

Development of High Performance Polyether Based Elastomers

Rui Xie

*The Dow Chemical Company
2301 N. Brazosport Blvd.
Freeport, TX 77541*

Laura Grier

*The Dow Chemical Company
2301 N. Brazosport Blvd.
Freeport, TX 77541*

Ben Buckley

*Dow Chemical Service UK Ltd
Station Road
Birch Vale, EN SK 22 1BR, UK*

Nita Xu

*The Dow Chemical Company
2301 N. Brazosport Blvd.
Freeport, TX 77541*

Gareth Faulkner

*Dow Chemical Service UK Ltd
Station Road
Birch Vale, EN SK 22 1BR, UK*

Andrew Davies

*Dow Chemical Service UK Ltd
Station Road
Birch Vale, EN SK 22 1BR, UK*

Rajat Duggal

*The Dow Chemical Company
2301 N. Brazosport Blvd.
Freeport, TX 77541*

ABSTRACT

Polyether polyols, and specially Polytetramethylene Ether Glycol (PTMEG), are widely used in the development of high performance elastomers. However, PTMEG polyols with molecular weight greater than 1000 are solid at room temperature, making them difficult to handle. Because of that, they are not suitable for applications that require low viscosity at the processing temperature, such as coatings. On the other hand, conventional polypropylene glycols (PPG) often suffer from low reactivity and poor mechanical strength due to high level of monol content. Over the last few years, Dow Polyurethanes has developed a new range of high performance polyether polyols. In this paper, we discuss and present new elastomer systems based on the new range of polyether polyols. Dow believes these elastomer systems have processing characteristics superior to PTMEG based systems, and improved mechanical strength and dynamic performance compared to conventional PPG based elastomers. As a result, we believe these elastomer systems will fulfill the need for a polyether elastomer system that possesses low viscosity as well as excellent mechanical strength.

INTRODUCTION

Polyols available for polyurethane elastomer synthesis include polyesters, polyethers, polycarbonates, polycaprolactones, and other hydroxyl and amine terminated compounds, such as hydroxyl and amine terminated polybutadiene. The most commonly used polyols are polyether or polyester polyols of molecular weight from 400 to 5000. Polyether polyurethanes exhibit a relatively high resistance to hydrolytic cleavage, improved resilience, better processing characteristics due to lower viscosity, and superior dynamic performance when compared with polyester polyurethanes. The polyether that results in a polyurethane with the best physical and dynamic properties is poly(tetramethylene ether glycol) (PTMEG). Urethanes prepared with this soft segment show a level of mechanical strength comparable to that of polyester polyurethanes and possess better hydrolytic stability, higher resilience, and improved dynamic performance [1]. These improvements are attributed to the linear backbone of repeating tetramethylene units connected by ether linkages that enables strain-induced crystallization in the soft segment. The primary hydroxyl functional groups and exact functionality of the PTMEG polyols

impart good reactivity and polymer network formation with isocyanates. However, PTMEG polyols with molecular weight greater than 1000 are solid or semi-solid at room temperature. As a result, PTMEG is limited in some room temperature applications, such as coating and is not cost effective in some applications.

Polyether polyols such as polypropylene glycol (PPG) offer superior hydrolysis resistance than polyester polyols but exhibit inferior mechanical properties. The loss of mechanical properties in conventional PPG-based polyurethanes for coatings, adhesives, sealants, and elastomers (CASE) compared to PTMEG-based systems is attributed to a lack of strain-induced crystallization in soft segments comprised of PPG, and the presence of unsaturation at chain ends in conventional PPG polyols, leading to reduced network formation. Conventional polypropylene glycols are produced via the base catalyzed propoxylation of glycol initiators such as propylene glycol. However, base catalyzes not only the addition of propylene oxide to the growing polyol molecule, but also a side reaction where propylene oxide isomerizes into allyl alcohol [2, 3, 4]. Allyl alcohol acts as a monofunctional starter resulting in propoxylated allyl alcohol referred as “monol”. This reaction is illustrated in Figure 1. In conventional PPG polyols, the level of unsaturation or monol content increases dramatically with increasing polyol molecular weight. For example, a typical 2000 Mw conventional PPG typically has an unsaturation level of 0.03 meq/gm, which corresponding to a functionality of 1.94, while a 4000 Mw diol with an unsaturation level of 0.085 would have a functionality of 1.69. In other words, a conventional 4000 Mw diol is a blend of about 70% diol and 30% monol on a mole percent basis [2]. Moreover, conventional PPG polyols possess significant amount of secondary hydroxyl groups. Primary hydroxyl groups react readily with isocyanates at mild temperatures, while secondary and tertiary hydroxyl groups are less reactive than primary hydroxyl groups by a factor of 0.3 and 0.005 respectively [5]. Slower reactivity of the secondary hydroxyl groups in PPG polyols makes this class of polyols more sensitive to trace amount of moisture in the chemicals as well as in the ambient environment.

