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# Fire retardancy and morphology of nylon 6-clay nanocomposite compositions

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Shanmuganathan, Kadhirava; Razdan, Sandeep; Dembsey, Nicholas; Fan, Qinguo; Kim, Yong K.; Calvert, Paul D.; Warner, Steven B.; and Patra, Prabir, "Fire retardancy and morphology of nylon 6-clay nanocomposite compositions" (2005). *Faculty of Science, Medicine and Health - Papers: part A*. 316. https://ro.uow.edu.au/smhpapers/316

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# Fire retardancy and morphology of nylon 6-clay nanocomposite compositions

## Abstract

We investigated the effect of organically modified clay on the thermal and flammability behavior of nylon 6 nanocomposites. We also used zinc borate along with layered silicate with an aim of achieving synergistic effect in flame retardancy. It is found that addition of 10 wt% clay reduced the onset decomposition (5% wt loss) temperature of nylon 6 by 20°C, while addition of 5 wt% zinc borate and 5 wt% clay in combination reduced it by around 10°C. Differential thermogravimetric analysis indicated that the peak decomposition temperature was not affected by the addition of clay, but the rate of weight loss decreased with increasing clay concentration. The horizontal burning behavior of the nanocomposite films of approximately 0.5mm thickness changed with additive concentration. The nanocomposites with 2.5 wt% and 5 wt% clay burned for almost the same duration as neat nylon 6 but dripping was reduced. The 10 wt% clay nanocomposite sample burned without any dripping and the flame spread rate was reduced by 25-30%. The burn rate of 5 wt % zinc borate/5 wt% clay nanocomposite sample was about 20% higher than that of 10 wt% clay nanocomposite sample, which could be attributed to varying char morphology. Scanning electron microscopy images of the 10wt% clay nanocomposite char surface and cross- section revealed an integrated layer of clay platelets with increasing density gradient from the center to the surface, while the 5 wt% zinc borate/5 wt% clay nanocomposite char appeared foamy and porous. The 5 wt% zinc borate and 5 wt% clay sample developed into a very good intumescent system in cone calorimeter test, swelling about 10-13mm height prior to ignition forming a cellular char structure. This was as effective as the 10wt% clay nanocomposite sample in reducing the heat release and mass loss rate of nylon 6 by around 65%. Fourier transform infrared spectroscopy of the 10 wt% clay nanocomposite char showed the presence of amides, indicating possible residual polymer within the shielded char.

## Keywords

nanocomposite, clay, 6, morphology, retardancy, compositions, fire, nylon

## Disciplines

Medicine and Health Sciences | Social and Behavioral Sciences

## **Publication Details**

Shanmuganathan, K., Razdan, S., Dembsey, N., Fan, Q., Kim, Y. K., Calvert, P. D., Warner, S. B. & Patra, P. (2005). Fire retardancy and morphology of nylon 6-clay nanocomposite compositions. MRS Online Proceedings, 887 pp.79-91.,

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Mater, Res. Soc. Symp. Proc. Vol. 887 © 2006 Materials Research Society

0887-Q02-01

Fire Retardancy and Morphology of Nylon 6-clay Nanocomposite Compositions.

