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The Development Of A High Temperature And Acid Resistant Fire Retardant Resin

ABSTRACT

Composites made with polyester and vinyl ester resins are resistant to acidic environments. However, most of these products can not be used to contain aqua regia, which is a blend of nitric acid and hydrochloric acid. Nitric acid is a powerful oxidizing acid; while hydrochloric is a very strong acid that removes the oxidation products. The combination produces a tough chemical environment. A newly developed resin that can handle aqua regia at elevated temperatures was tested according to ASTM C 581, ASTM E 84 and standard mechanical protocol. The test results will be presented.

INTRODUCTION

A chlorendic polyester (CPE) resin has been developed that meets the standard fire retardant requirements for chlorendic resins but has the added advantage of 2.4% elongation versus 1.1% for current product offering (CPO). This newly developed CPE has two formulations. One meets the requirements of ASTM E 84 class I with 3.0% antimony trioxide (CPEA), which is unique, and the other meets ASTM E 84 class II with 3.0% antimony trioxide (CPEB). In this paper CPEA and CPEB are compared to CPO. The paper reviews the cast and laminate mechanical properties, ASTM E 84 results, some corrosion resistant data and some applications for CPE.

EXPERIMENTAL

1) LIQUID PROPERTY TESTING

Liquid properties were tested with standard equipment and test methods that are typically used in the industry.

2) CASTING PREPARATION

Figure #1 details how the castings were prepared with respect to catalyst system and post cure schedule.

3) ASTM C 581 LAMINATE PREPARATION

ASTM C 581 laminates were prepared according to Figure #2.

Note that two layers of fiberglass C veil were used on each side of the coupon to provide extra protection.

4) STRUCTURAL LAMINATE PREPARATION

A structural laminate was made with glass veil, chopped strand mat and 24 ounce woven roving, according to Figure #3.

5) ASTM E 84 TESTING

Laminates were sent to an outside lab for testing. Panels made for the ASTM E 84 fire and smoke testing were prepared according to Figure # 4.

6) CHEMICAL ENVIRONMENTS

Aqua Regia is a very strong acid that was blended according to the formulation in Figure #5. Corrosion resistant testing was done at both 130°F and at ambient temperature. The ambient temperature study was started later and complete testing results will not be available until after the CFA convention in 2001. Tests that were recorded were flexural strength, % retention, flexural modulus, % retention, Barcol Hardness, % retention and weight gain. Antimony trioxide was not added to the resin for the chemical resistant testing. This is the normal industry practice.

PROPERTIES

1) RESIN LIQUID PROPERTIES

The liquid properties are listed in Figure # 6.

2) CAST MECHANICAL PROPERTIES

Cast properties are listed in Figure #7

3) STRUCTURAL LAMINATE MECHANICAL PROPERTIES

Laminate properties are listed in Figure #8. The laminate properties of CPEA will be deter-

- mined and made available at a future date.
- 4) **ASTM E 84 RESULTS**
The ASTM E 84 values obtained from outside laboratory testing are listed in Figure #9 with the exception of CPO. In this case values from technical data sheets were used.
 - 5) **CORROSION RESISTANT PROPERTIES**
The corrosion testing results are listed in Figures [10-13](#).

RESULTS

- 1) Laminates made with CPEA, CPEB and CPO are equally resistant to aqua regia at 130 °F.
- 2) Initial 3-month corrosion results of CPEA with aqua regia at ambient temperature are encouraging. (Final results will be available later.)
- 3) Cast and laminate mechanical properties of CPEB are excellent. Based on the heat distortion of 280 °F on the casting, CPEB can be used for applications where high heat resistance is required.
- 4) Cast mechanical properties of CPEA are typical of corrosion grade resins but the heat distortion is lower than CPEB (234 °F versus 280 °F).
- 5) Viscosity and gel properties of CPEA and CPEB are typical of general-purpose resins. Thus

these resins are user friendly and use standard laminating techniques.

APPLICATIONS

- 1) CPEA and/or CPEB have been used for acid applications such as:
 - a) Tanks and the mixing shafts that are used to contain and mix aqua regia.
 - b) Tanks for chrome plating plants
- 2) CPEB has been used for aerospace where fire resistance and high heat resistance are required.

