Thermal Stability of Poly(vinyl chloride)/Layered Double Hydroxide Nanocomposites

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ABSTRACT: Effects of nanoscale dispersed layered double hydroxides (LDHs) on thermal stability of poly(vinyl chloride) (PVC) in thermal and thermooxidative degradation processes are investigated by dynamic and isothermal thermogravimetric analysis (TGA), discoloration test, fourier transform infrared (FTIR), and ultraviolet-visible (UV-vis) spectroscopic techniques. During both stages of thermal degradation, the degradation temperatures, including onset degradation temperature and temperature of the maximum degradation rate, increase, and the final residue yield of the PVC/LDH nanocomposites reaches 14.7 wt %, more than double that for neat PVC. The thermooxidative degradation process is more complex. During the first two stages, the presence of nanoscale dispersed LDH particles enhances the thermal stability, whereas in the last stage accelerates the thermal degradation possibly due to the accumulation of heat released. Additionally, the studies of the isothermal thermooxidative degradation process by FTIR and UV-vis spectra indicate that both polyene backbone formation and some carbonyl groups are simultaneously developed. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 116: 2058–2064, 2010

Key words: nanocomposites; poly(vinyl chloride) (PVC); thermal properties

INTRODUCTION

It is well known that poly(vinyl chloride) (PVC) is very unstable and easy to degrade when exposed to heat, shear or radiation (such as Ultraviolet (UV) light) during melt-processing and practical applications due to an autocatalytic dehydrochlorination degradation reaction.[1,2] Therefore, it is always necessary to add various additives to improve its thermal stability.

Layered double hydroxides (LDHs), an important layered inorganic material, have an intrinsic hydrogen chloride (HCl) absorption capacity and thus can enhance the thermal stability of PVC resin.[3] However, LDHs have much higher charge densities in the interlayer than other common inorganic layered materials such as clay, which makes the intercalation of PVC into LDHs to prepare PVC/LDH nanocomposites rather difficult. As a consequence, in most of the previous publications, LDHs were usually used as micron-scale fillers when added into PVC and their enhancement of thermal stability are very limited.[4–8] Fortunately, in recent years, the addition of LDHs to PVC matrix to prepare PVC/LDH nanocomposites has attracted much interest. For example, Bao et al.[9,10] obtained PVC/LDH nanocomposites via *in situ* polymerization, and studied the effect of LDHs on the PVC thermal stability, smoke emission, and mechanical properties. We prepared PVC/LDH nanocomposites by melt intercalation or solution intercalation process,[11–14] and reviewed the recent research progress of PVC/LDH nanocomposites.[15] Although the present publications reveal the importance and prospect of the attractive PVC/LDH nanocomposites, their studies are obviously very scarce, especially when compared with the extensive publications of polymer/clay nanocomposites. Undoubtedly, further detailed studies are expected for the promising PVC/LDH nanocomposites, especially the important effect of nanoscale dispersed LDH particles on the thermal stability of PVC.

In our recent work,[13] we reported the synthesis and characterization of PVC/LDH nanocomposites via solution intercalation process. The thermal stability was simply studied by conventional Congo Red method. It was found that by addition of only 5 or 10 wt % of organo-LDHs, the thermal stability time increased to 15 or 20 times of that of the pure PVC,

respectively. As is well known, thermogravimetric analysis (TGA) is an important technique to study the thermal stability. In this article, the effect of the nanoscale dispersed LDH particles on PVC thermal stability is reported in detail by dynamic and static TGA in nitrogen or air atmosphere. As thermooxidative degradation process in air is more complex than thermal degradation in nitrogen, discoloration method, fourier transform infrared (FTIR), and UV-visible (UV-vis) spectra were employed to study the isothermal thermooxidative degradation mechanism.

