Mechanical Properties of Thermal and UV Polymerized Graphene-Reinforced Acrylic Resin: Effect of Post-treatment on Samples Obtained by Stereolithography

Sara Lopez de Armentia[1*], Yolanda Ballesteros[1], Juan Carlos del Real[1], Nicholas Dunne[2], Eva Paz[1]

[1] Institute for Research in Technology/Mechanical Engineering Dept., Universidad Pontificia Comillas, Madrid, Spain
[2]School of Mechanical and Manufacturing Engineering, Dublin City University, Dublin, Ireland
[*] sara.lopez@comillas.edu

INTRODUCTION

Due to their geometry, graphene (G) presents an interesting toughening effect. Additionally, G has shown great potential in the preparation of materials for biomedical applications thanks to its antimicrobial and osteoconductive capacity.

Additive manufacturing, especially VAT polymerization technologies (Stereolithography, SLA; Digital Light Processing, DLP and Liquid Crystal Display, LCD) have many advantages in the manufacturing of bone scaffolds since they allow to obtain controllable and customized designs with very high accuracy.

However, one of the main shortcomings of these technologies is the distortion caused by shrinkage during polymerization and manufacturing process. Residual stresses generated can cause cracks and delamination and decrease mechanical properties[1]. The presence of nanoparticles could impact on the residual stresses, printing process or mechanical properties; therefore, it is an important aspect to study. Mechanical properties of printed samples depend on many factors[2]: adhesion between the layers, possible internal stresses due to shrinkage, polymerization degree, postcuring process, etc. In a previous work[3], it was found that all these factors hinder the reinforcing effect of G. Therefore, for a better understanding of the effect that G has over all these factors, samples were prepared by molding to be able to study the effect of G on curing and reinforcement mechanism, leaving aside the possible effect that the 3D printing process has on the final properties.

EXPERIMENTAL METHODS

Materials: Acrylic resin Clear V4 (Formlabs) and G of 1-2- layers of thickness (Avanzare) were used.

Preparation of samples: Dog bone samples were manufactured (ISO 527-2:2012). Samples were obtained using different polymerization processes:(i) compression molding was carried out at 160°C for 11 min, (ii) UV molding was done in FormCure chamber for 11 min and (iii) SLA samples were obtained with a Form2 printer.

Post-treatments: Postcuring was applied in a FormCure chamber where heat and UV were applied. Temperature was set at 80°C for 90 minutes.

Annealing was carried out at atmospheric pressure and 100°C for 12 hours.

Mechanical testing: Tensile properties were obtained with a Universal Testing Machine IBTH/500 (Ibertest).

FTIR Analysis: Polymerization degree (X) was calculated using a Tensor 27 spectrophotometer (Bruker). The polymerization was determined analyzing the peaks at 1733 and 810 cm^{-1}, corresponding to C=O and C=C, respectively, and using Eq 1. I_{liquid} is the intensity of the peak in the unpolymerized resin.

$$X = \left[1 - \frac{I_{C=C}}{I_{liquid\ C=C}} \cdot \frac{I_{liquid\ C=O}}{I_{C=O}}\right] \cdot 100 \quad (1)$$

RESULTS AND DISCUSSION

Mechanical properties depended on the source of the polymerization process. The addition of G produced improvements when polymerization was carried out by heat, however a decrease of the tensile strength was observed with the addition of G when the resin was cured by UV light.

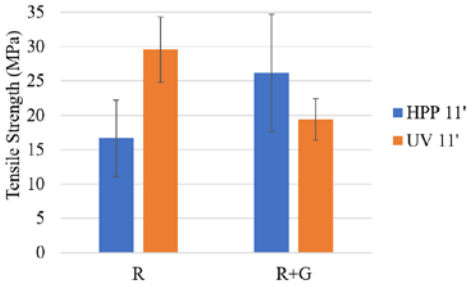

Figure 1. Tensile strength of samples obtained by compression molding (HPP 11') and UV molding (UV 11')

The presence of G and the source of polymerization affect the polymerization degree. In Table 1 it can be observed that G favors thermal polymerization and inhibits UV polymerization.

Table 1. Polymerization degree (%) obtained by FTIR

	R	***R+G***
HPP 11'	29.21	34.52
UV 11'	24.27	19.47

Different post-treatments were evaluated in order to allow the obtention of 3D printed parts with G, with acceptable mechanical properties. The results demonstrated that when postcuring and annealing are combined, the decrease in tensile strength due to the addition of G is corrected.

CONCLUSION

The addition of G and the polymerization process have a significant impact on mechanical properties of photocurable resins. The presence of G affects UV polymerization and reduces tensile strength. This reduction can be solved by post-treatments, releasing residual stresses. These post-treatments could be used to tailor the mechanical properties of printed structures, adapting it to the properties to the bone of interest in every particular case.

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