Clearly, there is a need to develop a class of cost effective polyether polyols with low viscosity, good functionality, and reactivity. Over the past several years, Dow Polyurethanes has devoted significant effort in the development of a new class of high performance polyether polyols that can meet the unmet needs in the market place. In this paper, we discuss and present new elastomer systems based on the newly developed polyether polyols. Dow believes these elastomer systems have processing characteristics superior to PTMEG based systems, and improved mechanical strength and dynamic performance compared to conventional PPG based elastomers.

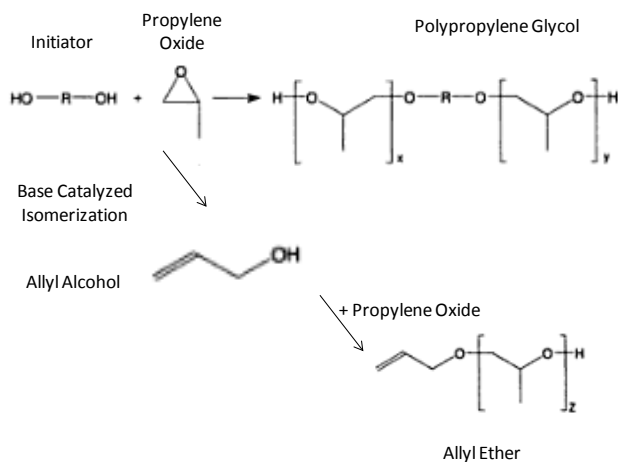


Figure 1. Isomerization of propylene oxide in base catalyzed propoxylation of glycol imitators to form diols and monol.

EXPERIMENTAL

An aromatic diamine, Aromatic Amine 300, was supplied by Albemarle Corporation. 1,4 butanediol was obtained from Aldrich. Polytetramethylene Ether Glycol (PTMEG) polyols were supplied by Invista. Toluene diisocyanate, TDI100 containing more than 98% of the 2,4 isomer and TDI80 containing 80% 2,4 isomer were supplied by Bayer Corporation. Other raw materials, such as Isonate® 125M, a 4,4-methylene diphenyl diisocyanate were supplied by the Dow Chemical Company.

Elastomer specimens were prepared via a two-step prepolymer process. Polyols were reacted with isocyanates first to produce the prepolymers. All the reactions were conducted in a 4-neck glass reactor under conditions similar to the state of the art. The extent of the reaction was determined by an isocyanate equivalent method. The NCO contents of the prepolymers are listed in the Table 1. The prepolymers were further degassed at 70°C under vacuum after the reaction was completed. The prepolymers were then mixed with the chain extenders, such as Aromatic Amine 300 or 1,4 butanediol using a SpeedMixer DAC 600 FVZ made by Hausechild, Germany at 2000 rpm for at least 1.5 minutes before cast into an aluminum mold preheated at 100°C. After post-cured at 100°C for 16 hours, the specimen were demolded and kept at room temperature for at least 2 weeks before they were subjected to further characterizations.

In the preparation of HYPERLAST™ 301 elastomers, both the isocyanateside and polyol side were preconditioned at 40°C before mixed with the SpeedMixer DAC 600FVZ. The mixtures were poured into an aluminum mold preheated at 80°C, and postcured at 80°C for 16 hours. The specimen was characterized after aged at room temperature for 2 weeks.

