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NewWave

D3.3 – Production and delivery of polyols

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Executive Summary

D3.3 is the last deliverable in a series of 3 describing the production and delivery of polyol samples. The deliverables are part of sub-task 3.1: Production of polyols (M1-M36). In D3.1 and D3.2, the polyol samples produced and delivered in M1-12 and M12-M24 were described. In D3.3 the polyols produced and delivered in M24-32 are added. The polyols were produced at BTG, by the hydrogenation of pyrolysis oil or pyrolytic sugars. The hydrogenation was executed in bench-scale or pilot-scale hydrotreaters applying the patented Ni-based PICULA™ catalyst. After hydrogenation of the feed, distillation was applied to separate the small and large polyols. Subsequently, different post-treatments/purification steps were applied to remove impurities. The 15 polyol samples produced were sent to partner AEP Polymers to be tested in different polyurethane applications.

Introduction

Introduction and Objectives of NewWave

NewWave will contribute to building a circular economy by introducing sustainable raw materials in different manufacturing lines (ML's), replacing toxic chemicals, and lowering the environmental footprint of the products. The four manufacturing lines (see Figure 1) will produce engineered wood panels, furan base-chemicals, polyols and polyurethanes, and modified/engineered wood. These lines are not chosen 'at random', rather they evolved from previous research and technology development work and will reach TRL-6 by the end of the project. The products produced in the manufacturing lines will be used to enhance the sustainability of building materials in the construction industry. NewWave aims at wood-based materials including Cross Laminated Timber (CLT) to replace steel and concrete as structural components, modified/engineered wood to replace tropical hardwood or chemically treated wood for outdoor use, and Medium Density Fibreboard (MDF) and plywood for interior usage. Toxic chemicals like formaldehyde and creosote will be replaced by non-toxic, bio-based alternatives. Within the project, a small demonstration project will be built with these materials to show the products and test their durability.

