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Properties and performance of Biosuccinium sustainable succinic acid in microcellular polyurethane elastomers

Biosuccinium sustainable succinic acid has been evaluated as alternative for adipic acid in the production of polyester polyols used in microcellular polyurethane (m-PU). It is a unique 100 % bio-based product from Reverdia, the use of which increases the renewable content and reduces the environmental footprint of polyurethane formulations, while maintaining the performance required in many applications. During this research, various 2,000 g/mol polyester polyols, all based on ethylene glycol and diethylene glycol mixtures, were synthesised using adipic acid, succinic acid or a succinic/sebacic acid blend. In addition, a commercially available adipate reference (Daltorez P716) was obtained from Huntsman. Synthesis of these polyester polyols was completed without any problems and the resulting properties were within expected ranges. The main difference observed was that the succinate polyol showed some degree of crystallinity, causing a melt temperature (T_m) of approximately 50 °C; in comparison, the other polyols were amorphous and showed no transitions other than T_a. Compared to adipate systems a higher viscosity of the EDS polyol was observed as well for the polyol based on succinic/sebacic acid blend. Microcellular PU elastomers were prepared based using an MDI prepolymer with an NCO content of 19 %. The ethylene glycol – diethylene glycol – succinate polyol required minor adjustments for higher processing temperature. Reactivity for all formulations was similar. For performance evaluations, moulded slabs were produced with densities in the range of 0.47–0.60 g/cm³). The succinate elastomer showed a higher hardness compared to the adipate (48 Shore A vs. 37 Shore A). Physical properties like tear strength and elongation at break as well as abrasion resistance were quite similar for all elastomers. Results from this evaluation indicate that Biosuccinium based polyester polyols can be successfully formulated into microcellular polyurethane systems, without the need for extensive revision of formulations to produce materials with very similar mechanical performance to non-bio-based systems.

1. Introduction

In the last decade a number of global megatrends have intensified the need for products made from "bio-based materials". First, the global upturn on sustainability is driven by the need to respect generations to come and to be careful with the resources that are available. This is leading to more efficient ways to use, reduce, re-use and recycle materials, and also to using renewable raw materials. Next, the strong growth in demand for oil due to the increasing world population has dominated national and international agendas for many years already. Policy making in several parts of the world is focusing on security of energy supply, now and in the future. Very recently, oil price volatility has

been particularly large, and has driven many purchasing organisations to search for alternatives.

Also, environmental concern has risen strongly in the past ten years. Concern for the planet's wellbeing is at the forefront of the attention of governments, corporations, customers and non-governmental organisations alike. Consumers ever more expect and demand sustainable products. Non-governmental organisations push brand owners to source responsibly and corporations have made sustainability an integral part of their strategies. Renewable materials offer a potential way of improving the sustainable characteristics of the products that are made from them. All these megatrends have come together, creating a unique situation propelling the growth of bio-based chemicals.

1.1 Biosuccinium

In this paper Biosuccinium is investigated as a bio-based alternative for adipic acid (AA) for use in production of polyester polyols and microcellular polyurethane elastomers with an improved environmental footprint.

Biosuccinium sustainable succinic acid (SA) is produced by Reverdia, a joint venture between DSM and Roquette, using a proprietary low pH yeast process. It is a 100 % biobased and renewable diacid. Typically, Bio-

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Tab. 1: Overview of polyester polyol formulations and properties

Label	Diols ¹	Diacids	Renewable carbon con- tent / %	Mn / g/mol	OH / mg KOH/g	AV / mg KOH/g	Oven tem- perature ³ / °C	Melt viscosity @ 75 °C / mPa·s
EDS	EG / DEG	SA	59	2,044	54.5	0.5	~70	1,405
EDSS	EG / DEG	SA / SebA ²	63	1,941	57.3	0.5	50	1,292
EDA	EG / DEG	AA	0	1,908	57.7	1.1	50	502
P716	EG / DEG	AA	0	~2,000	~56.1	< 0.5	50	520
¹ Molar ratio of EG/DEG = 60/40; ² Molar ratio of SA/SebA = 85/15; ³ Oven temperature used to melt the polyol								



succinium can be used to substitute adipic acid and could be regarded as a "near dropin" raw material for the production of polyester polyols and polyurethanes [1, 2].

Reverdia is the first company in the world to have a large scale facility for the commercial production of bio-based succinic acid, which is marketed under its Biosuccinium brand. It benefits from the best and most sustainable (yeast-based) fermentation technology [3] to produce bio-based succinic acid, which has been in development since 2008. The new production facility with a 10 kt/y capacity is located in Cassano Spinola, Italy, and started operations in December 2012.

