UV-LED Curable Acrylic Films Containing Phosphate Glass Powder: Effect of The Filler Loading on the Thermal, Optical, Mechanical and Flame Retardant Properties

UV-LED CURABLE ACRYLIC FILMS CONTAINING PHOSPHATE GLASS POWDER: EFFECT OF THE FILLER LOADING ON THE THERMAL, OPTICAL, MECHANICAL AND FLAME RETARDANT PROPERTIES

D. Pugliese[1], G. Malucelli[2]
[1] Politecnico di Torino, Dipartimento di Scienza Applicata e Tecnologia
c.so Duca degli Abruzzi 24, 10129 Torino, Italy
[2] Politecnico di Torino, Dipartimento di Scienza Applicata e Tecnologia
c.so Duca degli Abruzzi 24, 10129 Torino, Italy
e-mail: giulio.malucelli@polito.it

1. Introduction

During the last 10 to 15 years, photoinduced polymerization (i.e., UV-curing) has become a reliable and efficient technique suitable for industrial scale applications, thanks to its high curing kinetics, low toxicity (because of the absence of VOC – Volatile Organic Compounds), energy saving (energy is necessary just to activate the reaction process) and limited environmental impact. Despite of all these advantages, the standard UV-curing practice relies on the use of high-pressure Hg lamps as effective radiation sources. Conversely, the very high temperatures achieved during the irradiation process limit the appropriateness of the high-pressure Hg lamps for temperature-sensitive substrates, such as paper, wood, textiles and plastic films [1].

Quite recently, it has been demonstrated that it is possible to limit the heat release, while decreasing the energy consumption and keeping high curing efficacies by utilizing light emitting diodes (LEDs) as radiation sources, hence taking advantage of the so-called UV-LED curing processes [2].

Composite films based on polymer matrices containing inorganic fillers (at micro- or nano-scale) have been intensively studied in the last decades, as the incorporation of an inorganic phase homogeneously dispersed within the polymer allows obtaining a final system with improved performances (i.e., enhanced thermal and/or thermo-oxidative stability, mechanical and tribological behavior, barrier properties toward different gases, electrical features…).

In this context, micropowders of phosphate glasses may represent a good alternative to the aforementioned fillers, for the design of polymer composites with enhanced properties. Certainly, in recent years, phosphate glasses have demonstrated to be a sound alternative to silicate glasses as a host material for a wide range of photonic applications. In particular, their higher rare-earth ions solubility with respect to silicate glasses has allowed the development of more compact photonic devices, such as lasers and amplifiers [3]. Besides, the phosphate glasses are very well known for their homogeneity, good chemical durability and thermal stability and excellent optical properties.

To the best of our knowledge, the application of UV-LED curing processes to acrylic resins containing phosphate glass powders has never been investigated and reported in the scientific literature so far. Therefore, in the present work, we demonstrate the feasibility of the UV-LED curing process for obtaining free standing epoxy-acrylate films containing different amounts (namely, 10, 20, 30, 40 and 50 wt.%) of phosphate glass powder and showing enhanced thermal and flame retardant features. To this aim, a commercially available epoxy-acrylate resin, namely bisphenol-A-ethoxylate-diacrylate, was selected as a model system. Then, the effect of the filler on the overall thermal, optical, mechanical, and flame retardant behavior was thoroughly studied and correlated with the phosphate glass powder loading, establishing some noteworthy structure-property relationships.

2. Results and discussion

Figure 1 shows the typical FTIR-ATR spectrum of the UV-LED cured film containing the highest phosphate glass powder loading (i.e., 50 wt.%). After the UV-LED curing process, the C=C double bond peak totally disappears, hence confirming the completeness of the curing reaction, even in the presence of high filler amounts.

Figure 2 shows the typical morphology of the prepared UV-LED cured films, as assessed by SEM analysis: irrespective of the filler loading, the distribution of the filler is quite homogeneous within the polymer matrix; irregular particles (size: about few microns, though some larger aggregates are present) are well dispersed within the polymer phase.

Table 1 lists the T_g values of the UV-LED cured films, as measured by DSC analyses (2nd heating scan). It is worthy to note that the presence of increasing filler loadings slightly affects the glass transition temperature of the polymer network, which rises from 58.5 °C (unfilled cured system) to 65.1 °C (for the system containing 50 wt.% of phosphate glass powder). The observed T_g increase indicates a moderate effect exerted by the filler particles on the mobility of the polymer segments in the formed 3-dimensional (3D) network.

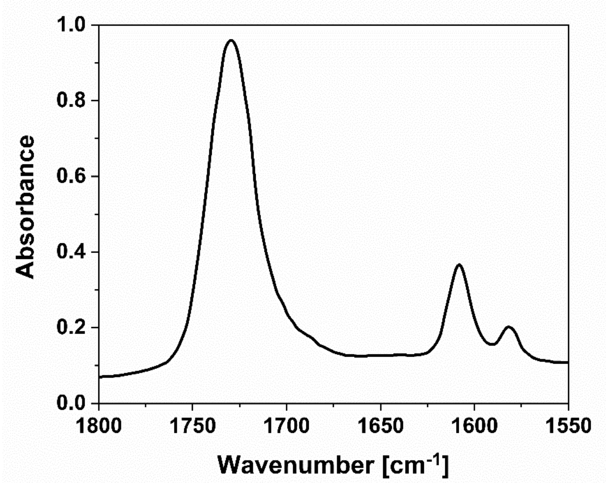

Figure 1: FTIR-ATR spectrum of a UV-LED cured film containing 50 wt.% of phosphate glass powder.

