EUROPEAN SYNCHROTRON RADIATION FACILITY
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Experiment Report Form

	Experiment title: Renwable thermotropic polyesters: optimizing molecular orientation for enhanced nucleating efficiency and reinforcement in polyester blends.	**Experiment number**: 26-02 856
Beamline: BM26B	**Date of experiment**: from: 03/02/2018 to: 06/02/2018	**Date of report**: 30/1/2019
Shifts: 9	**Local contact(s)**: Daniel Hermida Merino	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr Carolus H.R.M. Wilsens[1]*, Dr. Enrico Troisi[2], Prof. dr. Sanjay Rastogi[1], Prof. dr. Gerrit Peters[2], Dr Daniel H. Merino[3]

[1]Department of Biobased Materials, Faculty of Humanities and Sciences, Maastricht University, P.O. Box 616, 6200MD, Maastricht, The Netherlands.
[2]Department of Mechanical Engineering, Materials Technology Institute, Eindhoven University of Technology, P.O. Box 513, 5600 MB, Eindhoven, The [3]Netherlands3Netherlands Organisation for Scientific Research (NWO), DUBBLE@ESRF BP CS40220, 38043 Grenoble, France

Report:

In the allocated beamtime, we aimed to evaluated the effect of shear on blends of thermotropic polyesters dispersed in a polyester matrix, including polycaprolactone and polypentadecalactone. The experiments were prospected to be performed in a multi-pass rheometer, providing excellent control over shear, shear rate on the samples. Unfortunately, malfunctioning of the MPR device prevented us from performing the proposed experiments. Instead, the samples were mounted in the Linkam Shear-cell of the Dubble beamline, were shear experiments were performed, albeit under less controlled conditions.

Unfortunately, none of the evaluated shear protocols yielded any orientation of the LCP phase in the thermoplastic matrix. There are several reasons for this behavior; 1) The viscosity of the polyester matrix is relatively low, thereby decreasing its sensitivity to shear, 2) the sample slips on the kapton windows used in the shear cell (these are nessecary as the other windows interfere with the WAXD scattering), and 3) the LCP has a very fast relaxation in blends in the order of 2 second which coincides with the framerate of our

experiments. Examplary XRD patterns showing the LCP already almost in a fully relaxed state directly after the application of shear (right image) is displayed in Figure 1 together with the reference materials.

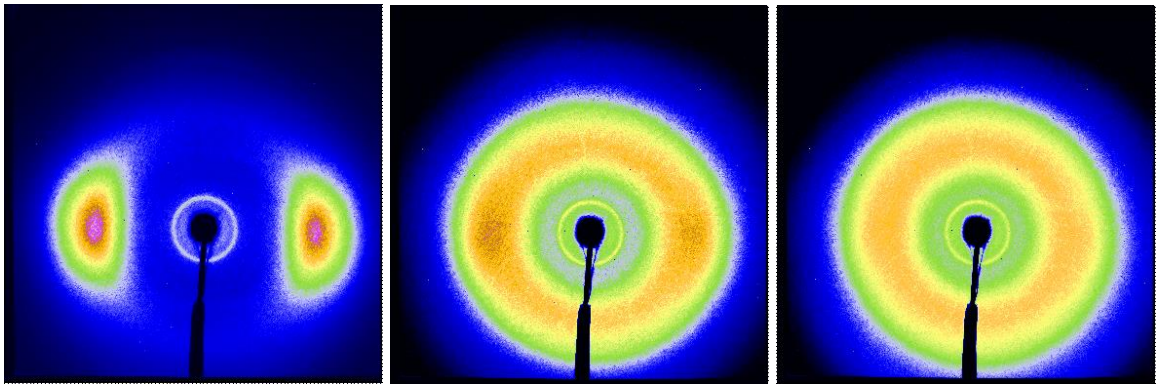

Figure 1. Left, WAXD pattern showing highly oriented inter-chain diffraction signals of pure injection molded LCP. Middle WAXD pattern of the same LCP after blending 30 wt% in a polyester matrix (after injection molding). Right, WAXD pattern of 30 wt% LCP in polyester mounted in the shear-cell (10 seconds of shear with 50 s^{-1}) directly after the application of shear, suggesting that LCP orientation is low and rapidly relaxes in the current set-up.

After trying to develop alternative protocols, we have resorted to evaluation of the crystallization behavior of the materials without the application of shear. Different samples have successfully been tested: PPDL, PCL, but also PLA with nucleating agent as reference. An example of the crystallization behavior of PLA with nucleating agent during cooling in the shear cell is displayed in Figure 2.

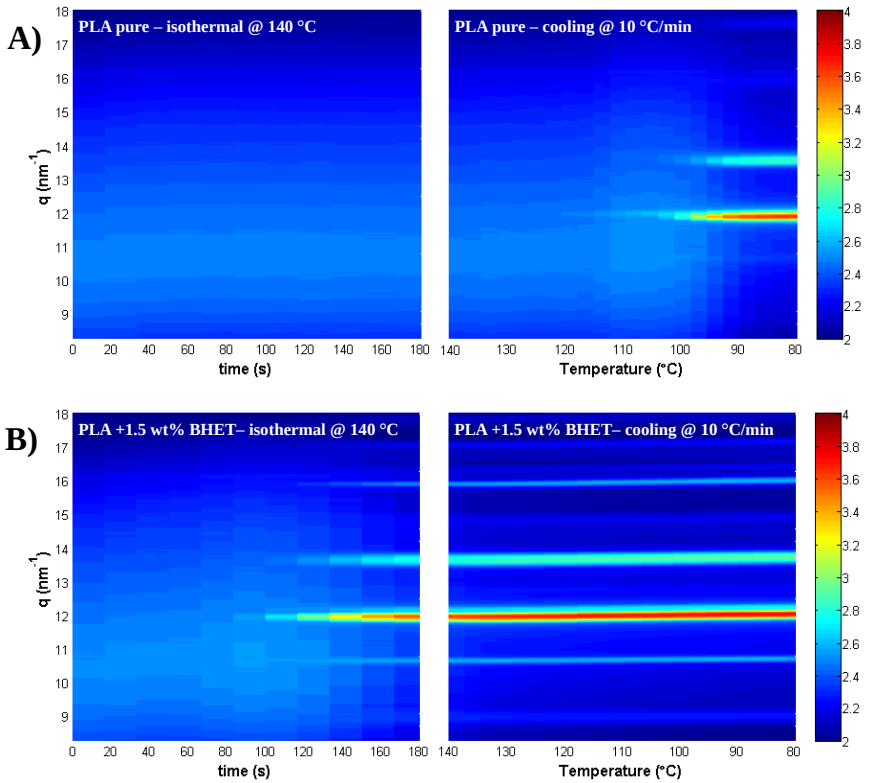

Figure 2. Crystallization of PLA with nucleating agent used as reference. The samples were heated to 200 °C, then cooled to 140 °C and subjected to a 3-minute isotherm. Next, the samples were cooled with 10 °C/min and the crystallization behavior was monitored.