EXPERIMENTAL

Materials

The PVC resin (SG-5 type, suspension grade) was kindly provided by Beijing Huaer Company. The preparation procedure and structure characterization of PVC/LDH-nitrate (PVC/LDH-NO$_3$) composite, PVC/LDH-dodecyl sulfate (PVC/LDH-DS), and PVC/LDH-stearate (PVC/LDH-st) nanocomposites have been reported in Ref. 13. The content of the LDH-NO$_3$, LDH-DS, and LDH-st in the aforementioned composite or nanocomposite is 5 wt %.

Measurements

Dynamic TGA experiments were performed over a temperature range of 40–800°C using a Perkin-Elmer Pyris-7 thermogravimetric analyzer under flowing nitrogen or air environment, respectively, at a temperature-increase rate of 20°C/min. Isothermal TGA was conducted at 180°C under a flowing air environment in the time range of 0–3.5 h.

The discoloration test was carried out in an oven maintained at 180°C and subjected to static thermal aging according to the standard ISO 305–1990[16]. Eight time periods were chosen to investigate the degradation process.

The FTIR spectra were recorded on a Perkin-Elmer System 2000 FTIR spectrophotometer ranged 4000-400 cm^{-1} at a nominal resolution of 4 cm^{-1}. The FTIR studies were conducted using film samples cast from tetrahydrofuran (THF).

The UV-vis spectra were collected on a UV-2501PC UV-vis spectrometer with the wavelength from 200 to 800 nm, and the slit width was set as 2 nm.

RESULTS AND DISCUSSION

Thermal degradation process in nitrogen by dynamic TGA

Generally, the TGA and the derivative TGA (DrTGA) curves of PVC in nitrogen, characteristic of thermal degradation process, can be divided into two stages: the first stage is attributed to the dehy-
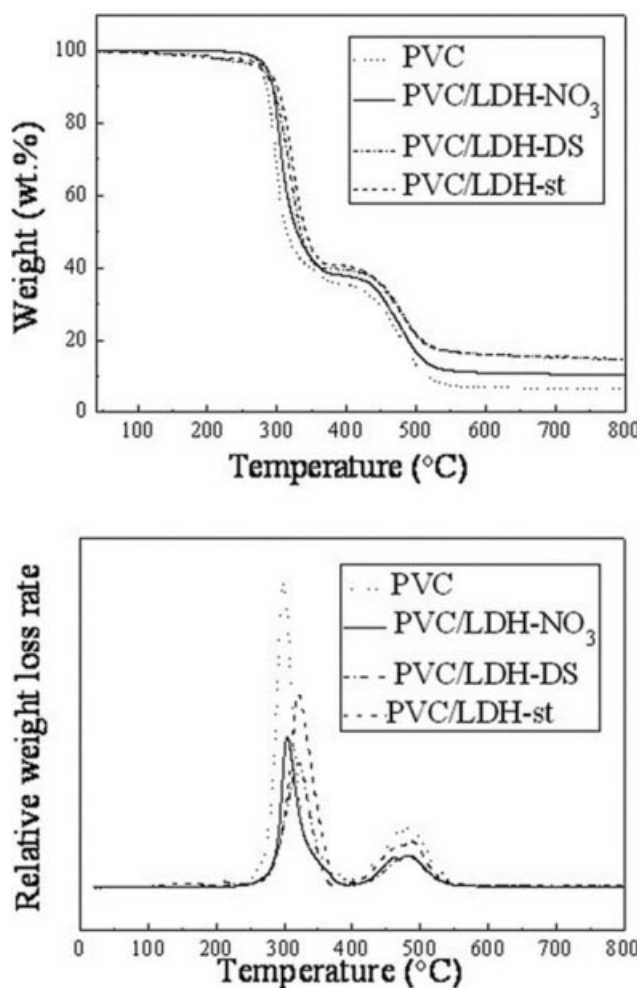

Figure 1 TGA and DrTGA curves for the degradation of pure PVC, PVC/LDH-NO$_3$ composite, PVC/LDH-DS, and PVC/LDH-st nanocomposites in nitrogen.

