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REPORT NO. NAEC AML 1636 DATE 13 MARCH 1963

FINAL REPORT

DEVELOPMENT AND FORMULATION OF EXPERIMENTAL DIISOCYANATE-BASED LAMINATING RESINS

PROBLEM ASSIGNMENT NO. C 09 RMA 31-1 UNDER BUREAU OF NAVAL WEAPONS WEPTASK RRMA 03 017/200 1/ROO7 04 01

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NAVAL AIR ENGINEERING CENTER
PHILADELPHIA 12, PENNSYLVANIA

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ABSTRACT

The successful synthesis of halogenated polyurethane resins for laminating is reported. The test results indicate that halogen groupings in a polyurethane resin, tend to increase the thermal stability of the resulting laminate and to increase the pot-life of the resin.

Future efforts will be directed toward improving stability above 300°F of the polyurethane structural laminate.

A patent pertaining to the technique of formulating polyurethane resins has been allowed to Mr. A. P. Bonanni. This patent will be issued soon.

I. INTRODUCTION

A. This is the final report under problem assignment C 09 RMA 31-1, WEPTASK RRMA 03 017/200 1/RO07 04 01 which was authorized by reference (a). Early experimentation (references (b), (c), (d) and (e)) indicated that the formulation of a polyurethane resin suitable for preparing a reinforced laminate was feasible. Also, that 2-butene-1,4-diol when reacted with 2,4-tolylene-diisocyanate (TDI) produced the best physical properties in a polyurethane structural laminate. This study pertains to the reaction of halogenated diols with TDI, to increase the pot-life of the polyurethane resin, and to increase the thermal stability of the structural laminates. This work was performed between February 1960 and February 1963.

II. SUMMARY OF RESULTS

A. Improved flexural modulus properties at elevated temperatures have been attained with the use of halogenated groupings in the polyurethane resins. The physical properties of these structural laminates could be improved with further experimentation.

B. The pot-life, a problem with un-halogenated diols, is extended to several days with the use of halogenated diols.

III. CONCLUSIONS

A. The thermal stability of a structural laminate, increases with the use of halogenated diols. Further improvement can be made by varying the concentration of TDI and Macconate 200. Although the flexural properties do not appear to be significantly high, they must not be considered as the laminate's ultimate flexural strength. In order to determine their ultimate flexural strength, a study must be made to determine the temperature, pressure and time of lamination.

B. The pot-life of a polyurethane resin can be extended by modifying the chemical structure of the experimental resin using halogenated diols.

IV. RECOMMENDATIONS

A. It is recommended that a new problem assignment be established to:

1. Determine optimum laminating parameters of a halogenated diol and a diisocyanate resin which will give superior physical properties in a structural laminate.

2. Ascertain if a peroxide will increase cross-linking in the unsaturated polymers.

3. Determine the molecular weight of a polyurethane resin.

4. Study the physical properties of structural laminates formulated with varying amounts of:

a. 2,4-tolylene-diisocyanate

b. 2-butene-1,4-diol

c. Halogenated diols

d. 3,3'-bitolylene-4,4'-diisocyanate and 3,3'-dimethyldiphenylmethane and the laminating parameters (temperature, pressure and time).

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V. DESCRIPTION, METHODS, RESULTS

A. Description

1. Materials used in this investigation were obtained from sources shown in Plate 1. All the materials reported herein are new to the investigation of polyurethane resins with the exception of TDI and 2-butene-1,4-diol.

B. Method

1. The reactants used in the formulation of the polyurethane resins reported herein are TDI, Macconate 200 (3,3'-bitolylene-4,4'-diisocyanate, Macconate 300 (diphenylmethane-4,4'-diisocyanate), 2-butene-1,4-diol, HFPD, DEBD and tetrachlorobutane diol (TCDB).

