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Data Article

Experimental data in support of characterization of the $CepO₄$ dispersion into transparent PMMA/PU IPNs by the sequential route

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ABSTRACT

This article is focused on the complementary data referring to the article "Dispersion of upconverting nanostructures of $CePO₄$ using rod and semi-spherical morphologies into transparent PMMA/PU IPNs by the sequential route". It contains the XPS data of $CePO₄$. photographs and DSC thermograms of transparent PMMA/PU IPNs as well as with $CepO₄$ dispersed in different wt.%, Confocal laser scanning micrographs, transmission electron microscopy (TEM), optical images of surface, and visual inspection (photographs) before and after aging of hybrid materials.

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Specifications table

Value of the data

- \bullet This data can be useful for comparing the dispersion of CePO₄ nanostructures in similar systems and how the morphology affects the structural, thermal and mechanical properties.
- \bullet Data highlights the influence of dispersed CePO₄ in different PMMA/PU ratios.
- \bullet This article will serve as a guideline to select the amounts adequate for dispersing CePO₄ into PMMA/PU.

1. Data

Plastic materials with good durability can be obtained from the synthesis of interpenetrating polymer networks (IPNs) while incorporating luminescent nanostructures such as Fluorofunctionalized nanostructured silica (FSiO₂), silica (SiO₂), carbon black (CB), barium titanate (BaTiO₃), polyaniline, and cerium phosphate (CePO₄) $[1-6]$ $[1-6]$ $[1-6]$.

The dataset of this article shows additional information about the dispersion of CePO₄, in rods and semi-spherical morphologies, into transparent PMMA/PU IPNs. Initially, this study provides facts of the elements in CePO₄. For this reason, [Fig. 1](#page-2-0) shows the X-ray spectroscopy (XPS) spectra of nanorods and semi-spherical particles of CePO₄. This data also include photographs of different ratios of PMMA/ PU that showed the transparency of the samples synthesized ([Fig. 2](#page-2-0)). The thermal properties of PMMA/PU IPNs are confirmed in thermograms of selected PMMA/PU IPNs in [Fig. 3](#page-3-0). In addition, visual examination after incorporation of nanostructure in the polymer was analyzed. The incorporation, 0.1, 0.5, and 1 wt.% of $CepO₄$ (nanorods and semi-spherical) in 50/50 ratio of PMMA/PU IPNs is shown in photographs ([Fig. 5](#page-3-0)). In this context, dispersion and emission properties of CePO₄/PMMA/PU IPNs is confirmed by confocal laser scanning images showed in [Figs. 6](#page-4-0) and [7.](#page-5-0) Dispersion of nanorods and semi-spherical CePO₄ was evaluated in a selected sample, 5O/50 PMMA/PU IPNs ([Fig. 8\)](#page-5-0). On the other hand, the texture of fractures from the tensile test is observed in [Fig. 9](#page-6-0). Finally, visual inspection before and after accelerated weathering test is seen in [Fig. 10.](#page-7-0)

Fig. 1. XPS spectra of nanorods and semi-spherical CePO₄ nanoparticles.

Fig. 2. Photographs of pure PMMA/PU IPNs in different ratios.

2. Experimental design, materials, and methods

2.1. Cerium phosphate (CePO₄) synthesis and characterization

CePO4 nanostructures in nanorod and semi-spherical morphologies were obtained at pH 1 and 11, respectively, by the microwave-assisted hydrothermal method following the procedure described in Ref. [\[7\].](#page-9-0)

Fig. 4. Scheme of polymerization process of hybrid PMMA/PU/CePO₄ IPNs.

Fig. 5. Photographs of pure PMMA/PU IPNs (50/50 system) and PMMA/PU/CePO₄ IPNs (50/50 system) with the addition of nanorods (on the left) and semi-spherical (on the right) type morphologies.

PMMA/PU (50/50)/CePO4 Nanorods (0.1 wt.%)

PMMA/PU (60/40)/CePO4 Nanorods (0.1 wt.%)

PMMA/PU (70/30)/CePO4 Nanorods (0.1 wt.%)

PMMA/PU (80/20)/CePO4 Nanorods (0.1 wt.%)

Fig. 6. CLSM images of CePO₄ nanorods in 0.1 wt.% into 50/50, 60/40, 70/30, and 80/20 ratio of PMMA/PU IPNs.

