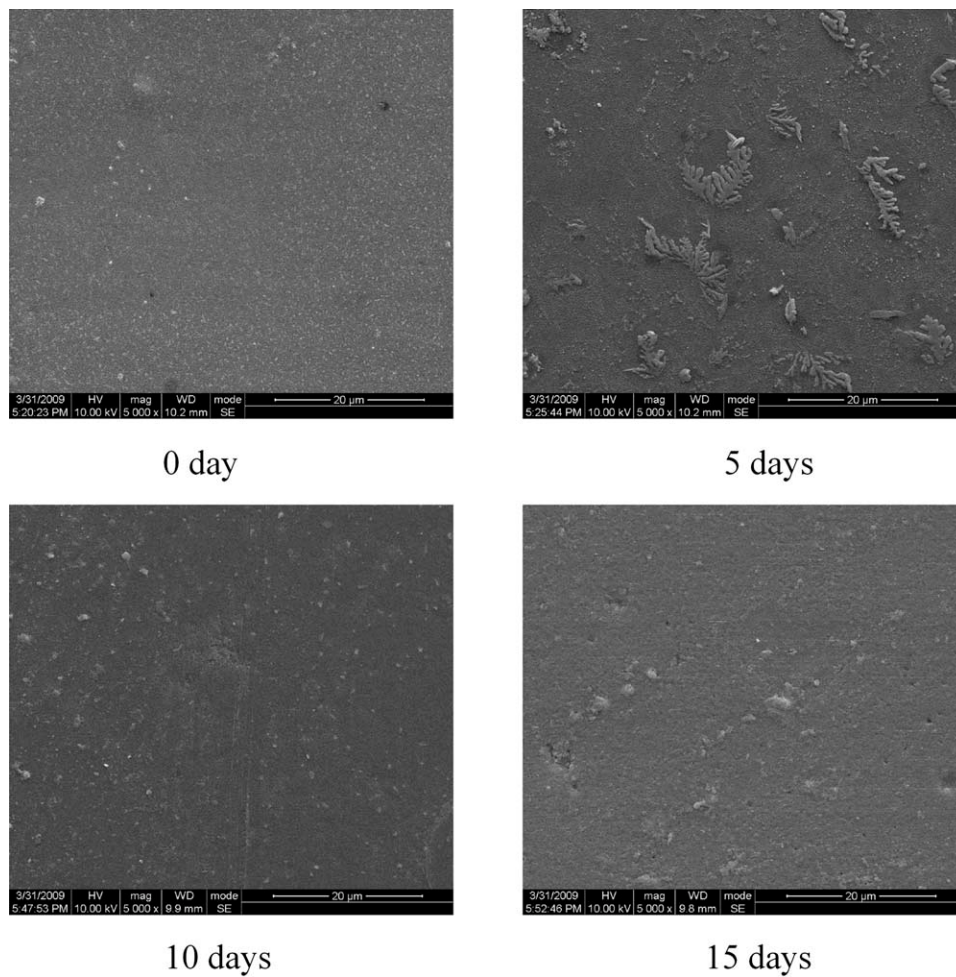
## RESULTS AND DISCUSSION

### Mechanical properties

Under UV irradiation, as the results of photo-degradation and photo-crosslinking reaction, the polymers become stiff and brittle, resulting in the poor mechanical properties.<sup>24,25</sup> The higher the absorbed energy is, the more quickly and deeply the properties drop. The data listed in Table I show that the elongation at break and impact strength of the PVC sheets gradually decrease with the increasing UV irradiation time. Obviously, the coated PVC sheets



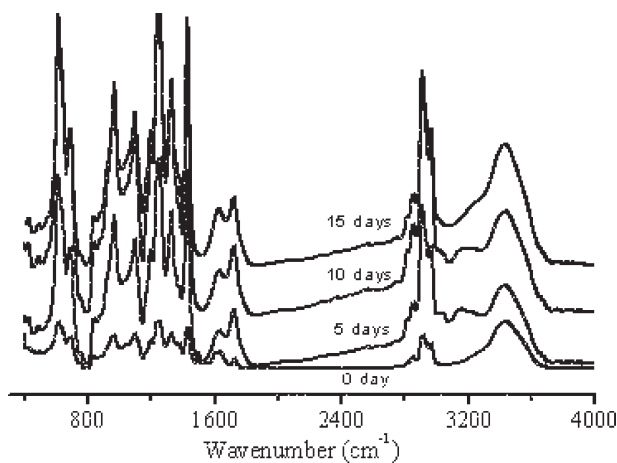
**Figure 2** The surface changes of uncoated samples during UV irradiation.