2. A study of the melting points of halogenated polyurethane resins using the Koffler Hot Stage (Plate 2) was used to indicate the thermal stability of the resin. These softening points indicated that the physical properties of the structural laminate should be investigated at elevated temperatures. These results are reported in Plates 3 to 11 and graphically presented on Plates 12 to 19. Tests for the evaluation of melting points were performed in accordance with WADC Technical Report 56-399 under the title "Test Methods for Estimation of Thermal Behavior of Polymers."

3. All the structural laminates were prepared using 181 Volan A glass cloth. The laminates were cured at 30 psig and 300°F for 30 minutes and post cured at 158°F for 16 hours. Flexural strength and flexural moduli tests were utilized as a screening measure to compare the different polyurethane formulations; also, to make a comparison of the polyurethane resins at room temperature and elevated temperatures (200°F). The flexural properties were conducted in accordance with Test Method Standard No. 406, Plastics: Method of Testing.

C. Results

1. An examination of the melting points, using the Koffler Hot Stage, indicates that as the molar concentration of TDI is increased and the alkene diol (2-butene-1,4-diol) is kept constant, the softening points of the resins increase to a maximum of 226°F with a molar construction of 1.2 moles of TDI. But when a halogenated diol is substituted for the alkene diol, the softening point increases steadily with the addition of more TDI. The softening points become more significant when polyurethane resins are synthesized using TDI, halogenated diols and Macconate 200. These resins do not soften, but begin to carbonize at 572°F. The results are tabulated and shown on Plate 2.

2. An examination of the flexural values at room temperature, (Plates 3, 5, 7, 9, and 11 to 19) and at 200°F after being exposed to 200°F for 1/2 hour (Plates 4, 6, 8, and 10 to 19) indicates that although some formulations show temperature stability, there are indications that the laminating parameters (curing temperature, pressure and time) used in this study are not the optimum for the laminates. On Plates 3, 11 and 12, the data show extremely low flexure values for a polyurethane resin using 1.0 mole of HFPD and 1.0 mole of TDI; but, with the increase of only a 0.10 of mole of TDI, a flexural value of 90,100 psi and flexural modulus of 4.07×10^6 is obtained. When exposed to 200°F for 1/2 hour and tested at 200°F (Plates 4, 11 and 12) a loss of more than 70% of the flexural strength is seen and a loss of only 10% of the flexural modulus. On Plates 5, 13 and 14, using 1.0 mole of DBBD and 1.1 moles of TDI a flexural strength of 93,900 psi and a modulus of 4.24×10^6 psi is obtained. When exposed to 200°F for 1/2 hour and tested at 200°F (Plates 6, 13 and 14) a loss of less than 3% is found in the flexural strength and a loss of approximately 5% in the flexural modulus. These values substantiate the theory that when halogenated diols are used, the thermal properties can be increased.

3. The pot-life of polyurethane resins is extended to several days when halogenated diols are used. In the formulation using HFPD and TDI, the resulting polyurethane resin remains a liquid for several days provided the resin is placed in an air-tight container free of moisture. The formulations using DBBD and TDI form a polyurethane resin which solidifies in 2 to 3 hours after synthesis but can be poured as a hot melt, provided the temperature of the resin is maintained between 190°F to 205°F. Formulating TDI with HFPD and DBBD and various concentrations of Nacconate 200 yielded resins having a pot-life of less than one hour. Formulating TDI with DBBD and Nacconate 300 yielded a resin which had a pot-life of approximately one-half hour. Once these resins are polymerized, they cannot be reheated to a melt.

