LET'S TALK PERFORMANCE

INFLUENCE OF POST-CURE ON MECHANICAL AND CHEMICAL RESISTANCE OF POLYESTERS AND VINYL ESTERS

Ronald Uitterdijk

FRP Unlimited
February 6-7, 2018

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QUALITY RESINS

END-USE PERFORMANCE REQUIREMENTS

- Resistance to chemicals
- Heat resistance
- Low maintenance
- Strength, stiffness, toughness
- Food contact (when required)
- Light weight, easy installation
- Design flexibility

Exposure determines resin choice
Different solutions available for heat and chemicals involved. Good chemical resistance means low maintenance and peace-of-mind on performance

High mechanical strength
Selection of right resin, reinforcement, and their interaction is key

Food contact
Resins made in line with GMP for good food quality and consumer safety

Key benefit vs. steel
Enabling technology for light weight constructions in corrosive environment

Optimized design
Shaping flexibility and part integration possibility is a key composites benefit.

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INDUSTRY STANDARDS HELP IN ACHIEVING GREAT PART PERFORMANCE

- Well-established international standards
  - Focus today: EN 13121 “GRP tanks and vessels for use above ground”
- Providing guidelines on part design, manufacturing and installation
  - Part 1: Raw materials, specifications, acceptance conditions
    - Resin chemistry linked to 8 classes with increasing corrosion resistance
  - Part 2: Composite materials – Chemical resistance
    - Media types according 3 groups of increasing chemical attack
    - Build-up of laminate for optimal performance
  - Part 3: Design and workmanship
    - Calculation of loads, design of supports, fittings
    - Quality control, including pressure testing
  - Part 4: Delivery, installation and maintenance
    - Handling and transport, installation guidelines
    - Ensuring installation quality and avoiding damage

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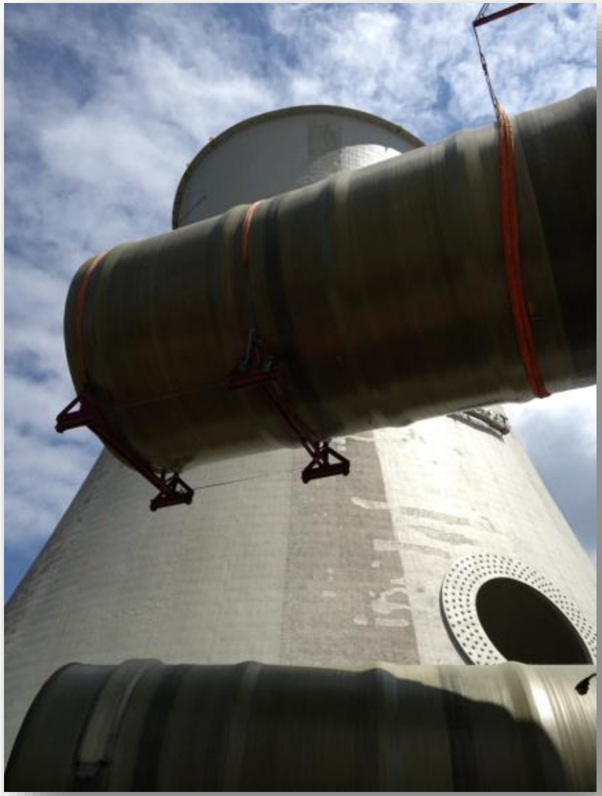
IMPORTANT FOR QUALITY OF COMPOSITE PART

- Resin selection
- Reinforcement selection
- Curing agent formulation
- Optimal curing

- Are determined by design standard, customer specifications, corrosion resistant lists, and/ or following Aliancys Chemical Resistance advice

- Proper part cure is very important for final part performance



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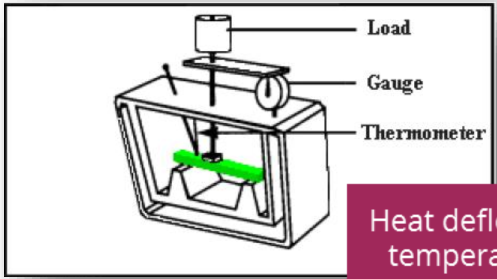
SELECTING THE RIGHT RESIN FOR ELEVATED TEMPERATURES

- Chemicals are mostly more corrosive at elevated temperatures
- EN 13121 standard prescribes that HDT of the resin should be at least 20°C higher than the exposure temperature of the part
- Component may loose mechanical integrity if use temperature gets too close or above HDT