CONCLUSION

Consider the use of:

- 1) CPEA or CPEB for acid environments such as aqua regia.
- 2) CPEB for high heat distortion applications.
- 3) CPEA if the laminate must meet ASTM E 84 class I with 3.0% antimony trioxide.
- 4) CPEB if the laminate must meet ASTM E 84 class II with 3.0% antimony trioxide.

Figure #1 - CASTING PREPARATION

Catalyst and Cure Schedule	Quantity and Time
Catalyst system	
Benzoyl Peroxide (BPO), %	1
Cure schedule	
60 °C/160 °F, hours	4
93 °C/200 °F, hours	1
116 °C/240 °F, hours	1
138 °C/280 °F, hours	2

Figure #2 - ASTM C 581 LAMINATE PREPARATION

Promoter system	None (Resins are prepromoted)
Catalyst system	1.25 %MEKP (9% active oxygen)
Laminate construction	2 plies of fiberglass Cveil
	2 plies of 1.5 ounce chopped strand glass mat
	2 plies of fiberglass Cveil
Postcure	3 hrs at 100°C/212°F

Figure #3 -STRUCTURAL LAMINATE PREPARATION

Promoter system	None (Resin is prepromoted)
Additive	3.0% Antimony trioxide added to the resin
Catalyst system	1.25 % MEKP (9% active oxygen)
Laminate construction	1 ply of fiberglass C veil
	2 plies of 1.5 ounce chopped strand glass mat
	1 ply of 24 ounce woven roving
	1 ply of 1.5 ounce chopped strand glass mat
	1 ply of 24 ounce woven roving
	1 ply of 1.5 ounce chopped strand glass mat
Postcure	3 hrs at 100°C/212°F

Figure #4 -ASTM E 84 LAMINATE PREPARATION

Laminate Preparation	
Promoter system	None (Resin is prepromoted)
Catalyst system	1.25 % MEKP (9% active oxygen)
Additive	3.0% Antimony trioxide was added to the resin.
Laminate construction	2 plies of 2.0 ounce chopped strand glass mat
Glass content, %	30
Postcure	3 hrs at 100°C/212°F

Figure #5 -CHEMICAL ENVIRONMENTS

CHEMICAL	FORMULATION, (Volume)	TEMPERATURE
Aqua Regia	60% Hydrochloric Acid 37% active	54°C/130°F
	20% Nitric Acid 70% active	
	20% Distilled Water	
Aqua Regia	Same as Above	Ambient

Figure #6 -RESIN LIQUID PROPERTIES

	C P E A	C P E B	C P O
25°C Brookfield Viscosity Spindle #2 and 20 rpm, cps	600	600	600
Thix Ratio 2/20	2	2	2
Gel Time with 1.25% DDM-9, minutes	15	15	15
Total Time, minutes	25	27	31
Peak Exotherm, °F	365	385	385
Specific Gravity	1.19	1.15	1.14

Figure #7 -CAST MECHANICAL PROPERTIES

	C P E A	C P E B	C P O
Flexural Strength, psi	15,600	16,000	13,500
Flexural Modulus, psi	610,000	550,000	530,000
Tensile Strength, psi	9,000	8,500	6,700
Tensile Modulus, psi	540,000	490,000	500,000
Elongation, %	1.9	2.4	1.5
Heat Distortion Temperature, °F	234	280	280
Barcol Hardness (934)	45	45	42

Figure #8 - STRUCTURAL LAMINATE MECHANICAL PROPERTIES

	C P E A	C P E B	C P O
Flexural Strength, psi	Not	31,300	18,000
Flexural Modulus, psi	Completed	1,300,000	990,000
Tensile Strength, psi	Yet	22,000	16,300
Tensile Modulus, psi		1,800,000	1,400,000
Elongation, %		2	--
Barcol Hardness (934)		52	--

TEST	RESULTS		
	CPEA	CPEB	CPO
ASTM E 84 test with 3.0% Antimony Trioxide			
Flame spread	20	34	--
Smoke development	330	445	--
ASTM E 84 test with 5.0% Antimony Trioxide			
Flame spread	--	--	<25