Faculty of Science and Engineering

Novel biobased nucleating agent for high-speed melt spinning of PLA

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Introduction

Poly(lactic-acid) (PLA) is well known for its renewable origin, its capability to degrade in nature, and for its bioactive properties. However, PLA does suffer a low crystallization rate, which limits its potential in high strength applications. Within AMIBM we develop and study organic molecules that can self-assemble during cooling and act as nucleating agent. So far, we have demonstrated that terephtalic acid based compounds are successful nucleating agents for PLA in injection molding process and quiescent melt (see Figure 2). In the next step, the efficiency of the nucleating agent in the melt spinning process for fiber production will be analysed.

Concept of Project BB100

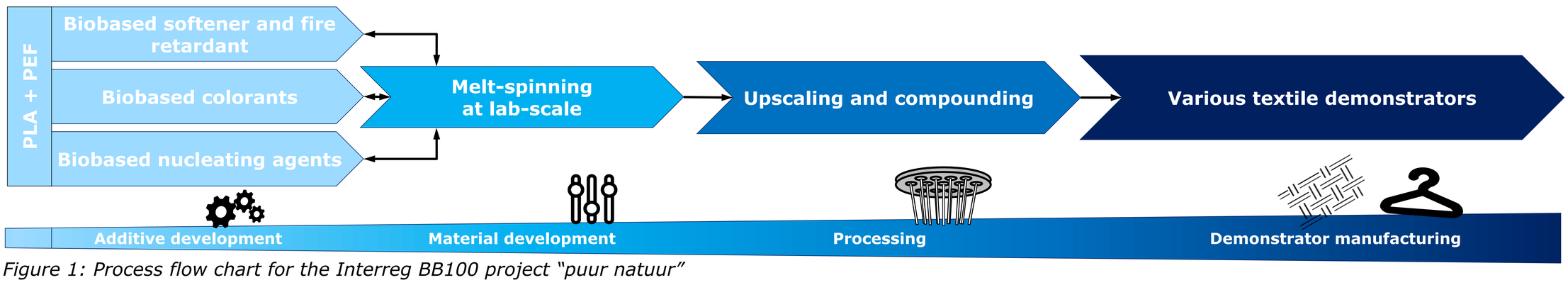
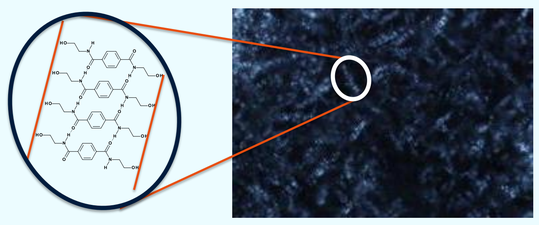


Figure 1: Process flow chart for the Interreg BB100 project "puur natuur"

Self-assembling biobased nucleating agent

The chemical synthesis is a one pot synthesis of terepthalic acid and ethanol amine, under refluxing condition, followed by purification steps. The nucleating agent self assembles during cooling from the melt into needle like structures acting as starting point for shish-kebab crystals.



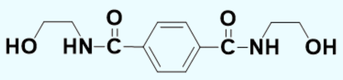
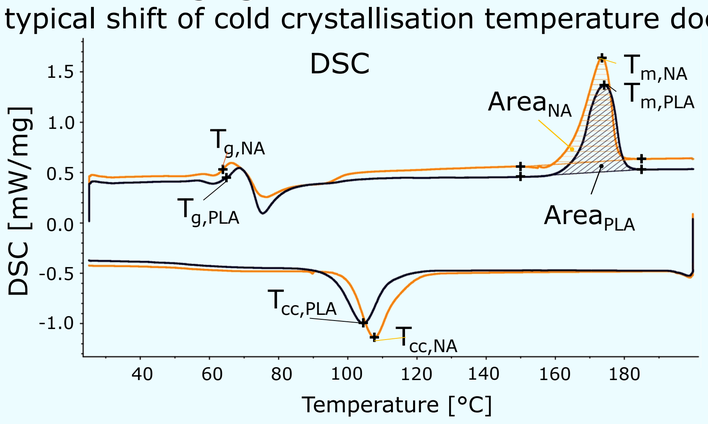


Figure 2: left: Crystal structure of self assembled nucleating agent in PLA
right: The chemical structure of terepthalic based nucleation agent [N1,N4-bis(2-hydroxyethyl) terephthalamide]

DSC and GPC analysis

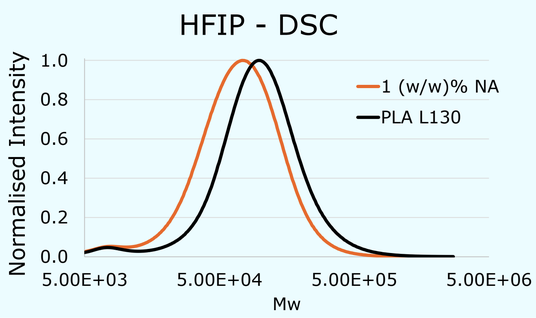
Thermal behavior of neat PLA and the compound with 1 w/w% NA is analysed with DSC and GPC measurements. The DSC curves indicate, that the nucleating agent did not come into effect in the used process as the typical shift of cold crystallisation temperature does not occur here.