Heat deflection temperature

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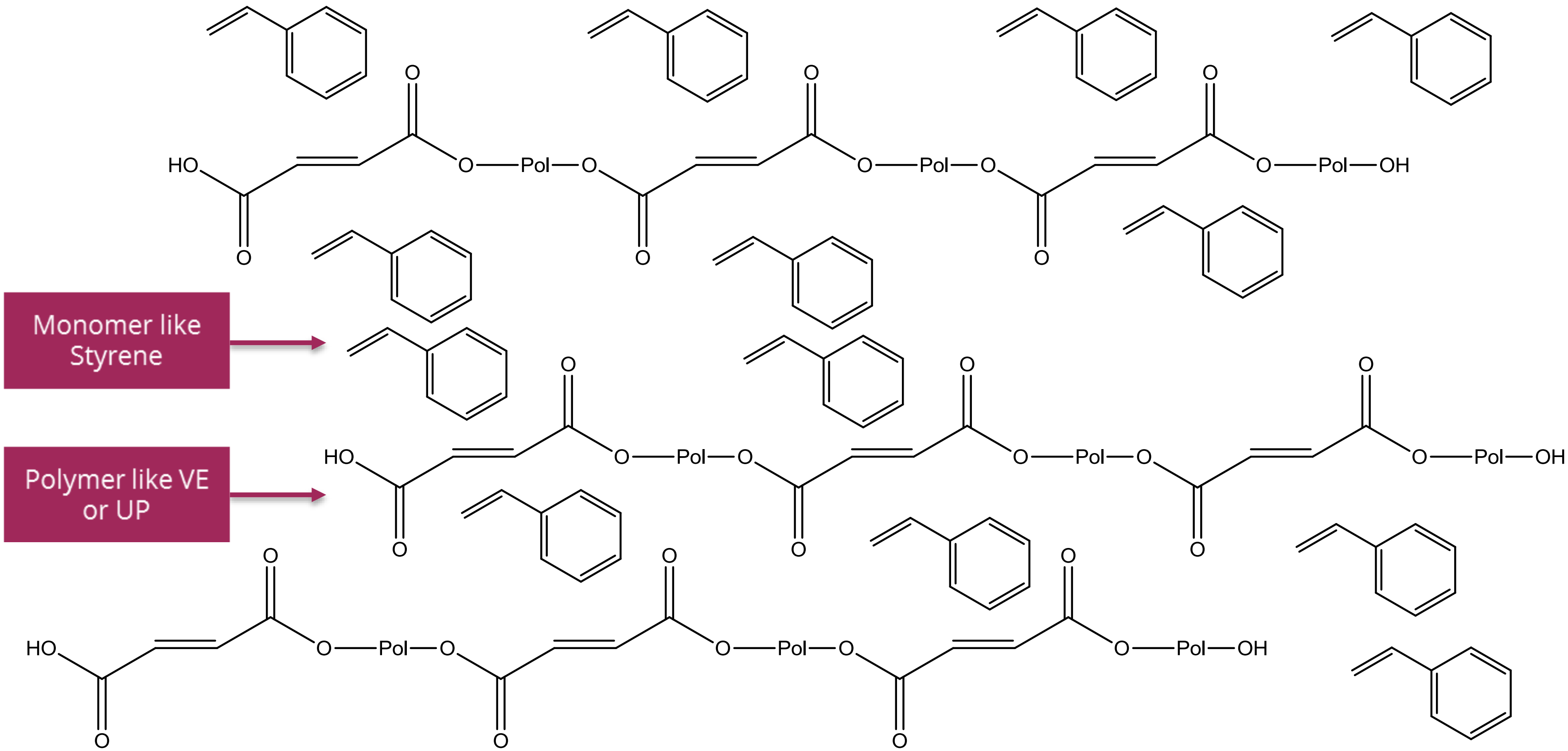
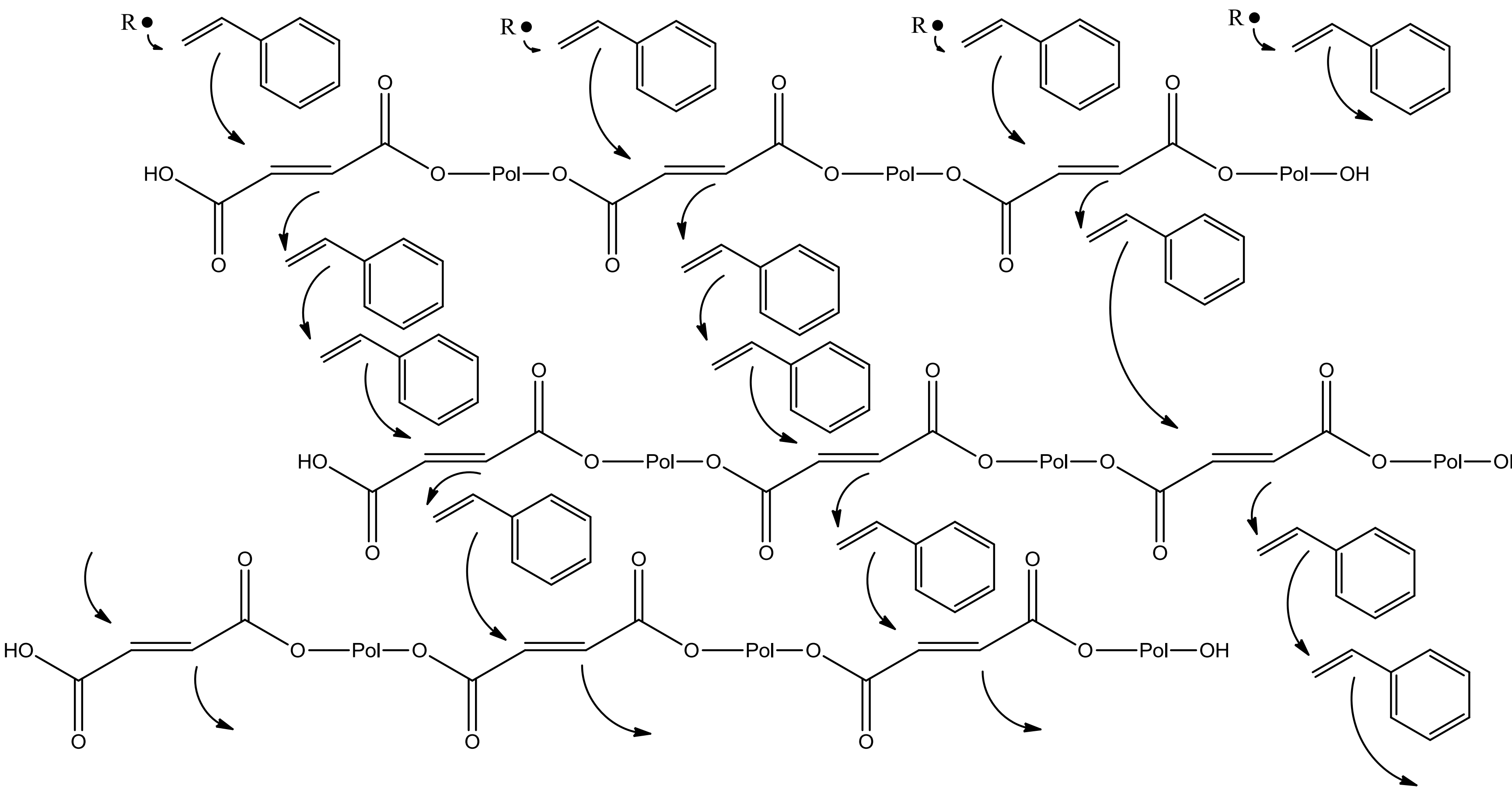
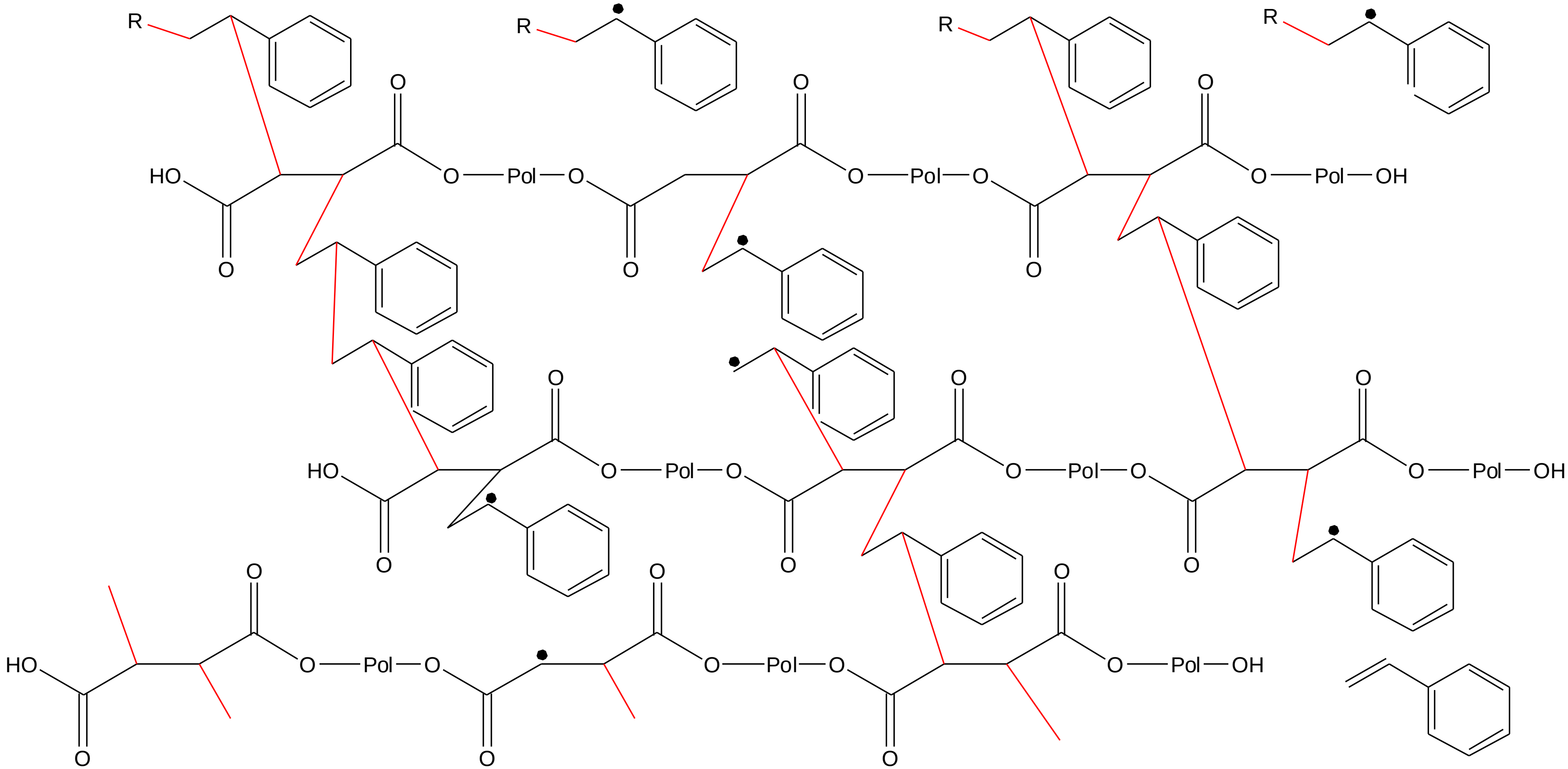
BASICS OF CURE THROUGH FREE RADICAL POLYMERIZATION