drochlorination process followed by the formation of conjugated polyene sequence, while the second stage corresponds to the thermal cracking of the carbonaceous conjugated polyene sequences.[17,18] The TGA and DrTGA curves of PVC, PVC/LDH-NO$_3$ composite, PVC/LDH-DS, and PVC/LDH-st nanocomposites in nitrogen are shown in Figure 1. Similar to that of pure PVC, there are two major weight loss stages for the PVC composite and nanocomposites. The first stage ranges from room temperature to about 400°C, and the second is in the range between 400 and 800°C. It is clearly observed that the thermal stability of PVC is obviously improved after the addition of the LDHs. From the TGA and DrTGA curves, the following thermal characteristics can be determined: the onset degradation temperature (T_{onset}); the temperature of the maximum degradation rate of each stage (T_{max1}, T_{max2}), the temperature at 20, 50, and 80 wt % weight loss ($T_{20\%}$, $T_{50\%}$, $T_{80\%}$), and the residue yield at each stage (W_{res1}, W_{res2}). The determined values are given in Table I.

TABLE I
Dynamic TGA Results for the Degradation of PVC, PVC/LDH-NO$_3$ Composite, PVC/LDH-DS
and PVC/LDH-st Nanocomposites in Nitrogen

Materials	T_{onset} (°C)	T_{max1} (°C)	T_{max2} (°C)	$T_{20\%}$ (°C)	$T_{50\%}$ (°C)	$T_{80\%}$ (°C)	W_{res1} (%)	W_{res2} (%)
PVC	279	298	479	293	314	481	36.2	6.4
PVC/LDH-NO$_3$	286	304	482	302	327	487	38.4	10.1
PVC/LDH-DS	289	321	484	307	332	505	39.8	14.7
PVC/LDH-st	298	323	488	315	344	505	41.1	14.7

T_{onset}: onset degradation temperature.
T_{max1}: temperature of the maximum degradation rate in the first degradation stage.
T_{max2}: temperature of the maximum degradation rate in the second degradation stage.

The first thermal degradation stage can be attributed to the volatilization of hydrochloride (HCl) molecules and the formation of conjugated polyene sequences. After the addition of 5 wt % of LDH-NO$_3$, the T_{onset} and T_{max1} increase from 279 and 298°C to 286 and 304°C, respectively. Furthermore, the residue yield of PVC/LDH-NO$_3$ composite is 2.2 wt % larger than that of the pure PVC. This phenomenon results from the fact that LDHs themselves could contribute to the HCl absorption and thus improve the thermal stability of PVC, although they are not exfoliated and dispersed very well. Interestingly, the thermal stability of the PVC nanocomposites is improved further due to the increased degree of exfoliation and dispersion of LDH platelets and particles even at the content of LDH-DS or LDH-st of only 5 wt %. The T_{onset}, T_{max1}, and W_{res1} of PVC/DS nanocomposite increase to 289°C, 321°C, and 39.8 wt %, respectively, much higher than the corresponding values of the neat PVC and the PVC/LDH-NO$_3$ composite. This strongly supports that the nanoscale dispersion structure has a significant improved effect on the PVC thermal stability. As for the PVC/LDH-st nanocomposite, it is interesting that the T_{onset}, T_{max1}, and W_{res1} increase further to 298°C, 323°C, and 41.1 wt %, being 19°C, 25°C, and 4.9 wt % higher than those of the pure PVC. This prominent enhancement of the thermal stability can be ascribed to the effects of both nanoscale dispersed LDHs and the stearate anions. These results are in consistent with that our recent Congo Red test[13] very well. Additionally, about 2 wt % weight loss in the temperature range from 100 to 210°C can be observed due to the dehydration of LDHs and the thermal decomposition of organic anions of DS$^-$ and stearate. A similar weight loss behavior has been reported in LLDPE/LDH-DS nanocomposites by Chen and Qu.[19] In addition, the temperatures at 20, 50, and 80 wt % weight loss were chosen for comparison in Table I. The results show that the $T_{20\%}$ and $T_{50\%}$ obviously increase as well, from 293, 314 and 481°C for PVC to 315, 344, and 505°C for the PVC/LDH-st nanocomposite, respectively. Based on the earlier results, we may conclude that LDHs, especially the nanoscale dispersed LDHs,

which can react with HCl and thus weaken its autocatalytic function, have an important effect on prevention or delay the degradation process in the first stage of thermal degradation. Moreover, the nanoscale LDH-st particles have the best thermal stability, as the stearate anions are a thermal stabilizer, whereas DS$^-$ anions are not.