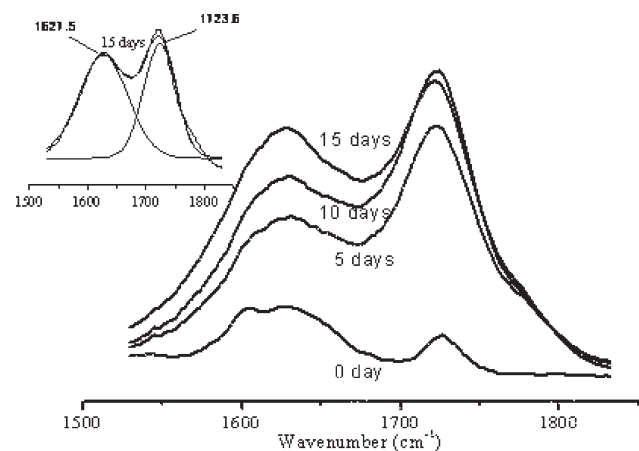
**Figure 3** The surface changes of coated samples during UV irradiation.

have the higher properties preservation. For example, the retained percentage of elongation at the break of the coated PVC sheet is 53.9% after UV irradiation for 15 days, whereas that of the uncoated one is only 32.3%. The former is 21% higher than the

latter. The retained percentage of impact strength of coated PVC sheet is 82.1%, which is 14% higher than the compared one's. This indicates that the acrylonitrile acrylate copolymer coating improves the UV resistance of the PVC sheets.



**Figure 4** FTIR spectra of uncoated samples during UV irradiation.



**Figure 5** FTIR spectra of uncoated samples during UV irradiation (1530–1830  $\text{cm}^{-1}$ ).

**TABLE II**  
Curve-Fitting Results for the FTIR Spectra of Uncoated Samples During UV Irradiation

Irradiation time (day)	Conjugated polyene sequence			Carbonyl group			Stretching vibration of CH <sub>3</sub>		
	$\nu_1$ (cm <sup>-1</sup> )	$w_1$ (cm <sup>-1</sup> )	$S_1$	$\nu_2$ (cm <sup>-1</sup> )	$w_2$ (cm <sup>-1</sup> )	$S_2$	$\nu_3$ (cm <sup>-1</sup> )	$w_3$ (cm <sup>-1</sup> )	$S_3$
0	1626.2	75.5	0.71	1724.4	23.9	0.49	1429.3	29.6	2.54
5	1628.6	71.0	9.64	1723.7	56.4	6.51	1429.2	30.9	14.74
10	1628.7	76.6	13.50	1723.2	54.0	8.93	1429.1	27.9	18.44
15	1627.5	83.2	10.36	1723.6	56.6	6.87	1428.9	26.1	13.27

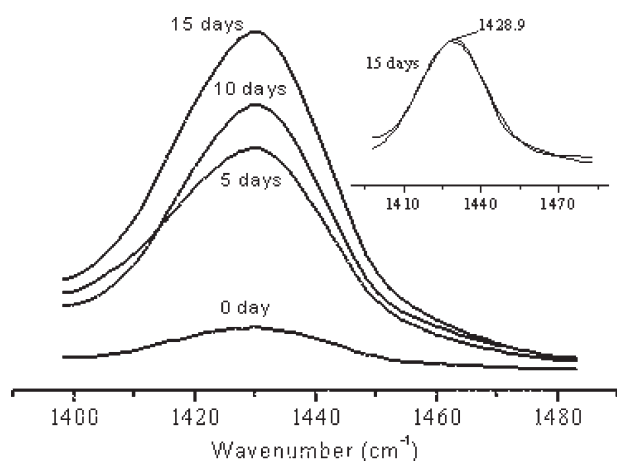
$\nu$ , wavenumber;  $w$ , width at half-height;  $S$ , intensity.