CURE MECHANISM



GLASSY STATE WITH UNREACTED RADICALS AND DOUBLE BONDS



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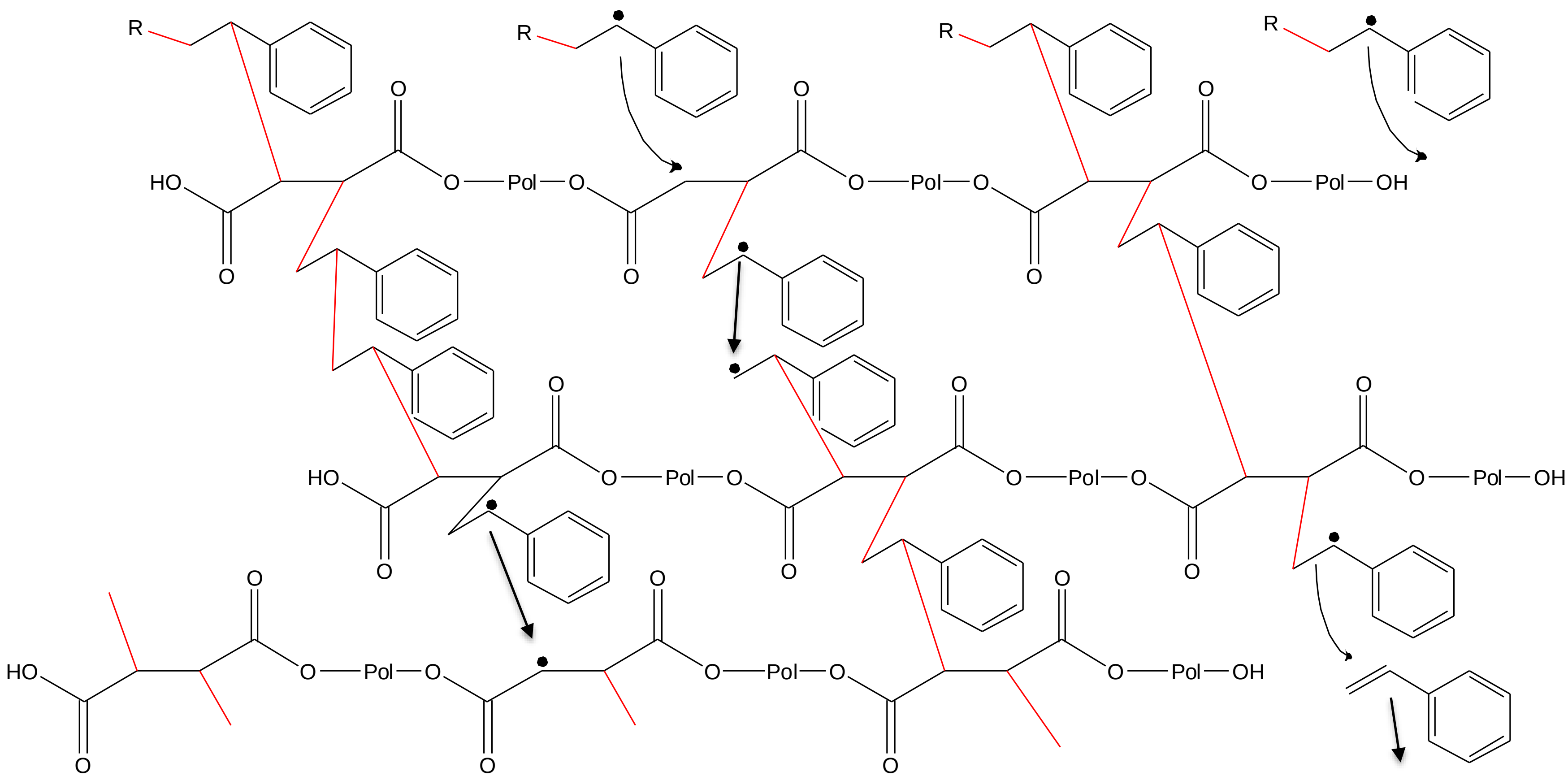
POST CURE: REINITIATING FREE RADICAL POLYMERIZATION



