

Improving flame retardancy and melt processability of polyethersulfone using low molecular weight additives

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ABSTRACT: The effect of a calcium stearate (CaSt₂) additive on the melt processability and flame retardancy of polyethersulfone (PES) was studied. Measurements of the viscosity of PES and its composites showed a marked decrease in viscosity with increase in the fraction of CaSt₂ additive. About 40% reduction in viscosity of PES was achieved with addition of 5 wt % CaSt₂. By decreasing the viscosity, the CaSt₂ additive enabled the melt extrusion of PES at lower temperatures up to 90 °C below that of conventional melt processing. The flammability was also investigated using a Pyrolysis Combustion Flow Calorimeter (PCFC). The CaSt₂ additive resulted in tremendous improvement in the flame retardancy of PES as evident in the reduction of the heat release capacity (HRC) of the composites by up to 37%. Moreover, the peak of heat release rate (pHRR) of PES in the composites was up to 84% lower than in the neat polymer. The remarkable improvement in flame retardancy of PES was demonstrated to be related to the rapid charring of the composites and the *in situ* formation of calcium carbonate/calcium oxide upon decomposition of CaSt₂. The CaSt₂ additive was also found to enhance the flame retardancy of thermoplastics including polyamide-6 and polypropylene (PP). © 2016 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2016**, *133*, 43525.

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INTRODUCTION

There is increasing demand for high performance polymers for various applications. Polyethersulfone (PES), a high performance polymer, is known for its superior mechanical strength, heat resistance, and flame retardancy.¹ These have secured its applications particularly in the aerospace industry for the manufacture of airplane interior and exterior components. PES is also used in the manufacture of some automotive parts. In the aerospace industry, fire safety is of utmost importance. Therefore, there is the need for improving the flame retardancy of PES without compromising the mechanical properties.

Despite the superior mechanical and chemical properties, the difficulty in processing of PES presents a major drawback to its use. Due to its high viscosity and high glass transition temperature, the melt processing of PES requires extreme thermal conditions² (>330 °C) which approaches the degradation temperatures of the polymer. PES is therefore commonly processed via solution casting.³ However, the solution processing typically involves excessive amounts of toxic solvents such as *N*-methylpyrrolidone and *N,N*-dimethylformamide since PES is

not soluble in many benign solvents. Therefore, there is ongoing demand for alternate approaches, which do not involve toxic materials in the PES processing. Particularly, modified melt processing approaches are desirable since they are readily adaptable to many current industrial processes. Such modification may be achieved with certain additives.

Interestingly, one strategy for improving the flame retardancy and thermal properties of certain polymers is the development of composite materials containing some inorganic additives.^{2,4-7} However, most of these additives are incapable of enhancing the melt processability via viscosity reduction. In a recent study, Lecouvet *et al.*² made PES/halloysite nanocomposites via a water assisted melt extrusion process. The thermal and flame retardancy improved in the composites with about 45% increase in the peak of heat release rate (pHRR) when the halloysite fraction was 6 wt %. However, the viscosity increased by almost ten-fold due to the crosslinking between the PES chains and the halloysite nanoparticles. Thus, the flame resistance was improved whereas the melt processability worsened, given the increased viscosity.

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There is active research to identify non-hazardous additives that can tune the viscosity of polymer melts. In their study, Yordem *et al.*⁸ used low molecular weight additives to obtain toughened polypropylene (PP) composites. Specifically, the addition of calcium stearate (CaSt₂) was reported to achieve decreased viscosity, increased Young's modulus, and increased fracture energy of the PP. Thus, the stearate, which is also a cheap material, served as a multifunctional additive. However, the effects of CaSt₂ on the flame properties of PP were not reported.

In this article, we report the use of CaSt₂ as a multifunctional additive to improve the flame retardancy and melt processability of PES. This additive is also non-toxic and non-halogenated. We coextruded the PES with low fractions of CaSt₂ and studied the rheology and flammability of the PES-CaSt₂ composites. Interestingly, the presence of CaSt₂ facilitates the melt processing of PES at significantly lower temperatures and synergistically enhances its flame resistance.

EXPERIMENTAL

Materials

Polyethersulfone pellets (Ultrason E3010) were provided by the BASF Company, Ludwigshafen, Germany. PES, PA6, and PP were used as received. Calcium stearate (Lot # B22T035) with a reported 2.50% moisture content was obtained from Alfa Aesar and was used as received.

Preparation of Composites

Composites were made via extrusion of polymer and CaSt₂ using a Brabender twin screw extruder D6/2 with 7:1 L/D (length and screw diameter, respectively). The zone temperatures used are provided in the Results and Discussion section. Typically, the PES and CaSt₂ powder were premixed in a 1.0 L glass beaker before introducing to the extruder feed zone via a hopper. The samples extruded at 20 rpm screw speed were immediately quenched in a cold water bath as they exited the extruder. The water was continually replenished to maintain a cold temperature below 20 °C.