1.2 Research objectives

The investigation will look at a standard formulation of polyester polyol and microcel-



Fig. 1: Reduction of the carbon footprint using Biosuccinium versus fossil-based adipic acid [3] lular polyurethane elastomers. The differences in manufacturing, processing and properties of the resulting materials versus the adipic acid-based benchmark are to be identified. No optimisation or improvements will be done at this stage, but where possible recommendations for optimisations will be given.

1.3 Enabling sustainable polyurethanes

Biosuccinium is made from renewable feedstocks, which requires less from the earth's limited fossil resources, and delivers a reduction in greenhouse gas (GHG) emissions. The use of this materials makes it possible to yield a polyester polyol with a renewable content of up to 60 % and a polyurethane product with a renewable content of up to 30 % (tab. 1 and 2). A calculation of the sustainability improvement potential has been performed for the formulations as prepared in this study. Additionally, an estimate has been made for further improvements by using bio-based raw materials for the diols as used in the polyester polyol, the prepolymer and the chain extender.

The Biosuccinium cradle-to-gate study was executed and published by the Copernicus Institute of Sustainable Development at Utrecht University, the Netherlands [3]. The Biosuccinium process uses non-fossil raw materials, sequesters carbon dioxide (CO_2), is energy efficient and the process does

Tab. 2: Physical properties of moulded PU elastomers

Elastomer (polyol)	PU1 (EDS)		PU2 (EDSS) PU3 (EDA)		PU4 (P716)	
Mix ratio (polyol : prepolymer = 100 :)	82	84	84	84	84	86
Bio-based content* / %	28	28	30	0	0	0
Density / g/cm ³	0.56	0.60	0.51	0.56	0.49	0.47
Hardness / Shore A	46	48	37	46	38	38
Stress / MPa @ 50 % strain @ 100 % strain @ 200 % strain @ break	1.8 2.4 3.3 3.5	2.0 2.6 4.0 5.0	1.3 1.8 2.7 3.7	1.5 2.0 2.8 4.3	1.3 1.6 2.4 2.7	1.4 1.7 2.7 3.7
Strain @ break / %	235	270	280	340	250	300
Tear strength / N/mm	26	31	22	32	21	24
Tear strain @ break / mm	65	80	80	110	95	100
Abrasion resistance / mg weight loss	15	n.d.	18.3	19.1	n.d.	18.4
* Bio-content originating from the dicarboxylic acids only. Using bio-based EG or DEG will further increase bio-content.						

not produce unnecessary by-products. **Figure 1** shows the carbon footprint of Biosuccinium and the large potential carbon footprint reduction – about 8 kg CO_2 -equivalent per kilogram of acid – that is possible when "substituting" fossil-based adipic acid with Biosuccinium. The adipic acid data have been executed by DSM for a best in class plant with 98 % N₂O abatement. DSM used SimaPro software with EcoInvent database 2.0.

Today, different technologies are being employed for the production of succinic acid derived from renewable feedstocks, and each of these has its own characteristics with regards to product quality, operational aspects, and environmental footprint.

The Copernicus Institute conducted a Life Cycle Assessment study [3] which in detail compared the various production methods, assuming all other things are equal (especially the energy mix and feedstock usage). They found that the yeast-based fermentation process at low pH, with direct crystallisation, as used by Reverdia to produce Biosuccinium, has significantly lower GHG emissions compared to other fermentation routes as well as petrochemical routes.

2. Experimental

2.1 Materials

The raw materials used in this study are shown in **table 3**.

2.2 Polyester polyol preparation

Polyester polyols with a molecular mass of 2,000 g/mol were synthesised on 3 kg scale in the laboratory by polycondensation of the diacid(s) with a mixture of ethylene glycol and diethylene glycol utilising TBT as the catalyst **(tab. 1)**.

The processes were carried out under nitrogen atmosphere by conventional means; first an esterification stage, under atmospheric pressure to 220 °C, followed by a



polycondensation stage under vacuum to achieve required molecular weight (as measured by product viscosity and hydroxyl value) and an acid value lower than 1 mg KOH/g.

2.2.1 Polyester polyol properties

All synthesised polyester polyols had a molecular mass closely matching the intended 2,000 g/mol. Hydroxyl values and acid values were very low as desired, except for the adipate polyol which showed a somewhat higher acid value of 1.1 mg KOH/g. This higher acid value may have an effect on processing and/or performance of polyurethane elastomers.