REFERENCES

- (a) BUAER ltr Aer-AE-44/127 of Aug 1955
- (b) MAXSTA ltr XM-52-SP:dsf J2-5 (3517) of Aug 1955
- (c) Report No. NAMC AML AE 4420, Part I of 2 Mar 1956
- (d) Report No. NAMC AML AE 1071, (AE 4420, Part II) of 16 Oct 1958
- (e) Report No. NAMC AML 1131 (Project No. TED NAM AE 4420, Part III) of 22 Jun 1960

SOURCES OF MATERIALS

<u>Compound</u>	<u>Source</u>
2,4-Tolylene-Diisocyanate	E. I. duPont de Nemours & Co., Inc. Wilmington 98, Delaware
3,3'-Bitolylene-4,4'-Diisocyanate	National Aniline Division Allied Chemical & Dye Corporation 40 Rector Street New York 6, New York
Diphenylmethane-4,4'-Diisocyanate	Same
2-Butene-1,4-Diol	General Aniline & Film Corporation Commercial Development Department 435 Hudson Street New York 14, New York
Hexafluoropentane Diol	Harker Chemical Corporation Niagara Falls, New York
Tetrachlorobutane Diol	General Aniline & Film Corporation Commercial Development Department 435 Hudson Street New York 14, New York
Dibromobutene Diol	Same

MELTING POINTS USING KOFFLER HOT STAGE (°F)

Sample	Moles of			Softening Point	Initial Flow	Complete Melt
	<u>TDI</u> (1)	<u>2B14D</u> (2)	---			
1	1.0	1.0				
2	1.1	1.0		212	230	473
3	1.2	1.0		248	293	426
4	1.3	1.0		226	248	392
	<u>TDI</u>	<u>HFPD</u> (3)				
1	1.0	1.0		---	381	419
2	1.1	1.0		241	284	340
3	1.2	1.0		248	401	473
4	1.3	1.0		365	388	410
	<u>TDI</u>	<u>DBBD</u> (4)				
1	1.0	1.0		203	214	221
2	1.1	1.0		207	234	273
3	1.2	1.0		248	374	410
4	1.3	1.0		---	---	---
	<u>TDI</u>	<u>TCBD</u> (5)				
1	1.0	1.0		347	392	424
	<u>TDI</u>	<u>Naconate 200</u>	<u>HFPD</u>			
1	1.0	0.5	1.5	158	169	190
2	0.75	0.75	1.5	255	324	Charred at 572
3	0.50	1.0	1.5	243	266	302
	<u>TDI</u>	<u>Naconate 200</u>	<u>DBBD</u>			
1	1.0	0.050	1.5	-----Sample Charred at 572-----		
2	0.75	0.75	1.5	262	313	360
3	0.50	1.0	1.5	257	293	Charred at 477

NOTES: (1) TDI - 2,4-Tolylene-Diisocyanate
 (2) 2B14D - 2-Butene-1,4-Diol
 (3) HFPD - Hexafluoropentane Diol
 (4) DBBD - Dibromobutene Diol
 (5) TCBD - Tetrachlorobutane Diol

FLEXURAL DATA FOR HEXAFLUOROPENTANE DIOL (HFPD)
AND 2,4-TOLYLENE-DIISOCYANATE (TDI)

Spec. No.	Moles		Width (in.)	Thick. (in.)	Ultimate Load (lbs.)	Flexural Strength (psi)	Avg.	E x 10 ⁻⁶	
	TDI	HFPD						(psi)	Avg.
1	1.0	1.0	0.498	0.124	56	21,904		--No Results--	
2			0.513	0.124	53	20,126			
3			0.502	0.125	54	20,690			
4			0.509	0.123	52	20,312			
5			0.516	0.124	52	19,623	20,500		
1	1.1	1.0	0.500	0.088	115	89,600		4.20	
2			0.499	0.087	115	91,000		4.17	
3			0.497	0.090	120	89,300		3.92	
4			0.050	0.088	116	90,400		4.11	
5			0.498	0.089	118	90,100	90,100	3.97	4.07
1	1.2	1.0	0.515	0.089	116	85,500		4.32	
2			0.516	0.088	114	86,200		4.48	
3			0.528	0.089	123	88,500		4.26	
4			0.521	0.090	122	86,700		4.14	
5			0.517	0.090	118	84,500	86,300	4.29	4.30
1	1.4	1.0	0.530	0.103	19	8,407		--No Results--	
2			0.505	0.111	18	8,696			
3			0.510	0.110	18	8,752			
4			0.510	0.112	13	6,113			
5			0.509	0.111	13	6,903	7,800		