Melt Rheology

Sample extrudates were initially compression molded using a minimum load of 25 tons at 300 °C to obtain 1 mm thick plaques. The rheology of disc-shaped samples (25 mm × 1 mm) was investigated using TA Instruments AR2000 Rheometer equipped with parallel plate geometry. Stepped flow measurements of viscosity as a function of shear rate were conducted in nitrogen atmosphere at 300 °C. Angular velocity of 0.1 to 10 rad sec⁻¹ at 10 points per decade was used for the rheology. Total measurement time per sample was limited to less than 20 mins to minimize the extent of degradation. New samples were used for each rheological measurement.

Fourier Transform Infrared (FTIR) Analysis

Solid samples (<5 mg) were analyzed using Perkin Elmer Spectrum 100 FTIR Spectrometer. All spectra were collected with attenuated total reflectance (ATR) sample accessory and for a spectral range of 650 to 4000 cm⁻¹. Generally, each spectrum was collected with an average of four scans requiring about 24 sec.

Microscopy

Sample extrudates were cryofractured orthogonal to the extruding axis. The fractured surfaces were imaged using High-Resolution Scanning Electron Microscope, FEI Magellan 400 XHR-SEM. Images were recorded with electron beam of 1 kV and 13 pA.

Thermal Analysis

Samples were analyzed with TA Instruments Q50 thermal gravimetric analyzer (TGA) and a nitrogen purged Q200 refrigeration cooled differential scanning calorimeter (DSC). Typically, about 5 mg sample was used for each analysis. The TGA was run in nitrogen atmosphere; a Platinum TGA pan and a 10 °C min⁻¹ heating rate from room temperature to 800 °C were used for every sample. For the DSC, Hermetic Aluminum pans were used with a 10 °C min⁻¹ heating and cooling rate between 20 °C and 300 °C. The samples were contained in hermetic pans and lids for the DSC experiments.

Flammability

Following ASTM D7309 the flammability characteristics of the neat polymer and composites were determined using a pyrolysis combustion flow calorimeter (PCFC). The PCFC is a custom-built instrument by workers at the United States Federal Aviation Administration (FAA). A detailed description and the working principles of the PCFC are provided elsewhere by Lyon and Walters.¹ Currently, there are three of these instruments in use. One at the FAA, one at the National Institute of Standards and Technology, and one at the University of Massachusetts, Amherst. Briefly, specimen gases are combusted following controlled thermal decomposition of samples in nitrogen atmosphere. The combustion temperature was maintained at 900 °C whereas pyrolysis chamber under nitrogen flow was heated at 1 °C sec⁻¹ from room temperature to 900 °C during each sample run. The oxygen and nitrogen flow rates were maintained at 20 and 80 cc min⁻¹, respectively. The heat release rate (HRR) curves were analyzed with a LabVIEW program using Asymmetric Gaussian-Lorentzian function. The PCFC provided flammability information as heat release capacity (HRC) and total heat released (THR) for the test samples. The HRC is the ratio of the maximum specific heat release rate to the heating rate.

RESULTS AND DISCUSSION

Formation PES-CaSt₂ Composites

The CaSt₂ additive plays an important role in both the decrease in viscosity and the increase in flame retardancy of PES. Therefore, it is necessary to characterize the extruded composites. The Fourier transform infrared (FTIR) spectra of PES, CaSt₂, and PES-CaSt₂ (20% CaSt₂) composite are plotted in Figure 1. The appearance of the peaks at 1575, 1482, and 1230 cm⁻¹ are noted for the PES (Curve a, Figure 1). The 1575 cm⁻¹ is indicative of aromatic C=C stretch and the peak at 1230 cm⁻¹ is representative of the S=O asymmetric stretch of the PES.⁹ For the CaSt₂ spectrum (Curve c, Figure 1), the peaks at 2921 and 2847 cm⁻¹ are of interest as this region is free of vibrations due to PES. These select peaks are due to the C-H stretches of the stearate alkyl tail. Thus, the presence of both PES and CaSt₂ are evidenced in the spectrum of PES-CaSt₂ composite (Curve b, Figure 1).

