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URETHANE ADHESIVES AS L-100/
MOCA SUBSTITUTES

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August 1974

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OAK RIDGE Y-12 PLANT
OAK RIDGE, TENNESSEE

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ABSTRACT

A number of urethane systems were evaluated as room-temperature curing substitutes for Adiprene L-100/MOCA because of restrictions placed upon the use of MOCA by the Occupational Safety and Health Administration. Adhesive and bulk physical properties were determined for two systems both of which appear to be suitable substitutes for the L-100/MOCA system for many applications.

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SUMMARY

A number of urethane systems were evaluated as room-temperature curing substitute materials for Adiprene L-100/MOCA as a result of restrictions placed upon the use of MOCA by the Occupational Safety and Health Administration. Two of these systems were evaluated for compatibility with certain materials and for variability as related to curing agent concentration, water content, cure time and temperature, component preparation, and usability. Tests utilized were: viscosity, hardness, tensile strength, adhesive tensile strength, creep and various analytical procedures.

The selected prepolymers (Adiprene L-315 and Adiprene LW-520) are both based on the same polyalkylene ether glycol used in Adiprene L-100, with the L-315 having an aromatic isocyanate termination and the LW-520 having a cycloaliphatic isocyanate termination. The L-315 prepolymer is cured with a catalyzed polyol blend; whereas, the LW-520 is cured with an aromatic diamine mix. Data indicate that both systems are suitable substitutes for the L-100/MOCA system for many applications. The cured LW-520 approximates the cured L-100 in physical properties. However, a shorter pot life and higher viscosity cause some complications in use. The L-315 system more nearly approximates the handling and use properties of the L-100 system. However, a slower cure rate and greater tendency to creep are properties that cause some difficulties in substituting this system for the L-100/MOCA.

INTRODUCTION

Adhesive bonding of a number of chemically sensitive or reactive materials has been accomplished in the past with a urethane adhesive—Adiprene L-100/MOCA.^(a) However, as a result of restrictions placed upon the use of MOCA by the Occupational Safety and Health Administration (OSHA), substitute systems are being evaluated at the Oak Ridge Y-12 Plant.^(b)

(a) MOCA is a product of the DuPont Company.

(b) Operated by the Union Carbide Corporation's Nuclear Division for the US Atomic Energy Commission.

URETHANE ADHESIVES AS L-100/MOCA SUBSTITUTES

ADHESIVE SELECTION AND PREPARATION

Evaluation of substitute materials for a MOCA-cured urethane rubber (Adiprene L-100) was initiated as a result of restraints placed upon the use of MOCA (4, 4'-methylene-bis-2 chloroaniline) by OSHA. Viscosity, cure time, physical properties, and handling characteristics of several urethane mixes were studied. As a result of these preliminary studies, two urethane prepolymers and curing agents were selected for further evaluation.

The selected prepolymers (Adiprene L-315 and Adiprene LW-520)^(c) are both based on the same polyalkylene ether glycol used in Adiprene L-100. L-315 contains the same isocyanate termination [toluene diisocyanate (TDI)], whereas LW-520 is terminated with 4,4'-methylene-bis (isocyanato cyclohexane). Formulas for these systems, as well as that for the L-100 system, are presented, with their respective cures, in Figure 1. The L-100 and L-315 prepolymers are essentially the same except for their molecular weight and respective isocyanate contents of 4.10 and 9.45%. They are, therefore, quite similar in reactivity. The LW-520 system, being terminated with an aliphatic isocyanate, has considerably reduced activity which permits it to be cured with simple aromatic diamines. (The slow cure provides for a convenient mixing and handling time.) The water reaction rate of the LW-520 prepolymer, evaluated by the cure time when exposed to atmospheric moisture, was found to be < 3% of the rate of the L-100 cure. This condition suggests that the LW-520 may not function as its own drying agent during the time required for mixing and processing.

