The effect of photo-oxidation on thermal and fire retardancy of polypropylene nanocomposites

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Introduction

✓ Nanocomposite technology has been the topic of severa scientific papers in the last years.

✓ The typical methods used to produce polymer nanocomposites are:

- intercalation of a suitable monomer followed by polymerisation,
- polymer intercalation from solution and direct polymer intercalation.

Materials

- ✓ The reference specimen was a commercial polypropylene containing 0.6% of maleic anhydride (designated as PP gMA) produced by Aldrich chemical company.

- ✓ Three montmorillonite (MMT) in which, different cations were used to change the hydrophilic nature of layered silicates to make them miscible with the hydrophobic character of PP

  - Closite 30B (MT2EtOH)
  - Closite 20A(2M2HT)
  - I28E (OD3MA)

Preparation of the nancomposites

- ✓ The melt compounding of PP with the modified clay was performed using an intermeshing twin-screw extruder Haake Rheomex TW100.
- ✓ These experimental conditions allowed the production of PPCNs with clay mass fraction of 5%.
- ✓ For the X-ray and TEM investigations, materials were pressed in a form of thin films (150– 300 lm).
- ✓ Samples for the cone calorimeter investigation (100 mm · 100 mm, 6 mm thick)

Characterization

The commun techniques that are used to evaluate the state of mixing of nanocomposites are X-ray diffraction (XRD) and transmission electron microscopy (TEM).

- XRD provides information on the registry between the clay layers as the polymer inserts.
- The second technique allows a direct observation of the state of org-MMT dispersion.

Photo-oxydation of the nanacomposites

- **UV irradiation**

For photo-oxidation operations, the nanocomposites were exposed to UV light:

  – under accelerated conditions using a SEPAP 12–24 unit that operates at 60 ° C with wavelength longer than 300 nm,

  – under natural conditions at Dakar (Senegal) located at 17° E longitude and 15° N latitude.

- **Thermal analysis**

Thermogravimetric analysis (TGA) was carried out using a Mettler/Toledo TGA/SDTA 851

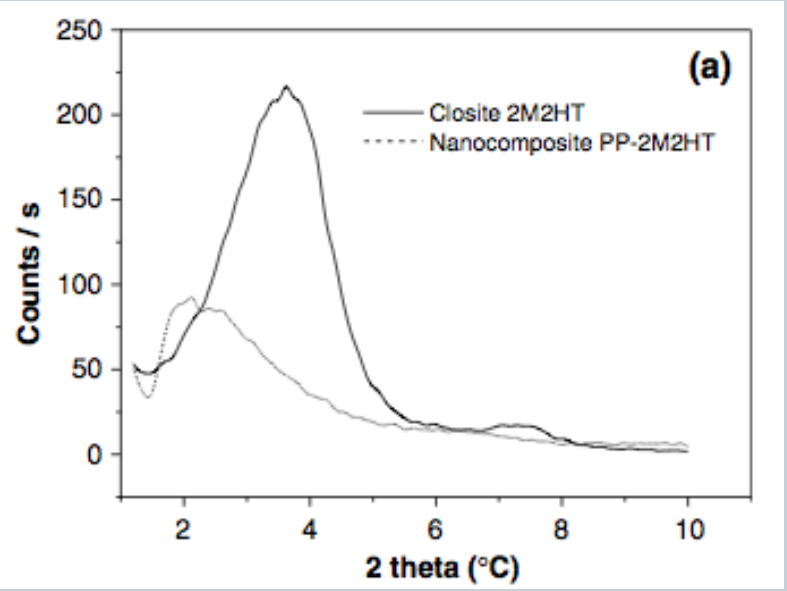
Fire retardancy of the nanocomposites

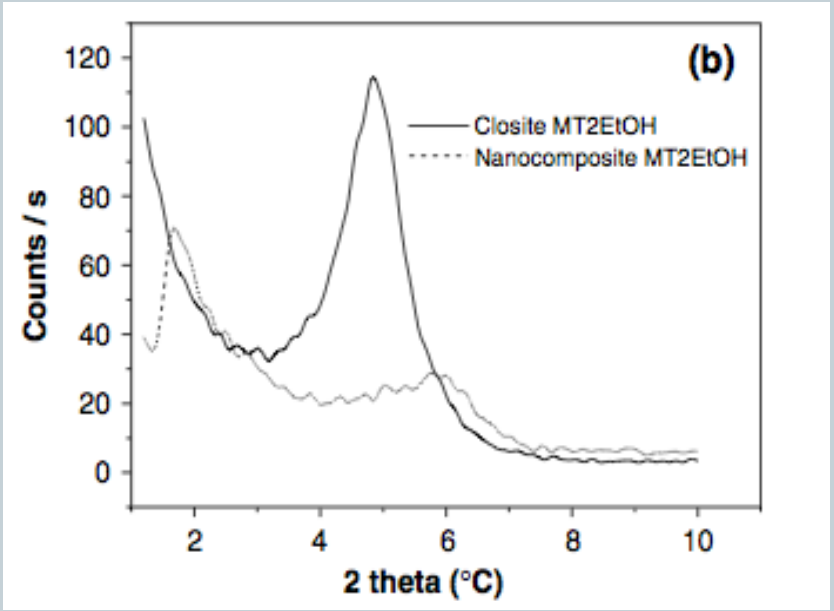
The cone calorimetry was used to study fire retardancy properties. It has the ability to measure:

- the time to ignition (tign),
- the heat release rate (HRR) and and the time to (tPHRR)
- the mass loss rate (MLR),
- the evolution of smoke
- the rate of production of various gas species within 10%.