The stress-strain properties of the elastomer specimens were measured according to ASTM D412, and tear resistance of the elastomer samples were measured by ASTM D 624. Viscosity of the prepolymer samples was measured by a TA Instrument AR 2000 with a temperature ramp rate at 3°C/min.

RESULTS AND DISCUSSIONS

1. Processing Characteristics of Prepolymers based on the New High Performance Polyols

Polyether polyol based prepolymers, such as those based on polypropylene glycols often exhibit low viscosity at processing temperatures. As a result, they are advantageous comparing to polyester based prepolymers in some applications, such as spray coatings. To understand the processing characteristics of the prepolymers based on the new class of high performance polyols, viscosity of these prepolymers was evaluated side-by-side against the benchmarks based on PTMEG and PPG polyols. Table 1 shows typical characteristics of TDI prepolymers based on the new high performance polyols and the conventional PTMEG and PPG polyols. It is evident that at similar NCO content, the prepolymers based on the new high performance polyols have achieved similar free TDI content, while possessing much lower viscosity comparing to the prepolymers based on PTMEG or PTMEG/PPG blends. On average, a 40% to 75% reduction in viscosity can be achieved by utilizing the new high performance polyols. At similar NCO content and free MDI content, the prepolymers based on the new polyols exhibit much lower viscosity comparing to the MDI PTMEG based prepolymers. On average, about 50% reduction in viscosity was observed for the MDI prepolymers based on the new polyols, as evident in Table 2.

Table 1. Characteristics of TDI based Prepolymers Evaluated in the Study

	95 Shore A Prepolymers		80 Shore A Prepolymers		85 Shore A Prepolymers		82 Shore A Prepolymer	
	Benchmark	HP Polyether Prepolymer	Benchmark	HP Polyether Prepolymer	Benchmark	HP Polyether Prepolymer	Benchmark	HP Polyether Prepolymer
Diisocyanate	TDI100	TDI100	TDI80	TDI80	TDI100	TDI80	TDI100	TDI100
Polyols	PTMEG 1000	New HP Polyols	PTMEG 2000	New HP Polyols	PTMEG 1000/PTMEG 2000 Blend	New HP Polyols	PTMEG 2000/PPG blend	New HP Polyols
NCO	6.20%	6.24%	3.20%	3.40%	3.70%	3.80%	3.30%	3.75%
Free TDI, %	<0.8%	<0.8%	<0.7%	<0.7%	<0.9%	<0.7%	<0.8%	<1.0%
Viscosity @ 50°C, cPS	3300	2000	6000	3000	4800	3150	1300 @ 80°C	300 @ 80°C

Table 2. Characteristics of MDI based Prepolymers Evaluated in the Study

	90 Shore A Prepolymers		85 Shore A Prepolymers	
	Benchmark	HP Polyether Prepolymer	Benchmark	HP Polyether Prepolymer
Diisocyanate	Isonate 125M	Isonate 125M	Isonate 125M	Isonate 125M
Polyols	PTMEG 2000	New HP Polyols	PTMEG 2000	New HP Polyols
NCO	8.20%	8.40%	6.60%	6.60%
*Free MDI, %	<25%	<25%	<15%	<15%
Viscosity @ 75°C, cPS	1800	800	2500	1000

*Estimated by theoretical calculation.

Figure 2 shows the viscosity curve of the 95 shore A prepolymers and the 85 shore A prepolymers based on PTMEG and the new polyols. Clearly, the new polyol based prepolymers demonstrated significant lower viscosity across the processing window of 25°C to 110°C. The data suggest that prepolymers based on the new polyols may be more advantageous in applications that lower viscosity is needed, such as coatings. Lower viscosity can be advantageous in hot casting applications as well because a lower viscosity allows the system to be processed at lower temperature, therefore, extend the pot life and improve consistency of the casting.