The EDS polyol showed some crystallinity, with dual melting peaks at 37 °C and 47 °C (DSC, first heating, see **fig. 2).** Therefore



Tab. 3:

Materials

Fig. 4: Reactivity evaluation using cup mouldings



this polyol required a higher oven temperature (70 °C) in order to be melted and processed. The other polyols are amorphous and do not show a melting point (**fig. 3**, EDSS) and could be melted without problems at the usual temperature of 50 °C.

Furthermore, both the polyester polyols containing succinic acid showed a viscosity that is higher than for the adipate reference. This is consistent with reported observations for Biosuccinium polyols for TPUs [1, 2, 4, 6, 7]. The higher viscosity of the EDS and EDSS polyols should be carefully monitored in order to ensure efficient raw material mixing. The higher melting point of the EDS polyol was not considered to be a problem and could easily be adjusted for.

Designation	Identification	Supplier	
Bio-based succinic acid	Biosuccinium (SA)	Reverdia	
Sebacic acid	Sebacic acid (SebA)	Dong Feng	
Adipic acid	Adipic acid (AA)	Rhodia	
Ethylene glycol	EG	Sabic	
Diethylene glycol	DEG	Sigma-Aldrich	
Tetra n-butyl titanate	TBT catalyst	Acros	
Adipate polyol (Mn=2000)	Daltorez P716	Huntsman	
MDI prepolymer (NCO=19)	Suprasec 2980	Huntsman	
Activator blend	Proprietary	Apple Polyurethanes	





▼ Fig. 5: Moulded slab after sample punching





3. Microcellular PU elastomer preparation

Using the above-mentioned polyols, polyurethane elastomers were made with a formulation typically used for a man's shoe, single density unit sole.

First, an activator blend was made in bulk by blending chain extender, catalyst, surfactant and water. This activator blend was blended with each polyol using a ratio of 86 parts polyol to 14 parts activator blend.

In a next step, a mix was prepared of the polyol blend (heated to 40–45 °C) and the MDI prepolymer (heated to 35–40 °C). Initially, a mixing ratio of 100:86 w/w was used, followed by experiments in which this ratio was reduced in 2 % steps. Reactivity was compared using 200 ml cup mouldings (fig. 4), with components mixed using an electric mixer for 10 s. These cup mouldings were used to determine the ratio at which the polyurethanes showed the best foaming behaviour. For performance evaluations, moulded slabs were produced from which test specimens were taken, using the

Tab. 4: Overview of PU elastomer compositions and reactivity

Mix ratio (polyol : prepolymer = 100 : ...) Elastomer/ Cure parameter polyol 80 82 84 86 88 50 45 45 45 Tack-free / s Rise time / s 50 45 45 45 80 65 70 80 PU1/EDS Pinch time / s Cup cure @ 2 min Slightly soft Good Quite good Slightly soft Slab demould @ 4 min Quite good Good Quite good Soft 50 Tack-free / s 45 50 50 Rise time / s 45 50 50 50 PU2/EDSS Pinch time / s 85 85 120 120 Cup cure @ 2 min Slightly soft Good Slightly soft Soft Slab demould @ 4 min Quite good Good Good Slightly soft Tack-free / s 50 40 40 Rise time / s 50 40 40 PU3/EDA 150+ 90 Pinch time / s 95 Cup cure @ 2 min Very soft Quite good Slightly soft Slab demould @ 4 min Soft Quite good Soft 50 60 60 Tack-free / s 50 Rise time / s 50 40 40 50 105 PU4/P716 Pinch time / s 85 80 85 Cup cure @ 2 min Soft Good Good Slightly soft Slab demould @ 4 min Slightly soft Good Quite good

ratio that gave best reactivity and ± 2 %, targeting a density of 0.6 g/cm³. These slabs had a size of 295 x 97 x ~6 mm (fig. 5), and a theoretical weight of approximately 103 g.

Tack-free time, rise time and pinch times (in seconds) were recorded. No cream times were recorded as samples started to cream during or immediately after the 10 s mixing time. Cup cure is assessed by relative permanent indentation at 2 min. Slab cure is assessed by relative indentation using a ballpoint pen tip at 4 min. Both cup cure and slab cure are rated according to the following scale:

Full recovery \rightarrow		Good cure		
		Quite good cure		
		Slightly soft		
No re	covery \rightarrow	Soft		

Suitability for de-moulding of product was tested after 4 min by bending all slabs on one corner over 90 degrees to observe any surface cracking. For none of the combinations of polyol and mix ratios any cracks were observed. In this study all moulded slabs were demoulded after 4 min. Recent patent literature [5] reports that in some cases succinatebased PU elastomers allow for shorter demoulding times but this effect was not investigated as part of this study.