### Yellowness index

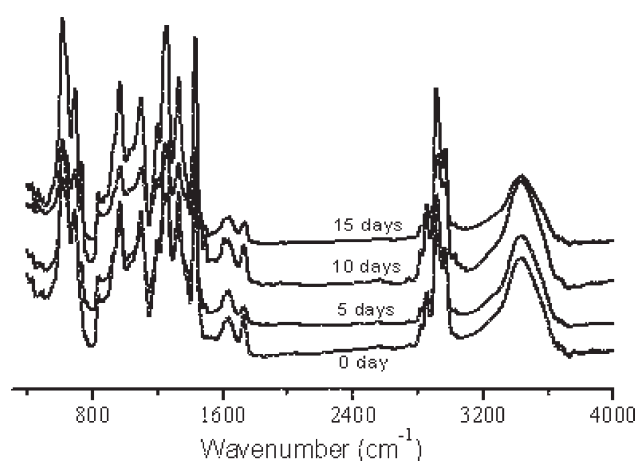
The discoloration of PVC under UV radiation is a result of the absorption capacity of the plastic in the UV range and the destructive action of the energy absorbed. This particular property of PVC cannot be altered, even by the application of a coating. However, the coating can efficiently prevent PVC sheets from damage by absorption of the UV energy and converting it into thermal energy. Figure 1 shows the changes in yellowness index with UV irradiation time. In general, the yellowness index increases for both of the samples when the UV irradiation time increases. It is obvious, however, that the coated PVC sheets show lower values and slower increasing rate of the yellowness index than the uncoated one. Furthermore, the yellowness index of the coated PVC sheets gradually levels off after the irradiation time reaches 10 days, while the index of the uncoated one is still rising. That means that the structure of the coated PVC sheet is on a relative balance state after 10-day UV irradiation, indicating that the coating can greatly filter the UV energy so that weak UV intensity could not cause the breakage of chemical bond in PVC. The coated PVC sheets show a greater UV resistance.

### Surface morphology

Figure 2 shows the surface changes of uncoated samples during UV irradiation. It can be seen that the surface of uncoated samples is chalked, and there are many small granules on the surface after 5-day irradiation. These granules become larger further after 10-day irradiation. When the UV irradiation time is up to 15 days, many holes and other new granules appear on the surfaces of PVC sheets. These phenomena are ascribed to the chalking and granular effect caused by the serious chemical reactions such as dehydrochlorination, oxidation, and chain scission and crosslinking under the intensive UV irradiation. When the top layer of PVC sheets has been eaten away, the sub-top layer is eroded to heel. The removed granules leave behind many holes. While for acrylonitrile acrylate copolymer coated PVC sheets, the situation is quite different. As shown in Figure 3, the smooth surface still keeps after 10-day irradiation. After 15-day irradiation, only few small crannies and chalked granules appear on the surface, indicating that the acrylonitrile acrylate copolymer coating greatly protects PVC sheets from UV degradation and crosslinking.



**Figure 6** FTIR spectra of uncoated samples during UV irradiation (1398–1483 cm<sup>-1</sup>).



**Figure 7** FTIR spectra of coated samples during UV irradiation (Coating was removed in toluene).

**TABLE III**  
Curve-Fitting Results for the FTIR Spectra of Coated Samples During UV Irradiation (Coating Was Removed in Toluene)

Irradiation time (day)	Conjugated polyene sequence			Carbonyl group			Stretching vibration of CH <sub>3</sub>		
	$\nu_1$ (cm <sup>-1</sup> )	$w_1$ (cm <sup>-1</sup> )	$S_1$	$\nu_2$ (cm <sup>-1</sup> )	$w_2$ (cm <sup>-1</sup> )	$S_2$	$\nu_3$ (cm <sup>-1</sup> )	$w_3$ (cm <sup>-1</sup> )	$S_3$
0	1623.3	110.5	6.10	1729.5	27.1	1.26	1429.0	25.9	6.86
5	1622.1	91.3	4.26	1729.4	30.5	1.19	1429.0	26.9	4.21
10	1621.9	113.2	7.26	1730.7	29.4	1.91	1429.0	26.0	6.10
15	1623.9	104.7	4.09	1731.9	32.4	1.16	1429.2	27.3	3.22

$\nu$ , wavenumber;  $w$ , width at half-height;  $S$ , intensity.