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### ABSTRACT

We investigated the effect of organically modified clay on the thermal and flammability behavior of nylon 6 nanocomposites. We also used zinc borate along with layered silicate with an aim of achieving synergistic effect in flame retardancy. It is found that addition of 10 wt% clay reduced the onset decomposition (5% wt loss) temperature of nylon 6 by 20°C, while addition of 5 wt% zinc borate and 5 wt% clay in combination reduced it by around 10°C. Differential thermogravimetric analysis indicated that the peak decomposition temperature was not affected by the addition of clay, but the rate of weight loss decreased with increasing clay concentration. The horizontal burning behavior of the nanocomposite films of approximately 0.5mm thickness changed with additive concentration. The nanocomposites with 2.5 wt% and 5 wt% clay burned for almost the same duration as neat nylon 6 but dripping was reduced. The 10 wt% clay nanocomposite sample burned without any dripping and the flame spread rate was reduced by 25-30%. The burn rate of 5 wt % zinc borate/5 wt% clay nanocomposite sample was about 20% higher than that of 10 wt% clay nanocomposite sample, which could be attributed to varying char morphology. Scanning electron microscopy images of the 10wt% clay nanocomposite char surface and cross- section revealed an integrated layer of clay platelets with increasing density gradient from the center to the surface, while the 5 wt% zinc borate/5 wt% clay nanocomposite char appeared foamy and porous. The 5 wt% zinc borate and 5 wt% clay sample developed into a very good intumescent system in cone calorimeter test, swelling about 10-13mm height prior to ignition forming a cellular char structure. This was as effective as the 10wt% clay nanocomposite sample in reducing the heat release and mass loss rate of nylon 6 by around 65%. Fourier transform infrared spectroscopy of the 10 wt% clay nanocomposite char showed the presence of amides, indicating possible residual polymer within the shielded char.

#### INTRODUCTION

Nanocomposites are a distinct class of composite materials showing impressive performance for multifunctional applications at a very low filler level of 2-5 wt%. They are defined by the particle size of the dispersed phase having at least one dimension less than 100 nm [1]. Polymer-layered silicate nanocomposites (PLSN) have been in the realm of research since 1961 [2] owing to their enhanced mechanical [3], thermal [4] and barrier properties [5] over conventional composites with micron size fillers. The enhancement in properties is largely attributed to the aspect ratio and intercalation ability of the layered silicates, resulting in high specific surface area and hence more potential for polymer filler interaction [6]. Nanocomposites show great promise as effective fire retardants for many polymers [7-12]. Because of the upcoming

restrictions on some of the current popular flame retardants, the flame retardancy of nanoparticle filled systems or any non-halogen alternatives need to be improved to meet the new standards. As a result interest in this area of research has recently increased. Though the significant reduction in heat release rate of nanocomposites was reported for quite some time [2], the flame retardancy of polymer filled with nanoparticles such as clay, carbon nanotubes or silica has not further improved to achieve industrial acceptance. Also the effect of nano fillers on thermal stability and flame retardancy of nanocomposite fibers is not clear.

Work done in the previous years by our group and others were focused on understanding the mechanism behind the flame retardancy of polymer layered silicate nanocomposites. It has been found that the addition of 2-5 wt% of montmorillonite reduces the peak heat release rate by 50. 60% compared to neat nylon 6 as found by cone calorimeter studies on slabs of 4-5 mm thickness. The formation of a surface char which acts as a thermal insulation and mass barrier reduces the mass loss rate and hence the rate of heat release [13, 14]. The heat of combustion, CO2 and CO yields are not changed significantly, which indicates the predominant role of layered silicates in the condensed phase rather than the gas phase. Since the flame retardant effects of layered silicates is more physical than chemical the sample geometry would strongly influence the burning behavior of nanocomposite films. The nanocomposite films of thickness less than 1mm or a fabric of nanocomposite fibers would behave much different when subjected to a flame. As the sample becomes thinner, the concentration of layered silicates would become critical in providing a significant effect. In that case we need to either increase the concentration of layered silicates to a level sufficient to form a network of platelets and hence a strong mass barrier or alternatively the flame retardant effect of layered silicates has to be augmented by another flame retardant additive. We studied the effect of clay concentration on the flame retardant behavior of nanocomposites in relation to polymer layered silicate interactions and char morphology. We also investigated the effect of zinc borate in enhancing the flame retardancy of polymer-layered silicate nanocomposites.

We chose zinc borate in order to reinforce the flame retardant effect of layered silicates through the formation of a stable char. Decomposition of zinc borate releases  $B_2O_3$  moiety, a low melting glass that can stabilize the char. It also releases its water of hydration (about 13-15%) at 290-450 °C which can blow the char to a foamy structure [15]. Thus it could significantly influence the char morphology and hence the flame retardancy of polymer layered silicate nanocomposites. Also it is thermally stable in the processing temperature of nylon 6.