4. Results and discussion

4.1 Reactivity of polyester polyols

4.1.1 EDS polyol

Melting the EDS polyol required a temperature of about 70 °C. The higher viscosity made it difficult to achieve efficient mixing within the cream time, so moulded slabs have mixing defaults on them. This viscosity is probably difficult to process at 50 °C on most normal PU shoe soling machines. Reactivity however was slightly faster when compared to the P716 reference **(tab. 4).**

4.1.2 EDSS polyol

Also the EDSS polyester polyol has a higher viscosity than P716, but it was mouldable using the process as used in this project. As with the EDS polyol, the viscosity of the polyol blend should be carefully considered to ensure commercial production machine processing and efficient mixing. The reactivity of this polyester polyol is comparable but slightly lower than P716 **(tab. 4)**.

4.1.3 EDA polyol

The elastomers based on polyester polyol EDA gave a lower reactivity with longer pinch times and softer cures relative to P716 (also adipate-based). It is considered that this may well be due to the higher acid value of the polyol **(tab. 4)**.

4.1.4 P716 polyol (commercial adipate reference)

An overview of the adipate PU elastomer composition and the reactivity of reference polyester polyol P716 can be found in **table 4**.



5. Microcellular PU elastomer properties

From each of these elastomers, 6 mm thick slabs were moulded from which test specimens were taken for evaluation of hardness, density, and mechanical properties.

5.1 Hardness and density

The target moulded density of the slabs was ~0.6 g/cm³, but since some of the slabs where thicker than 6 mm, the actual density of the moulded slabs varied between 0.47 g/cm³ and 0.60 g/cm³. The hardness of the resulting slabs also varied between 37 Shore A and 48 Shore A. As expected there is a correlation between these two, where the higher density slabs also show a higher hardness.

From the moulded slabs test samples were taken for evaluation of physical properties, according to the following standardised test methods:

- Density ISO 1183 ٠
- Hardness ISO 868 Shore A
- Tensile behaviour ISO 52 Type 5
- Tear strength ISO 34 Trouser
- Abrasion resistance ISO 9352 H-18 wheel, 1,000 g/wheel, 1,000 cycles

As indicated above, both the density and the hardness of the various slabs varied significantly, which in turn also has an influence on the other (mechanical) properties. In order to be able to draw valid conclusions, it is suggested to compare only slabs of similar density/hardness. Hence, PU3 (EDA) will be used as reference to compare with PU1 (EDS), and PU4 (P716) will be used as a reference to compare with PU2 (EDSS).

When comparing PU1 and PU3 at similar hardness, it can be seen from tensile measurements that the succinic acid-based elastomer has a slightly higher stiffness. The strain at break and the strength, measured in both tensile and tear mode, are slightly lower for the succinic acid-based elastomer. Values are still at practically acceptable lev-

els though. The abrasion resistance of the succinic acid-based elastomer is better compared to the reference, which is obviously highly relevant to wear-resistant, single density unit soles. When slightly increasing the mix ratio of polyol-to-prepolymer for PU1, both density and hardness increase, and overall mechanical properties change accordingly.

PU2 (EDSS) is obtained by incorporating a small amount of sebacic acid into the polyester polyol. The influence of the incorporation of this sebacic acid is difficult to detect from above results since samples of similar hardness are not available. However, an adipic acid-based reference with similar hardness is available (PU4).

When comparing PU2 and PU4, at similar hardness, it can be seen that mechanical properties are almost exactly the same. The differences are very small, and when desired can easily be adjusted for by optimising the polyol-to-prepolymer mix ratio.

Based on the above, it is to be expected that both the EDS and the EDSS formulations will lead to practically feasible elastomers, whereas the EDSS formulation will be most comparable to commonly used adipic acidbased formulations.

The differences in properties that are observed seem to correlate guite well with the expected influence of different density/hardness of the elastomers, except for the stiffness of PU1, which is higher compared to the other formulations (fig. 6).

Fig. 6:

In terms of abrasion resistance, PU1 (EDSbased) seems to outperform the other elastomers, even when compared to the adipate reference at similar hardness (PU3). For PU2 (based on EDSS) it is comparable to the adipate reference.

