NOVEL QUANTIFICATION METHODS OF FILLERS DISPERSION IN POLYMER COMPOSITES AND NANOCOMPOSITES BASED ON HIGH RESOLUTION SYNCHROTRON X-RAY \( \mu \)-CT

S. Pérez-Tamarit\(^1\), E. Solórzano\(^1\), E. Laguna-Gutierrez\(^1\), B. Notario\(^1\), A. Hilger\(^2\), I. Manke\(^2\), M. A. Rodriguez-Pérez\(^1\)

\(^1\) CellMat Laboratory, University of Valladolid, Paseo de Belén 7 47011, Valladolid, Spain.
\(^2\) Helmholtz-Zentrum Berlin für Materialien und Energie, Lise-Meitner-Campus, Hahn-Meitner-Platz 1 14109, Berlin, Germany.

* Presenting author saul.perez@fmc.uva.es

Keywords: synchrotron x-ray tomography, polymer composites, particle dispersion analysis, particle morphology analysis.

Summary: The aim of this work was quantifying the dispersion quality of different fillers (geometry and size) with concentration ranging 1-3 %Vol. in two different polymeric matrixes (amorphous and semicrystalline) by using synchrotron X-ray \( \mu \)-CT. In addition, several morphological features of the selected additives have been analysed.

1. INTRODUCTION

The addition of fillers and nanofillers in polymeric materials is a common practice to accomplish determined requirements for these kinds of materials. As an example, certain physical properties such as mechanical or thermal properties could be enhanced by the addition of fillers [1]. Nonetheless, one of the critical aspects in these composites or nanocomposites to achieve the desired effect is to guarantee a good dispersion of the particles added.

Determining quantitatively the degree of dispersion of these fillers, especially in the case of nanofillers, is not a simple task. Different methods, all of them with advantages and disadvantages, have been used such as SEM, TEM, rheology, etc [2-3]. In this contribution, we propose an alternative approach with 3D resolution, avoiding any 2D skew, to evaluate the degree of fillers dispersion in polymeric systems.

2. EXPERIMENTAL METHOD

Synchrotron X-ray micro-tomography (\( \mu \)-CT) represents one of the most powerful techniques for the structural characterization in materials science [4]. It allows the reconstruction in 3D of the structure of the analysed samples with a very high spatial resolution (up to 0.4 micrometres) (Fig. 1). In our particular case, the high coherence of the synchrotron beam used permitted obtaining projections with valuable contrast. The synchrotron X-ray experiments were carried out at BAM-line, imaging beamline located in Bessy II synchrotron (Berlin, Germany).

Furthermore, two different groups of polymer composites were included in the study. On one hand, three different additives (sepiolite, talc and nanosilica) were included in a polystyrene (PS) matrix and on the other hand, particles of azodicarbonamide were added to three different polypropylene (PP) matrixes.

3. RESULTS

The image analysis of the 3D images comprised several parameters such as total volume of added filler, effective particle size, particle orientation, number of particles per unit volume and inter-particle distance distribution. In addition, quantitative indicators of the particle dispersion quality were calculated and analysed for the first time in three dimensional images.
Acknowledgments

Financial assistance from MINECO (MAT2015-69234-R and the Junta of Castile and Leon (VA011U16) is gratefully acknowledged. In addition, predoctoral contract of S. Perez-Tamarit by University of Valladolid (E-47-2015-0094701) and co-financed by Banco Santander is also acknowledged.

References


Figure 1: Example of a 3D reconstruction of a polymer composite (2% Vol.) with 350x350x350 µm³ of volume in which it is shown that the fillers were accurately resolved by using synchrotron X-ray µ-CT