The second thermal degradation stage of PVC, PVC/LDH-NO$_3$ composite, PVC/LDH-DS, and PVC/LDH-st nanocomposites in nitrogen ranges from 400 to 800°C. This stage involves the scission of polyene sequences by cracking, cross-linking, aromatization, dehydrocarbonation, and charring, evolving a wide range of hydrocarbons, such as benzene, toluene, xylene, naphthalene, ethylbenzene, styrene, 1-butene, butane, pentane, and hexane.[20,21] It can be observed in Figure 1 and Table I that the thermal stability of PVC/LDH-NO$_3$ composite is much higher than that of the pure PVC, similar to that in the first stage. For the PVC/LDH-DS and PVC/LDH-st nanocomposites, their TGA curves are almost superposition and still higher than those of PVC and PVC/LDH-NO$_3$ composite, which suggests that it is the nanoscale dispersed LDHs but not the DS$^-$ or stearate anions influence the thermal stability of PVC in this stage. One possible reason may be that the organic anions might have been completely decomposed during the first degradation stage, and the degree of exfoliation and dispersion of LDHs in the PVC/LDH-DS and PVC/LDH-st nanocomposites is similar. In addition, the residue yields of PVC, PVC/LDH-NO$_3$ composite, PVC/LDH-DS, and PVC/LDH-st nanocomposites at 800°C are 6.4, 10.1, 14.7, and 14.7 wt %, respectively. Thus, it may be concluded that the addition of LDHs can promote the charring during the degradation process, and the charring amounts of the nanocomposites are larger than that of the PVC/LDH-NO$_3$ composite.

Thermooxidative degradation process in air by dynamic TGA

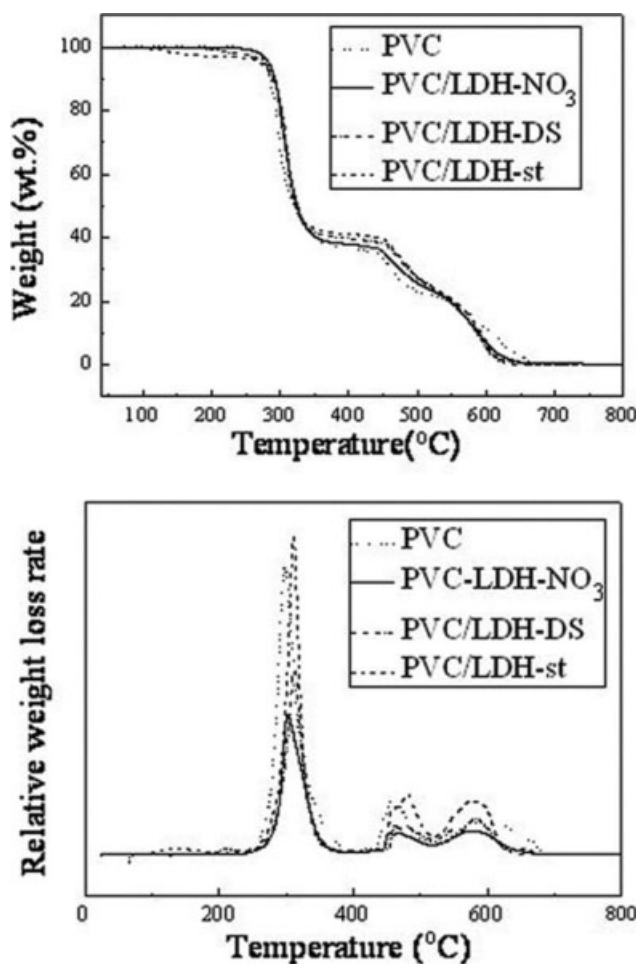The dynamic TGA study of the thermooxidative degradation of PVC, PVC/LDH-NO$_3$ composite,

Figure 2 TGA and DrTGA curves for the degradation of pure PVC, PVC/LDH-NO$_3$ composite, PVC/LDH-DS, and PVC/LDH-st nanocomposites in air.