**FLEXURAL DATA FOR HEXAFLUOROPENTANE DIOL (HFPD)
AND 2,4-TOLYLENE-DIISOCYANATE (TDI),
TESTED AT 200°F AFTER EXPOSURE TO 200°F FOR 1/2 HOUR**

Spec. No.	Moles		Width (in.)	Thick. (in.)	Ultimate Load (lbs.)	Flexural Strength (psi)	E x 10 ⁻⁶			
	TDI	HFPD					Avg.	(psi)	Avg.	
1	1.0	1.0	-----Not Tested Because of Delamination-----							
2										
3										
4										
5										
1	1.1	1.0	0.502	0.087	32	25,100		---		
2			0.512	0.090	39	28,200		3.43		
3			0.495	0.089	35	26,800		3.55		
4			0.503	0.088	36	27,900		3.59		
5			0.500	0.089	34	25,800	26,800	3.43		3.50
1	1.2	1.0	0.530	0.090	52	36,400		2.95		
2			0.527	0.090	49	34,400		3.35		
3			0.512	0.088	43	32,700		3.77		
4			0.518	0.089	46	33,700		3.70		
5			0.519	0.088	44	33,000	34,100	3.87		3.49
1	1.4	1.0	-----Not Tested Because of Delamination-----							
2										
3										
4										
5										

FLEXURAL DATA FOR DIBROMOBUTENE DIOL (DBBD) AND
2,4-TOLYLENE-DIISOCYANATE (TDI) TESTED AT ROOM TEMPERATURE

Spec. No.	Moles		Width (in.)	Thick. (in.)	Ultimate Load (lbs.)	Flexural Strength (psi)	Avg.	E x 10 ⁻⁶ (psi)	Avg.
	TDI	DBBD							
1	1.0	1.0	0.512	0.123	95	59,200		3.95	
2			0.501	0.123	95	56,900		3.66	
3			0.511	0.123	95	58,000		3.75	
4			0.497	0.123	98	63,000		3.90	
5			0.513	0.123	88	54,800	58,400	3.97	3.85
1	1.1	1.0	0.495	0.095	140	94,200		4.23	
2			0.520	0.098	154	92,600		4.08	
3			0.511	0.098	150	91,600		4.23	
4			0.495	0.094	139	95,600		4.41	
5			0.522	0.097	155	94,700	93,900	4.23	4.24
1	1.2	1.0	0.526	0.101	101	56,400		3.75	
2			0.506	0.096	93	59,900		4.03	
3			0.528	0.097	105	63,500		4.08	
4			0.519	0.100	90	52,000		3.97	
5			0.532	0.101	93	51,400	56,700	3.92	3.95
1	1.3	1.0	0.507	0.100	58	34,800		3.30	
2			0.525	0.096	61	38,200		3.47	
3			0.486	0.096	55	36,900		3.95	
4			0.504	0.095	67	44,300		3.90	
5			0.505	0.098	63	43,100	39,500	3.77	3.68

**FLEXURAL DATA FOR DIBROMOBUTENE DIOL (DBBD) AND
2,4-TOLYLENE-DIISOCYANATE (TDI),
TESTED AT 200°F AFTER EXPOSURE TO 200°F FOR 1/2 HOUR**