Preliminary evaluation of curing agents for LW-520 involved viscosity-time-temperature^(d) relationships using methylenedianiline (MDA) and meta phenylenediamine (MPDA). Their cure rates are quite similar, with the MDA being slightly more reactive. Incorporation of molten MDA into LW-520 at 70° F (293 K) is extremely difficult, due to the rapid crystallization rate of the MDA. As a consequence, its unmodified use at or near room temperature would not appear to be desirable. Incorporation of low levels of other amines or resinous modifiers, such as epoxy, permits its use. A eutectic mix of MPDA/MDA (60/40 wt %) was selected primarily because of its desirable handling properties. This formulation is given in Table 1.

The eutectic normally remains liquid for several days at 293 K and poses no use problems other than those associated with the high viscosity of the prepolymer and the short working time of the mix. Viscosity of the LW-520/eutectic increases from $\sim 6 \times 10^4$ - $\sim 1 \times 10^5$ cPs ($6 \text{ E}+01$ - $1 \text{ E}+02$ Pa·s) in 35 minutes. A typical curve is given in Figure 2. Depending upon its application, its use beyond a 50-minute mix time becomes quite difficult. Because of the high viscosity of the LW-520/eutectic, machine mixing and/or a slight warming would, perhaps, be required for mixing large batches.

(c) Products of the DuPont Company.

(d) Spindle 7 was used with all viscosity determinations made with a Brookfield RVT viscometer.

of the L-315 system. Trimethylolpropane (TMP) and one lot of 1,4-butanediol (BDO) failed to meet the vendor's water specifications of 500 and 400 ppm, respectively. A dry-nitrogen sparge at 95° C (368 K) for 48 hours was found to be adequate for reducing the water content to ~ 100 ppm. Room-temperature sparging equilibrated after 2 to 3 days at ~ 400 ppm water. Nuclear magnetic resonance (NMR) analysis of a mix of BDO and TMP which had been sparged together for 48 hours at 368 K revealed no change in the ratio of the components, indicating the practicality of jointly sparging the materials. Some typical water analyses are listed in Table 2.

Table 1
ADIPRENE L-100/MOCA ADHESIVE SUBSTITUTES

	Parts by Weight
<u>Urethane/Polyol</u>	
Adiprene L-315	100
1,4-Butanediol (BDO)	7
Trimethylolpropane (TMP)	2
Ferric Acetyl Acetonate (FAA)	0.03
<u>Urethane/Amine</u>	
Adiprene LW-520	100
Methylenedianiline (MDA) (40 pbw)	7.5
Metaphenylenediamine (MPDA) (60 pbw)

Figure 2. TYPICAL VISCOSITY CURVES FOR URETHANE FORMULATIONS.

An attempt was made to dry a polyol mix with the catalyst (FAA) incorporated by sparging with dry nitrogen at 368 K. This treatment overnight apparently converted the iron from

the ferric to the ferrous state as the color changed from red to green. As a result of this problem, procedures were established to prepare the catalyzed polyol component at a maximum temperature of 65° C (338 K) with a minimum exposure time.

Table 2
TYPICAL WATER-CONTENT ANALYSIS

Material	History	Water Content (ppm)(1)
BDO, Lot 1	Old Material on Hand	639
BDO, Lot 2	Anhydrous, As Received	975
BDO, Lot 3	Anhydrous, As Received	117
BDO, Lot 4	Anhydrous, As Received	147
BDO, Lot 2	N ₂ Sparge - 24 Hr at 368 K	89
BDO, Lot 2	N ₂ Sparge - 48 Hr at 368 K	99
BDO, Lot 2	N ₂ Sparge - 48 Hr at 368 K	22
BDO-TMP Mix	N ₂ Sparge - 69 Hr at RT	386
TMP, Lot 1	As Received	1100
TMP, Lot 2	As Received	876
TMP, Lot 1	N ₂ Sparge - 13 Hr at 368 K	265
TMP, Lot 1	N ₂ Sparge - 24 Hr at 368 K	184
TMP, Lot 1	N ₂ Sparge - 48 Hr at 368 K	128
FAA	As Received	2800
MDA/MPDA		
Eutectic	N ₂ Sparge - 16 Hr at 383 K	273(2)
L-100	As Received	118
LW-520	As Received	181

(1) By the Karl Fischer method.

(2) By neutron activation analysis.