An interesting finding is that no matter the coated or uncoated PVC sheets, the variation trends of the mechanical properties, yellowness index, and surface morphologic character are corresponding to each other.

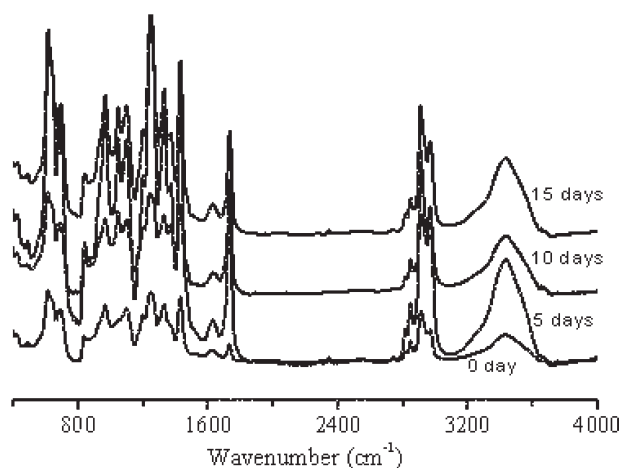
### FTIR analysis

FTIR is used to investigate the effect of the acrylonitrile acrylate copolymer coating on microstructures development of PVC sheets during UV irradiations. Figure 4 shows FTIR spectra of uncoated PVC sheets during UV irradiation. The intensities of the characteristic bands at around 1630 cm<sup>-1</sup> and 1726 cm<sup>-1</sup>, which are attributed to conjugated polyene sequence and carbonyl group, respectively, increase gradually with irradiation time. Here, the conjugated polyene sequence comes from the dehydrochlorination reaction, and the carbonyl group is the product of oxidation reaction.

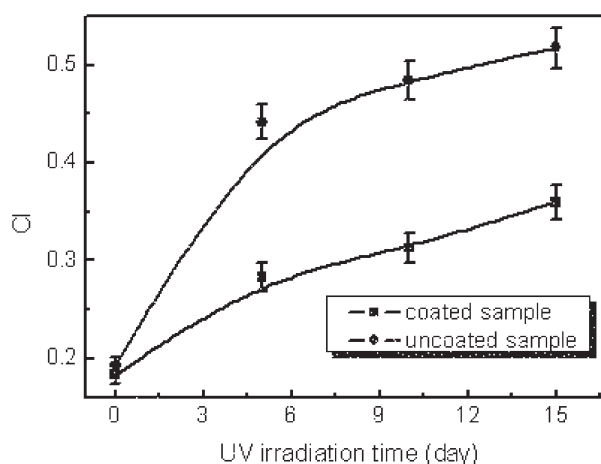
When the PVC sheet is subjected to UV irradiation, some weak chemical bonds or defected structures are first broken up, forming alkyl radicals and chlorine radicals. At the presence of oxygen, the alkyl radicals can be scavenged and lead to the formation of peroxy and alkoxy radicals with low activ-

ity. The following hydrogen abstraction reactions results in the generation of oxygenated structures and conjugated polyene sequence on the PVC backbone. These structures eventually initiate the chain scission and crosslinking reactions. This demonstrates that the main photo-degradation processes of PVC are dehydrochlorination and simultaneous oxygen reactions. So, the discussions of this section are emphasized on the changes of conjugated polyene sequence and carbonyl group.