	neat PLA	1 (w/w)% NA
T_g [°C]	64.3	63.7
T_m [°C]	174.1	173.6
T_{cc} [°C]	104.3	107.6
Area [J/g]	50.16	53.84

The GPC analysis* visualizes degradation that occurred during processing of the NA masterbatch. $M_{w,NA}$ (8.543E4) is reduced by 30% referred to $M_{w,PLA}$ (1.2186E5).

*relative to PMMA reference sample



Melt spinning of multifilament fibres

The synthesized nucleating agent is incorporated into PLA as a masterbatch with a final ratio of 1 w/w%.

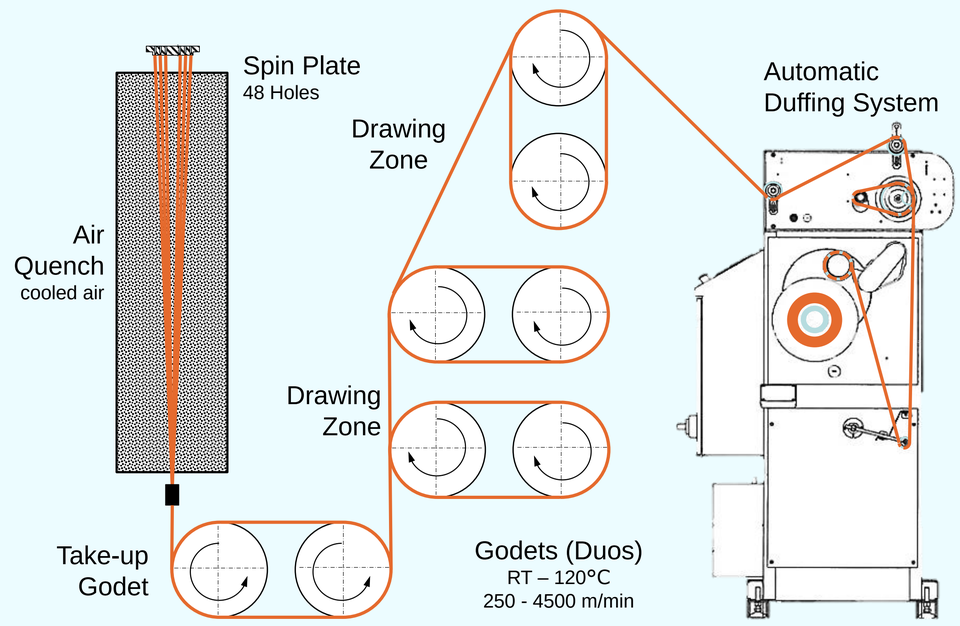
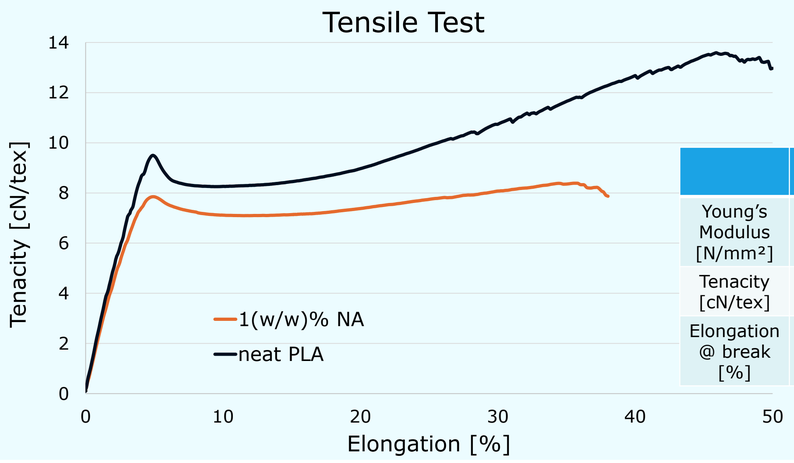


Figure 3: Industrial melt spinning setup with FET 101 multifilament line (left) and Sahm Twinstar II (X382E) winder (right)

Tensile testing of as-spun fibres

Tensile testing of the as-spun fibres with capstan clamps on a ZwickiLine 2.5kN.



	neat PLA	1 (w/w)% NA
Young's Modulus [N/mm²]	66,667	55,814
Tenacity [cN/tex]	14	8,7
Elongation @ break [%]	50	38

Conclusion & outlook

- Incorporation of the nucleating agent changed the crystallisation behaviour
- Melt spinning parameter have to be optimized to take full advantage of the nucleating agent
- Analysis of the crystal morphology via WAXD and SAXS for better understanding

Founding

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