

The method of obtaining linear polyester bio-polyols

National Polish patent number 234556

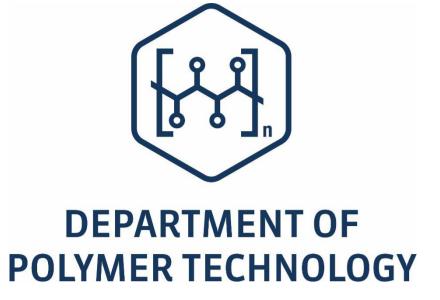
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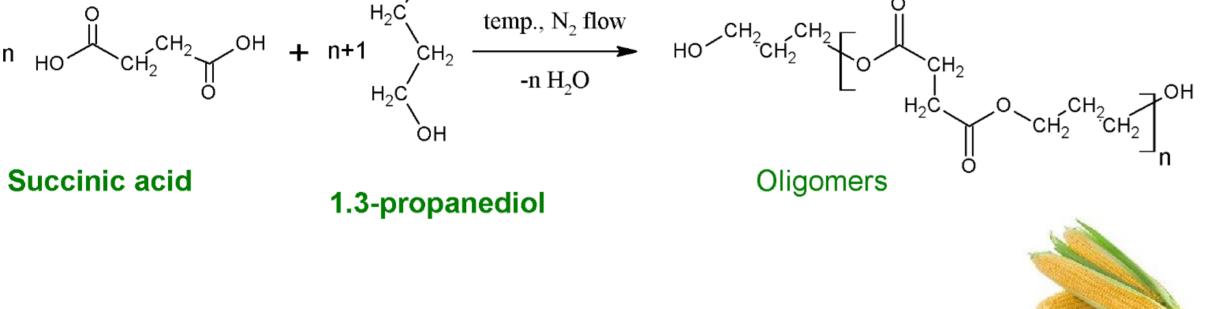


Linear polyester bio-polyols and the method of their preparation

Patent application in the Polish Patent Office No. P.438914

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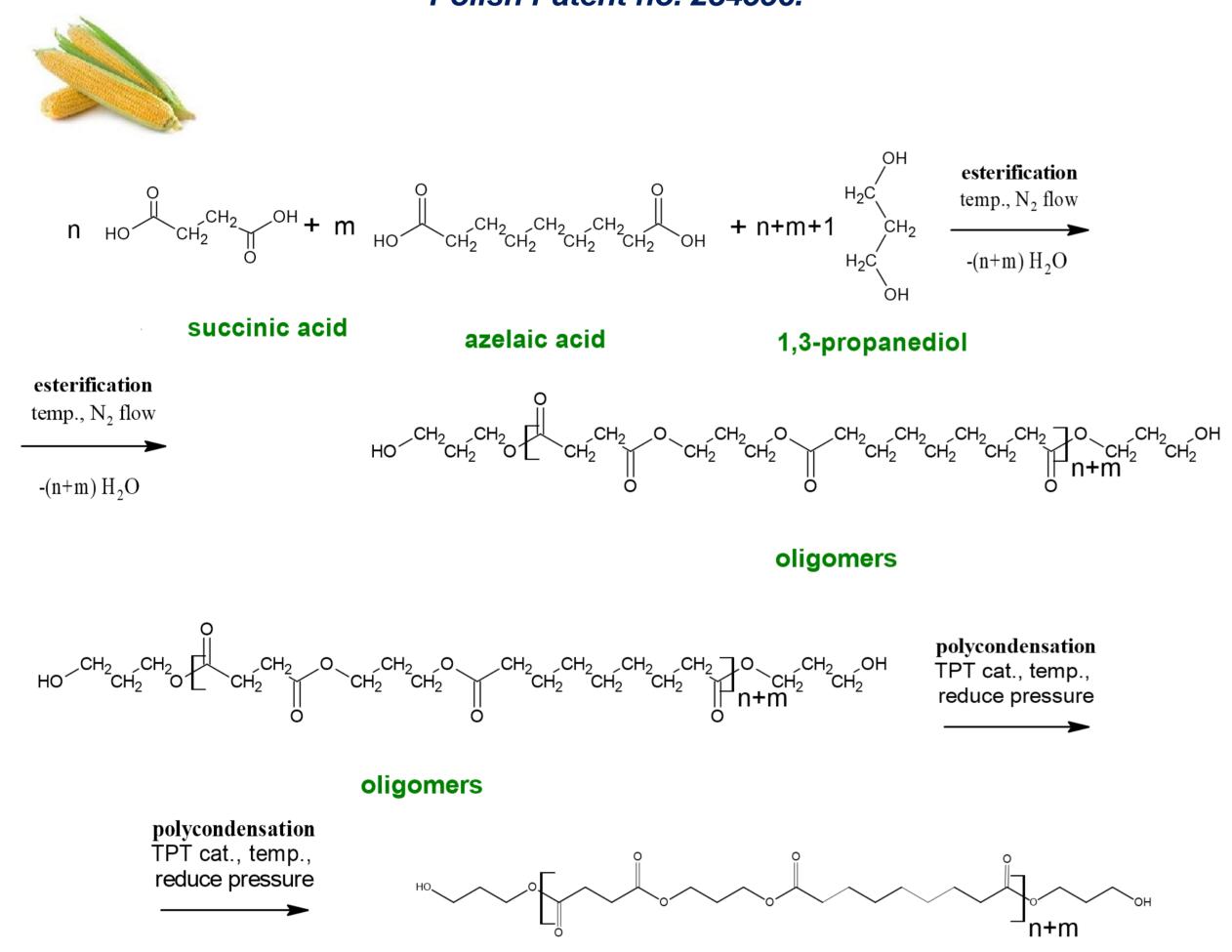


Oligomers

Polycondensation TPT cat., temp., reduce pressure

$$Ho \stackrel{CH_2}{\leftarrow} CH_2 \stackrel{CH_2}{\leftarrow}$$

Fig. 1 Scheme of poly(propylene succinate) polyol obtaining. Polish Patent no. 234556.



poly(proplene siccinate-co-propylene azelate)

Fig. 2 Scheme of poly(propylene succinate-co-propylene azelate) polyol obtaining. Polish Patent Application no. P.438914.