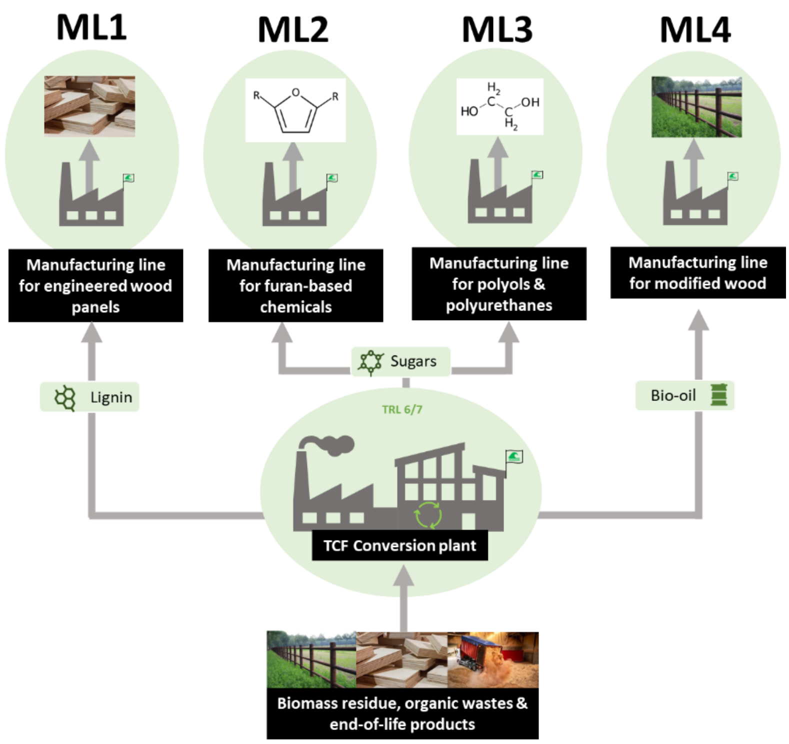

Figure 1: Schematic overview of the NewWave ML's

Thermo-Chemical Fractionation & sugar hydrogenation

The sustainable raw materials used in the manufacturing lines are obtained by Thermo-Chemical Fractionation (TCF) of biomass residues and end-of-life products. TCF is an innovative, two-step conversion process to transform different bio-resources into sustainable raw materials and is currently at a TRL-6/7. In the TCF process biomass is first converted by fast pyrolysis into Fast Pyrolysis Bio-Oil (FPBO). Subsequently, the FPBO is fractionated -based on chemical functionality- yielding a reactive lignin fraction and a sugar-rich fraction, both being excellent starting materials to produce sustainable, bio-based chemicals & materials. The selected product lines fully exploit the unique chemical functionalities already present in the biomass feeds. Moreover, the lines are interlinked, and output from one line will further improve the sustainability of the other. Wastewater treatment and water re-use is integral part of the NewWave concept. Combined with end-of-life recycling options and efficient use of byproducts this results in essentially waste-free manufacturing processes.

Figure 2: On the left TCF pilot plant, on the right HDO pilot plant

FPBO, and the pyrolytic sugar can be used as a raw material for the production of green polyols. In TCF, the pyrolytic sugars (PS) are obtained as a watery solution that can directly be processed in hydrogenation. Furthermore, the pyrolytic lignin can be used in other added value applications such as various resin systems, minimalizing the production of waste residues. At BTG, the TCF process is performed on pilot-scale (see Figure 2). The pyrolytic sugar consists of small sugar derived molecules such as glycoaldehyde, levoglucosan, cellobiosan, large polymer fractions of cellulose but also other organic components such as carboxylic acids and lignin derived phenolics etc.

Production of polyol samples

The polyols are produced by the hydrogenation of fast pyrolysis bio-oil (FPBO) or the pyrolytic sugars (PS). The hydrogenation is executed in bench-scale or pilot-scale hydrotreaters applying the patented Ni-based PICULA^{TM} catalyst. In addition, alternative inhouse prepared catalysts are tested at various process conditions in an endeavor to increase the yield of small polyols. After hydrogenation of the feed, post-treated and/or further purification will be applied to make the polyols suitable to be used as a green additive or as raw materials for the production of polyurethanes. The mixed polyols, the MEG (mono ethylene glycol) and MPG (mono propylene glycol) will be used by AEP Polymers as the starting polyols or as chain extenders in polyurethane production and as an additive in formulations to modify wood.

Results

Polyol samples prepared and shipped to AEP Polymers

A large amount of SPO (stabilized pyrolysis oil) was produced by the hydrogenation of FPBO applying the PICULA™ catalyst under elevated but standard T and PH_2. A smaller amount of SPS (stabilized pyrolytic sugars) was produced by the hydrogenation of PS under similar conditions in a bench-scale hydrotreater. Both The SPO and SPS contain a large fraction of polyols and were subsequently post-treated to remove impurities such as residual lignin. An overview of the samples produced and sent to AEP Polymers for testing in PUR-systems is given in Table 1. The samples produced contain both the large/heavy and small/light polyols (mixed polyols), only the small/light polyols (MEG + MPG), or only the large/heavy (oligomeric & polymeric) polyols. The small polyols are separated from the large polyols by distillation. Typically, the yield of large polyols from the mixed polyols is 65-75 wt.% and the yield of small polyols 25-35 wt.%. Figure 3, shows a photo of a PUR test specimen prepared by AEP Polymers using one of the light polyol sample.