PMMA/PU (60/40)/CePO4 Semi-spherical (0.1 wt.%)

PMMA/PU (70/30)/CePO4 Semi-spherical (0.1 wt.%)

PMMA/PU (80/20)/CePO4 Semi-spherical (0.1 wt.%)

Fig. 7. CLSM images of CePO₄ semi-spherical in 0.1 wt.% into 60/40, 70/30, and 80/20 ratio of PMMA/PU IPNs.

Fig. 8. TEM images of nanorods and semi-spherical CePO₄ nanoparticles dispersed in PMMA/PU 50/50 system.

Fig. 9. Images of selected PMMA/PU/CePO₄ IPNs.

2.1.1. X-Ray photoelectron spectroscopy (XPS) test was assessed in an Alpha 110, ThermoFisher Scientific XPS spectra of CePO₄ powders at two different pH are shown in [Fig. 1](#page-2-0). XPS spectra show typical

binding energies of Ce^{3+} and Ce^{4+} , which are forming the chemical state of as-prepared powders. The results were used to confirm the chemical composition.

2.2. Sequential synthesis of PMMA/PU/IPNs and CePO₄/PMMA/PU/IPNs

IPNs were prepared following the procedure in Ref. [\[1\]](#page-8-0). The transparency of the different ratios of PMMA/PU was evaluated by visual examination of samples synthesized, as it is shown in [Fig. 2.](#page-2-0)

Thermal properties of selected PMMA/PU pure samples were evaluated in Differential scanning calorimetry (DSC), conducted using a simultaneous Labsys Evo, Setaram TGA/DSC. Samples of DSC were tested at a heating rate of 10 K/min over the temperature range from 30 to 250 °C, under nitrogen atmosphere. Approximately 10–20 mg of each sample was placed in aluminium crucibles and maintained at 30 °C for 2 min, heated from 30 °C to 250 °C, maintained again at 250 °C for 2 min,

Fig. 10. Images of selected PMMA/PU/CePO₄ IPNs before (on the right) and after (on the left) aging during 500 h under heat and humidity conditions.

and cooled from 250 °C to 50 °C. PMMA/PU. [Fig. 3](#page-3-0) shows the thermograms corresponding to 60/40, 70/30, and 80/20 ratios of PMMA/PU.

Different amounts (0.1, 0.5, and 1.0 wt.%) of both nanostructures were incorporated and sonicated in the raw materials for PMMA and PU using two mixing times: 3 h and 10 min. DBTDL catalyst was added into the solution and sonicated for 10 min ([Fig. 4](#page-3-0)). The final solution was poured into a PTFE mould and kept for 18 h and 3 h in UV lamp (363 nm).

Selected sample, PMMA/PU 50/50 ratio, is displayed in [Fig. 5](#page-3-0) showing the differences for each addition of nanorod and semi-spherical particles, respectively.

Dispersion of CePO₄ nanostructures in rod and semi-spherical morphologies within the PMMA/PU IPNs was performed by confocal laser scanning microscopy (CLSM) using a Carl ZEISS microscope (Carl Zeiss, Jena, Germany). The fluorescence intensity measurements were performed using the built-in software ZEN of the LSM 710. Results are shown in [Figs. 6](#page-4-0) and [7](#page-5-0).

Transmission electron microscopy (TEM) was used to observe the dispersion of nanostructures in selected PMMA/PU/CePO₄ (50/50/1 wt.%). Ultra-thin sections were microtomed in epoxy resin. The sample was embedded and cured in R1078 Agar low viscosity resin (Agar scientific). The sample was shaped into a pyramidal shape and cut with a diamond knife into slices. A slice was put onto a Holey Carbon film 200 mesh Copper (HC200Cu). TEM observations were recorded in a JEM-2000 FX electron microscope (JEOL). Results are shown in [Fig. 8](#page-5-0).

The surface of PMMA/PU/CePO₄ IPNs in 80/20 and 50/50 ratio with 0.1, 0.5, and 1 wt.% of CePO₄ were analyzed in a were evaluated using an Olympus BX51 microscope. Results are shown in [Fig. 9](#page-6-0).

Selected dog-bone specimen of $PMMA/PU/CEPO₄$ were subjected to aging under humid conditions using a climate chamber (Memmer GmbH equipment) at 55 °C and 85% relative humidity for 500 h. Photographs of physical changes are shown in [Fig. 10.](#page-7-0)

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Transparency document. Supplementary material

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