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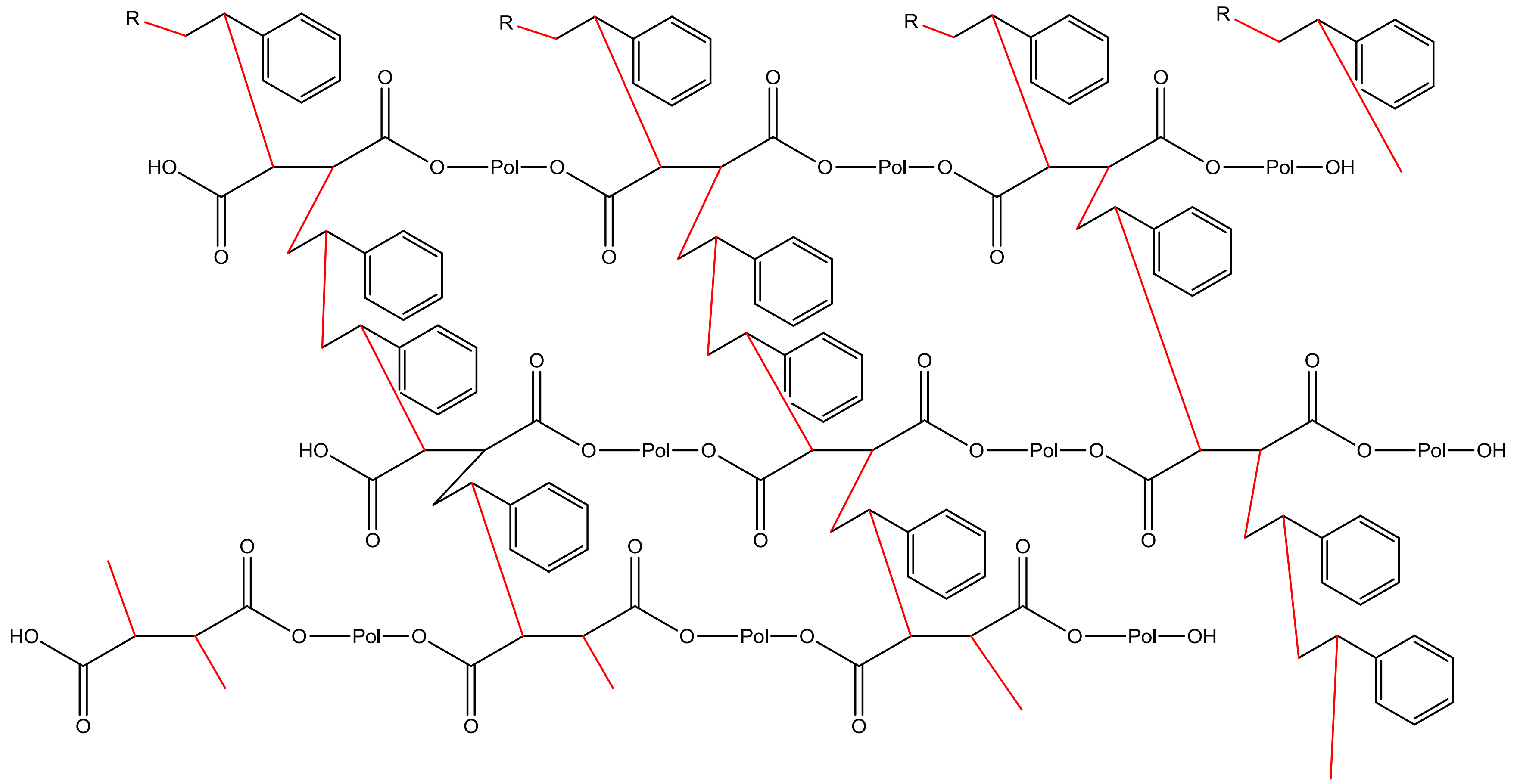
COMPLETE CURE ACHIEVED



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SOME CONSIDERATIONS ON POST-CURE

TIME AND TEMPERATURE

- Norm suggests post-cure of 1 hour per mm of laminate, at a temperature close to HDT
- According EN13121-2 at least 4 hours at 80°C or HDT (for certain media)
- According DIN18820: 1 hour per mm laminate thickness, at maximum 100°C for 15 hours, but at least 5 hours at minimum 80°C, and slow cooling down

IMPORTANT

- Post-cure is strongly recommended in BPO/ amine cure system and should be done within 2 weeks after construction
- Obviously, post-curing temperature should also reach the inner part of the tank

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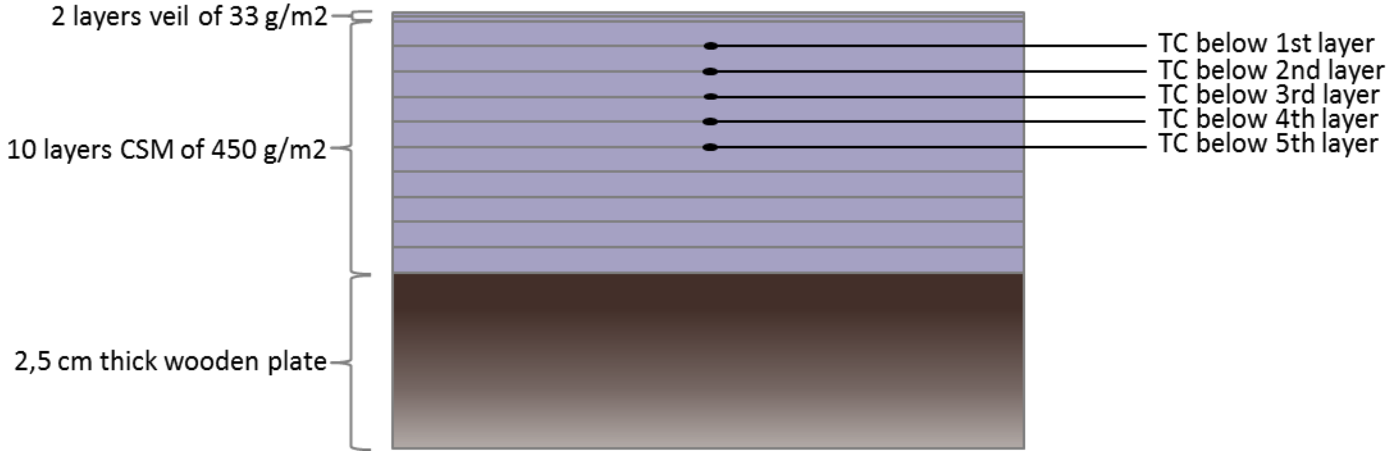
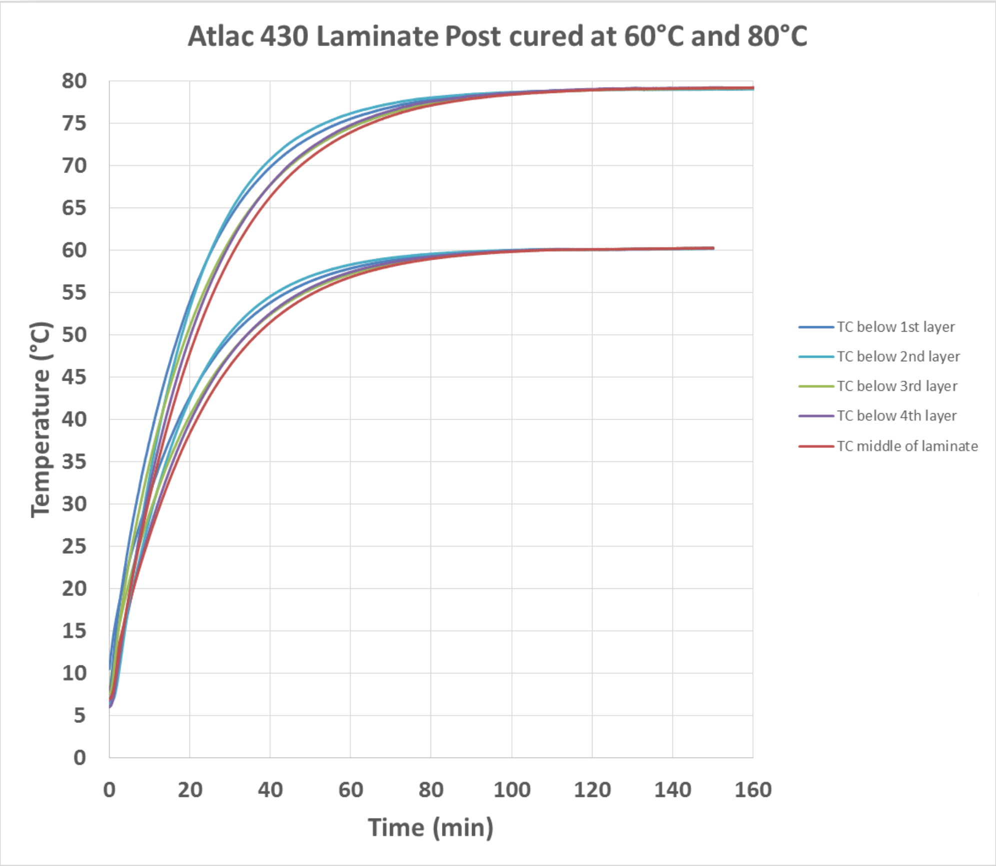
HEAT TRANSFER OVEN TEST