#### **EXPERIMENTAL**

#### <u>Materials</u>

Nylon 6 was supplied by Honeywell Inc and montmorillonite (1.34 TCN), modified with methyl dihydroxyethyl hydrogenated tallow ammonium was provided by Nanocor Inc. Hence the term layered silicate or clay used in our literature refers to organically modified montmorillonite (OMMT). The specific surface area of OMMT is around 750 m<sup>2</sup>/g and the cation exchange capacity is 92 meq/100g (as per manufacturer). Nylon 6 pellets were dried in a vacuum chamber at 80°C for 16 hours and OMMT was dried for 4 hours at 100°C prior to melt mixing. Nanocomposite hybrids with 2.5 wt% (NCH 2.5), 5 wt% (NCH 5), and 10 wt% (NCH 10) of OMMT were prepared by melt compounding using Brabender twin blade roller mixer. Also,

nanocomposite with 5 wt% OMMT and 5 wt % zinc borate (NCH OMMT/ZnB) and nanocomposite with 5wt% ZnB were prepared. Melt compounding was done at 240°C for 10 minutes at 90 rpm. The samples were pressed into films of approx 0.5 mm thickness using Carver hot press under a pressure of 10000 psi at 240°C for 2 min.

#### **Characterization**

#### Thermal Analysis

The pyrolysis behavior of nanocomposites and neat samples was analyzed using TA instruments model Q500 Thermo gravimetric analyzer (TGA). The samples were heated in a nitrogen atmosphere from room temperature to 600°C at a heating rate of 20°C/min. The kinetics of the decomposition process was studied by differential thermo gravimetric analysis and the activation energy of the decomposition process was determined assuming a first order reaction for polymer pyrolysis using the Broido equation [16],

$$\ln[\ln(\frac{1}{x})] = \frac{-E_a}{RT} + \cos\tan t$$
<sup>(1)</sup>

where Ea = activation energy for the reaction,

T = absolute temperature in KelvinR = 8.314 I/mole °K

$$x = \frac{W_t - W_{\infty}}{W_0 - W_{\infty}}$$
<sup>(2)</sup>

where  $W_t$  is the weight of the sample at any time t,  $W_o$  and  $W_{\infty}$  are initial and final weight of sample respectively.

#### Flammability testing

The nanocomposites films of approx 0.5mm thickness were subjected to flame testing (FMVSS 302) using an Atlas horizontal flame spread tester. The flame height was set to about 1.5 inch and the ignition source was applied for 15 sec.

Flammability testing was also done on selected samples using cone calorimeter. Nanocomposite slabs of 100 mm X 100 mm and about  $3.0\pm0.1$  mm thickness were subjected to an incident heat flux of 35 kW/m<sup>2</sup> and the ignition and heat release characteristics were observed.

Char morphology was imaged using scanning electron microscope (JEOL 2000) with an accelerating voltage of 15 kV. Also Fourier transform infrared spectroscopy was done on the char using Digilab Excalibur series FTS 3000 Max in diffuse reflectance mode.

#### **RESULTS AND DISCUSSIONS**

#### **Thermal Behavior**

Sample	5% wt loss temp (°C)	10% wt loss temp (°C)	Peak decomposition temp (°C)	Peak decomposition rate (%/min)	Residue (%)	Activation energy X 10 <sup>5</sup>
Neat nylon 6	432	447	488	45.59	1.009	2.24
NCH 2.5	427	444	486	45.87	2.242	2.25
NCH 5	422	440	490	37.10	5.506	1.85
NCH 10	412	436	487	39.24	8.769	1.59
NCH Zn B	420	438	484	41.36	11.03	1.82

## Table I: TGA and DTGA data of nylon 6 and nanocomposites

The onset decomposition temperatures (5% and 10% wt loss temperature), peak decomposition temperature, peak decomposition rate, % residue, activation energy of decomposition of nanocomposites and neat samples are given in Table I. The values reported are average of 3 tests The TGA graphs and data showed reduction in the onset decomposition temperature of nanocomposite as compared to neat nylon 6 (Figure 1a) and the difference increased with increasing concentration of OMMT. However, the peak decomposition temperature was almost same and decomposition rate reduced with increasing concentration of OMMT (Figure 1b). The amount of additional carbonaceous residue was negligible accounting for the OMMT content.