Table 1 Selected properties of the synthesized polyester polyols.

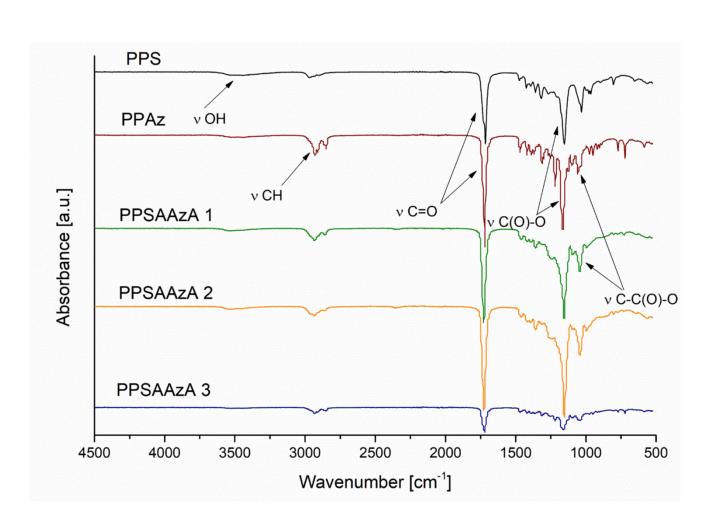
SAMPLE	SA:PDO / SAAAz: PDO	CAT. [wt.%]	Synthesis temperature [°C]		Sa:AAz	Acid number [mg	Hydroxyl number [mg	Viscosity [80°C, Pas]
			I step	II step		KOH/g]	KOH/g]	
PPS	1:1.2	0.25	140	160	-	0.83	52	3.4
PPSAAzA 1					1:1	0.76	148	4.8
PPSAAzA 2					3:1	0.90	128	6.2
PPSAAzA 3					1:3	0.92	120	5.7

Table 3 Termal decomposition characteristic of the poly(ester-urethane) elastomers.

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Bio-based TPU		Thermal decomposition characteristics					
		T _{5%} [°C]	T _{50%} [°C]	T _{max} [°C] [Istep / II step]	Residue at 600°C [%]		
E1 (PPS_MDI)	BDO	319.0	404.0	384.0 / 426.1	7.8		
	PDO	326.4	408.0	383.5 / 427.4	9.9		
K1 (PPS_MDI)	BDO	322.5	400.0	381.1 / 423.1	10.8		
	PDO	322.7	408.8	385.0 / 426.1	11.0		

The subject of the inventions is a method of obtaining linear polyester bio-polyols using monomers of natural origin. The invention is applicable in the plastics industry for the production of various types of polyurethanes, e.g. flexible foams, elastomers, thermoplastic elastomers, paints, varnishes, adhesives etc.

The synthesis of polyols from renewable substances as an alternative for petrochemical-based polyols plays important matter in the polyurethane industry. The polyester polyols with 100 % bio-carbon content were synthesized with the use of succinic acid (SA), azelaic acid (AzA), 1,3-propanediol (PDO) and 1,4-butanediol (BDO) all with natural origin. The syntheses were conducted via polycondensation reaction to obtain products with planned chemical structure, average molecular weight and functionality, which indicate their application in polyurethane materials. The obtained bio-based polyester polyols revealed similar properties to the commercially used petrochemical-based counterpart. Bio-based polyurethane materials were also synthesized. Their selected properties confirmed similarities to commercially used petrochemical-based polyurethane materials.



-5OLD
-10OLD
-11OLD
-11-

Fig. 3 FTIR results of the synthesized polyester polyols and poly(ester-urethane) prepolymer and elastomers.

Fig. 4 DTG results of the obtained polyester polyols.













Table 2 Thermal decomposition characteristic of the synthesized polyester polyols.

POLIOL	Themal degradation characteristics						
	T _{2%} [°C]	T _{5%} [°C]	T _{50%} [°C]	T _{max} [°C]	Char residue at 800°C [%]		
PPS	194,7	251,3	394,2	402,4	1,66		
PPAz	212,1	272,2	412,4	414,0	2,33		
PPSAz 1 (1:1)	195,6	257,2	405,3	415,8	1,73		
PPSAz 2 (3:1)	193,2	251,6	399,7	408,5	1,53		
PPSAz 3 (1:3)	213,6	277,0	409,6	416,0	2,36		

Table 4 Selected mechanical properties of the synthesized poly(ester-urethane) elastomers.

		[NCO]/[OH] = 1.00 [-]				
Thermoplastic polyurethane elastomer		Tensile strength [MPa]	Relative elongation at break [%]	Permanent elongation after breaking [%]	Hardness [°Sh D]	
PU_PPS _MDI	BDO	17,4±1,3	438±17	64±5	39,7±3,3	
	PDO	21,4±3,2	521±57	84±4	39,2±1,7	
PU_POLIOS	BDO	37,5±1,3	656±20	44±4	41,7±0,3	
55/20_MDI	PDO	41,4±0,5	651±9	39±1	40,0±2,6	