- Atlac 430 10 layer CSM laminate made with 5 thermocouples inside
- Hand lay-up
- Oven test at 60°C and 80°C



HEAT TRANSFER TAKES 2-2.5 HOURS FOR 4-5 MM THICKNESS



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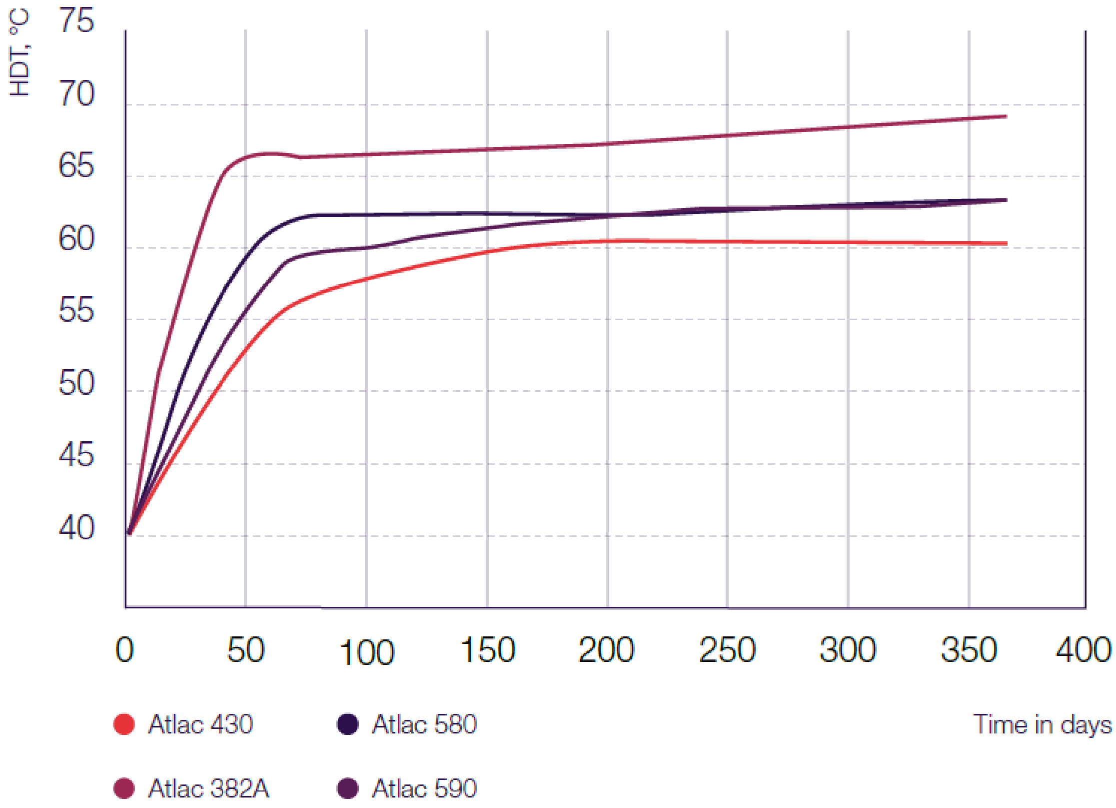
HDT LOWER WHEN CURED AT AMBIENT TEMPERATURES



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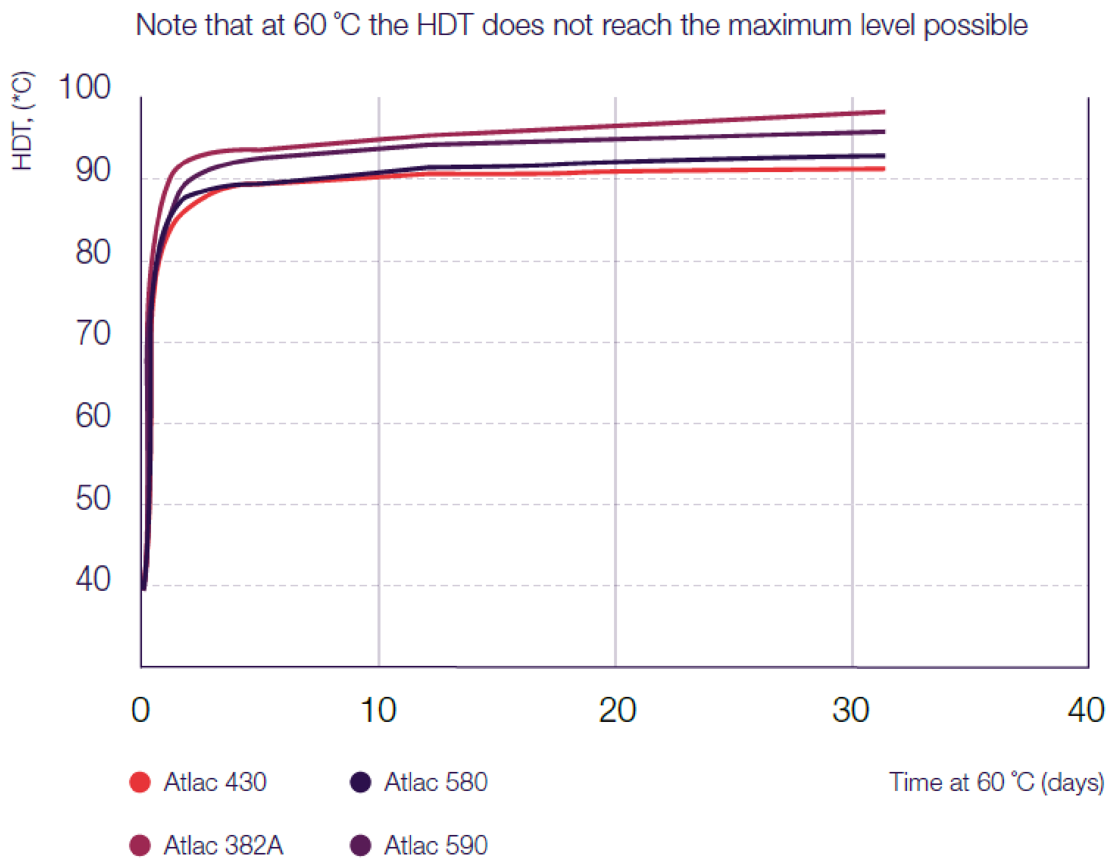
POST-CURE HELPS TO BUILD HDT