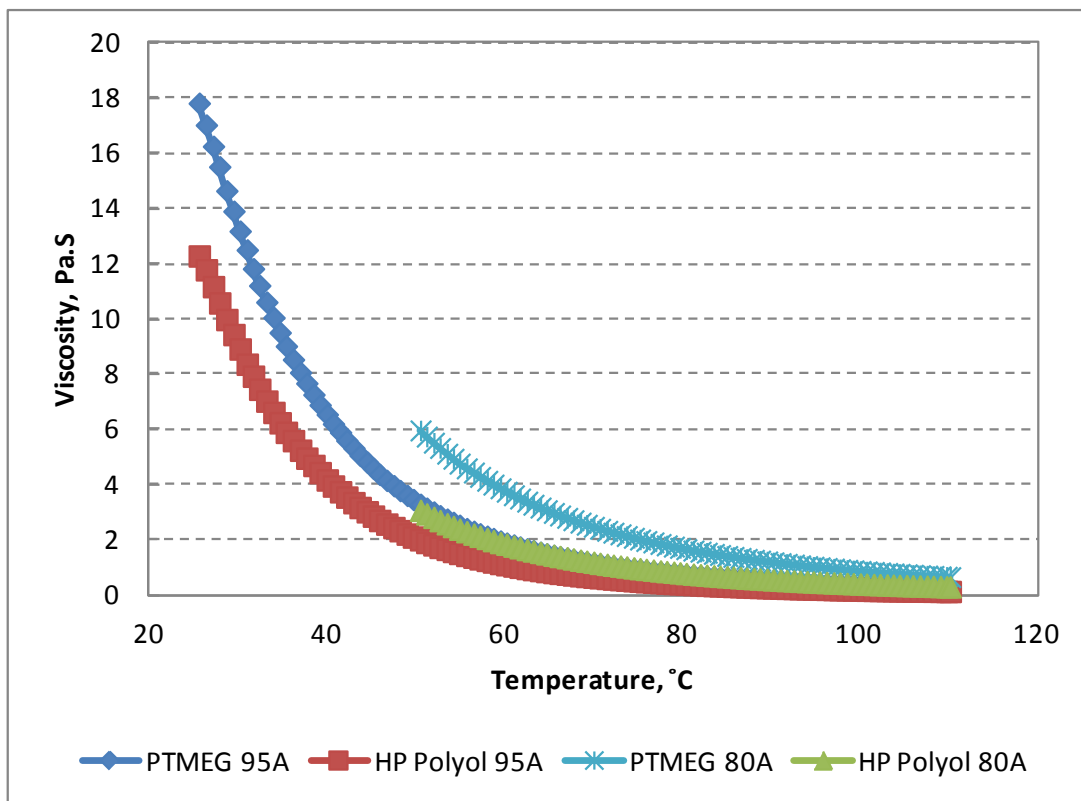


Figure 2. Viscosity of the benchmark 80A and 95A TDI prepolymers versus viscosity of the 80A and 95A TDI prepolymers based on the high performance polyether polyols.

2. Mechanical Properties of Elastomers based on the New Polyols

2.1 Full TDI Prepolymer Based Elastomers

Table 3 shows side by side studies of elastomers based on TDI prepolymers of PTMEG and the new high performance polyols. Mechanical properties of elastomers at 95 Shore A, 85 Shore A, and 80 Shore A were evaluated. In order to have an apples-to-apples comparison, NCO content of prepolymers was kept the same (within the error of the NCO measurement) for the elastomers of the same hardness, and Aromatic Amine 300 was used as the chain extenders in preparation of all the elastomers. As a result, any performance difference exhibited between the two elastomers at the same hardness, one based on PTMEG and the other one based on the new polyols, can only be attributed to the performance attributes of the polyols. At 95 Shore A hardness, the elastomer based on the new polyol exhibited higher elongation at break, but slightly lower tensile strength, tear strength, resilience, and slightly higher compression set compared to the PTMEG based elastomer. Nevertheless, the difference between the two elastomers is insignificant. At 80 Shore A hardness, the elastomer based on the new polyol exhibited similar elongation and 100% and 300% modulus (stress), but slightly lower tensile strength, resilience, and slightly higher compression set. Again, the difference in performance between the two elastomers is insignificant. In the case of the 85 shore A elastomer, except the slightly lower resilience demonstrated by the elastomer based on the new polyol, other properties were very similar. The results clearly indicate that by way of formulating, one can achieve mechanical properties similar to PTMEG based elastomers using the new class of high performance polyether polyols. In addition to the excellent mechanical properties, Dow believes these prepolymers demonstrate enhanced processing attributes over PTMEG based prepolymers, such as lower viscosity as discussed in section 1.