The influence of layered silicates (OMMT) on the decomposition behavior of nanocomposites can be considered in dual aspects. The presence of enhanced polymer layered silicate interactions favor the thermal stability of the polymer. On the other hand the organic modifier present in the montmorillonite decomposes between 200-300°C and causes significant polymer matrix degradation during the melt processing of polymer organoclay mixtures (17). It is found that OMMT and water combine to catalyze the degradation of polymer (18). Hence, the decomposition behavior of nanocomposites would be influenced by the concentration of OMMT. From the values of decomposition temperature it is difficult to confirm the degradation effects of OMMT as a function of concentration. Therefore the estimated weight loss of nanocomposites was compared with the actual weight loss as a function of temperature (Table II). The estimated weight loss was calculated using the formula,

(3)

$$M_{c}(T) = W_{p} * M_{p}(T) + W_{f} * M_{f}(T)$$



**Figure 1**: (a) TGA thermograms of nylon 6 and nanocomposites (b) DTGA thermograms of nylon 6 and nanocomposites.

Table II: Weight loss data of nylon 6 and nanocomposites

Temp	NCH 2.5 M <sub>c</sub> (%)		NCH 5 M <sub>c</sub> (%)		NCH 10 M <sub>c</sub> (%)	
°C						
	Estimated	Observed	Estimated	Observed	Estimated	Observed
300	99.27	99.30	98.98	99.16	98.40	99.46
350	98.99	99.03	98.48	98.79	97.59	98.74
400	98.06	98.15	97.52	97.64	96.40	96.13
450	88.24	86.59	87.9	84.70	87.20	83.76

where  $M_c(T)$ ,  $M_p(T)$  and  $M_f(T)$  are respectively the percentage mass of nanocomposite, nylon 6 and OMMT not yet decomposed at a particular temperature T, and  $W_p$  and  $W_f$  are the weight fractions of nylon 6 and OMMT in the nanocomposite.

It is clear from the table that up to 400°C, the observed % fraction of nanocomposite not yet decomposed is higher than the estimated values based on weight fraction of polymer and OMMT. Above that temperature the observed values are less than the estimated values and the difference increases with increase in concentration of clay. This clearly shows the polymer - clay interactions and degradation effects of clay working against each other on the decomposition behavior of nylon 6. The values of activation energy for the decomposition process also decreased with increasing concentration of clay. However the values of decomposition temperature and activation energy for 5 wt % Zinc borate/5 wt% clay nanocomposite was higher than 10 wt% clay nanocomposite and almost close to 5 wt % clay nanocomposite showing the dominance of degradation effect of clay at higher concentrations.

In the case of nanocomposites with a lower clay loading (upto 5 wt %), which has partially exfoliated /intercalated structure, the polymer layered silicate interaction is enhanced which compensates the degradation effects in the polymer. With a 10 wt% loading the clay particles are not sufficiently intercalated and there is less interaction between the polymer and layered silicates. This causes the degradation effect to dominate and result in lower onset decomposition temperature. Nevertheless the peak decomposition temperature is almost same and the decomposition rate decreases with increasing concentration of OMMT.

#### Flammability Behavior

The flame spread behavior of nylon 6/OMMT polymer films with different concentrations of OMMT (2.5, 5 and 10wt %) is given in table III where the reported values are average of 3  $t_{ests}$ The 2.5 wt% and 5 wt % OMMT nanocomposite films burned much the same way as neat nylon films, though the dripping tendency was reduced. However the 10 wt% OMMT nanocomposite film burned without any dripping and the flame spread rate was reduced by 20-30%. This assumes significant importance in practical applications. Dripping initiates flame spread to surrounding materials and also increases risk of injuries during fire hazards. Reduced dripping or no dripping would be beneficial from safety point of view. The nanocomposite with 5wt% OMMT and 5wt% zinc borate showed very less dripping. However the flame spreading rate was higher than 10wt% OMMT nanocomposite film (Table III).