Figure 5 shows the FTIR spectra of uncoated PVC sheets during UV irradiation in the region of 1530–1830 cm<sup>-1</sup>, which includes the conjugated polyene sequence and carbonyl group stretching band. To quantitatively analyze the effects of UV irradiation time on the conjugated polyene sequence and carbonyl group, the respective area method is used. With the aid of computer, the curve-fitting spectra of the region are executed. An iterative least-squares computer program is adopted to obtain the best fit of spectra of the region by varying the wavenumber ( $\nu$ ), width at half-height ( $w$ ), and area ( $S$ ) of the several bands. There are two Gaussian bands in the region to be confirmed by the curve-fitting procedure. Typical example is displayed in the iconograph of Figure 5.



**Figure 8** FTIR spectra of coated samples during UV irradiation (Coating was removed in ethyl acetate).



**Figure 9** Change of carbonyl groups of samples during UV irradiation.

**TABLE IV**  
Curve-Fitting Results for the FTIR Spectra of Coated Samples During UV Irradiation (Coating Was Removed in Ethyl Acetate)

Irradiation time (day)	Conjugated polyene sequence			Carbonyl group			Stretching vibration of CH <sub>3</sub>		
	$\nu_1$ (cm <sup>-1</sup> )	$w_1$ (cm <sup>-1</sup> )	$S_1$	$\nu_2$ (cm <sup>-1</sup> )	$w_2$ (cm <sup>-1</sup> )	$S_2$	$\nu_3$ (cm <sup>-1</sup> )	$w_3$ (cm <sup>-1</sup> )	$S_3$
0	1630.9	46.3	0.78	1732.2	22.3	0.59	1428.9	28.4	2.91
5	1632.0	70.2	4.37	1733.2	25.7	8.20	1429.6	24.5	10.87
10	1633.6	57.0	2.09	1731.8	31.9	4.78	1429.7	28.1	4.34
15	1632.9	67.8	3.35	1733.1	26.7	4.79	1429.7	24.2	6.35

$\nu$ , wavenumber;  $w$ , width at half-height;  $S$ , intensity.

The iconograph of Figure 5 shows that there are two bands in the region: the first band at 1627.5 cm<sup>-1</sup> is attributed to the conjugated polyene sequence stretching vibration; the second band at 1723.6 cm<sup>-1</sup> belongs to the carbonyl group stretching vibration. The curve-fitting results for spectra of the region are listed in Table II.

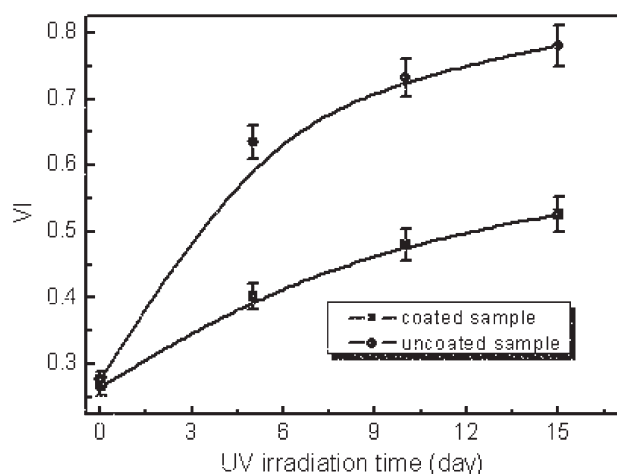
Under the UV irradiation, the structure of CH<sub>3</sub> is relatively sure and stable in PVC. Based on this premise, the CH<sub>3</sub> stretching band (at around 1430 cm<sup>-1</sup>) can be regarded as an inner standard for calculating the carbonyl index (CI) and the vinyl index (VI).

Figure 6 shows the FTIR spectra of uncoated PVC sheets during UV irradiation in the region of 1398–1483 cm<sup>-1</sup>, which is attributed to the CH<sub>3</sub> stretching band. To obtain the best fit of spectra of the band, an iterative least-squares computer program is adopted again. The curve-fitting results for spectra of the region are also listed in Table II.