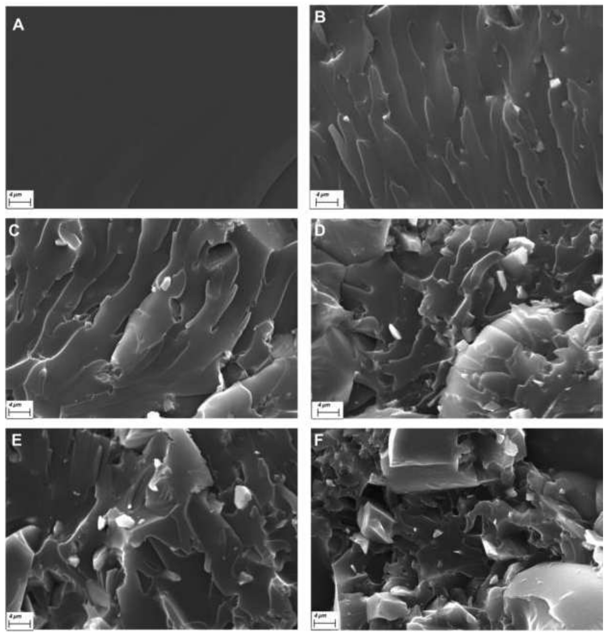

Figure 2: Typical SEM micrographs (at 5000 X) of UV-LED cured films: Eb150 (A), Eb150+10%G (B), Eb150+20%G (C), Eb150+30%G (D), Eb150+40%G (E), Eb150+50%G (F).

As expectable, the transparency of the samples decreases progressively as the amount of glass dispersed in the epoxy-acrylate resin increases, due to scattering phenomena exerted by the dispersed inorganic phase. This result is in agreement with the transmittance (*T%*) spectra shown in Figure 3 and the photos of the films shown in Figure 4.

Additionally, the incorporation of increasing amounts of phosphate glass powders enhances the thermal and thermo-oxidative stability of the polymer network, as revealed by the increase of both the degradation onsets observed in TG analyses and of the final residues. Finally, the inorganic filler is also responsible for an overall improvement of the flame retardance of the UV-LED cured films, as assessed by forced combustion tests [4].

Table 1. Glass transition temperatures of the UV-LED cured films.

Sample	T_g [°C]
Eb150	58.5
Eb150+10%G	59.3
Eb150+20%G	59.5
Eb150+30%G	61.0
Eb150+40%G	62.1
Eb150+50%G	65.1

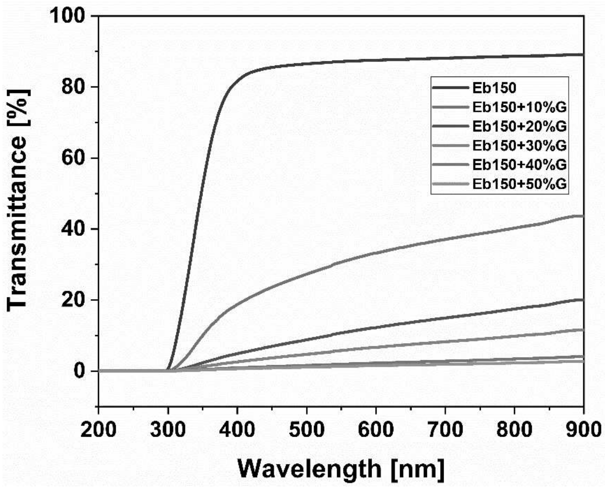

Figure 3: Transmittance spectra of UV-LED cured films loaded with different amounts of phosphate glass particles.

Figure 4: Pictures of UV-LED cured films prepared with different loadings of phosphate glass particles.

Conclusions

In the present work, the effects of the incorporation of different amounts of phosphate glass powder (ranging from 10 to 50 wt.%) on the morphology, as well as on the thermal, optical, mechanical and flame retardant properties of a UV-LED curable acrylic resin have been thoroughly investigated, establishing interesting structure-property relationships.

References

[1] C. Dietlin, S. Schweizer, P. Xiao, J. Zhang, F. Morlet-Savary, B. Graff, J.-P. Fouassier, J. Lalevée, *Polym. Chem.*, 6, 3895–3912 (2015).

[2] Y. Yagci, S. Jockusch, N.J. Turro, *Macromolecules*, 43, 6245–6260 (2010).

[3] D. Pugliese, N.G. Boetti, J. Lousteau, E. Ceci-Ginistrelli, E. Bertone, F. Geobaldo, D. Milanese, *J. Alloys Compd.*, 657, 678–683 (2016).

[4] D. Pugliese, G. Malucelli, *Polymers*, 14, 1899 (2022).