Results and discussion

XRD





TEM

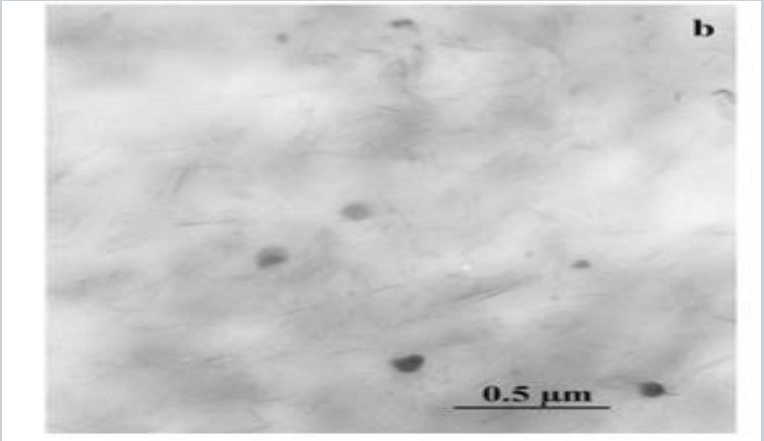
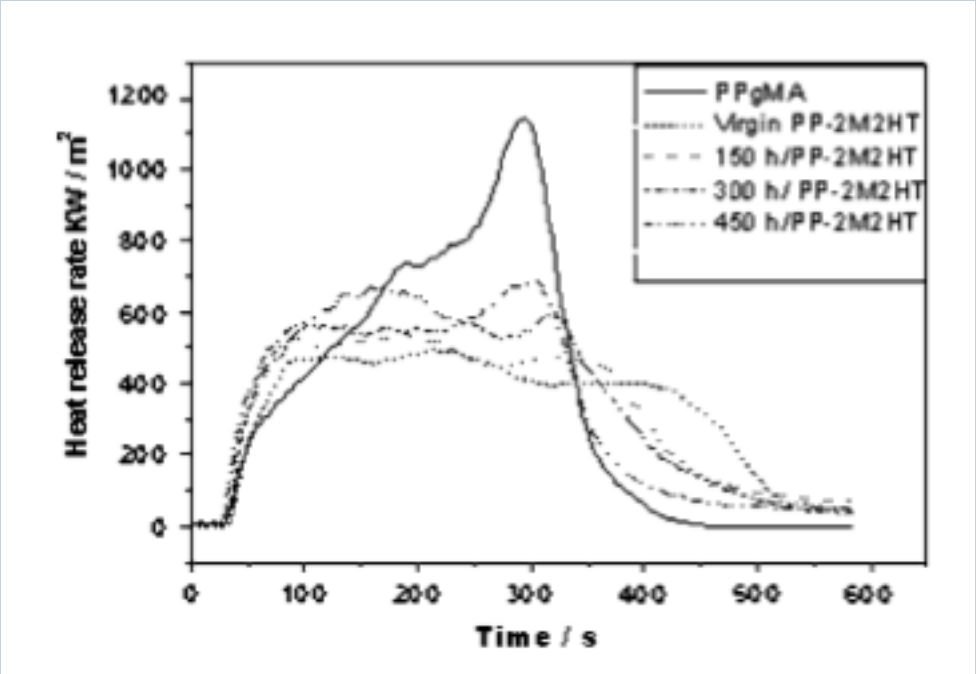


Table 1: XRD and TEM results of the nanocomposites produced

Experimental	PP-2M2HT	PP-MT2ETOH	PP-OD3MA
XRD	Diffuse peak	Decrease *d*-spacing	Diffuse peak
TEM	Homogenous exfoliation	Tactoid	Tactoid exfoliation

Cone calorimetry (1)

Sample	TI (s)	PHRR (kW m^{-2}) (% reduction)	TPHRR (s)	Mean Hc (MJ/kg)	SEA	Visual observations
PP-g-MA	35	1010	294	34.66	580	Dripping
PP-OD3MA	27	609 (40)	258	38.14	667	Swelling/bubling
PP-2M2HT	29	491 (52)	211	36.52	681	Swelling/bubling
PP-MT2EtOH	26	776 (23)	295	38.92	665	Dripping



Cone calorimetry (2)