Table 4 shows side by side study of elastomers based on TDI prepolymers of the new polyol and a blend of PTMEG and polypropylene glycol (PPG). The blend contained about 20wt% of a difunctional polypropylene glycol. Both prepolymers reached a hardness about 80 shore A when chain extended by Aromatic Amine 300. However, the elastomer based on the new polyols demonstrated better stress-strain properties, such as elongation and tensile strength, while maintaining similar tear strength and resilience comparing to the elastomer based on PTMEG and PPG blend. The lower stress strain properties of the elastomer based on the PTMEG and PPG blend may be attributed to the unsaturation (monol content) of the conventional polypropylene glycol. The results suggest that the new polyols can be advantageous when compared to the popular approach of blending PTMEG and PPG for improved cost performance ratio.

Table 3. Mechanical Properties of Elastomers based on TDI Prepolymers of PTMEG and the High Performance Polyols

	PTMEG 95A	HP Polyol 95A	PTMEG 80A	HP Polyol 80A	PTMEG 85A	HP Polyol 85A
NCO, %	6.0	6.1	3.2	3.4	3.7	3.8
Curative	Aromatic Amine 300	Aromatic Amine 300	Aromatic Amine 300	Aromatic Amine 300	Aromatic Amine 300	Aromatic Amine 300
Hardness, A	95	95	82	83	85	87
Tensile Strength, psi	6165	5430	4500	3900	5020	5040
Elongation, %	415	506	645	650	645	720
100% Modulus, psi	2015	1965	815	840	910	1080
300% Modulus, psi			1570	1420	1650	1700
Tear Strength, pli (ASTM D624)	660	560	385	400	450	465
Bashore rebound, %	43	35	64	59	64	51
Compression set, ASTM D395 Method B	36	40	27	32	36	36

Table 4. Mechanical Properties of Elastomers Based on TDI Prepolymers of PTMEG/PPG Blend and the New High Performance Polyols

	PTMEG/PPG blend 80A	New Polyol
NCO, %	3.3	3.75
Curative	Aromatic Amine 300	Aromatic Amine 300
Hardness, A	80	82
Tensile Strength, psi	2755	3080
Elongation, %	790	1000
100% Modulus, psi	490	780
Tear Strength, pli (ASTM D624)	388	370
Bashore Rebound, %	67	65

2.2 Full MDI Prepolymer Based Elastomers

Table 5 shows mechanical properties of elastomers based on MDI prepolymers of PTMEG and the new high performance polyols. Mechanical properties of elastomers at 90 Shore A and 85 Shore A were evaluated. NCO content of prepolymers was kept the same (within the error of the NCO measurement) for the elastomers with similar hardness. 1,4 butanediol was used as the chain extenders for all the elastomers. At 8.2% NCO content, the MDI prepolymer based on the new polyol yielded a 88 Shore A elastomer, slightly lower than the prepolymer based on PTMEG. However, the elastomer demonstrated similar elongation at break, tear resistance, and 100% modulus (stress). Lower tensile strength and resilience were observed. Both of the 6.6% NCO prepolymers yielded elastomers at 85 Shore A hardness. The elastomer based on the new polyols exhibited similar elongation at break, resilience, and 100% modulus, but slightly lower tensile strength and tear strength. The data suggest that by way of formulating, it is possible to achieve a high level of performance close to that of PTMEG based elastomers via employing the new class of polyols.