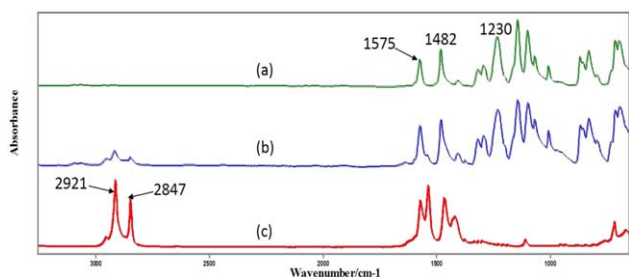


Figure 1. FT-IR spectra of (a) PES, (b) CaSt₂, and (c) PES-CaSt₂ (20% CaSt₂). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Figure 2 shows the SEM images of the PES-CaSt₂ composites containing 5%, 10%, and 20% CaSt₂. Each of the images for the composites displays a uniform dispersion of the CaSt₂ particles with sizes up to 2 μm . Yordem *et al.*⁸ observed a similar dispersed morphology of CaSt₂ in PP in which the toughness (based on Izod values) was improved by up to 24%. In the present study, the uniformly dispersed CaSt₂ would be expected to change the viscosity of the PES.

Figure 3 shows thermogravimetric (TGA) curves of PES, CaSt₂ and their extruded composites at 5%, 10%, and 20% CaSt₂ loadings. The PES decomposition occurs at 571 $^{\circ}\text{C}$ whereas CaSt₂ shows marked weight loss at 441 $^{\circ}\text{C}$ and 662 $^{\circ}\text{C}$. It is noted that in the case of CaSt₂, the approximately 3% weight loss at 82 $^{\circ}\text{C}$ is due to moisture as these samples were not thermally pretreated. However, a 20 h pretreatment of a CaSt₂ sample in vacuum oven at 50 $^{\circ}\text{C}$ did not completely remove the bound water content. The stearates and other carboxylate salts of calcium are known to show such weight loss in TGA.¹⁰ Moreover, this moisture content is consistent with the 2.5% reported in the calcium stearate data sheet by Alfa Aesar. The decomposition of CaSt₂ gives a residue of 10% (above 700 $^{\circ}\text{C}$).

The TG curves (Figure 3) for the extruded composites show the presence of CaSt₂ and PES corresponding to the two major inflections between 441 $^{\circ}\text{C}$ and 571 $^{\circ}\text{C}$, respectively. It is noted that the decomposition showing up at 662 $^{\circ}\text{C}$ for neat CaSt₂ is

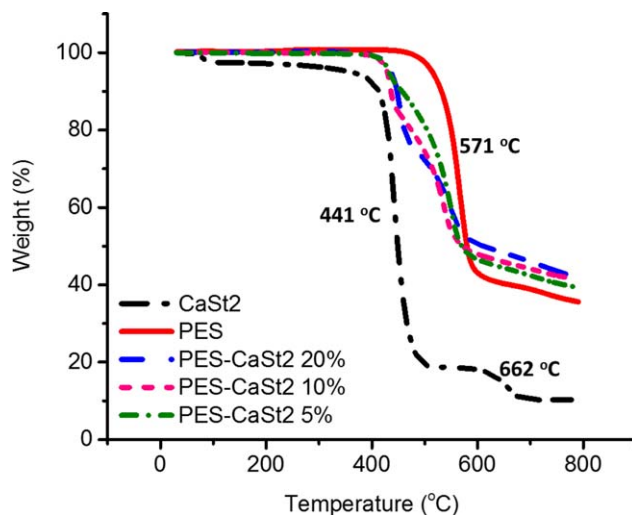


Figure 3. TGA of PES, CaSt₂ and their composites of 5% CaSt₂, 10% CaSt₂, and 20% CaSt₂. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

inconspicuous in the case of the composites. This might be due to the expected small amount of weight change (up to 1.6% in the case of 20 wt % CaSt₂) in this temperature region. It is also possible that the composites uniquely do not show any weight loss in this region.

The fraction of CaSt₂ in the composites was verified from their TGA and DSC. Figure 4 shows the percentage of CaSt₂ derived from TGA and DSC of the composites. For the TGA values the weight change in Figure 3 at 440–460 $^{\circ}\text{C}$ were used. An 80% correction was applied based on the weight change in this temperature range for the neat CaSt₂. The plot (Figure 4) shows a linear dependence on the fraction of CaSt₂ in the composites with an R-squared value of 0.9809. However, the y-intercept at 4.58 may be indicative of the lower sensitivity of the DSC values. The DSC values of percent CaSt₂ were obtained from the enthalpy of the crystalline-smectic transition at 121 $^{\circ}\text{C}$.^{11,12} It is known that excessive heat treatment can alter crystallinity of the stearates¹³ and hence the enthalpy of crystalline-smectic