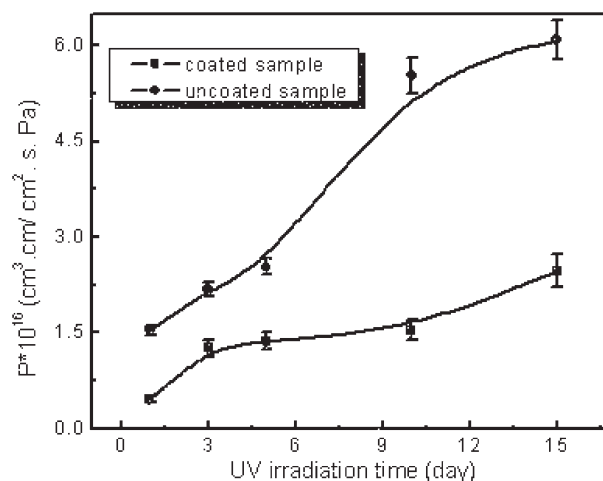
For the coated PVC sheets, two kinds of solvent are adopted to remove the coating from the baseplate to quantitatively detect the effects of UV irradiation on PVC matrix. The analysis results of the carbonyl group are shown in Figure 7 and Table III and that of the conjugated polyene sequence list in Figure 8 and Table IV.

According to eqs. (1) and (2), the CI and the VI of samples can be calculated. The curves of the CI and the VI versus the irradiation time are shown in Figures 9 and 10, respectively. From Figure 9, the CI of PVC without or with the coating increased with increasing UV irradiation time. However, the CI of coated PVC sheet is smaller than that of uncoated one for all irradiation time. The increasing rate of the CI for coated PVC is slower than that of compared one. The change tendency of the VI is similar to that of the CI. The coated PVC sheets show higher UV resistance than the uncoated one.

Moreover, from Figures 9 and 10, it can be also found that the microstructures development is consistent with the change of mechanical properties and the yellowness index of PVC sheets during the UV irradiation. The reason is that the degree of molecular degradation is in direct proportion to the degree of oxidation reaction. The higher the CI value is, the more greatly the oxidation reaction occurs, which results in higher degree of molecular degradation. The mechanical properties fall more quickly correspondingly. For the yellowness index, it is primarily controlled by the amount of conjugated polyene sequences. The high value of VI is corresponding to the high value of the yellowness index.



**Figure 10** Change of vinyl groups of samples during UV irradiation.



**Figure 11** The oxygen permeability coefficient of samples during UV irradiation.

### Mechanism

Improved stabilization and depressed degradation of PVC sheets through the acrylonitrile acrylate copolymer coating during UV irradiation is validated from above analysis. It is ascribed that the functional groups in the acrylonitrile acrylate copolymer, such as  $C\equiv N$ ,  $-CO-$ ,  $-COO-$ , absorb the UV energy more easily than PVC. They can convert the energy into harmless heat by molecules auto-rotation or/and recombination. So, when it is coated on the PVC surface, it can absorb most of UV energy and weaken the ability of photochemical reactions so as to avoid the breakage of chemical bond in PVC matrix.

On the other hand, the acrylonitrile acrylate copolymer can obstruct oxygen to immerge into the PVC matrix, as shown in Figure 11. The oxygen permeability coefficient of both PVC sheets increase with increasing UV irradiation time, but the values of the coated one are less than those of uncoated one. Moreover, the oxygen permeability coefficient of coated PVC sheets increases more slowly than that of uncoated one. It means that the coating can obviously hinder the oxygen diffusion. As a result of that the photochemical reaction rate decreases and reaction products reduce, such as carbonyl groups and conjugated polyene sequences, when the PVC sheets are covered with this coating.

### CONCLUSIONS

As the acrylonitrile acrylate copolymer can screen the UV irradiation and obstruct oxygen immerge into the matrix, it can effectively protect PVC sheets from photoageing during the UV irradiation. The coated PVC sheets have the higher mechanical properties preservation after UV irradiation for 15 days. The retained percentage of elongation at the break of the coated PVC sheet is 53.9%, whereas the uncoated one's is only 32.3%. The value of the former is about 21% higher than that of latter. The retained percentage

of impact strength of coated PVC sheet is 14% higher than the compared one's.

During UV irradiation, the coated PVC sheets shows lower yellowness index values, VI and CI, as well as the smoother surfaces than the uncoated PVC sheets.

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