A viscosity test of a catalyzed polyol mix aged for 17 days indicated no deterioration in catalyst activity with L-315 (see Figure 3).

A series of viscosity tests were made with the LW-520 system using MDA, MPDA, and the eutectic at different temperature levels. The temperatures and formulations are summarized in Figure 4. Temperatures were initial and not maintained. MDA appears to be slightly more reactive than MPDA. Temperature effects are more pronounced, however, than the curing-agent effect.

A number of tests were made with the L-315/polyol system to determine the effects of water concentration and the FAA from two suppliers (Pfaltz & Bauer and Aldrich Chemical Company). Water content was evaluated at four levels (100,400,700,

and 1000 ppm) with no significant effect on viscosity or hardness. Likewise, no difference was observed in the vendors' FAA. Slight differences were noted in adhesive tensile strength at both 24 and 48-hour cure times; however, strengths were so low in all cases that the data are of questionable consequence. Water-content tests, for visual evaluation, were made at higher concentrations (ppm levels of 100, 1000, 2000, 3000, and 4000) to determine the bubble-causing effects. Though rates of reaction do not appear to be affected, disastrous results regarding porosity are evident. A view of the castings is provided in Figure 5.

FAA catalyst concentrations at four levels (0.02, 0.03, 0.04, and 0.05 phr) were evaluated with L-315. Increasing concentration yielded increasing viscosity and hardness with time. Figures 6 and 7 display the marked effects. Adhesive tensile tests were made after a 24-hour cure with inconclusive results.

Polyol concentration effects with L-315 have been evaluated at five levels with a single prepared mix of the polyol component (BDO/TMP/FAA in a 7/2/0.03 ratio). Varying the concentration of polyol consequently resulted in a similar variation in FAA concentration. Viscosity effects (reported in Figure 8) probably reflect the influence of the FAA concentrations as well as the polyol levels. Hardness levels, on the other hand, reflect

Figure 3. AGING EFFECTS OF CATALYZED POLYOL MIX WITH L-315.

principally the polyol concentration effects. The data are plotted in Figure 9 as a function of the cure time and in Figure 10 as a function of concentration. Polyol concentrations of 10 to 11 phr appear to yield the maximum hardness. A concentration of 8 phr or less results in a composition with properties of a viscous liquid rather than a cured rubber.

PROPERTIES OF SELECTED SYSTEMS

Tests were made to determine some basic properties of both selected urethane systems. A series of hardness tests, made with a Shore A meter, established limits for immediate hardness (~ 1 -sec reading) and hardness after one-minute indentation. The one-minute reading was taken for its greater sensitivity, reproducibility, and implication regarding creep. These data are plotted in Figures 11 and 12 for the L-315 and LW-520 systems, respectively. Immediate hardness readings indicate that full cures (90 Shore A) are attained in 200 hours with L-315 and in 150 hours with LW-520. The one-minute readings indicate a full cure at 300 hours with LW-520 and a rather indefinite cure time with L-315. Samples of the L-315 system, cured three hours at 212°F (373 K), had immediate hardnesses of ~ 90 Shore A and one-minute readings of ~ 56 Shore A.

Bulk tensile properties were determined from specimens cut from cast sheet, 0.25 inch (6.4 mm) thick. The results (see Table 3) were obtained for cure times of 4, 7, 14, and 28 days. An ultimate strength of 1980 psi (13.7 MPa) for L-315 does not represent a full cure, as shown by the curve in Figure 13. The LW-520, however, appears to have reached a full cure

Figure 4. TYPICAL LW-520 VISCOSITY CURVES.

after 14 days. These results represent only one series of experiments and provide no statistical limits. Tensile modulus values, calculated using the initial and very-low-elongation part of the curve, are only approximations, as are the numbers calculated for Poisson's ratio. The creep and rate-sensitive nature of these materials make it difficult to obtain precise and meaningful data.

Adhesive tensile strengths for L-315 and LW-520, determined with aluminum coupons having 20-mil (0.5 mm) bondlines, are reported in Table 4. These data (plotted in Figure 14) indicate that the L-315 has not reached maximum strength after a cure of 56 days. The LW-520, however, appears to have leveled off in strength after 14 to 28 days, with the last specimens tested (cured 91 days) exhibiting a considerably reduced strength. It is not known whether this reduction is real or the result of some mishandling.