PVC/LDH-DS, and PVC/LDH-st nanocomposites in air are shown in Figure 2 and Table II. The thermooxidative degradation process in air is more complicated than the thermal degradation in nitrogen atmosphere. The process can be divided into three major weight loss stages: lower than 390°C, between 390 and 550°C, and higher than 550°C. The T_{onset}, T_{max1}, and T_{max2} of the PVC nanocomposites and composite are higher than the corresponding tem-

peratures of the pure PVC, whereas T_{max3} (the temperature of the maximum degradation rate in the third stage) of the PVC nanocomposites and composite is lower than that of pure PVC. In this sense, the addition of LDHs improves the thermal stability of PVC in the first and second stages, whereas benefits little in the third stage. Indeed, the thermooxidative degradation is very complex, and will be discussed in detail in the following.

The first thermooxidative degradation stage can be divided into three parts. The first part is between 100 and 278°C. Similar to the earlier thermal degradation in nitrogen, the increased weight loss for the nanocomposites may be due to the dehydration of LDHs and the thermal decomposition of the organic DS$^-$ and stearate anions. Then, with the increase of temperature, the thermal stability of the PVC nanocomposites is higher than that of the pure PVC. It is worthy of noting that there is an intersection point at 332°C. In the range of 278–332°C, the difference of the weight loss between the PVC nanocomposites and composite is little. At temperature higher than 332°C, the thermal stability follows the sequence of PVC/LDH-st nanocomposite > PVC/LDH-DS nanocomposite > PVC/LDH-NO$_3$ composite > pure PVC. The mechanism of the first stage might be very complex. On the one hand, LDHs have the ability to absorb HCl, and delay or reduce the autocatalytic reaction, which increases the thermal stability. On the other hand, the decomposition of the organo anions results in heat release, and accelerates the thermooxidative degradation. The observed results may be the overall effects of these two contradictive factors.

The second thermooxidative degradation stage is between 390 and 550°C. This stage corresponds to the thermal cracking of the carbonaceous conjugated polyene sequences. From Table II, it can be seen that the T_{max2} increases from 456°C of PVC to 462°C after the addition of LDH-NO$_3$, and even increases to 470 and 482°C for the PVC/LDH-DS and PVC/LDH-st nanocomposite, respectively. When the 70 wt % weight loss is chosen for comparison, the thermooxidative degradation temperatures for the pure PVC, PVC/LDH-NO$_3$ composite, PVC/LDH-DS, and PVC/LDH-st

TABLE II
Dynamic TGA Results of PVC, PVC/LDH-NO$_3$ Composite, PVC/LDH-DS, and PVC/LDH-st Nanocomposites in Air

Materials	T_{onset} (°C)	T_{max1} (°C)	T_{max2} (°C)	T_{max3} (°C)	$T_{20\%}$ (°C)	$T_{70\%}$ (°C)	$T_{80\%}$ (°C)	W_{res} (%)
PVC	276	297	456	591	294	462	543	0.3
PVC/LDH-NO$_3$	284	304	462	578	299	475	546	0.3
PVC/LDH-DS	284	312	470	581	302	487	548	0
PVC/LDH-st	283	309	482	582	300	489	541	0

T_{onset}: onset degradation temperature.
T_{max1}: temperature of the maximum degradation rate in the first degradation stage.
T_{max2}: temperature of the maximum degradation rate in the second degradation stage.
T_{max3}: temperature of the maximum degradation rate in the third degradation stage.

nanocomposites were determined to be 462, 475, 487, and 489°C, respectively. These phenomena indicate that the LDHs, especially the nanoscalely dispersed LDHs, can delay or clag the scission of polyene and thus improve the PVC thermooxidative stability.