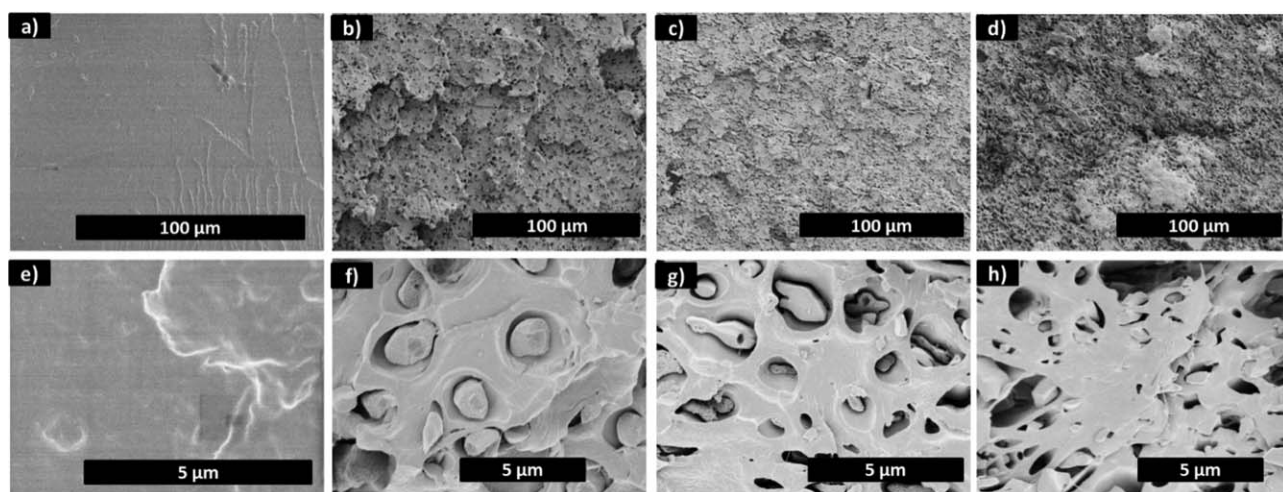


Figure 2. SEM images of the neat PES (a,e) and PES-CaSt₂ composites containing 5% (b,f), 10% (c,g), and 20% (d,h).

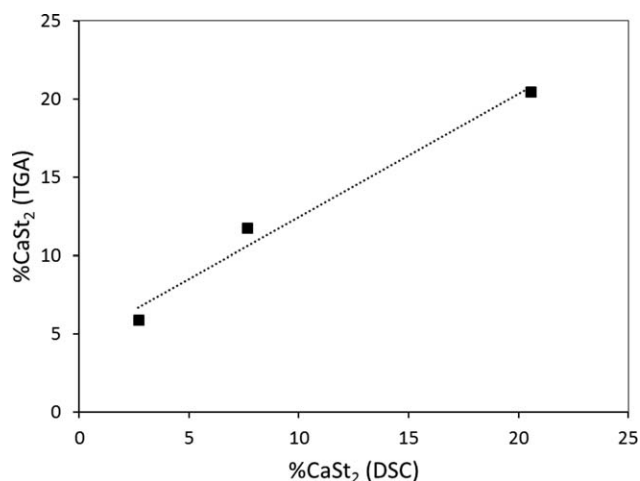


Figure 4. Percentage of CaSt₂ derived from TGA and DSC of the composites.

transition. Notwithstanding, the enthalpy value increases linearly with the fraction of CaSt₂.

Table I shows the nominal %CaSt₂ compared with the average %CaSt₂ calculated from the TGA and DSC results in Figure 4. Based on Table I, a calculated R-squared value of 1 is obtained indicating high accuracy of the amount of CaSt₂ in the composites.

Rheology of PES-CaSt₂ Composites

Table II shows the temperature for the melt processing of PES and PES-CaSt₂ composites. In the presence of CaSt₂ (at all %CaSt₂ loading), PES were extruded at significantly lower temperatures up to 90 °C reduction in temperature of the Feed zone, for example. This suggests a reduction in the viscosity of the PES by the CaSt₂ additive. Moreover, the CaSt₂, being a lubricant,¹⁴ might reduce the shear stress at the extrusion wall where shear stresses are typically high.

Figure 5 shows the viscosity as a function of shear rate for the melt processed PES and the PES-CaSt₂ composites. Interestingly, 5% CaSt₂ additive reduces the viscosity by more than 40%. Furthermore, the viscosity decreases as a function of %CaSt₂ and up to 75% reduction is realized with 20% CaSt₂. This reduction in the viscosity is the principal cause for the significantly lower temperatures at which PES was extruded in the presence of the CaSt₂. Yordem *et al.*⁸ also observed a similar decreased viscosity, from melt flow rate measurements, upon addition of the CaSt₂ to PP. However, the significant decrease in the processing

Table I. Nominal %CaSt₂ Compared with the Average %CaSt₂ from the TGA and DSC

%CaSt ₂	
Nominal	Average (TGA and DSC)
5	4.3
10	9.7
20	20.5

Table II. Temperature of the Melt Processing of PES and PES-CaSt₂ Composites

Parameter	PES	PES + CaSt ₂
Feed zone/°C	330-370	280-290
Middle zone/°C	330-370	300
Metering/°C	300	260
Screw speed/min ⁻¹	20-40	20

temperatures of PES as noted in Table II were not previously noted for PP which by itself does not require excessive processing temperatures.