Table 1: Polyol samples prepared and shipped to AEP

Sample no.	Sample name	Sample description[1]	Shipment code	Amount (g)	Shipment date
1	BTG_NW_SPOF	Mixed polyols derived from SPO	2022-2567	300 g	24/05/22
2	BTG_NW_SPOL	Light polyols derived from SPO	2022-2566	300 g	24/05/22
3	BTG_NW_SPOH	Heavy polyols derived from SPO	2022-2565	800 g	24/05/22
4	BTG_NW_SPSL	Light polyols derived from SPS	2022-2619	90 ml	16/11/22
5	BTG_NW_SPSH	Heavy polyols derived from SPS	2022-2621	350 ml	16/11/22
6	BTG_NW_SPSL	Light polyols derived from SPS	2022-2618	50 ml	16/11/22
7	BTG_NW_SPOFE	Extracted mixed polyols derived from SPO	2022-2620	175 ml	16/11/22
8	BTG_NW_SPOLE	Extracted light polyols derived from SPO	2022-2622	50 ml	16/11/22
9	BTG_NW_SPSL	Light polyols derived from SPS	2023-2697	100 ml	11/05/23
10	BTG_NW_SPOFE	Extracted mixed polyols derived from SPO	2023-2698	250 ml	11/05/23
11	BTG_NW_SPOLE	Extracted light polyols derived from SPO	2023-2707	200 ml	13/06/23
12	BTG_NW_SPOLE	Extracted light polyols derived from SPO	2023-2770	150 ml	16/11/23
13	BTG_NW_SPSL	Light polyols derived from SPS	2023-2771	150 ml	16/11/23
14	BTG_NW_SPOLE	Extracted light polyols derived from SPO	2024-2864	400 ml	17/06/24
15	BTG_NW_SPSL	Light polyols derived from SPS	2024-2881	250 ml	03/07/24