Table 5. Mechanical Properties of Elastomers based on MDI Prepolymers of PTMEG and the High Performance Polyols

	PTMEG 90A	HP Polyol 90A	PTMEG 85A	HP Polyol 85A
NCO, %	8.2	8.2	6.6	6.6
Curative	BDO	BDO	BDO	BDO
Hardness, A	90	88	85	85
Tensile Strength, psi	5100	4445	4800	4165
Elongation, %	500	600	540	550
100% Modulus, psi	1110	1220	830	900
300% Modulus, psi		2235		1965
Tear Strength, pli (ASTM D624)	540	530	500	393
Bashore Rebound, %	64	48	70	67

2.3 Quasi MDI System --HYPERLAST™ 301 System

Table 6 shows typical physical performance of the HYPERLAST™ 301, a MDI quasi system based on the new polyols at 90, 85, and 70 Shore A. The elastomers demonstrate good stress strain properties, excellent tear strength, and outstanding abrasion resistance. These performance enhancements, in addition to the processing advantages, such as low viscosity, low temperature casting and cure, and convenience of achieving a wide hardness range (70A to 95A) with a single prepolymer make HYPERLAST™ 301 a good choice for general castings of a variety of applications. These applications can include wheels, rollers, mining screens, mechanical parts, agricultural parts, and replacement of other materials, such as rubber in industrial applications.

Table 6. Typical Physical Properties of HYPERLAST™ 301

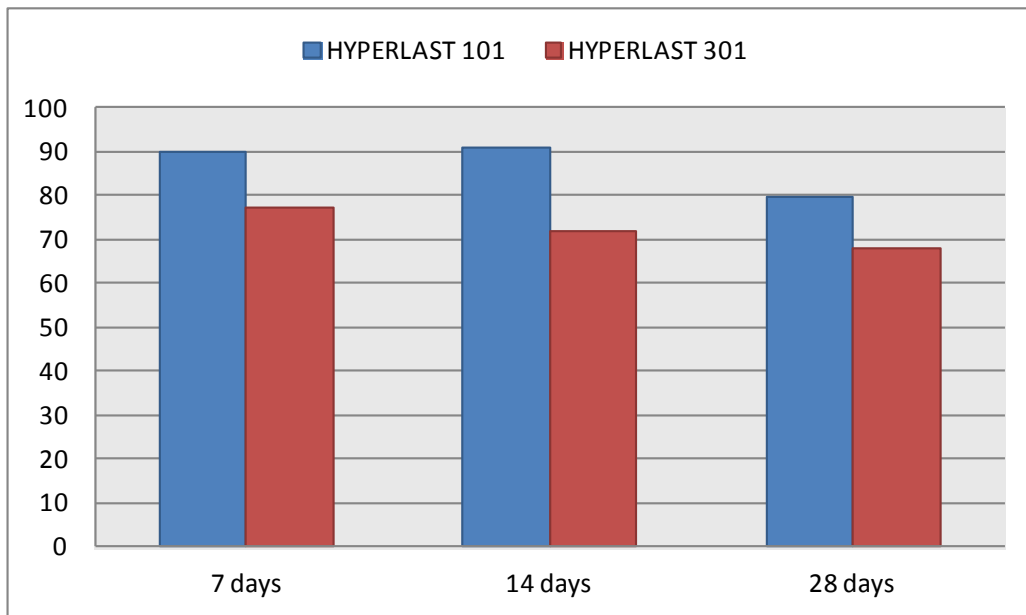
Hardness, Shore A	90	85	70
Mixing Ratio, polyol/Iso by weight	23.8/100	34.1/100	62.5/100
100%, Modulus, psi (BS 903/A)	1116	826	478
Tensile Strength, psi (BS 903/A)	4200	3200	3200
Elongation @ break, % (BS 903A)	483	678	691
Tear Strength, pli (BS 903 Pt A3)	525	440	320
Abrasion, mm (DIN 53516)	59	79	67
Resilience, % (Luke Pendulum)	32	44	49
Gel time, minutes	4	4	4.5

Table 7 shows performance comparison of HYPERLAST™ 301 to HYPERLAST™ 101, a PTMEG based system, and HYPERLAST™ 201, a conventional PPG based system at 85 Shore A hardness. Comparing to HYPERLAST™ 201™, HYPERLAST™ 301 demonstrates improved stress-strain properties, superior tear strength, abrasion resistance, and resilience. While its tensile strength, resilience, and abrasion resistance are lower than HYPERLAST™ 101, the difference is relatively small. Tear strength of the system is similar to HYPERLAST™ 10. Clearly, from mechanical property point of view, HYPERLAST™ 301 represents a significant improvement over conventional PPG based systems, and a closer match to the performance of PTMEG based systems.