Flammability of PES and PES-CaSt₂ Composites

Revisiting the TGA results in Figure 3, the residual weights at 800 °C were higher in the composites by up to 8% suggesting improvement of thermal properties of the PES composites. This is because a maximum of 2% increment would be expected for the 20% CaSt₂ composite based on the 10 wt % char residue of the neat CaSt₂ (Figure 3). Thus the observed 8% increment might be related to a synergistic effect of the PES and CaSt₂ on the thermal and/or flammability properties of the composite. Pyrolysis combustion flow calorimetry was used to analyze the flammability properties of the PES-CaSt₂ composites. The PCFC, a state-of-the-art instrument for studying the flammability of materials,¹⁵⁻¹⁸ provides valuable information including the heat release capacity (HRC) and the total heat released (THR) for the combustion of pyrolyzed products. A good correlation has been reported between the HRC and heat released in PCFC and the peak heat release rate in cone calorimetry.¹⁷

Figure 6 shows the HRC sum as a function of weight percent CaSt₂. Using HRC of 178 and 515 J g⁻¹ K⁻¹ for neat PES and CaSt₂, respectively, the estimated HRC sum for the composites were obtained for various the CaSt₂ fractions [Figure 6(a)]. First, the HRC observed (from PCFC) are up to 37% less than the estimated values. Thus confirming the enhanced flammability of the PES-CaSt₂ composites. It is noted that reduced HRC sum values were also observed for polyamide-6 and polypropylene composites with 5% CaSt₂ (see Supporting Information). Second, the 2% CaSt₂ seems to be in the region of the minimum of the HRC sum. Below 2% CaSt₂, the HRC sum

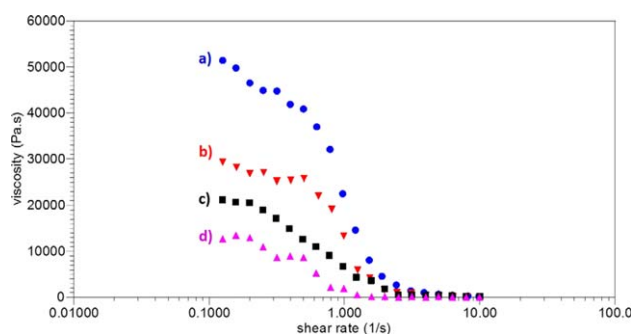


Figure 5. Viscosity as a function of shear rate for (a) PES and the PES-CaSt₂ composites containing (b) 5%, (c) 10%, and (d) 20% CaSt₂. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

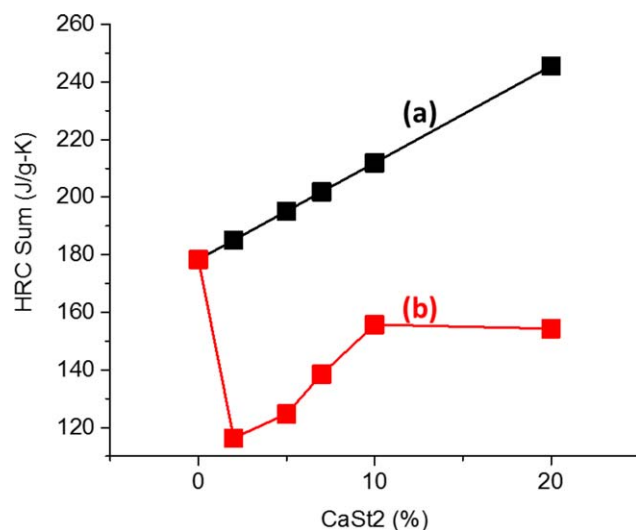


Figure 6. HRC sum as a function of weight percent CaSt₂. The estimated HRC sum (a) are based on HRC of 178 and 515 J g⁻¹ K⁻¹ for neat PES and CaSt₂, respectively. The observed HRC sum (b) are measured directly using PCFC. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

increases again as the PES fraction increases towards 100%. However, composites with less than 2% CaSt₂ were not explored as their extrusion required higher temperatures above 300 °C because of the increase in viscosity with the excessive PES fraction. Notwithstanding, the observed apparent minimum HRC sum around 2% CaSt₂ is revisited in a later paragraph.