5.2 Microcellular PU sustainability characteristics

5.2.1 Bio-based carbon content of polyols and elastomers

In this study, bio-based raw materials have only been applied in the polyester polyols.

The use of Biosuccinium succinic acid (in combination with sebacic acid for the EDSS polyol and elastomer) leads to polyester polyols that have a bio-based carbon content as shown in table 5. Mixing this polyol with activator blend (to obtain the A component), and mixing the A component with prepolymer (B component) to obtain the final elastomer, will dilute the bio-based carbon content in the final polyurethane elastomer to 28 % (EDS) and 30 % (EDSS) respectively.

The reduction in carbon footprint of the PU elastomer due to the incorporation of Biosuccinium amounts to ~2.3 kg CO₂-eq/kg PU.

5.2.2 **Opportunities for further** increasing the bio-based carbon content

When also using Biosuccinium-based polyol in the prepolymer, and using renewable diols





and chain extender, the bio-based carbon content of the elastomers can be stepwise increased as indicated below. Bio-based carbon content in final PU elastomer (**fig. 7**):

Bio-polyol in pre-polymer 46 % (+17 %) Bio-EG/DEG in polyester polyol 64 % (+18 %) Bio-BDO as chain extender 70 % (+6 %)

This further increase in the bio-based carbon content of the final elastomer is almost independent of the formulation being used (EDS or EDSS). Furthermore, this further increase in the use of renewable raw materials is already feasible today, since bio-based versions of EG, DEG and BDO are available already on the market.

6. Conclusion

The work done in this paper shows that Biosuccinium is a feasible alternative for (fossilbased) adipic acid as raw material for polyester polyols and polyurethanes. This enables a potential improvement of the sustainability characteristics of polyester polyols and polyurethane materials because Biosuccinium is a 100 % bio-based and renewable raw material. Moreover, Biosuccinium has a much lower carbon footprint [3] than fossilbased adipic acid – about 8 kg of CO_2 -eq/kg of acid – which also leads to a substantial decrease of the carbon footprint of the polyurethanes and products made of it.

The work presented here is intended to be a reference for a first technical evaluation of



use of Biosuccinium in a standard formulation of polyester polyol and microcellular polyurethane elastomers. The differences in processing and properties of the resulting elastomers versus the benchmark, adipic acid-based polyesters and thermoplastic polyurethanes, were identified. No attempt was made in any case to optimise or to improve the products, but this is of course very well possible.

6.1 Polyester polyols

Various polyester polyols, all based on EG/ DEG glycol mixtures, were synthesised using either adipic acid, succinic acid and succinic/sebacic acid blend. In addition, a commercially available adipate reference was obtained.

Synthesis of these polyester polyols of 2,000 g/mol molecular weight were completed without any problems using a standard process for synthesis of adipate polyester polyols. The main difference observed is that the succinate polyol showed some degree of crystallinity, causing a melt temperature (T_m) of approximately 50 °C; in comparison, the other polyols were amorphous and showed no transitions other than T_g . Compared to adipate systems a higher viscosity of the EDS polyol was observed as well for the polyol based on succinic/sebacic acid blend.

6.2 Polyurethane elastomers

6.2.1 Manufacturing of elastomers

The crystallinity of the EDS polyol required a slightly higher processing temperature than typical, but that could be adjusted for without problems. The higher viscosity however led to difficulties in (hand) mixing, and

Tab. 5: Bio-based carbon content in polyester polyol▼and PU elastomer

	EDS formulation	EDSS formulation
Bio-based carbon content in polyester polyol	59 %	63 %
Bio-based carbon content in final PU elastomer	28 %	30 %

should be carefully monitored when using this polyol in commercial production equipment to ensure proper mixing. The EDSS and adipate polyols could be handled without problems.

Reactivity for all formulations was similar, with EDS elastomer being slightly faster than the adipate references.

6.2.2 Properties of elastomers

The achieved densities ranged between 0.47 g/cm³ and 0.60 g/cm³ with the succinate polyols having a higher density and a higher hardness. The hardness of the resulting slabs varied between 37 Shore A and 48 Shore A.

Overall, the physical properties of the elastomers are quite similar. The succinate/sebacate and the adipate-based elastomers are quite similar, the pure succinate elastomers are slightly different on density (higher), hardness (higher), strength and abrasion resistance (both higher).

In general, it is expected that experts in the field will find a technically feasible alternative in Biosuccinium sustainable succinic acid compared to fossil-based raw materials like adipic acid.

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Biosuccinium is a registered trademark for bio-succinic acid from Reverdia.

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