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Cyclodextrin nanosponges as flame retardant for polymers

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Abstract

Among various halogen-free flame retardants (FRs), intumescent flame retardants (IFRs), which develop a carbonaceous shield on the burning surface of the polymer, thus limit the heat, fuel and oxygen transfer between polymer and flame are noticeable because they provide flame retardancy properties to polymers with contemporary low toxicity, however for IFRs be efficient a great load is still required.

Some efforts have been carried out using cyclodextrins which show a twofold advantage due both to their nanosized and polysaccharide nature. Cyclodextrin nanosponges (NS) in particular have been successfully used in combination with phosphorus molecules to enhance the thermal stability of an ethylene-vinyl-acetate (EVA) copolymer, PP, LLDPE and Polyamides [1, 2]. The resulting NS network contains internal and external cavities, where phosphorus species can be easily entrapped: the great advantage of such green flame retardants is that the NS embeds the phosphorus molecules allowing generation of phosphoric acid *in situ* during heating with consequent NS dehydration, water vapour evolution and char formation. NS can be designed with different cavities dimensions using tailored crosslinking agents with the aim of opportunely tune the flame retardant activity of the polymer/NS compounds.

Dextrin based NS (β -cyclodextrin, BCD and line caps, LC) were crosslinked with an epoxy derivative (N,N-diglycidyl-4-glycidyl-oxyaniline, DGGOA) or with pyromellitic anhydride (Pyro) in different ratios. Triethylphosphate (TEP) has been introduced either during the NS synthesis (*in synthesis route*) or on pulverized NS (*after synthesis route*). NS and NS/TEP systems have been fully characterized by FTIR, Thermal analysis, Pyrolysis-GC/MS and tested for fire performance through an on-purpose-modified Limiting Oxygen Index evaluation. Successful BCD-NS have been obtained with both hardeners, whereas LC only react with Pyro. Thermogravimetric curves shows that NS have a thermal stability lower than NS/TEP which also form a larger amount of charred residue at 700°C. Pyrolysis-GC/MS indicates that NS/TEP systems obtained via *in synthesis* route retains a larger amount of embedded TEP than those obtained via *post synthesis* route (Fig 1).

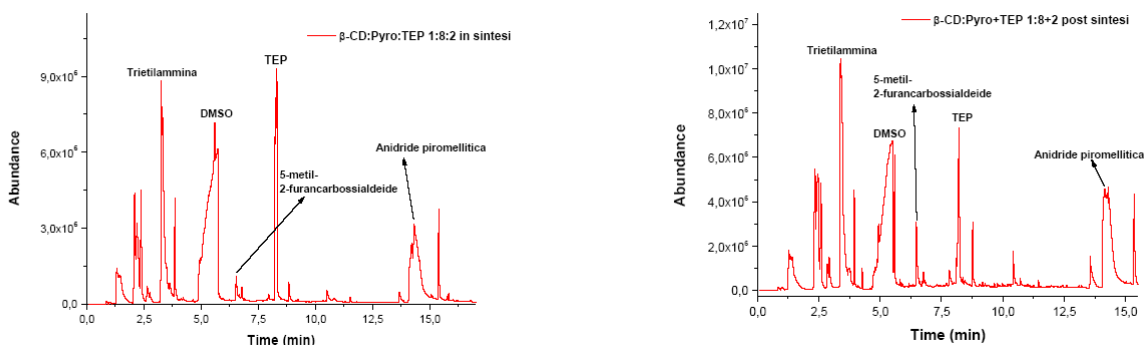


Fig 1 Pyrolysis GC/MS of BCD-Pyro TEP system obtained by in synthesis (left) and post synthesis route (right)

However in the case of DGGOA hardened NS systems, the *in synthesis* route leads to NS having a different structure than those from the *post synthesis* route, possibly due to autocrosslinking of the DGGOA itself in the synthesis conditions. On the contrary the same structure was highlighted for BCD-based NS or LC-based NS crosslinked with Pyro, no matter the synthetic route adopted. Combustion behaviour seems to be excellent however residual solvents strongly impair fire retardance performances.

References

- [1] Alongi, J. Poskovic, M. Visakh, P.M., Frache, A., Trotta F (2010). Novel flame retardants containing cyclodextrin nanosponges and phosphorus compounds to enhance EVA combustion properties. *Polymer Degradation and Stability* 95 2093-2100
- [2] Alongi, J. Poskovic, M. Visakh, P.M., Frache, A., Malucelli G. (2012). Cyclodextrin nanosponges as novel green flame retardants for PP, LLDPE and PA6. *Carbohydrate Polymers*, 88, 1387-1394.