Cure-time studies were performed with both systems as a function of creep and compared to the L-100/MOCA system. A creep test fixture (Figure 15) was designed which permitted the extrusion of the liquid system into a 25-mil (0.63-mm) gap of a precise area and further permitted loading to ~ 25 psi (~ 173 kPa) at desired cure-time intervals. Typical creep behavior is illustrated in Figures 16 through 18. The L-315 system required a cure of ~ 72 hours at 70°F (293 K) (per this test) to attain a creep-resistant cure level. The LW-520 system attained this level within 17 hours and was not tested at lesser periods of time. In

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Figure 5. EFFECTS OF WATER CONTENT ON THE L-315/POLYOL FORMULATION.

comparison, Adiprene L-100/MOCA required a cure time of 20 hours. Further comparison of creep phenomena, using a hardness gage, is portrayed in Figure 19. Some creep occurs, especially with the L-315 system, after a one-month cure.

A spectrographic analysis was made of both urethane systems. The results are reported in Table 5. The calculated elemental content of the cured systems is reported in Table 6. Except for the chlorine content of the L-100/MOCA system, no significant differences are apparent.

CONCLUSIONS

The following conclusions may be stated as a result of this study:

1. Both the LW-520 and L-315 adhesive systems appear to be interchangeable with L-100/MOCA from the standpoint of surface compatibility with the metals and materials that were tested.
2. The LW-520 system resembles very closely the L-100/MOCA system in physical properties. However, the shorter pot life and higher viscosity cause some complications in use.

Table 3
BULK TENSILE PROPERTIES OF
L-315 AND LW-520

Cure Time (days)	L-315(1)		LW-520(1)	
	(psi)	(MPa)	(psi)	(MPa)
Average Tensile Strength				
4	36(5)	0.25	830(4)	5.7
7	320(5)	2.24	2610(5)	18.0
14	880(5)	6.07	4130(5)	28.5
28	1980(5)	13.70	4010(4)	27.7
Total Elongation				
	(%)		(%)	
4	740(2)		320(2)	
7	510(3)			
14	370(5)		450(2)	
28	270(2)		370(2)	
Modulus of Elasticity				
	(psi)	(MPa)	(psi)	(MPa)
4			2400(4)	16.6
7	170(3)	1.2	5400(2)	37.3
14	220(5)	1.5	8100(5)	55.9
28	1700(5)	11.7	7600(5)	52.4
Poisson's Ratio				
4			0.46(2)	
7			0.41(2)	
14	0.43(2)		0.47(3)	
28	0.44(3)		0.49(3)	

(1) The numbers in parentheses indicate the sample population.

Table 4
ADHESIVE TENSILE STRENGTHS
(Aluminum to Aluminum)

Cure Time (days)	Average Tensile Strength(1)		Standard Deviation
	(psi)	(MPa)	
<u>L-315/Polyol</u>			
0.67	16	0.11	0.2
0.83	24	0.17	1.9
1	33	0.23	1.4
1.5	61	0.42	10
2	120	0.83	14
4	890	6.14	21
7	1270	8.76	130
14	1750	12.07	31
28	1950	13.45	180
56	2510	17.32	140
86	2480	17.11	150
182	2670	18.42	70
<u>LW-520/Eutectic</u>			
0.67	200	1.38	6
0.83	280	1.93	7
1	400	2.76	6
1.5	550	3.80	4
2	850	5.86	50
4	1040	7.18	65
7	1910	13.18	50
14	2220	15.32	26
28	2410	16.63	115
59	2460	16.97	70
91	1870	12.90	260
184	1940	13.39	120

(1) The average value was calculated on a sample population of three.