HDT VS. POST-CURE TIME AT 60 °C



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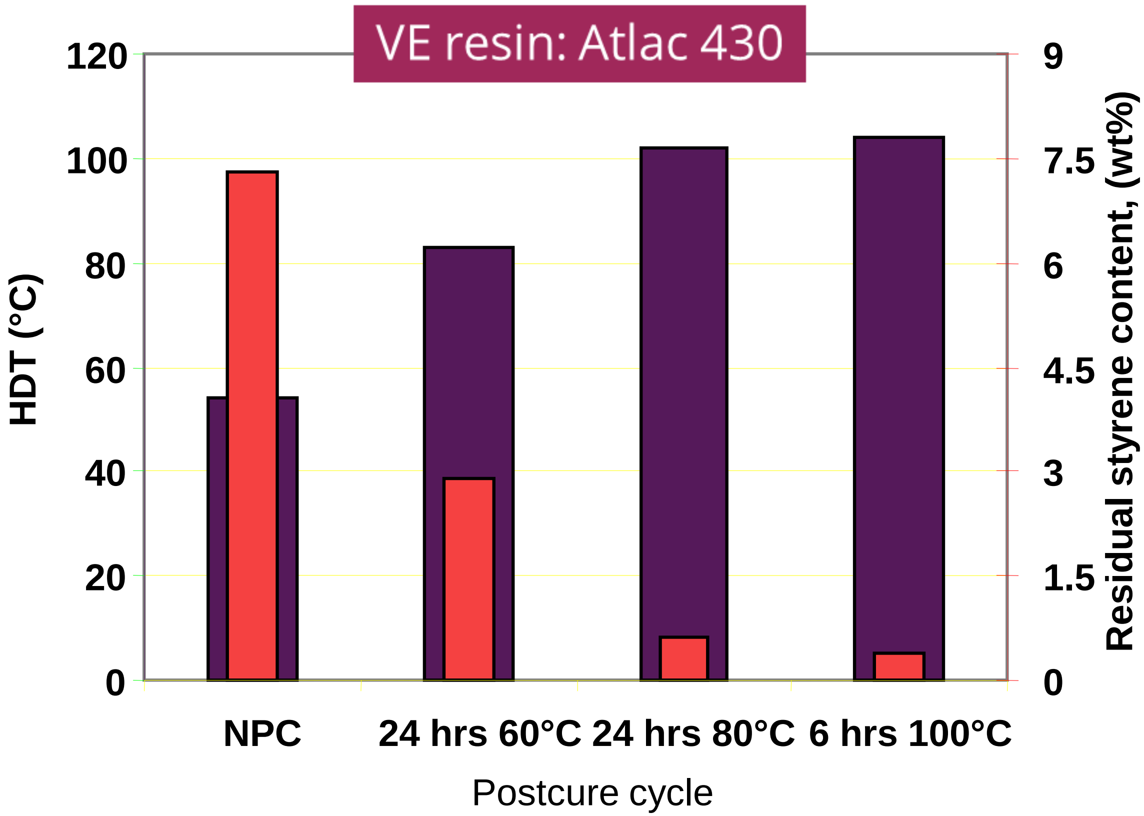
POST-CURE HAS MAJOR EFFECT ON HDT AND RESIDUAL STYRENE CONTENT



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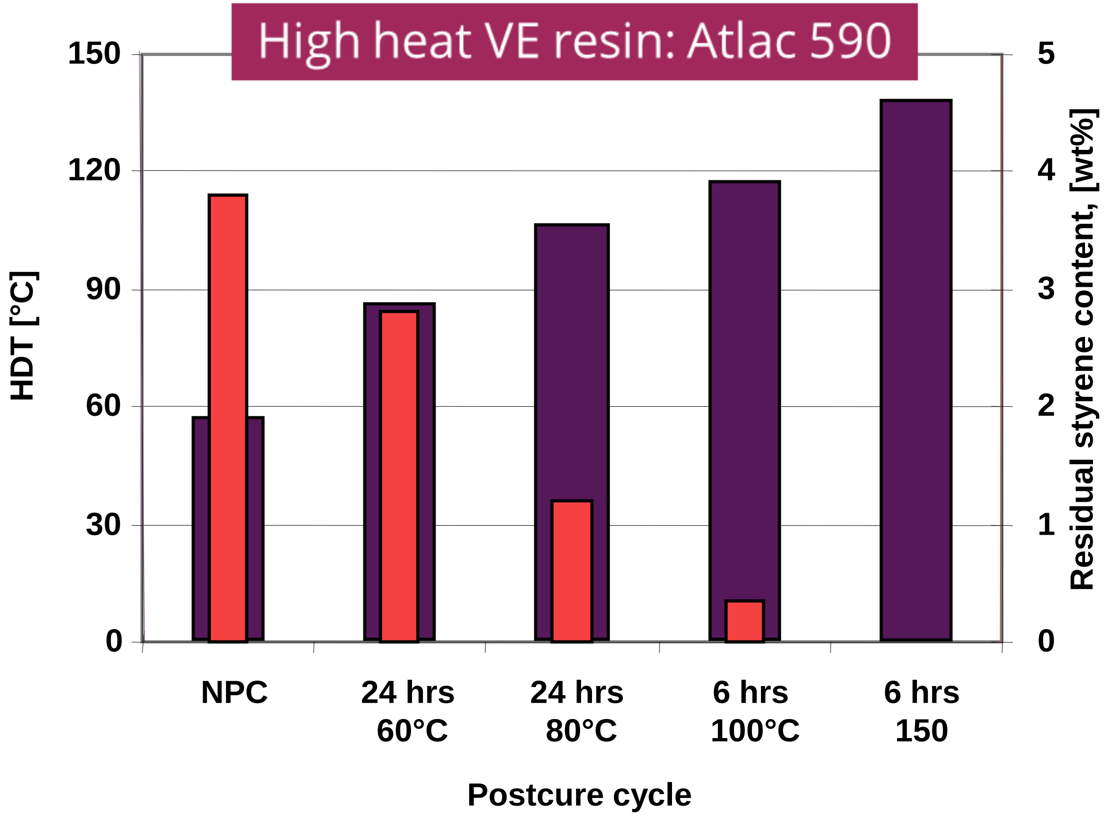

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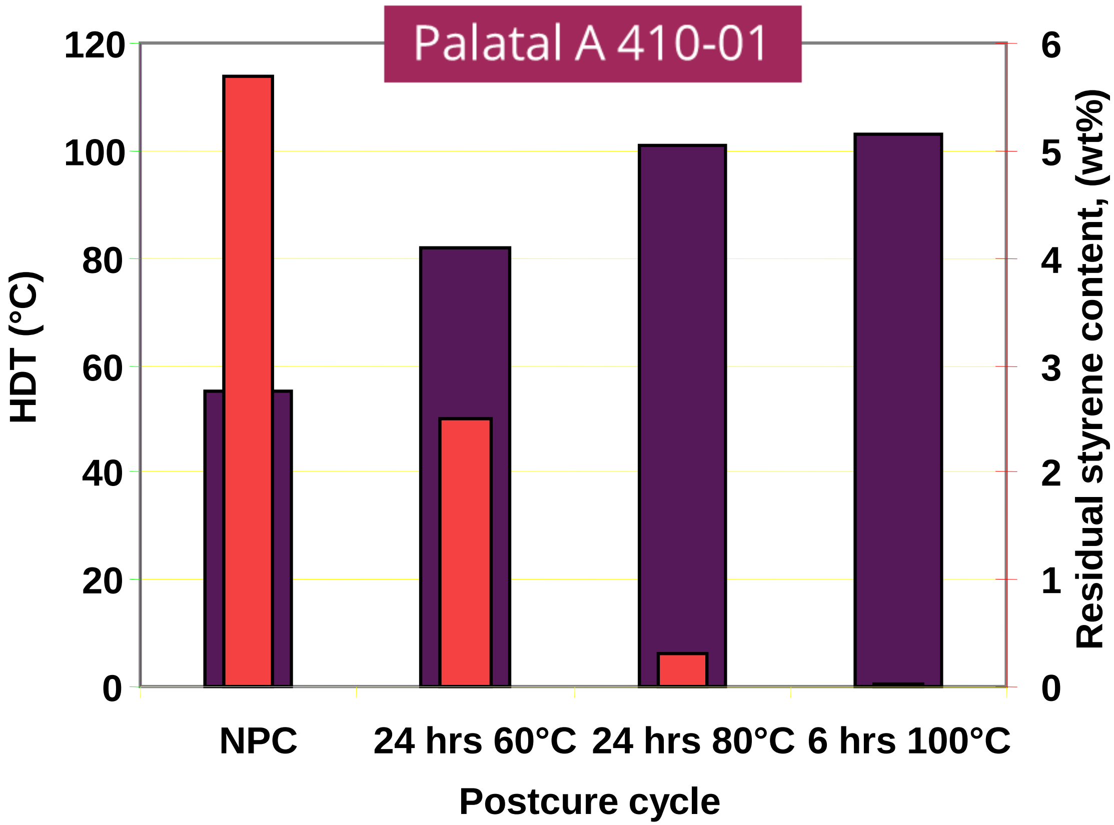
SAME FOR ISOPHTHALIC RESIN