It is noted that the peak decomposition temperature of PES decreased slightly for the composites. Table III gives the heat release capacity sum of the composites and the PES peaks, and the total heat release obtained from the PCFC. The peaks due to neat CaSt₂ and neat PES showed up at 474 and 603 °C, respectively. The PES peak temperature is suppressed by about 10 °C in the composites. However, the heat release rates at 400 °C are comparable for both neat PES and composites. Therefore, over 400 °C is required before significant heat release rates would occur in both PES and the composites. In addition, there is a general decrease of up to 24% in the THR of the composites.

Revisiting Figure 6, the observation of a minimum around 2% CaSt₂ composition and the nonlinear decrease in HRC sum with weight percent of CaSt₂ suggests that there is a combination of effects on the flammability of the composites. Figure 7

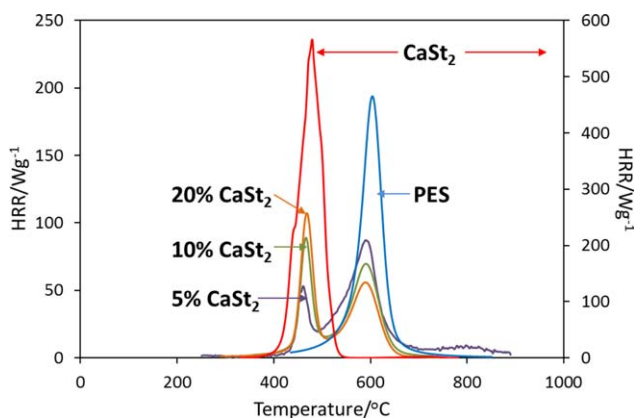


Figure 7. HRR as a function of temperature obtained from the PCFC of PES, CaSt₂, and PES-CaSt₂ composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

is a plot of the HRR as a function of temperature obtained from the PCFC of PES and PES-CaSt₂ composites. As the CaSt₂ increases from 5% to 20% the peaks due to CaSt₂ and PES increase and decrease, respectively. Conversely, 2% or lower CaSt₂ is not effective at significantly reducing the PES peak HRR. Hence, the general decrease of the HRC sum of the composites is dependent on at least two factors: (i) the extent at which the CaSt₂ HRR increases, and (ii) the extent at which the PES HRR decreases in the presence of CaSt₂ additive.

Therefore, the apparent similar HRC sum of 10% and 20% CaSt₂ in Figure 6 is not necessarily indicative of an upper limit in the extent of flammability reduction. Moreover, there is continual decrease in the HRR of PES peak for all CaSt₂ studied. From Table III, the 5%, 10%, and 20% CaSt₂ concentrations resulted in reduction of PES HRC by 53%, 65%, and 84%, respectively. Thus, the 5% CaSt₂ leads to greater HRC reduction among the three composites per weight of CaSt₂. Using a smaller amount of additive to achieve a greater flame resistance is very desirable, as other bulk properties of the matrix polymer would be retained.

Mechanism of Flame Retardation

Figure 8 shows the FTIR spectra of CaO, CaSt₂ residue after PCFC, and CaCO₃. At 1404 cm⁻¹, the CaCO₃ shows an intense peak whereas the CaO shows a short peak. However, the sharp and intense absorption at 3636 cm⁻¹ is unique to CaO and is assigned to the O–H stretching frequency, ν(OH) of the surface OH groups. The 1404 cm⁻¹ absorption in CaO reflects the presence of CaCO₃¹⁹ as CO₂ may be adsorbed on the CaO.²⁰

Table III. Heat Release Capacity Sum, Total Heat Released and Peak Decomposition Temperatures of the Composites, CaSt₂ and PES

Sample	HRC sum (J g ⁻¹ K ⁻¹)	HRC PES (J g ⁻¹ K ⁻¹)	THR (kJ g ⁻¹)	Peak temperature (°C)
PES	178.3	178.3	11.0	603
5% CaSt ₂	124.7	84	8.7	465 and 594
10% CaSt ₂	155.7	65	8.8	469 and 590
20% CaSt ₂	154.3	53	8.4	465 and 590
CaSt ₂	514.5	-	32.1	474

