



## Exploring composites based on PPO blend as ablative thermal protection systems – Part I: The role of layered fillers

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### ABSTRACT

An ablative Thermal Protection System (TPS) is a sacrificial shield able to insulate aerospace vehicles during atmospheric re-entry. In this paper, PPO-based composites were investigated in view of their potential exploitation as innovative ablative TPS. Their ability to degrade undergoing endothermic reactions and yielding a char layer was evaluated by means of thermal analyses and cone calorimeter tests. This first part discusses composites containing layered particles, in particular a cationically modified clay, a hydrotalcite and a sepiolite. Among them, sepiolite-added PPO composites showed the most promising behavior. In Part II spherical oxide and non-oxide fillers in the same matrix are studied.

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### 1. Introduction

During atmospheric re-entry, aerospace vehicles can experience very high temperatures and require a Thermal Protection System (TPS) to insulate them from the hyperthermal environment. Reusable or ablative TPS were developed and used in space missions for relatively mild re-entry environments (e.g. Shuttle) or for high heating rates and particular conditions (e.g. NASA planetary entry probes), respectively [1].

An ablative TPS is a sacrificial shield able to dissipate the relevant heat flux through various endothermic processes, such as phase transitions (melting, vaporization) and/or chemical reactions.

Most ablative materials during use undergo thermal degradation when their surface reaches a temperature, usually ranging between 250 and 600 °C, as a function of the local pressure and of the heating rate. Decomposition yields gaseous by-products leaving a porous carbonaceous layer, called char, which positively affects the protective features of the ablative TPS [2,3]. In fact, from that moment on, the ablative TPS is made up of three different zones: at the surface, the char and the developed gases are present. In the middle area, pyrolysis takes place and hot gases diffuse through the char, consuming energy to continue to react and partially blocking the incoming convective heat. Finally, the underlying zone is made of un-reacted material [3,4]. The char behaves like an insulation layer, reducing heat flux and gas diffusion from the surface boundary layer to the bulk, thus preserving the un-reacted ablator [3,5].

In the last few years, research has been focused on the development of new ablative TPS materials made of polymer nanocomposites [3]. Many polymers are effective candidates for matrices thanks to their highly endothermic degradation in non-oxidative atmospheres, low density, low thermal conductivity and high specific heat coupled to suitable mechanical properties. The dispersion of fillers in an organic matrix may enhance thermal and flame resistance, chemical and moisture resistance, barrier properties, mechanical behavior and the char tendency during ablation [6–10]. In the case of nanocomposites, the improvement in performances can be obtained at very low filler content, without affecting the lightness of the polymeric matrix [11].

In this study, a thermoplastic polymer, a commercial poly(phenylene oxide)/polystyrene (PPO/PS) blend (Noryl®), was employed as a matrix. Compared with thermosets, thermoplastic polymers present good processability, easy maintenance and repair, and recycling capabilities [12]. In addition, the chosen PPO/PS blend is characterized by a promising behavior in char formation, due to the presence of aromatic ring structures [13].

A systematic study was performed in which several different inorganic fillers were added to the polymeric matrix. The influence of the morphology, the chemical nature and the content of the fillers on the thermal and combustion behavior of Noryl® resin was investigated. For the sake of clarity, the overall research is here presented in two parts: Part I discusses the results referred to composites containing platelet-like and needle-like layered particles, while Part II is mainly focused on the effect of equiaxial oxide and non-oxide fillers. This preliminary investigation is aimed to select the best performing compositions comparing the behavior of dense composite materials, all having Noryl® resin as matrix. In a

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following step, low-density components produced by expansion of softened composite systems will be developed and tested.

Considering the complexity of the ablative processes (e.g. thermal degradation, endothermic reactions, char formation, etc.) it is not possible to simulate it with a single standard test facility [14]. To preliminarily characterize these composites, several lab-scale tests were performed: thermal degradation was evaluated by thermogravimetric analyses (TGA) [14]; the enthalpy of the reactions was measured by differential thermal analyses (DTA); the effect of an external, constant heat flux (as occurs during re-entry) was estimated by means of cone calorimeter tests [15].

## 2. Experimental

### 2.1. Materials

#### 2.1.1. Polymeric matrix

The PPO/PS matrix is the grade Noryl® 914 (produced by GE Plastics), and presents a density of 1.09 g/cm<sup>3</sup> and a melting temperature of 290 °C [16].