Table III: Burning behavior of nylon 6 and nanocomposites

Sample	Burning behavior	Flame spreading rate	
NT ( 1		(inch/min)	
Neat nylon	Pronounced dripping	1.803	
2.5 wt% OMMT nanocomposite	Drips	1.723	
5 wt% OMMT nanocomposite	Reduced dripping	1.380	
10 wt% OMMT nanocomposite	No dripping	1.222	
5wt% OMMT/ 5wt% Zn B nanocomposite	Very less dripping	1.625	

5wt% NCH film

10wt% NCH film

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Before burning



During burning

Figure 2: Schematic representation of horizontal burning behavior of films.

When the concentration of MMT is high enough to form a network structure and the material doesn't drip during burning, then the flame has to advance through the burnt clay. With rasification of the polymer near the flame front, the concentration of OMMT becomes high enough to form a significant mass barrier hindering the flow of volatile gases. This would significantly reduce the mass loss rate and heat release rate. This phenomenon can be schematically represented as in figure 2.

#### Morphology of Char

The microstructure of the char surface of the burnt nanocomposite films observed using scanning electron microscopy seems to explain well the difference in burning behavior of the nanocomposite films (Figure 3).

The neat nylon and the NCH 2.5 film did not form an interconnected char and dripped pronouncedly during burning. The NCH 5 and NCH 10 films formed significant char on burning. However a clear difference in char morphology could be observed between the NCH 5 and NCH 10 films. The NCH 5 film produced significant char but the char surface revealed lot of cracks and pores (figure 3a). However the NCH 10 film with 10 wt % OMMT formed a consolidated char (figure 3b) with very few cracks or pores. SEM image of the char cross section revealed densely accumulated clay platelets on both surfaces of char with density gradient from the center to the surface (figure 3c). The high aspect ratio and the platelet geometry of the OMMT led to a well interconnected network of platelets forming a protective shield. This interconnected network of platelets and viscosity effects could have restricted the dripping of the 10 wt % OMMT nanocomposite film during burning. Also this could result in significant mass barrier effect compared to NCH 5 film, which could explain the difference in burning rate of the films. The addition of 5wt% zinc borate along with 5wt% OMMT instead of 10wt% OMMT resulted in a significant change in char morphology (figure 3d). The char appeared to be foamy which might be due to water released during the decomposition of zinc borate. Most zinc borates in commercial use are hydrates (xZnO.yB2O3.nH2O) which release their water of hydration (about 13-15%) at 290-450°C. In addition to absorption of heat and dilution of fuel the release of water serves to blow the char to foam [9]. The difference in char morphology could have a significant effect on the thermal insulation and barrier properties. However cone calorimeter studies would be required to obtain more information and correlate char morphology and burning behavior.





(3b)



Figure 3: SEM micrographs of (a) NCH 5 char (b) NCH 10 char (c) NCH 10 char cross section (d) NCH ZnB char

#### **Cone Calorimeter Studies**

The cone calorimeter is being used extensively for flammability testing nowadays as the test reveals a lot of data useful for interpreting the combustion behavior of materials in real fires [2] The burning behavior of nanocomposite slabs is shown in figure 4. It is found that the burning behavior of 10 wt% OMMT nanocomposite slab differs much from the nanocomposite with 5wt% OMMT and 5wt% zinc borate. In the 10 wt% OMMT nanocomposite the specimen surface started to granulate before ignition and ignition flash occurred prior to full ignition. After ignition, specimen surface burned evenly across entire surface and a well consolidated layer of char was obtained (figure 4a and 4b). However in the case of nanocomposite with 5wt% OMMT and 5wt% zinc borate, the specimen surface started to bubble before ignition and intumesced approximately 5mm before ignition. After ignition, specimen surface burned unevenly across surface for several seconds before burning occurred over entire surface. Also the specimen intumesced significantly to about 10-13mm almost hitting the spark igniter. At the end a cellular char structure of about 10mm in height was obtained as shown in figure 4c and 4d. In order to distinguish the role of zinc borate and OMMT on char formation process one sample with nylon 6 and 5wt% zinc borate was also studied. Here the specimen surface began to bubble and intumesce in a localized manner and ignition occurred unevenly for 7-10 seconds before flames were present over (figure 4e and 4f). Some fragments of char were observed after burning instead of a consolidated layer. Thus it was believed that the release of water during the decomposition of zinc borate leads to bubbling and intumescence. However in the absence of char forming agent the intumescence could not develop into a well blown char structure and affect flame retardancy. The montmorillonite platelets forming a network and covering the surface serves a scaffold for forming a stable and cellular char structure along with the intumescent action of zinc borate which could not be obtained with zinc borate alone.