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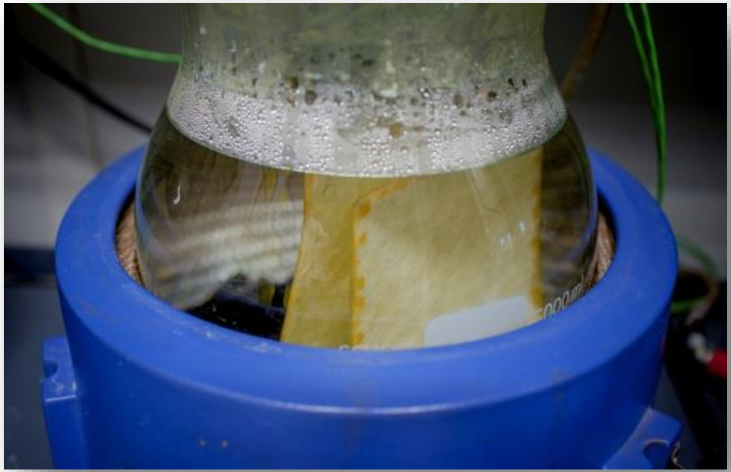
IN-HOUSE CHEMICAL RESISTANCE TESTING CAPABILITY

According to:
ASTM C 581and DIN 53393
(EN 977 - EN 13121-2)



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ASSESSMENT OF CHEMICAL RESISTANCE

Test Criteria

Aliancys interpretation method (based on ASTM C581 and EN13121):

$$\text{Retained Flexural Strength}\ [\%] = \frac{\text{Flexural Strength of specimen after test period}}{\text{Flexural Strenght of specimen after cure}} \times 100\%$$

$$\text{Retained Flexural Modulus}\ [\%] = \frac{\text{Flexural Modulus of specimen after test period}}{\text{Flexural Modulus of specimen after cure}} \times 100\%$$

$$\text{retention of Chemical Strength}\ [\%] = \frac{\text{retention of Flexural Strength} + \text{retention of Flexural Modulus}}{2}$$

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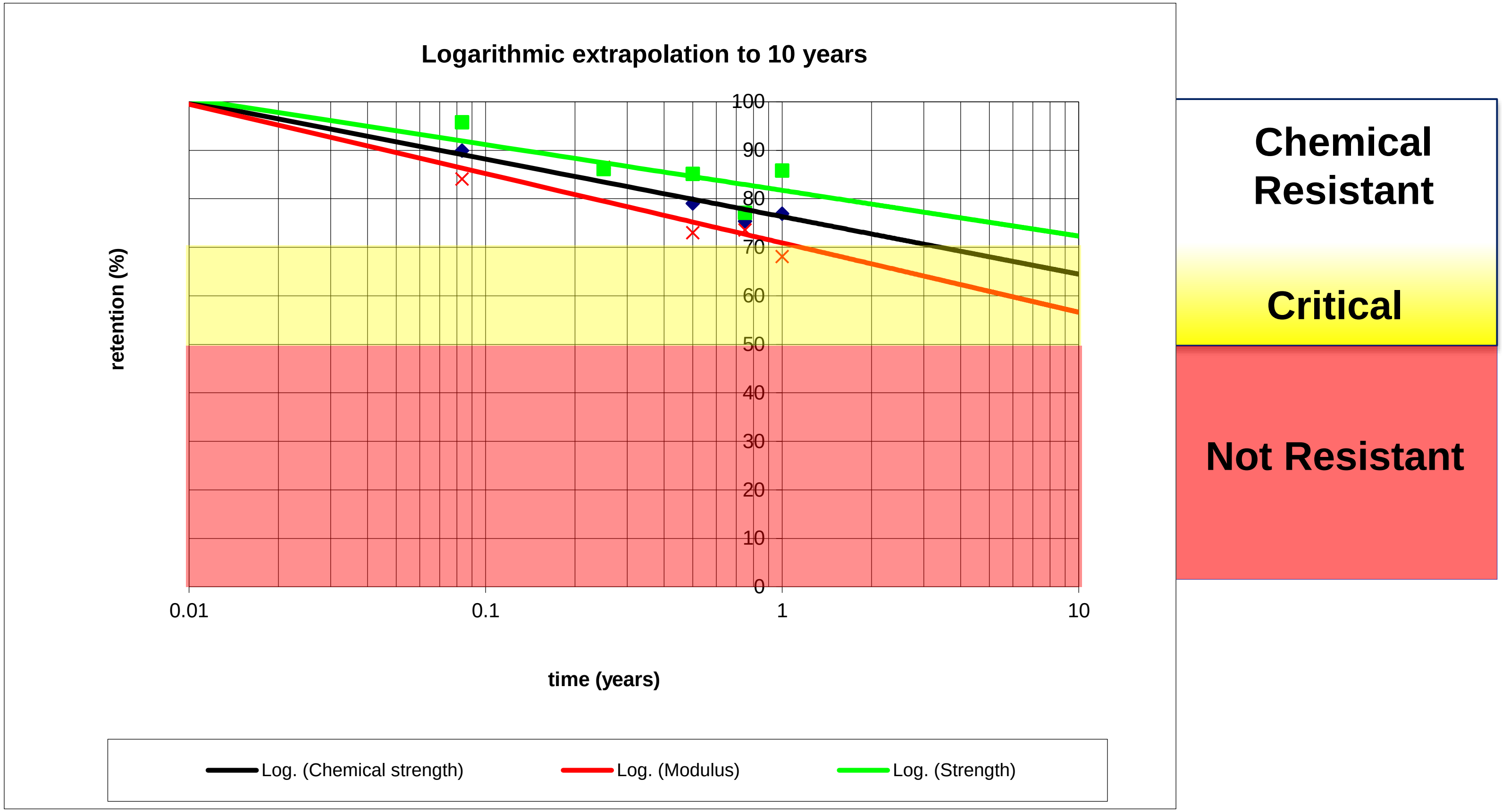
EXTRAPOLATION TO ASSESS LONG TERM PERFORMANCE