Spec. No.	Moles		Width (in.)	Thick. (in.)	Ultimate Load (lbs.)	Flexural Strength (psi)	Avg.	E x 10 ⁻⁶	
	TDI	DBBD						(psi)	Avg.
1	1.0	1.0	0.500	0.097	87	55,500		3.85	
2			0.497	0.097	84	54,000		3.90	
3			0.514	0.098	79	48,100		3.64	
4			0.499	0.096	94	58,900		4.03	
5			0.489	0.097	87	56,700	54,600	4.08	3.90
1	1.1	1.0	0.518	0.094	112	69,000		4.14	
2			0.514	0.095	104	67,400		4.14	
3			0.525	0.098	117	69,600		3.64	
4			0.528	0.097	121	73,900		4.11	
5			0.510	0.098	116	71,000	70,200	3.90	3.99
1	1.2	1.0	0.519	0.099	108	63,600		3.77	
2			0.503	0.099	93	56,600		3.95	
3			0.517	0.096	102	64,300		3.80	
4			0.523	0.097	103	62,800		3.92	
5			0.530	0.102	113	61,500	61,800	3.35	3.76
1	1.3	1.0	0.500	0.100	59	35,400		3.97	
2			0.495	0.099	62	38,400		3.85	
3			0.498	0.095	78	52,200		4.32	
4			0.504	0.095	77	50,900		3.97	
5			0.518	0.098	78	47,100	44,700	3.70	3.96

FLEXURAL DATA FOR HEXAFLUOROPENTANE DIOL (HFPD), MACCONATE 200 AND
2,4-TOLYLENE-DIISOCYANATE (TDI) TESTED AT ROOM TEMPERATURE

Spec. No.	Moles			Width (in.)	Thick. (in.)	Ultimate Load (lbs.)	Flexural Strength (psi)	E x 10 ⁻⁶ (psi)	Avg.
	TDI	HFPD	200						
1	1.0	1.5	0.5	0.532	0.073	42	44,400	3.97	
2				0.530	0.075	43	43,300	3.70	
3				0.533	0.073	38	40,100	3.80	
4				0.536	0.075	42	41,700	3.55	
5				0.532	0.072	38	37,100	4.03	3.81
1	0.75	1.5	0.75	0.507	0.085	63	51,800	4.41	
2				0.513	0.085	65	52,800	4.05	
3				0.513	0.086	72	56,800	3.97	
4				0.519	0.084	66	57,900	4.23	
5				0.514	0.085	62	50,300	4.00	4.13
1	0.5	1.5	1.0	0.473	0.087	50	41,900	3.68	
2				0.472	0.087	53	44,500	3.73	
3				0.471	0.089	51	41,000	3.69	
4				0.474	0.090	53	41,400	3.77	
5				0.471	0.087	50	42,100	3.62	3.70

FLEXURAL DATA FOR HEXAFLUOROPENTANE DIOL (HFPD), MACCONATE 200 AND
2,4-TOLYLENE-DIISOCYANATE TESTED AT 200°F AFTER EXPOSURE TO 200°F FOR 1/2 HOUR

Spec. No.	Moles			Width (in.)	Thick. (in.)	Ultimate Load (lbs.)	Flexural Strength (psi)	E x 10 ⁻⁶	
	TDI	200						Avg.	Avg.
1	1.0	1.5	0.5	0.539	0.075	7	6,900	--No Results--	
2				0.536	0.072	8	8,600		
3				0.533	0.075	7	7,000		
4				0.532	0.071	6	6,700		
5				0.526	0.077	7	6,700	7,200	
1	0.75	1.5	0.75	0.485	0.084	14	12,300	--No Results--	
2				0.519	0.085	22	17,600		
3				0.515	0.086	17	13,400		
4				0.509	0.084	21	17,500		
5				0.517	0.085	21	16,800	15,500	
1	0.50	1.5	1.0	0.473	0.089	9	7,200	--No Results--	
2				0.471	0.096	12	9,400		
3				0.470	0.089	14	11,300		
4				0.470	0.087	15	12,600		
5				0.470	0.087	15	12,600	10,600	

FLEXURAL DATA FOR DIBROMOBUTENE DIOL (DBBD), MACCONATE 200, AND
2,4-TOLYLENE-DIISOCYANATE (TDI) TESTED AT ROOM TEMPERATURE