The range from 550 to 800°C can be considered as the third thermooxidative degradation stage. In this stage, the presence of LDHs accelerates the thermooxidative degradation. This reverse trend of thermooxidative stability has also been found in other polymer/clay nanocomposites,[22,23] however, the exact mechanism is not yet clear. A possible explanation is that the LDHs sheets or particles become a heat source, and hold accumulated heat in this degradation stage.

Isothermal thermooxidative process in air

Isothermal TGA in air

To acquire a clear understanding of the static thermooxidative degradation of pure PVC, PVC/LDH-NO_3 composite, PVC/DS, and PVC/LDH-st nanocomposites, the isothermal TGA in air was conducted (Fig. 3). Temperature of the earlier discoloration method) (180°C) is chosen as the isothermal temperature. After 3.5 h, the weight loss amounts of the pure PVC, PVC/LDH-NO_3 composite, PVC/DS, and PVC/LDH-st nanocomposites are about 0.9, 2.3, 4.6, and 4.2 wt %, respectively. As the weight loss of pure PVC is due to the dehydrochlorination reaction, we may conclude that only a small amount of HCl is released during the thermooxidative degradation process at 180°C. For the PVC/LDH-NO_3 composite, both dehydrochlorination of PVC and the dehydration of LDHs contribute to the increased weight loss. As for the PVC/LDH-DS and PVC/
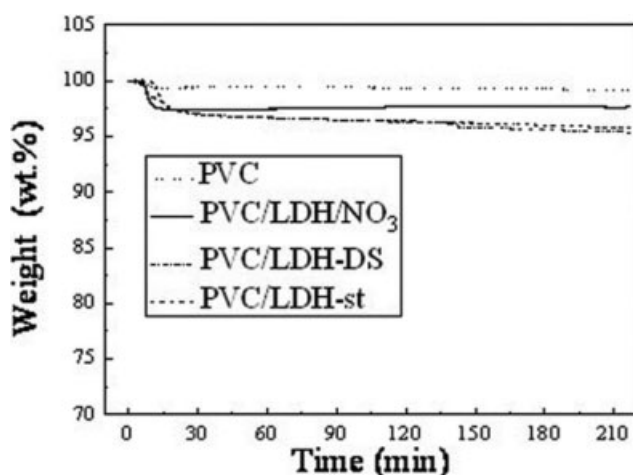

Figure 3 Isothermal TGA curves for the degradation of pure PVC, PVC/LDH-NO_3 composite, PVC/LDH-DS, and PVC/LDH-st nanocomposites in air.

LDH-st nanocomposites, the weight loss is owing to the dehydrochlorination of PVC, the dehydration of LDHs and the thermal decomposition of DS^- or stearate anions. Because the time was sustained long enough (3.5 h), the dehydration of LDHs and the thermal decomposition of organic anions were almost completed. Consequently, the quantities of dehydrochlorination for PVC/LDH-NO_3 composite, PVC/LDH-DS, and PVC/LDH-st nanocomposites are about 1.8, 2.1, and 2.1 wt %. These results suggest that the addition of LDHs accelerated the dehydrochlorination at the beginning of the thermooxidative degradation, and is in agreement with the results of the above dynamic TGA in air. The reason may lie in that the LDHs absorb the HCl and thus accelerate the release of HCl at the initial stage of the thermooxidative degradation.