Table 5
TRACE ELEMENTS IN LW-520 AND L-315 BY SPECTROGRAPHIC ANALYSIS

Element	Amount (ppm)	Element	Amount (ppm)	Element	Amount (ppm)
Adiprene LW-520/MDA/MPDA					
Ag	< 0.01	Cu	< 0.1	P	< 1
Al	0.3	Fe	0.6	Pb	< 0.01
B	0.01	K	< 0.2	Si	2
Ba	< 0.01	Li	< 0.1	Sn	< 0.01
Be	< 0.01	Mg	0.3	Ti	< 0.06
Ca	0.6	Mn	< 0.01	V	< 0.01
Cd	< 0.01	Mo	< 0.01	W	< 0.1
Co	< 0.2	Na	1	Zn	< 0.6
Cr	0.01	Ni	< 0.04	Zr	< 0.02
Adiprene L-315/Polyol					
Ag	< 0.02	Cu	0.4	P	< 2
Al	1	Fe	24	Pb	< 0.02
B	< 0.02	K	< 0.4	Si	6
Ba	0.02	Li	< 0.2	Sn	0.02
Be	< 0.02	Mg	1	Ti	< 0.1
Ca	1	Mn	0.02	V	< 0.02
Cd	< 0.02	Mo	< 0.02	W	< 0.2
Co	< 0.4	Na	2	Zn	< 1
Cr	0.04	Ni	0.2	Zr	< 0.04

Figure 6. VISCOSITY EFFECTS OF VARIOUS FERRIC ACETYLACETONATE CONCENTRATIONS IN ADIPRENE L-315.

Figure 7. HARDNESS EFFECTS OF VARIOUS FERRIC ACETYLACETONATE CONCENTRATIONS IN ADIPRENE L-315. [L-315(100)/BDO(7)/TMP(2)/FAA(as noted); TMP Sparged; BDO Sparged]

Figure 8. VISCOSITY EFFECTS OF VARIOUS POLYOL CONCENTRATIONS WITH ADIPRENE L-315.

Figure 9. HARDNESS EFFECTS OF VARIOUS POLYOL CONCENTRATIONS IN ADIPRENE L-315. [L-315(100)/BDO(7)/TMP(2)/FAA(0.03); TMP Sparged; BDO as Received]

Figure 10. HARDNESS EFFECTS OF VARIOUS POLYOL CONCENTRATIONS WITH L-315.

Figure 11. SHORE "A" HARDNESS VALUES AS A FUNCTION OF THE CURE TIME. (L-315/Polyol System)

Figure 12. SHORE "A" HARDNESS VALUES AS A FUNCTION OF THE CURE TIME. (LW-520/Eutectic System)

Figure 13. BULK TENSILE PROPERTIES OF L-315 AND LW-520.

Figure 14. ADHESIVE TENSILE STRENGTHS. (Aluminum to Aluminum)

Figure 15. FIXTURE FOR MEASURING CREEP UNDER A LOAD (Load of 25 psi (172.5 kPa))

Figure 16. CREEP OF L-315. [Indicated Curve Hours Represent Time Cured Prior to Loading at 25 psi (172.5 kPa); Creep is the Thickness Reduction in Mils of a 25-Mil-Thick Casting]

Figure 17. CREEP OF LW-520. [Indicated Curve Hours Represent Time Cured Prior to Loading at 25 psi (172.5 kPa); Creep is the Thickness Reduction in Mils of a 25-Mil-Thick Casting]

Figure 18. CREEP OF L-100. [Indicated Curve Hours Represent Time Cured Prior to Loading at 25 psi (172.5 kPa); Creep is the Thickness Reduction in Mils of a 25-Mil-Thick Casting]

Figure 19. CREEP AS A FUNCTION OF THE SHORE "A" HARDNESS VALUE. (L-315/BDO/TMP/FAA was Cured for One Month; LW-520/MDA/MPDA was Cured for Three Weeks; All Samples were Cured at Room Temperature)

3. The L-315 system more nearly approximates the handling and use properties of the L-100 system. However, a slower cure rate and greater tendency to creep are properties that cause some difficulties in substituting this system for the L-100/MOCA.
4. Both systems ultimately attain a good cure at room temperature.

Table 6
ELEMENTAL CONTENT OF CURED SYSTEMS
(All Values in Percent)

Element	L-315/Polyol	LW-520/Eutectic	L-100/MOCA
C	63	67	64
H	8	10	9
O	23	19	20
N	6	4	4
Cl	-	-	3

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