[1]: SPO: Stabilized pyrolysis oil, [2]: Stabilized pyrolytic sugars

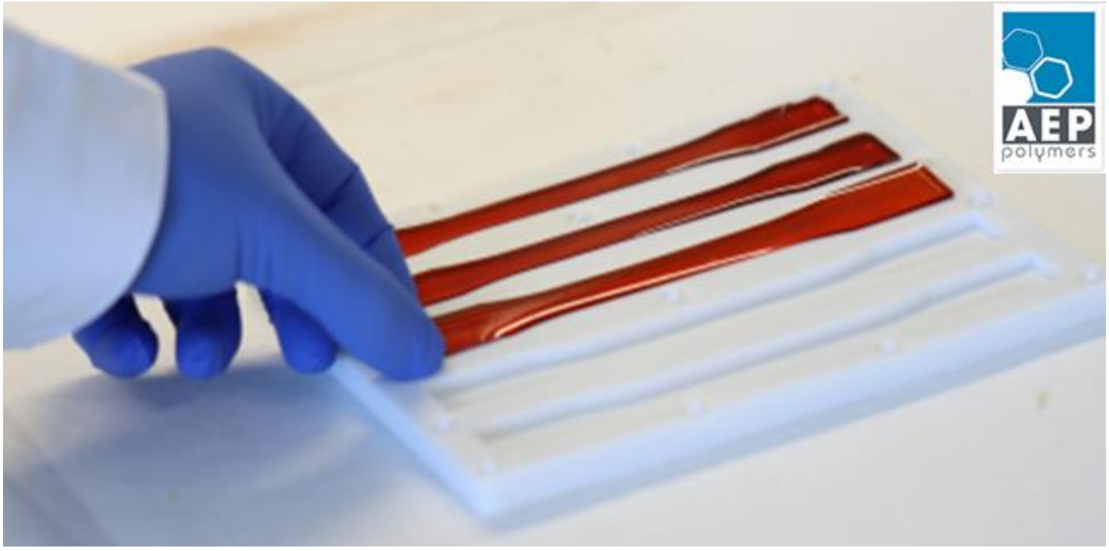

Figure 3: PUR test specimen made by AEP Polymers

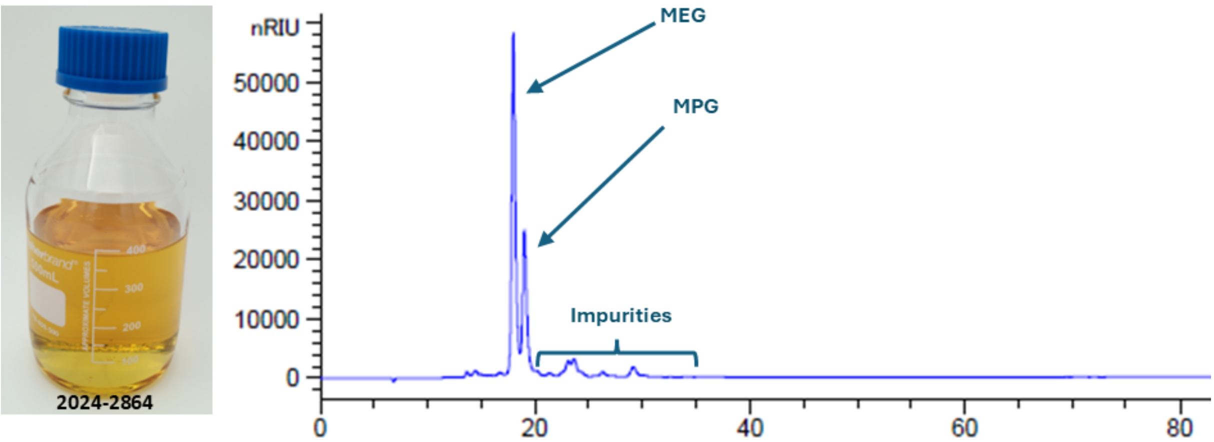

Figure 4: Photo & HPLC chromatogram of sample 2024-2864

Analysis of polyols samples

For all samples, standard analysis was performed including HPLC-analysis (if possible) to determine the light/small polyol content. Results of these analysis are given in Table 2.

A picture of sample 2864 and its HPLC chromatogram are illustrted in Figure 4.

Table 2. Analysis of samples prepared.

	2022-2565	2022-2566	2022-2567	2022-2618	2022-2619	2022-2620	2022-2621	2022-2622	2023-2697	2032-2698	2023-2707	2023-2770	2023-2771	2024-2864	2024-2881
HPLC[1]															
MEG (wt%)	-[2]	35,6	21,9	26,90	35,2	28,8	-[2]	35,0	27,9	28,2	55,3	54,0	31,8	49,5	29,7
MPG (wt%)	-[2]	15,9	11,3	10,3	14,7	12,5	-[2]	14,2	11,7	12,8	23,3	22,7	11,3	22,2	7,6
Standard analysis															
C/H/N (wt%)	63,1/7,8/,0,0	49,0/9,6/0,0	49,0/9,3/0,0	51,5/9,4/0,2	50,0/9,1/0,1	46,2/8,7/0,0	58,4/6,8/0,5	48,3/9,8/0,0	52,1/8,9/0,2	46,2/8,9/0,3	44,8/10,0/0,1	43,9/9,7/0,1	51,0/8,3/0,2	44,3/10,1/0,1	51,8/9,0/0,1
Karl Fischer H_2O (wt%)	0,6	1,0	2,1	1,7	1,9	2,5	0,5	1,1	0,9	1,1	1,2	0,5	0,8	1,6	0,8
TAN (mg KOH/g)[3]	11,6	40,7	37,1	61,4	64,6	15,2	11,5	12,6	61,2	19,5	11,9	7,2	50,6	14,6	34,9
CAN (mmol/g)[4]	1,9	0,2	2,6	1,7	1,9	0,5	2,4	0,0	1,4	1,6	0,6	0,0	1,6	0,0	1,3
pH	4,5	4,4	4,0	4,2	4,3	3,8	4,6	4,4	4,8	4,1	4,4	4,1	4,2	3,8	4,4
CR (wt%)[5]	15,6	0,0	3,9	1,1	0,6	3,2	25,4	0,0	0,3	3,8	0,0	0,2	0,5	0,0	0,2

Heavy polyols; oligomers & polymers (orange)

Light polyols; a.o. MEG & MPG (green)

Mixed polyols; Heavy + light polyols (blue)

[1]: HPLC analysis: MEG = Mono ethylene glycol, MPG = Mono propylene glycol. [2]: could not be determined by HPLC. [3]: Total acid number. [4]: Carbonyl number. [5]: Carbon residue.

Conclusions

D3.3 is a follow-up/update of D3.2 & D3.1 and is the third of three deliverables on the production and delivery of polyol samples to partner AEP Polymers. The polyols were produced at BTG, by the hydrogenation of pyrolysis oil or pyrolytic sugars. The hydrogenation was executed in bench-scale or pilot-scale hydrotreaters applying the patented Ni-based PICULA^{TM} catalyst. After hydrogenation of the feed, the small polyols and large polyols were separated by distillation. Subsequently different post-treatments/purification steps were applied to remove impurities. The 15 polyol samples produced were analyzed and sent to partner AEP Polymers to be tested in different polyurethane applications.

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