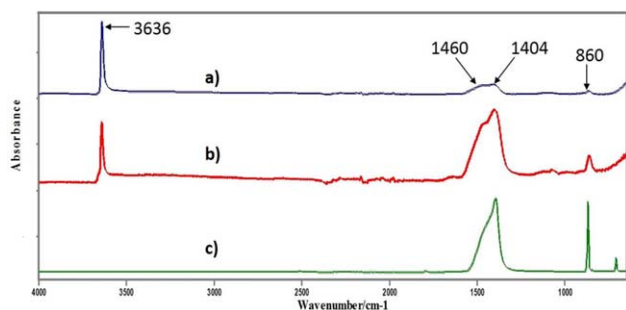


Figure 8. FTIR spectra of (a) standard CaO, (b) CaSt₂ residue after PFCF, and (c) standard CaCO₃. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Thus, the CaSt₂ residue is likely a CaO doped with some amount of CaCO₃ (Figure 8, Curve b). This shows that the decomposition of the CaSt₂ results in the inorganic CaCO₃/CaO residue. It is known that the CaCO₃/CaO mixture may be produced from the pyrolysis of organic acid salts of calcium.^{6,21} Wang *et al.*⁶ reported of a residue containing CaCO₃ and CaO mixture from TGA of calcium maleate.

Further evidence for the formation of CaCO₃ and CaO may be obtained from the TGA of CaSt₂. Revisiting Figure 3, the inflection at 662 °C is likely due to the CO₂ expulsion from the CaCO₃. The 8.28% weight loss (Figure 3) associated with this step is equivalent to the 44% molecular weight decrease upon loss of CO₂ from CaCO₃ to give CaO. Deodhar *et al.*⁵ used TGA-FTIR to confirm the release of CO₂ from CaCO₃ around 550 °C. In our case, it is possible that there is a decrease in the rate of the CO₂ expulsion from the *in situ* generated CaCO₃ until above 550 °C. Since the CO₂ expulsion is an endothermic

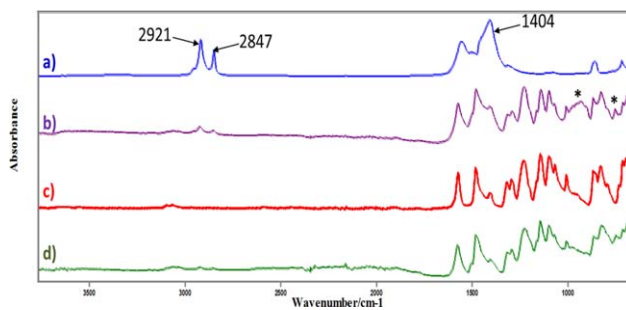


Figure 9. FTIR spectra of residue from pyrolysis under N₂ gas of (a) CaSt₂ (up to 460 °C), (b) PES-CaSt₂ (up to 460 °C), (c) PES (up to 460 °C), and (d) PES-CaSt₂ (up to 550 °C). The peaks marked with * are 940 and 750 cm⁻¹ and the 940 cm⁻¹ peak disappears in the residue pyrolyzed up to 550 °C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

process and occurs almost at the PES decomposition temperature, it is possible that this mechanism is competing with the PES degradation for the total heat supplied. Thus, the HRR for PES are decreased accordingly.

Figure 9 shows the FTIR spectra of residues from pyrolysis of CaSt₂ (up to 460 °C), PES-CaSt₂ (up to 460 °C), PES (up to 460 °C), and PES-CaSt₂ (up to 550 °C), respectively. The shoulder at 1404 cm⁻¹ in the residues indicates the presence of CaCO₃ (Figure 9, Curves a–c). The peaks due to the PES (1575, 1482, and 1230 cm⁻¹), as showed in Figure 1, are still present in the composites after heating to 460 and 550 °C (Figure 9, Curves b and d). This shows that the PES is not significantly degraded up to 550 °C at which approximately 20% weight loss may be expected given the TGA of neat PES (Figure 3). The small extent of PES degradation