Discoloration test

The powder samples of the pure PVC, PVC/LDH-NO_3 composite, PVC/LDH-DS, and PVC/LDH-st nanocomposites were placed on a glass slide and put into a blast oven at 180°C to carry out static thermal aging test by observing their color change. In Figure 4(a,b), both samples become darken with time, and the speed of color darkening for PVC/LDH-NO_3 is slower than that for pure PVC, indicating the inorganic LDHs could delay the thermooxidative degradation of PVC. As for PVC/LDH-DS nanocomposite [Fig. 4(c)], the color becomes brown in the beginning and then black after 70 min. After that, the color becomes thinner with time. A similar phenomenon happens to the PVC/LDH-st nanocomposite, as shown in Figure 3(d). When the color at 210 min is chosen for comparison, it can be observed that the PVC/LDH-NO_3 composite, PVC/LDH-DS, and PVC/LDH-st nanocomposites have the approximate color and all are lighter than that of the pure PVC. The reason for these phenomena may be that the initiative color change of rapidly becoming black, of PVC/LDH-DS and PVC/LDH-st nanocomposites mainly results from the decomposition of the DS^- and stearate anions, and char formation. The DS^- and stearate anions become carbonaceous char and formed char-like materials, which darkens the color of PVC/LDH-DS and PVC/LDH-st nanocomposites. Then, the char-like materials is further oxidized to afford carbon dioxide and released. As a result, the color of the PVC nanocomposites becomes thinner finally.

FTIR spectroscopic study

The FTIR spectra of pure PVC, PVC/LDH-NO_3, PVC/LDH-DS, and PVC/LDH-st nanocomposites before and after heating at 180°C for 3.5 h in air

have been studied. Besides the absorption bands of pure PVC and LDHs, there is an obviously different absorption band at 1710 cm^{-1} $\upsilon_{(C=O)}$ for the PVC/LDH-NO$_3$ composite, PVC/LDH-DS, and PVC/LDH-st nanocomposites before heat treatment. The appearance of this band may result from cyclohexanone, which is the dispersing solvent of LDHs in the preparation of PVC/LDH composite or nanocomposites via solution intercalation. After the heat treatment, there is a newly multiple absorption bands around 1720 cm^{-1}, which may be due to the β-chlorooketone or α-chlorocarboxylic groups produced during the thermooxidative degradation. Additionally,
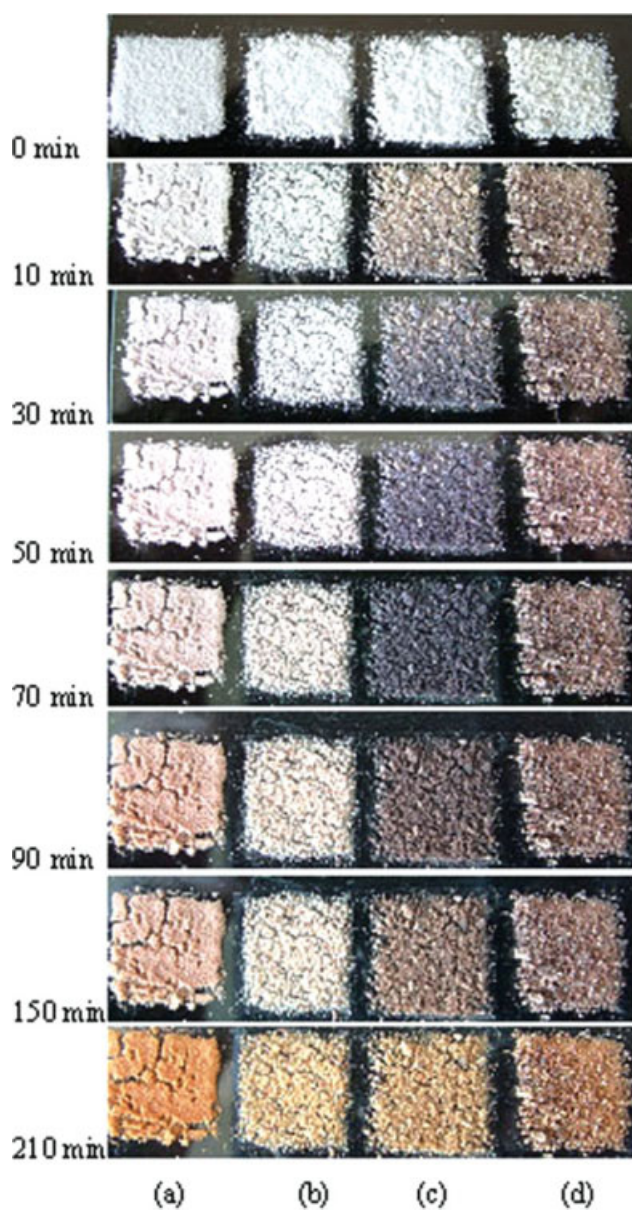

