TECHNICAL BULLETIN

Susterra™ Propanediol

A Renewable Resource for Unsaturated Polyester Resins

WILMINGTON, Del., July 9, 2008 – Opportunities for Susterra™ Propanediol (1,3-propanediol) in unsaturated polyester resins (UPR's) can be derived from a technical value proposition and physical / mechanical tests as presented in this study. Susterra renewably-sourced propanediol from DuPont Tate & Lyle Bio Products is a 100-percent renewably sourced ingredient made from corn sugar.

Formulation experiments led to a general recommended formulation window for Susterra in UPR's. Less reactive resins (PA/MA = 1.5/1.0) may be based on Susterra alone. In resins of intermediate or high reactivity (PA/MA = 1.0/1.0 to 1.0/1.5), about 20-40 wt% 1,2-propanediol (1,2-PG) on total diol is necessary to keep the UPR/styrene products non-crystalline and shelf stable. This corresponds to a level of 10-25 wt% Susterra in the final thermoset product. The exact level depends on the specific recipe, including types and levels of glycols and phthalic fragments.

Three recipes were selected for reactivity studies (60 wt% in styrene), and physical and mechanical property testing. Two recipes have a PA/MA ratio of 1.5 (Table 1). In these two recipes, Susterra and 1,2-PG are compared directly (UPR-Susterra) and (UPR-1,2-PG). A third recipe (UPR-80/20 mix) is more reactive (PA/MA = 1) and contains a mixture of diols (Susterra/1,2-PG = 8/2). Complimentary properties of these three recipes point towards unique opportunities and applications of Susterra, and are summarized as follows:

Processing of UPR's: Susterra vs. 1,2-PG

- The higher boiling point of Susterra gives production benefits such as shorter batch times or less loss of glycol.
- Susterra slows down the viscosity build of the UPR polymer during synthesis.

- Susterra decreases the isomerization rate of maleic units in the UPR. This effect is amended by additives or procedural optimizations. Thus, negative impact on end-use properties (e.g.; hydrolytic stability) is prevented.
- Susterra lowers the glass transition T_g of the non-crosslinked UPR to a value well below room temperature, rendering the Susterra based UPR sticky, rather than glassy.
- No differences in viscosity have been observed between UPR's based on Susterra or 1,2-PG at 60 wt% in methylcellosolve or styrene.
- Susterra slightly reduces the SPI gel and cure times and increases the peak exotherm.

<u>End-use properties of UPR's: Susterra and 1,2-PG</u>

- Susterra gives crosslinked UPR casts that are colorless (no yellowing observed) and clear.
- The glass transitions (T_g's) of Susterra and 1,2-PG based UPR's are similar. However, 1,2-PG containing UPR's show a small melt transition, absent in UPR-Susterra.
- No differences in the Barcol hardness of UPR-1,2-PG, -Susterra, and -80/20 mix were observed
- Susterra lowers the heat deflection temperature of a UPR/styrene thermoset.
- Susterra lowers the tensile modulus, while corresponding thermosets show higher values for tensile strength and elongation.
- Similarly, Susterra lowers the flexural modulus, while a higher value for flexural strength is obtained.
- Susterra lowers the compressive strength of a UPR/styrene material.
- Susterra slightly decreases the stiffness and increases the flexibility of a cross linked UPR without being detrimental to other mechanical properties.

Table 1: Overview of UPR Processing Properties

Property	Unit	UPR Description		
		UPR-1,2-PG	UPR-Susterra	UPR-80/20 mix
Recipe				
PA	mole %	1.2	1.2	1.0
MA	mole %	0.8	0.8	1.0
Susterra	mole %	0	2.2	1.76
PG	mole %	2.2	0	0.44
Analysis				
Acid Number	mg_{KOH}/g	12	10	11
Molecular Weight M_n	g/mol	2285	2554	4572
Molecular Weight M_w	g/mol	8094	9454	15088
Polydispersity DP		3.5	3.7	3.3
Isomerization	%	96	91	90
Glass Transition T_g of UPR	°C	33	0.25	6.9
Viscosity, 60 wt% in methylcellosolve	cP	366 ± 2.7	375 ± 2.2	774 ± 5.4
Reactivity at 60 wt% in styrene				
Solids Level	wt%	61.0	59.9	59.7
Garner Holdt Viscosity	-	P	P^2-Q	P-Q
Viscosity	cP	316 ± 2.6	358 ± 2.5	660 ± 4.9
SPI gel time	min:sec	3:47 ± 0:08	3:39 ± 0:03	3:08 ± 0:06
SPI cure time	min:sec	3:19 ± 0:02	2:24 ± 0:04	1:45 ± 0:03
SPI total time	min:sec	7:06 ± 0:08	6:03 ± 0:01	4:53 ± 0:03
SPI exotherm	°C	195.1 ± 2.1	215.2 ± 0.2	230.6 ± 0.6

Table 2: Overview of UPR End-Use Properties

Property	Unit	UPR Description		
		UPR-1,2-PG	UPR-Susterra	UPR-80/20 mix
Recipe				
PA	mole eq.	1.2	1.2	1.0
MA	mole eq.	0.8	0.8	1.0
Susterra	mole eq.	0	2.2	1.76
PG	mole eq.	2.2	0	0.44
Physical Properties (crosslinked at 60 wt% with styrene using 1 wt% BPO crystals)				
Yellowness Index YI	-	5.97 ± 0.10	1.61 ± 0.10	2.10 ± 0.16
mp and ΔH	°C , J/g	-44.1, 0.090	none observed	-45.0, 0.087
Glass Transition T_g	°C	-19.7, 86.3	-19.3, 85.1	-19.4, 110.8
Mechanical Properties (crosslinked at 60 wt% with styrene using 1 wt% BPO crystals)				
Barcol Hardness	-	64.9 ± 1.5	63.8 ± 1.8	65.0 ± 1.4
Heat Deflection Temperature	°C	75.2 ± 0.42	67.2 ± 0.90	91.4 ± 0.71
Tensile strength	MPa*	44.4 ± 2.8	70.0 ± 2.2	50.7 ± 5.9
Tensile modulus	MPa	3,866 ± 341	3,223 ± 282	3,810 ± 200
Elongation	%	1.21 ± 0.16	2.88 ± 0.20	1.57 ± 0.26
Flexural strength	MPa	67.1 ± 5.6	112.7 ± 6.5	95.9 ± 7.4
Flexural modulus	MPa	4,161 ± 164	3,548 ± 177	3,638 ± 187
Compressive strength	MPa	131.3 ± 4.2	103.7 ± 3.3	112.7 ± 1.5
Hydrolytic Stability		Only preliminary experiment performed		

* 1 MPa = 1.45 kpsi

DuPont Tate & Lyle Bio Products is a joint venture between DuPont, a global science company, and Tate & Lyle, a world leader in corn, wheat and sugar derived ingredients. By bringing together the unrivaled track record of DuPont in the integration of biology, engineering and materials science, and the technical excellence of Tate & Lyle in fermentation of natural products, scientists and engineers from the joint venture have developed a process that uses corn instead of petroleum-based feedstocks to produce 1,3 propanediol, or Bio-PDO. DuPont Tate & Lyle Bio Products provides renewably sourced ingredients that do not compromise product performance.

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