ESRF	**Experiment title:** Crystallization behavior of novel biobased random copolyesters	**Experiment number**: SC-5479
Beamline: BM26	**Date of experiment**: from: 12/09/2023 to: 15/09/2023	**Date of report**: 18/09/2023
Shifts: 9	**Local contact(s)**: Martin ROSENTHAL	*Received at ESRF:* *19/09/2023*

Names and affiliations of applicants (* indicates experimentalists):

Leire Sangroniz[1,2]*, Zakarya Baouch[2]*, Dario Cavallo[2]*

[1]POLYMAT and Department of Polymers and Advanced Materials: Physics, Chemistry and Technology, Faculty of Chemistry, University of the Basque Country UPV/EHU, Paseo Manuel de Lardizábal, 3, 20018 Donostia-San Sebastián, Spain.

[2]Department of Chemistry and Industrial Chemistry, University of Genova, Via Dodecaneso 31, 16146, Genova, Italy

Report:

3 copolymer families based on polyesters, which combine amide and additional ester groups, have been investigated. The samples were studied at room temperature and during heating and cooling scans to understand the crystalline structure and the thermal behavior.

In Figure 1 the DSC results and X-ray diffraction patterns collected at room temperature for one copolymer family are shown as an example. The DSC scans shows the complex melting behavior of some of the copolymers which could indicate the presence of several crystalline phases, each of one corresponding to the homopolymers. The WAXS patterns acquired at room temperature show that the copolymers display a similar trend to that of the corresponding homopolymer. The copolymers rich in aliphatic diol (AlD) show a similar pattern with two distinctive peaks, whereas the copolymers rich in amido diol (AD) show two broad peaks. The position of the main peak (around 15 nm^{-1}) shifts slightly with the composition, which indicates that crystalline unit cell depends on the copolymer composition. This is a characteristic of isodimorphic copolymers.

The SAXS patterns show that the maxima in the intensity is composition dependent, as could be expected for isodimorphic copolymers. The q value at which the maximum appears is shifted from 0.25 to 0.7 nm^{-1} from aliphatic polyester (AlD) to polyester amide (AD), which reflects that the long period is bigger for aliphatic polyester than for polyester amide. For copolymers rich in amido diol, this component dominates having only small variations in the long period or q_{max} parameter.

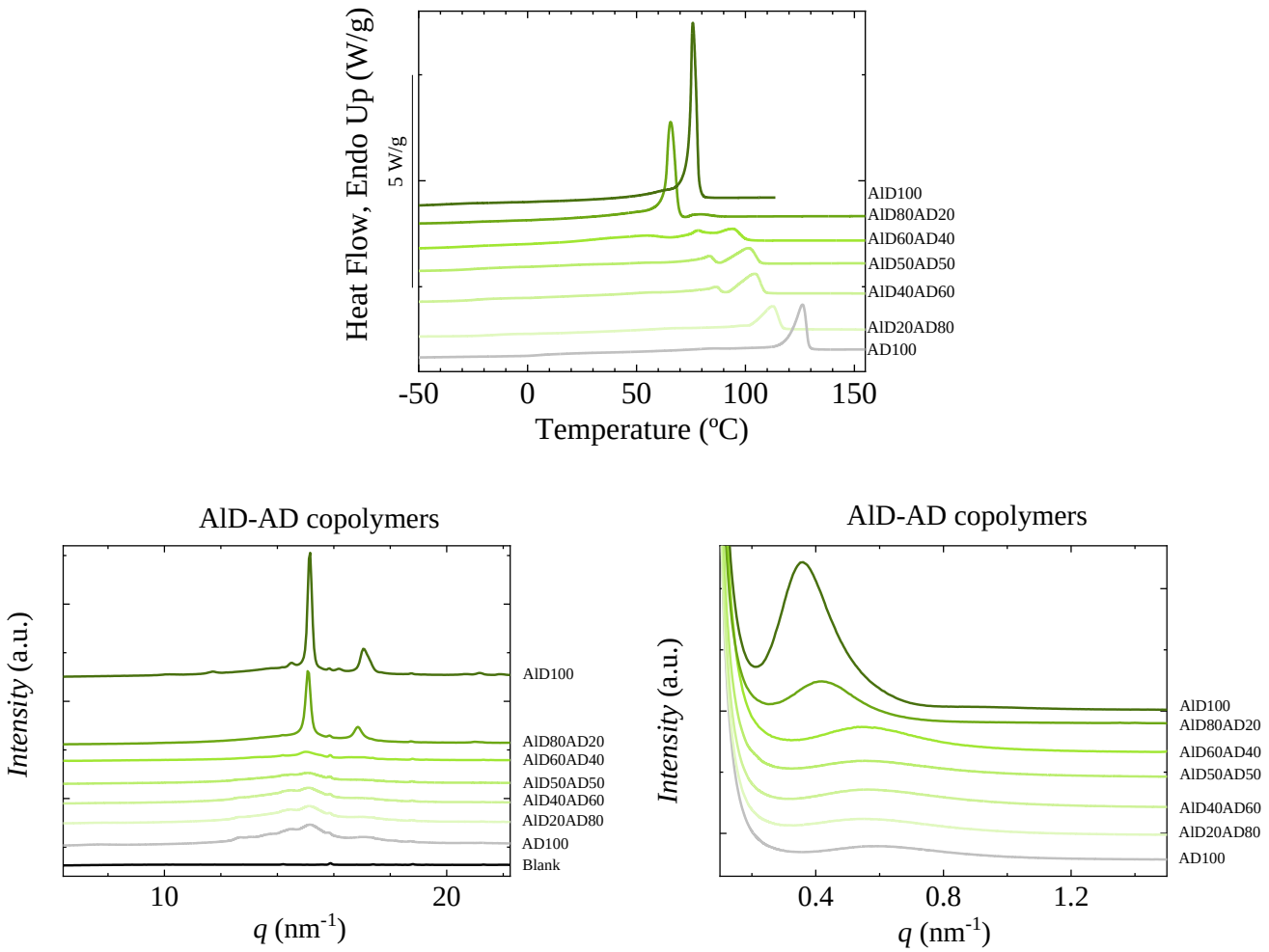

Figure 1. DSC heating scans for homopolymers and copolymers based on aliphatic polyester and polyester amide. WAXS and SAXS patterns collected at room temperature.

Due to the complexity of the materials, X-ray experiments acquired heating and cooling the material are needed to understand what happens at each endothermic peak of the DSC. Those experiments have been performed and the analysis is being carried out. The acquisition of WAXS and SAXS will allow us to understand if the several melting peaks correspond to the melting of several crystalline phases, each of one could correspond to crystalline phases resembling the one of the homopolymers, or if there is a reorganization of the crystals during heating.

In addition to this copolymer family, two other families were investigated which have additional ester groups. X-ray studies were carried out with all the samples at room temperature and during heating and cooling. The analysis of the results is being carried out. This study will allow us to understand the role of intermolecular interactions on the crystallization of isodimorphic copolymers.