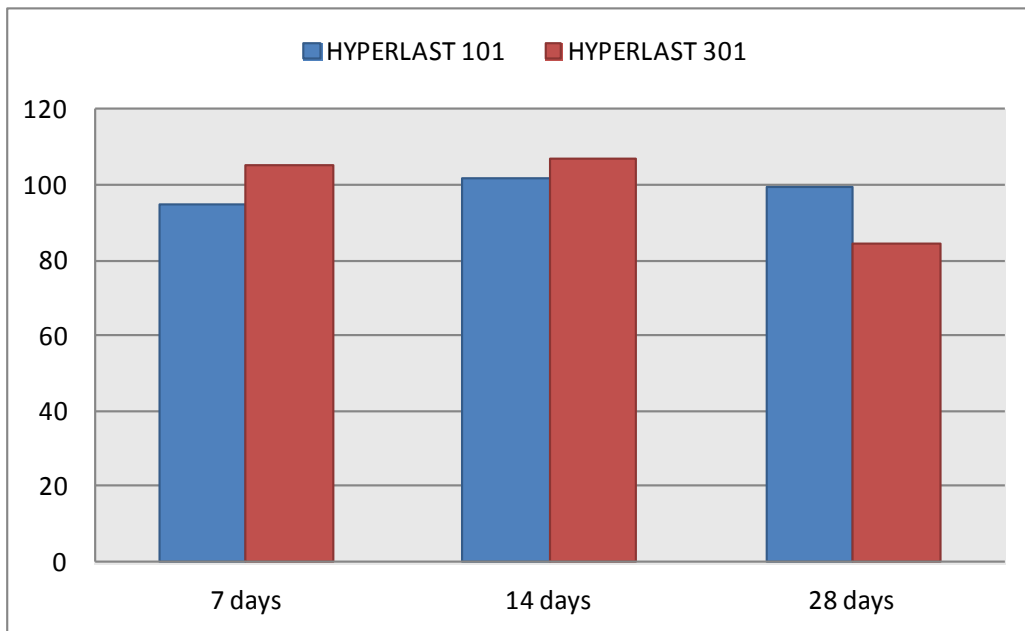
Table 7. Comparison of Typical Physical Properties of HYPERLAST™ 301, 201 and 101 at 85 Shore A Hardness

	HYPERLAST™ 101	HYPERLAST™ 201	HYPERLAST™ 301
Hardness, Shore A	85	85	85
Polyol Type	PTMEG	PPG	New Polyol
Mixing Ratio, polyol/Iso by weight	82/100	21/100	34/100
100%, Modulus, psi (BS 903/A)	899	725	826
Tensile Strength, psi (BS 903/A)	4930	1885	3200
Elongation @ break, % (BS 903A)	450	325	678
Tear Strength, pli (BS 903 Pt A3)	457	285	440
Abrasion, mm (DIN 53516)	45		79
Resilience, % (Luke Pendulum)	50		44
Gel time, minutes	5	8	4

Figure 3 shows the hydrolytic stability of elastomers based on HYPERLAST™ 101 and HYPERLAST™ 301. Specimens of both elastomers were aged in water at 70C for a given period of time before their stress-strain properties were measured. HYPERLAST™ 101 demonstrates better hydrolytic stability than HYPERLAST™ 301, as evident by retention of the tensile properties after aging. However, the difference is not large, and it can be attributed to the lower hardness of the HYPERLAST™ 301 specimen. Hardness of the HYPERLAST™ 101 elastomer was at 95 Shore A, and that of the HYPERLAST™ 301 elastomer was at 80 Shore A. Properties of a softer elastomer are more prone to hydrolysis comparing to a harder elastomer based on the same backbones, as widely reported by others.



