Abstract for oral presentations

Flame retardant effectiveness of nanodispersed organophosphorus-derivative in polyamide 6 textile filament yarns

Jelena Vasiljević[1*], Marija Čolović[2], Matic Šobak[2], Ivan Jerman[2], Barbara Simončič[1], Andrej Demšar[1], Nataša Čelan Korošin[3], Giulio Malucelli[4]

[1]University of Ljubljana, Faculty of Natural Sciences and Engineering, Aškerčeva 12, 1000 Ljubljana, Slovenia
[2]National Institute of Chemistry, Hajdrihova 19, 1000 Ljubljana, Slovenia
[3]University of Ljubljana, Faculty of Chemistry and Chemical Technology, Večna pot 113, 1000 Ljubljana, Slovenia
[4]Politecnico di Torino, Department of Applied Science and Technology, Viale Teresa Michel 5, 15121 Alessandria, Italy
Author for correspondence (e-mail: jelena.vasiljevic@ntf.uni-lj.si)

Keywords: polyamide 6 nanocomposite filament yarns, organophosphorus-derivative, *in situ* polymerization, flame retardance.

The halogen-free flame retardant (FR) polyamide 6 (PA6) composite textile filament yarns fabricated via melt-compounding have never achieved effective flame retardancy [1-6]. The main reason for that is related to the agglomeration of FR additives due to their poor compatibility with PA6 polymer chains. The formed FR agglomerates substantially reduce the flame retardant effectiveness, disable continuous melt-spinning process due to clogging of the filters and spinnerets, and significantly impair fibre tensile properties [2,4,5]. Therefore, the goal of this study was to investigate how lowering of the FR additive size to the nanoscopic level affects melt-spinning process and flame retardancy of the nanocomposite PA6 filament yarns.

To this aim, we established a new scalable approach for the production of new-generation PA6/FR nanocomposite filament yarns with uniformly distributed nano-dispersed halogen-free FR. So, instead of mixing FR additive with PA6 melt, we rather chose a step back, i.e. mixing of FR additive with ε-caprolactam melt and performing the *in situ* polymerization. The successful realization of this goal was closely dependent on the careful selection of the FR additive and realization of the next objectives:

(1) to achieve maximal matching between the pyrolysis characteristics of PA6 and FR,
(2) to achieve good miscibility between FR and ε-caprolactam melt,
(3) to achieve minimal influence on polymerization process,
(4) to achieve the production of nanocomposite stable enough to retain the nanocomposite structure after the melt-spinning process.

In accordance with the set objectives, three different organophosphorus-derivatives were selected. The organo-phosphorus FRs were selected as they are currently accepted as the more sustainable FRs, which will have the highest growth rate in the global flame retardant market up to 2025. The selected FRs were incorporated in the PA6 matric *in situ* during the water-catalysed ring-opening polymerization of the ε-caprolactam monomer. Afterwards, the melt-spinning was conducted on the laboratory-scale melt-spinning and drawing device The extruded textile filaments were wound using a winder operating at a 120 m/min speed with no filament breakage or clogging the filters and spinnerets.

Figure 1. FR PA6 nanocomposite filament yarns and knitted fabric (left) and SEM images of the knitted fabric (right).

The achieved outcomes of this work are:

(1) Undisturbed conduction of the *in situ* polymerization of PA6 with simultaneous uniform incorporation of the FR additive was achieved only for the specific FR structure, which minimally affected crystallization and melting temperatures.

(2) Continuous melt-spinning of the nanocomposite textile multi filament yarns from the selected nanocomposites was successfully conducted without clogging the filters and spinnerets. The tensile properties of the filaments were of efficient quality to be wounded and knitted into fabrics.

(3) Successful preservation of the nanocomposite structure after the melt-spinning process, which was confirmed by the EDX elemental mapping of the uniformly distributed phosphorus on the tested filament surface and cross-section;

(4) Increased thermo-oxidative stability of the nanocomposite filaments with approximately 43 % higher residue compared to that of PA6 at 500 °C;

(5) self-extinguishment of the nanocomposite fibre strand and knitted fabric samples at FR concentration of 15 wt. % within 1 s in standard vertical flame spread tests (ASTM D6413), followed by the complete reduction of the melt-drop flammability, whilst the melt-dripping was significantly reduced.

The obtained results confirm that this novel approach represents a powerful tool for the successful production of the PA6 filament yarns with uniformly distributed nanodispersed FR. Maximised FR effectiveness enabled the achievement of the balance between the effective FR concentration and good PA6 fibre spinnability and good tensile properties. Moreover, the achieved V0 rating in the UL94 vertical burning testing of the 1.2 mm-tick bar samples at FR concentration of 10 wt. % confirmed the highly effective flame retardant action of the incorporated nanodispersed FR additive.

[1] S. Bourbigot et al. 2002, *Polym Degrad Stab* **75**(2), 397–402.

[2] R. Horrocks et al. 2016, *Polymers* **8**(8), 288.

[3] M. Coquelle et al. 2014, *Polym Degrad Stab*, **106**, 150–157.

[4] H. Xiang et al. 2017, *Pro Nat Sci-Mater*, **27**(3), 369–373.

[5] A. Šehić et al. 2018, *Fiber Polym* **19**(6), 1194–1206.

[6] A. Šehić et al. 2018, *J Appl Polym Sci* **136**(5), 47007.