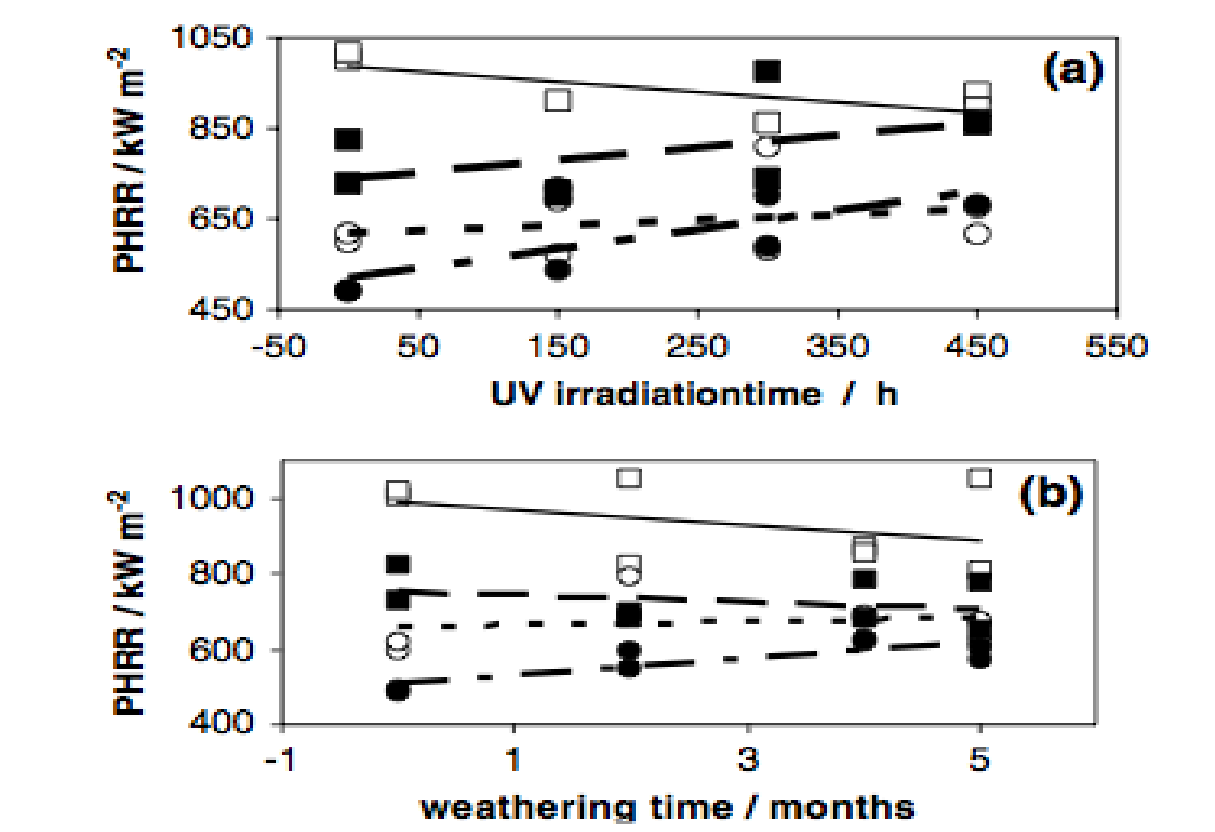


Fig. 1 PHRR changes of PP-g-MA and its nanocomposites degraded under accelerated (a) and natural conditions (b). Correspondance of symbols: PP-g-MA (Δ), PP-MT2EtOH (■), PP-OD3MA (O), PP- 2M2HT (◆)

Thermal degradation

Samples	Time	$T_{10\%}$	$T_{50\%}$	DTG	Residue
PP-g-MA	0 h (virgin)	310	376	411	0.81
	2 months	322	389	418	0.82
	4 months	314	380	392	0.74
	300 h	311	387	415	0.844
	450 h	304	378	387	0.81
PP-OD3MA	0 h (virgin)	330 (20)	401 (24)	419 (8)	1.02
	2 months	328	401	419	1.11
	4 months	318	395	417	0.97
	300 h	312	390	409	1.10
	450 h	307	387	405	1.08
PP-2M2HT	0 h (virgin)	337 (27)	408 (32)	427 (17)	0.97
	2 months	324	401	419	1.0
	4 months	329	407	425	1.03
	300 h	328	395	412	1.00
	450 h	317	393	414	0.95
PP-MT2EtOH	0 h (virgin)	313 (4)	387 (11)	401 (–10)	1.15
	2 months	319	389	409	1.02
	4 months	322	394	412	0.93
	300 h	308	390	411	1.15
	450 h	301	388	406	1.26

Table 3: TGA data in air atmosphere for PP-g-MA and its nanocomposites

Conclusion

- ✓ The true PP/clay nanocomposites exhibited improved thermal stability and fire retardancy properties
- ✓ The most interesting finding of this study was the dramatic change of these properties observed in the UV degraded nanocomposites.
- ✓ On the other hand an outstanding improvement of fire properties was achieved on UV irradiated PP-g-MA. This was due to crosslinking reactions that generate a compact structure, which is less easy volatilizable.

Thank you for attention