Remaining Tensile Strength over Time



Remaining Elongation over Time

Figure 3. Remaining tensile strength and elongation of HYPERLAST™ 301 and HYPERLAST™ 101 after aging in water at 70C for extended period of time. Y axis shows remaining properties in percentage. Hardness of the HYPERLAST™ 101 elastomer was at 95 Shore A, and hardness of the HYPERLAST™ 301 elastomer was at 80 Shore A.

CONCLUSIONS

In this paper, we have demonstrated by way of formulating, one can utilize a new class of polyols manufactured by the Dow Chemical Company to develop TDI and MDI prepolymer compositions with improved processing characteristics, such as lower viscosity, while achieving a performance level close to the PTMEG elastomers over a wide hardness range. The lower viscosity of these prepolymers can improve flow of the systems, therefore, minimize defects and improve quality consistency. These performance attributes open up possibilities to use this new class of polyols in replacement of PTMEG polyols for conventional casting and coating applications, especially those applications where improved processing characteristics are required, such as spray coatings, room temperature castings, and casting of large intricate parts that requires low viscosity and extended pot life.

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BIOGRAPHIES

Rui Xie

Rui Xie is a Technical Leader for the Laminating Adhesives business in the Dow Chemical Company. In this role, Rui is responsible for developing the global R&D pipelines and driving innovation growth for the Business. Rui joined Dow in 2001, and has taken a number of technical roles with several Dow businesses since then. Rui earned a Ph.D. in Polymer Science from Chinese Academy of Sciences, China in 1994. He has published over 30 papers in peer reviewed journals on polyurethane, epoxy, and structure-property of polymers. Rui is a regular contributor to a number of industry technical conferences. He has over 30 conference papers/presentations over the years. Rui has authored/co-authored 15 patents/patent applications.

Laura Grier

Mrs. Laura Grier is currently a Technologist Leader in the Elastomers R&D group in Dow Polyurethanes. Laura has 33 years of experience within R&D and is currently responsible for developing cast and spray elastomer systems. She has 7 external publications and has authored/co-authored 8 patent applications.

Ben Buckley

Mr. Ben Buckley is a Senior Technical Service Chemist for Dow Polyurethanes in EMEA. With 11 years of experience within R&D and Technical Service in the Polyurethane Business, he is currently responsible for the technical service and development activities for Engineering Elastomers in the industrial market segment.

Nita Xu

Nita (Qiuyun) Xu graduated from Shandong University, China, in 1987 with B.Sc. in Chemistry. After the graduation, she spent 8 years at Shandong Institute of Building Materials as Teaching Assistant and Lecturer to teach Analytical Chemistry. In 1995, she joined the Department of Chemistry, National University of Singapore, as Lab Technician. In 1999, she joined Croda Singapore as Production Chemist. From 2003 to 2010, she worked for Michigan Molecular Institute, Midland, Michigan as Chemist and Research Chemist focusing on the synthesis, characterization, and application of hyperbranched polymers. In 2010, she started to work in Polyurethane Business R&D, The Dow Chemical Company, first as a Contract Professional and then as a Lab Technologist to support the new product development.

Rajat Duggal

Dr. Rajat Duggal, currently Sr. R&D Manager, leads the Elastomers R&D in Dow Polyurethanes. In his role Rajat is responsible for application development in the field of cast and spray elastomers utilizing chemistry and material science expertise. Rajat has been at Dow since August 2007. His research has focused on material science of non-mercury catalyzed cast elastomers and new material development in the area of sub-sea wet insulation for flow assurance. Prior to joining Dow, Rajat worked as a Process Engineer at General Electric (GE) Plastics in the Display and Optical Films Technology Division developing coating and microreplication process for LCD films. He received his Bachelor in Technology degree in Chemical Engineering from Indian Institute of Technology (IIT) Bombay in 2000. He earned a Ph.D. in Chemical and Biomolecular Engineering from Rice University, Houston in 2006. He has 5 external publications in notable peer reviewed journals and has given a number of conference presentations. He has also co-authored 19 patent applications.

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