Figure 5 UV-vis spectra for the degradation of PVC, PVC/LDH-NO$_3$ composite, PVC/LDH-DS, and PVC/LDH-st nanocomposites after treatment at 180°C for 3.5 h.

the bands at 690 and 615 cm^{-1}, which can be assigned to the C—Cl stretching vibrations of the PVC chains, remain strong. And there are no obvious absorption band at 1640 cm^{-1}$\upsilon_{(C=C)}$, suggesting the main reaction is the formation of carbonyl groups during the thermooxidative degradation of the PVC and its composite or nanocomposites. This result agrees with that of isothermal TGA result given earlier that only a small amount of weight loss was observed.

UV-vis spectroscopic study

The UV-vis spectroscopic study was also carried out to investigate the dehydrochlorination reaction, which would form conjugated double bonds. It has been reported that the UV–vis absorption wavelength corresponds to the conjugated chain length of PVC degradation products.[24] Figure 5 shows the UV-vis spectra of pure PVC, PVC/LDH-NO$_3$ composite, PVC/LDH-DS, and PVC/LDH-st nanocomposites after heat treatment at 180°C for 3.5 h. The main absorption wavelengths of all of the four samples are around 230 nm, which corresponds to the conjugated double bonds with chain length of 2. This indicates that the reaction of dehydrochlorination exists and mostly forms the conjugated double bonds with chain length of 2 during the thermooxidative degradation at 180°C, although the main reaction is the formation of carbonyl groups confirmed by the earlier FTIR spectroscopic studies.

Figure 4 Discoloration test for the degradation of (a) pure PVC, (b) PVC/LDH-NO$_3$ composite, (c) PVC/LDH-DS nanocomposite, and (d) PVC/LDH-st nanocomposite. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
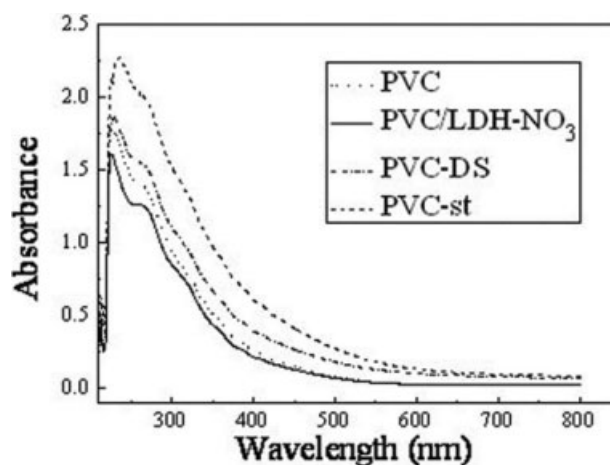
CONCLUSIONS

The thermal stability of PVC, PVC/LDH-NO$_3$ composite, PVC/LDH-DS, and PVC/LDH-st nanocomposites has been investigated. The presence of nanoscale dispersed LDH particles can greatly affect the

thermal stability of PVC during thermal and thermooxidative degradation processes. The PVC/LDH nanocomposites exhibit obviously enhanced the onset decomposition temperature, the maximum degradation rate temperature, and the residue yields during the thermal degradation in nitrogen. The dynamic thermooxidative degradation process in air is more complex. During the first two stages, the presence of the nanoscale dispersed LDHs particles enhances the thermal stability, whereas the thermal degradation is accelerated in the last stage. During the isothermal thermooxidative degradation process in air, the nanoscale dispersed LDH particles accelerate the dehydrochlorination and lower the thermal stability, the organic anions decompose and become charred, and the oxidative degradation reaction forming carbonyl groups is the main reaction accompanied by the simultaneous dehydrochlorination procedure.

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