#### 2.1.2. Inorganic fillers

Several platelet-like and needle-like commercial fillers were investigated.

- Cloisite® 15A (supplied by Southern Clay Products) is a platelet-like, overexchanged organomontmorillonite [17–19].
- Perkalite® A100 (produced by AkzoNobel Polymer Chemicals) is a platelet-like, organically modified hydrotalcite [20].
- Sepiolite (provided by Tolsa) is a hydrated magnesium silicate. Sepiolite has a similar structure to the layered structure of montmorillonite except that the layers lack continuous octahedral sheets [21]. The acicular morphology of sepiolite generally improves adhesion/compatibility with polymeric matrices and provides an excellent reinforcing effect on polymers [22].

The main features of the above-mentioned inorganic fillers are shown in Table 1, together with their designation.

As commonly reported for nanocrystalline ceramic powders [23], the as-received fillers were affected by a significant soft/hard agglomeration, as illustrated by the granulometric curves reported in Fig. 1.

Scanning Electron Microscope (SEM, Hitachi S2300) observations confirmed the presence of agglomerates (Fig. 2). In addition, concerning powder morphology, platelets were visible in C and S powders (Fig. 2a and c, respectively), even if in the latter case (Fig. 2d) the agglomerates were shown to be made up of nanometric needles at higher magnification. As declared by the supplier [20], H powder was made up of very fine platelets (not visible at the achieved magnifications) grouped in large equiaxial agglomerates (Fig. 2b).

#### 2.1.3. Preparation of the composites

Before mixing with the inorganic fillers, PPO/PS was dried in a Piovan dryer for 8 h at 120 °C. The organic and inorganic phases were then processed by using a Brabender internal mixer W48E.

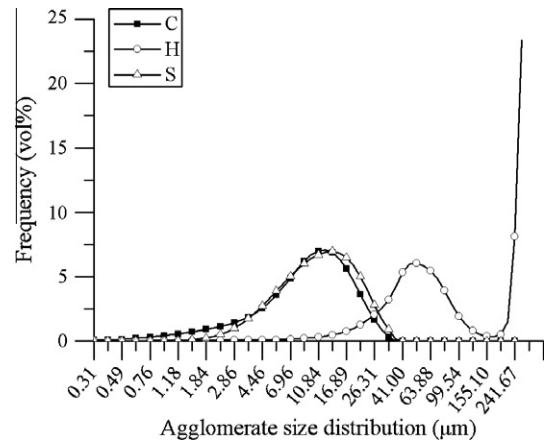


Fig. 1. Agglomerate size distributions of the as-received fillers.

The rotor speed was kept constant at 44 rpm for 90 s. The composites were prepared by mixing the polymer matrix with 2.5, 5.0 and 7.5 wt.% of each filler at 290 °C. In order to give preliminary information about the role of higher filler contents, the PPO + S composites were also prepared at 10 and 20 wt.%. As a reference, pure PPO/PS was also processed as previously described and it will be indicated as PPO.

Finally, for the sake of clarity, the composite materials are also referred to with abbreviations, based on their composition. For example, a composite containing 2.5 wt.% Cloisite will be referred as PPO + 2.5% C.

### 2.2. Methods

All the above characterisations were performed on three samples for each composition and the mean results are here reported, being the deviations negligible.

#### 2.2.1. Characterization of the composites

The evolution of the composite density as a function of the filler amount was evaluated by the Archimede method using distilled water.

The quality of the dispersion of the fillers in the matrix was investigated by SEM (Hitachi S2300).

#### 2.2.2. Ablation performances

In order to evaluate the effect of the fillers on the ablative behavior of the polymeric composites, accepted lab-scale tests were used [2,3]. Even if the characterization was carried out in operative conditions that were far from those of the potential application (in terms of temperature, heating rate, heat flux, etc.), a feasible pre-screening was carried out by comparing the composites behavior with that of the pristine blend.

Thermogravimetric analyses (Mettler-Toledo TGA analyzer) were carried out in an inert atmosphere (Argon flux of 50 ml/min), heating up to 1500 °C with a heating rate of 20 °C/min to evaluate

Table 1  
Summary of the fillers and their designation.

Filler	Designation	Chemical nature	Morphology	Density (g/cm <sup>3</sup> )
Cloisite® 15A	C	Cationic clay	Platelets	1.66
Perkalite® A100	H	Hydrotalcite (anionic clay)	Platelets	1.39
Sepiolite	S	Hydrated magnesium silicate	Needles	2.15

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