Design and Thermal and Spectroscopic Characterization of novel architecture's POSS nanoparticles

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Background

Our research group at the University of Catania, in collaboration with eminent researchers in the field, tested the stabilization of different polymers, synthetics or naturals, such as PES, EPDM, PEO and chitosan by the incorporation of POSSs [1-4]. Represented in their most common form by the symbol T_8, with a diameter usually falling in 1.5–3 nm, POSSs comprise a Si/O cage completed by organic groups covalently bonded with silicon atoms [5].

Methods and Materials

$$\ln\left(\frac{\Phi}{T_m^2}\right) = \ln\left(\frac{nRAW_m^{n-1}}{E_a}\right) - \frac{E_a}{RT_m}$$

Kissinger equation

$$\ln = ln\left(\frac{AE_\alpha}{g(\alpha)R}\right) - 5.3305 - 1.052\frac{E_\alpha}{RT_\alpha}$$

FWO equation

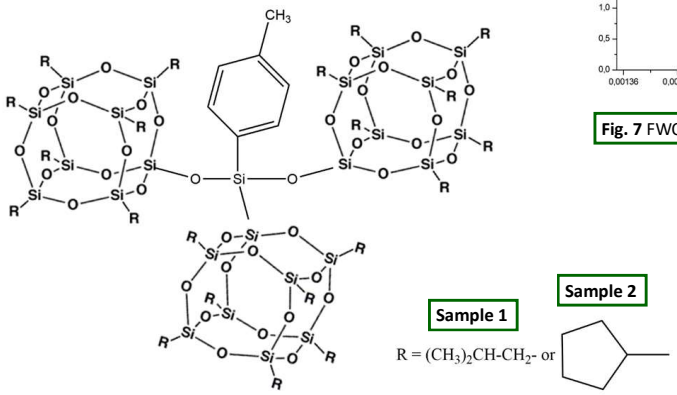

Fig. 1 Molecular structure of the multiple Polyhedral Oligomeric Silsesquioxanes cages

Fig. 2 DTA-TG Shimadzu DTG-60

Fig. 3 FTIR Perkin Elmer Spectrum 100

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Aim

PS composites were synthesized by in situ polymerization of styrene in the presence of 5% of multiple cages POSS. After a spectroscopic investigation their thermal behavior in term of resistance to the thermal degradation and rate of degradation were evaluated by the means of TGA and kinetics literature methods. The temperature at 5% mass loss ($T_{5\%}$) and apparent activation energy (E_a) of degradation, were compared with each other and with those of control PS to evaluate the differences in thermal behavior.

Experimental

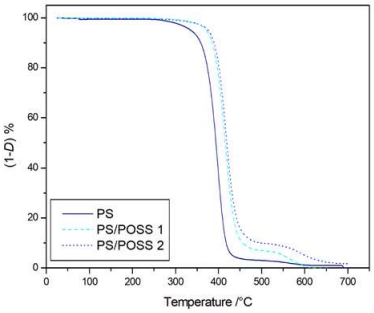

Fig. 5 TG curves, at 10 K·min^{-1}, in flowing nitrogen of samples 1 and 2 and control PS.

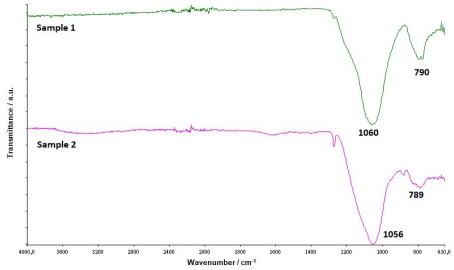

Fig. 6 FTIR of the residues at 700 °C for the samples 1 and 2

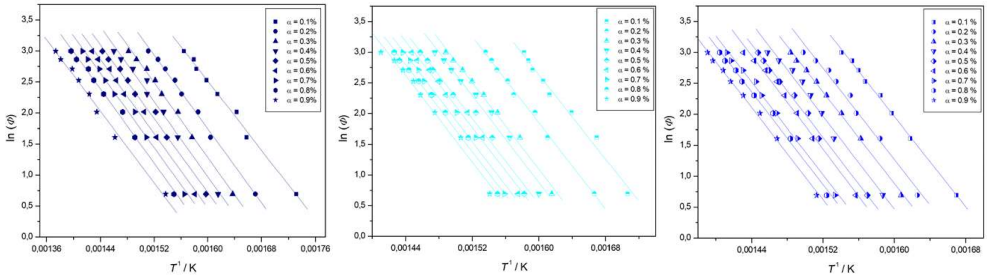

Fig. 7 FWO straight lines for PS, sample **1**, sample **2** (left to right) in flowing nitrogen at various degrees of conversion (α).

$T_{5\%}$ and E_a of degradation for PS and synthesized POSS/PS nanocomposites obtained by the Kissinger and Flynn-Wall-Ozawa (FWO) equation, in static air atmosphere and in flowing nitrogen.

Samples	Air			Nitrogen		
		Kissinger	FWO		Kissinger	FWO
	$T_{5\%}$ / K	E_a / (kJ·mol^{-1})	E_a /(kJ·mol^{-1})	$T_{5\%}$ /K	E_a / (kJ·mol^{-1})	E_a / (kJ·mol^{-1})
PS	582	126 (± 7)	123 (± 4)	614	182 (± 8)	183 (± 4)
1	608	142 (± 7)	146 (± 7)	645	193 (± 6)	197 (± 4)
2	614	150 (± 12)	154 (± 5)	649	197 (±8)	196 (± 6)

Results

The composites showed an increase of the thermal stabilization with respect PS, but that was not comparable with that obtained in the past for similar systems with one or at least two POSS cages. This result was attributed to the increase in steric hindrance of the multiple cages POSS that lead to a difficult dispersion at molecular level, reducing the interaction with the matrix and its reinforcement action.

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