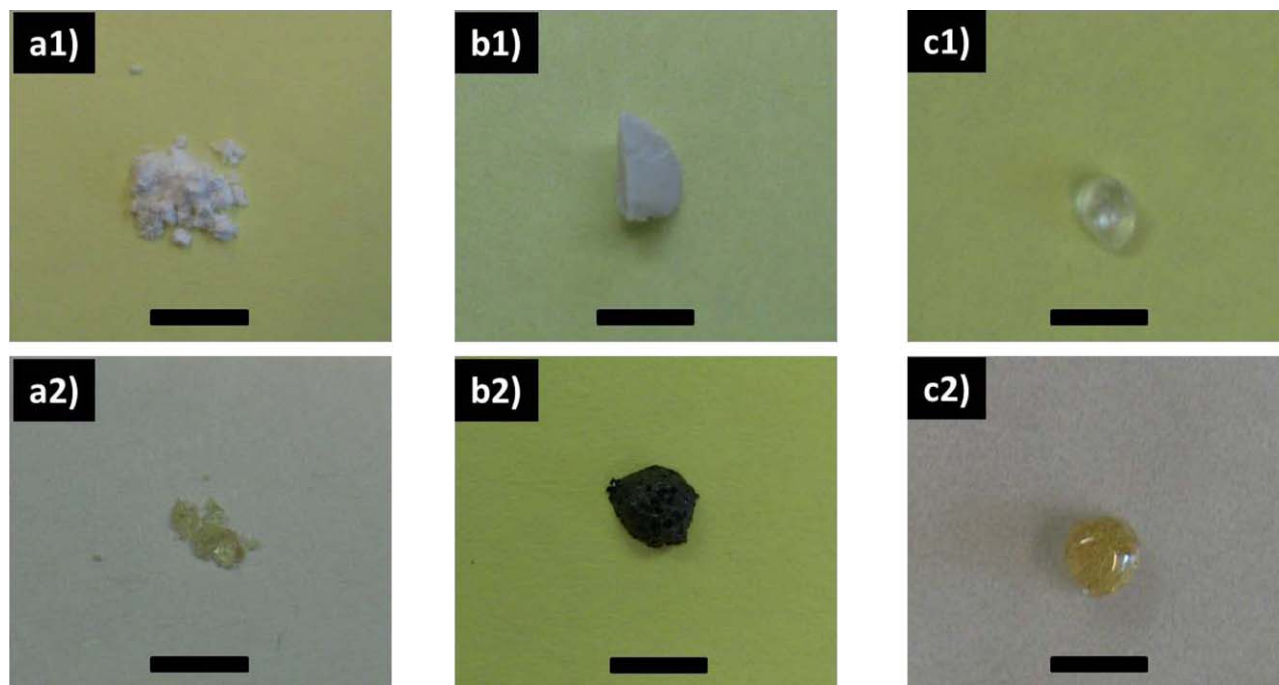


Figure 10. Pictures for before and after the pyrolysis up to 460 °C of CaSt₂ (a1,a2), PES-CaSt₂ (b1,b2), and PES (c1,c2). The black rectangular bars represent a 4 mm scale reference. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

based on the FTIR is consistent with the over 50% reduction in HRC of PES in the composites (see Table III). The peaks at 940 and 750 cm^{-1} in Curve b (Figure 9) are characteristic of alkenyl and aromatic groups, respectively. These peaks however do not appear in the residue of PES. This indicates that these alkenes and aromatic groups are only formed in the condensed phase of the composites and may be related to the flame resistance.

Figure 10 shows pictures of the CaSt_2 , PES- CaSt_2 , and PES before and after the pyrolysis up to 460 °C. At 460 °C, the composite has already formed black char whereas the CaSt_2 and PES produce solid residue with yellowish hue. This early char formation likely contributes to the flammability improvement of the PES- CaSt_2 composites. Char formation is well known mode of flame retardancy of polymers.^{22,23} The black char [Figure 10(b2)] is indicative of a PES composite containing carbonaceous residue obtained from the decomposition of the CaSt_2 . The carbonaceous residue is attributed to the partial decomposition products of the CaSt_2 as evidenced by the appearance of bending modes of CH groups (alkenyl)²⁴ and the out-of-plane modes of aromatic functionalities around 940 cm^{-1} and 750 cm^{-1} (Figure 9, Curve b), respectively. It is noted that the condensed phase reaction leading to the enhanced flame resistance is consistent with the up to 24% reduction in the THR (Table III).

A couple of factors may account for the slow decomposition rate of CaSt_2 in the composites. Particularly, entrapment or barrier to mass transport of the decomposition products caused by the viscous PES melt that might be mechanically reinforced by the *in situ* generated CaCO_3 . Such entrapment has been observed in PES-halloysite composites.² As a result, the entrapped products may undergo a series of recombination-decomposition reactions as they traverse a tortuous path through the composites. Consequently, the PES promotes the charring of CaSt_2 component. On the other hand, the CaCO_3 also contributes to the stability of the char solid structure and ultimately to the improved flame retardancy of PES in the composites via intumescence. Moreover, CaCO_3 is also a known flame retardant used with some polymers such as polyvinyl chloride and polypropylene.^{5,25}

CONCLUSIONS

PES- CaSt_2 composites were successfully prepared using melt extrusion. The PES- CaSt_2 composites had lower viscosities up to 75% less than that of the neat PES. This effect on viscosity allowed for melt extrusion of PES at approximately 90 °C below conventional melt processing temperatures. More importantly, the presence of CaSt_2 in the composites significantly decreased the HRC by up to 37%. A couple of factors were proposed responsible for the improved flame resistance; an early char formation mechanism, the mechanical strengthening of char by CaCO_3/CaO , and the release of CO_2 as a diluent of combustibles were identified to potentially improve the flame retardancy of the PES- CaSt_2 composites.

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