The heat release rate, mass loss rate, heat of combustion and smoke release data are presented in table IV and table V with values in parentheses corresponding to % change. The average values for each sample were recalculated after truncating the data corresponding to the tail portions of the respective heat release and mass loss curves. This is to ensure that signals and

noise in the beginning due to flashes and those corresponding to burning of samples in the edges are eliminated and the values represent the burning behavior of samples in the stable burning regime.

The peak heat release rates (HRR) of the nanocomposites were significantly lower than that of nylon 6. The addition of 10wt% OMMT reduced the peak HRR by about 67% from 948  $kW/m^2$  to 310  $kW/m^2$  whereas addition of 5wt % OMMT and 5wt% ZnB reduced the peak HRR by 69%. This is significantly higher than the reduction obtained with 5wt% OMMT (54%) and 5wt% ZnB (34%) alone.

The average HRR values which are more consistent and meaningful for comparing the burning behavior of the samples also concur well with peak HRR data and revealed almost similar percentage reduction. The average mass loss rate was also reduced by more than 60% by the addition of 10wt% OMMT or 5wt% OMMT and 5wt% ZnB which mirrored the trend observed in HRR data. On the other hand the effective heat of combustion was almost same for all samples except the 10wt% OMMT nanocomposite sample showing slightly lesser heat of combustion. The heat release rate is a function of mass loss rate and heat of combustion. With the heat of combustion remaining same it is the significant change in the rate of mass loss that has resulted in a drastic reduction in heat release rate.



(4c)

(4d)





**Figure 4**: Digital images of burning behavior and char formation (a & b) NCH 10, (c & d) NCH ZnB, (e & f) Nylon6/5wt% ZnB composite.

Table IV: Heat release and mass loss data of nylon 6 and nanocomposites

Sample	Peak Heat Release Rate (kW/m <sup>2</sup> )	Avg Heat Release Rate (kW)	Avg Mass Loss Rate (g/s)	Avg Heat of Combustion (kJ/g)	Sp. Extinction Area $(m^2/r)$
Nylon 6	948	5.04	0.18	28.04	(m/g)
Swt% OMMT	433 (54)	2.58 (49)	0.09 (50)	28.62	0.099
nanocomposite				20.02	0.177
10wt% OMMT	310.01 (67)	1.79 (65)	0.07 (61)	25 77 (8)	0.192
nanocomposite				25.11 (6)	0.182
5wt%ZnB	624.80 (34)	2.92 (42)	0.10(44)	27.82 (0.8)	0.104
nanocomposite			0.10 (14)	27.82 (0.8)	0.184
5wt% OMMT/	289.25 (69)	1.81 (64)	0.06 (66)	27.27 (2.2)	0.10.5
5wt%ZnB			0.00 (00)	21.51 (2.3)	0.196
nanocomposite					

#### Char analysis

The SEM image of the nanocomposite char led us to anticipate entrapment of polymer fragments between the densely accumulated clay platelets on both surfaces of char. Fourier transform infrared spectroscopy (FTIR) was conducted on clay, NCH 5 and NCH 10 char obtained from the flammability tests. In addition to Si peaks at 3670 cm<sup>-1</sup> and 1031 cm<sup>-1</sup>, which represents stretching vibration of Si-OH and Si-O, the NCH 10 char shows two additional distinct peaks at 1630 cm<sup>-1</sup> and 1520 cm<sup>-1</sup> which were not observed in clay and NCH 5 as shown in figure 5. The peaks at 1630 cm<sup>-1</sup> and 1520 cm<sup>-1</sup> could be assigned to amide I and amide II respectively [19] which might indicate possible unburnt polymer fragments or recession within the shielded