Spec. No.	Moles			Width (in.)	Thick. (in.)	Ultimate Load (lbs.)	Flexural Strength (psi)	Avg.	E x 10 ⁻⁶	
	TDI	DBBD	200						(psi)	Avg.
1	1.0	1.5	0.5	0.468	0.096	80	55,700		3.55	
2				0.468	0.096	78	54,300		4.01	
3				0.465	0.097	73	50,000		3.57	
4				0.469	0.096	83	57,600		3.62	
5				0.465	0.096	80	55,900	54,700	3.64	3.68
1	0.75	1.5	0.75	0.475	0.093	55	40,100		3.72	
2				0.485	0.095	53	36,400		3.69	
3				0.487	0.096	58	38,800		3.55	
4				0.470	0.093	57	42,000		4.06	
5				0.477	0.093	57	41,400	39,700	3.79	3.76
1	0.5	1.5	1.0	0.508	0.096	66	42,300		3.60	
2				0.508	0.097	65	40,800		3.76	
3				0.509	0.095	65	42,500		3.71	
4				0.516	0.088	60	45,100		4.30	
5				0.509	0.099	66	39,700	42,100	4.70	4.01

**FLEXURAL DATA FOR DIBROMOBUTENE DIOL (DBBD), HACCONATE 200 AND
2,4-TOLYLENE-DIISOCYANATE TESTED AT 200°F
AFTER EXPOSURE TO 200°F FOR 1/2 HOUR**

Spec. No.	Moles			Width (in.)	Thick. (in.)	Ultimate Load (lbs.)	Flexural Strength (psi)	E x 10 ⁻⁶ Avg.	E x 10 ⁻⁶ (psi)	Avg.
	TDI	DBBD	200							
1	1.0	1.5	0.5	0.469	0.096	80	55,600		3.25	
2				0.465	0.096	72	50,400		3.40	
3				0.468	0.094	78	56,500		3.67	
4				0.464	0.095	79	56,600		3.47	
5				0.469	0.097	78	53,100	54,400	3.35	3.43
1	0.75	1.5	0.75	0.486	0.098	59	39,500		3.48	
2				0.482	0.096	57	38,500		3.45	
3				0.475	0.096	58	39,700		3.85	
4				0.469	0.096	55	38,200		3.54	
5				0.471	0.095	56	39,500	39,100	3.72	3.61
1	0.5	1.5	1.0	0.513	0.095	58	37,600		3.20	
2				0.516	0.094	57	37,500		3.50	
3				0.505	0.098	60	46,800		3.21	
4				0.508	0.098	58	35,600		3.06	
5				0.511	0.097	54	33,700	38,400	3.47	3.29

FLEXURAL DATA FOR DIBROMOBUTENE DIOL (DBBD), MACCONATE 300, AND
2,4-TOLYLENE-DIISOCYANATE TESTED AT ROOM TEMPERATURE

Spec. No.	Moles			Width (in.)	Thick. (in.)	Ultimate Load (lbs.)	Flexural Strength (psi)	E x 10 ⁻⁶		
	TDI	DBBD	300					Avg.	(psi)	Avg.
1	1.0	1.5	0.5	0.464	0.105	156	91,800		4.18	
2				0.465	0.106	149	85,800		3.96	
3				0.465	0.106	161	92,700		3.69	
4				0.457	0.106	155	90,800		3.61	
5				0.450	0.106	158	94,000	91,000	3.67	3.82

TESTED AT 200°F AFTER EXPOSURE TO 200°F FOR 1/2 HOUR

1	1.0	1.5	0.5	0.470	0.105	110	63,800		3.09	
2				0.458	0.107	103	59,200		3.09	
3				0.468	0.105	117	68,155		3.40	
4				0.463	0.106	110	63,600		3.53	
5				0.454	0.106	126	70,900	65,100	3.41	3.36