Figure 5: FTIR spectra of nanocomposite char

char. At a higher concentration, the OMMT platelets collapse to form a well integrated shield in the case of NCH 10 char as compared to NCH 5 char (SEM micrograph, figure 3). This might have caused entrapment of polymer fragments and retention of unburnt chain fragments within the shielded char. These spectra are obtained from burnt char of nanocomposite film of thickness of 0.5mm. With increase in film thickness the shielding effect of the char would be more pronounced and could result in significant retention of polymer. This would reduce the amount of combustible polymer available for burning and might influence the burning behavior strongly.

#### CONCLUSIONS

1

We investigated the effect of layered silicate concentration and combined effect of zinc borate and layered silicates on the thermal stability and flame resistance of Nylon6/layered silicate nanocomposites. We focused our investigation on the effectiveness of the char formation process on the flame spread behavior of thin nanocomposite films of approximately 0.5 mm thickness. The onset of decomposition (5% wt loss temperature) decreased with increasing concentration of OMMT. The addition of 5 wt% zinc borate and 5 wt% OMMT in combination minimized the degradation effect of nylon6 as compared to the addition of 10 wt% OMMT. However the peak decomposition temperature was almost same for all nanocomposites but the rate of weight loss decreased with increase in OMMT concentration. It is found that in thin films, with increase in concentration of OMMT from 5 wt% to 10 wt%, the dripping of polymer is completely suppressed and the nanocomposite film remains intact during burning. While the nanocomposites with 2.5 wt% and 5 wt% clay burned for almost the same time as neat nylon 6 with reduced dripping tendency, the 10 wt% clay nanocomposite sample burned without dripping and the flame spreading rate was reduced by 25-30%. Addition of 5 wt% zinc borate and 5 wt% clay restricted the dripping of nylon 6 but not completely and also the flame spreading rate was slightly higher than that of 10 wt% nanocomposite sample. SEM of the burnt char showed a completely different char morphology for the two samples which could be the reason behind the difference in burning behavior. This demonstrates the effectiveness of the high aspect ratio OMMT platelets in forming a significant mass barrier and affecting the burning behavior in the

case of thin films. The reinforcing effect of zinc borate on char formation of Nylon 6/OMMT system was however evident in cone calorimeter studies. The 5 wt% zinc borate and 5 wt% clay sample intumesced significantly, swelling about 10-13mm height prior to ignition and resulted in a cellular char structure after burning. This char structure was not observed either with OMMT or zinc borate alone and resulted in a significant reduction in heat release and mass loss rate of nylon 6 (about 65%) comparable to 10 wt% OMMT nanocomposite sample. It could be concluded that though decomposition of zinc borate releases water leading to bubbling and intumescence, a char forming agent is needed to develop the intumescence into a well blown char structure and affect flame retardancy. Thus the zinc borate/OMMT system could be an effective intumescent flame retardant for nylon 6 in form of slabs. Fourier transform infrared spectroscopy (FTIR) of the 10 wt% OMMT nanocomposite char showed the presence of amides which might indicate possible unburnt polymer or recession within the shielded char. This was not observed in other chars. Thus 10wt% OMMT is sufficient enough to restrict the dripping of polymer and form a protective shield like char even in thin films of about 0.5mm thickness. Further enhancement of flame retardancy could be achieved by combining the physical effect of OMMT with chemical action either through coating or intercalating layered silicates with flame retardant additives.

#### ACKNOWLEDGEMENTS

We thank Mr.Leo Barish and Mithun Shah for their help and discussions with SEM studies, Randall Harris (WPI) and Frank Kozekowich (Quaker fabrics) for helping with flammability tests. We also thank Nanocor Inc for providing the layered silicates and Honeywell Inc for providing Nylon 6. Our special thanks are due to National Textile Center for funding this study through US Department of Commerce grant 02-0740 under the project M02-MD08.

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