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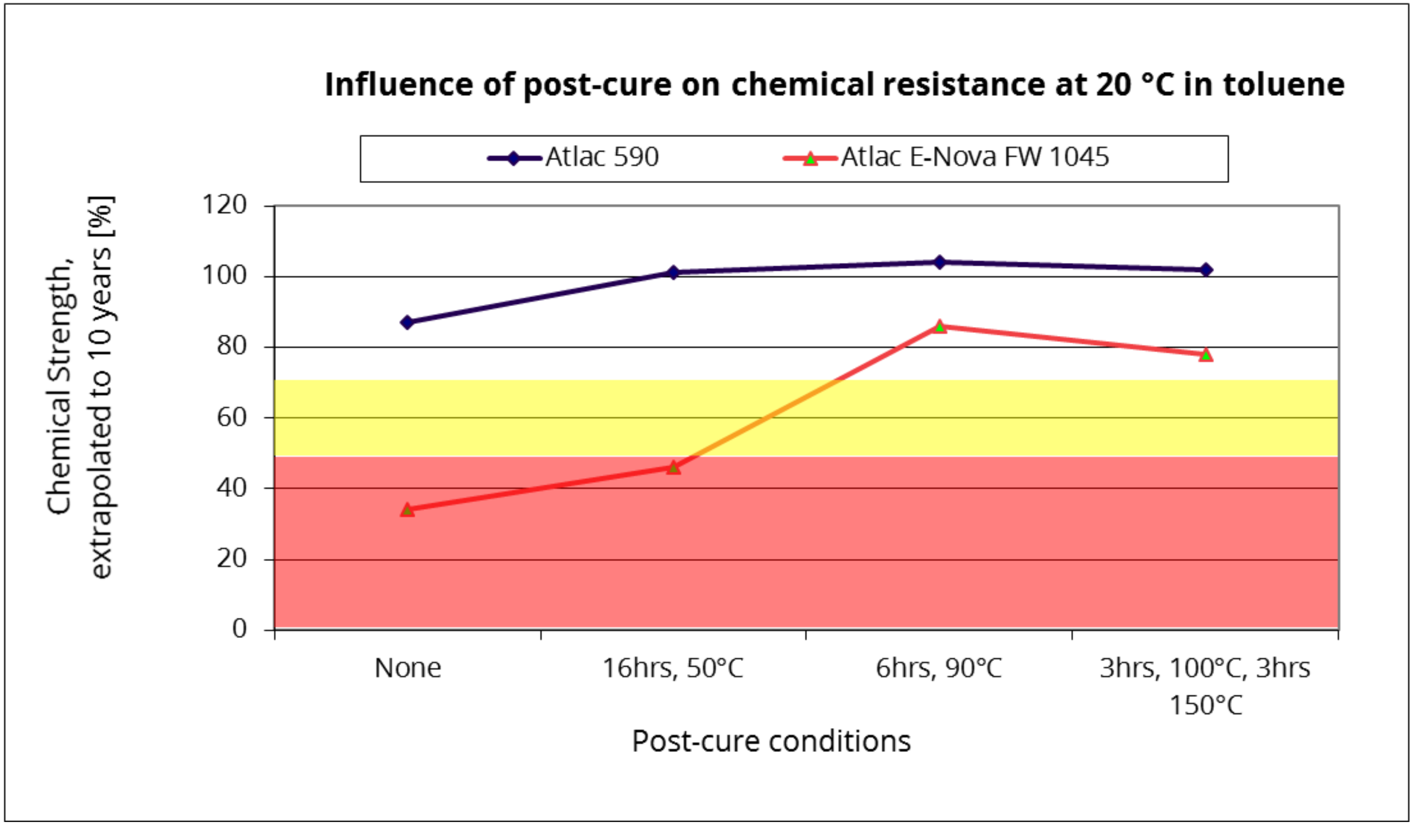
AT LOWER EXPOSURE TEMPERATURE EFFECT OF POST-CURE ALREADY VISIBLE



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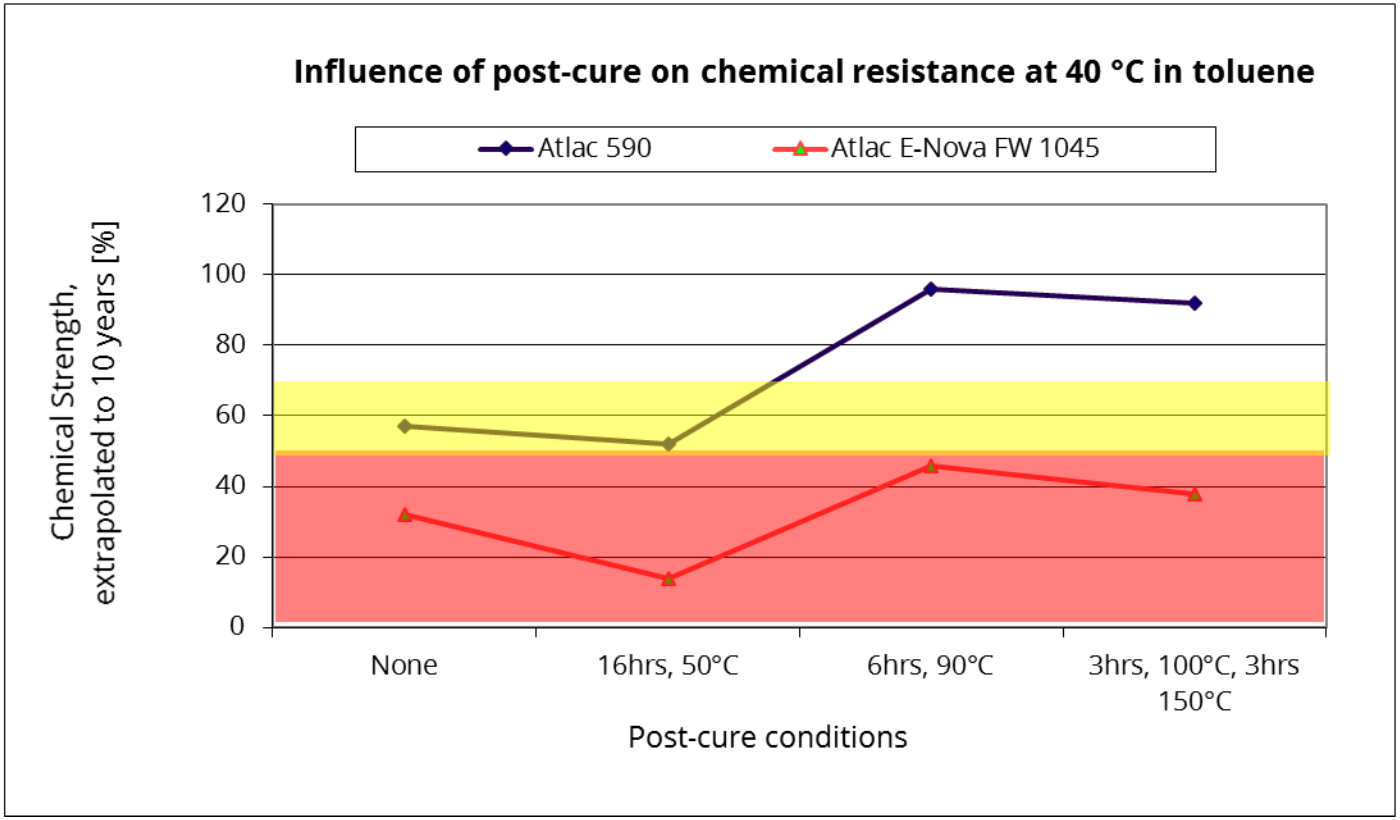
POST-CURE NECESSARY FOR BETTER RESISTANCE TO SOLVENTS



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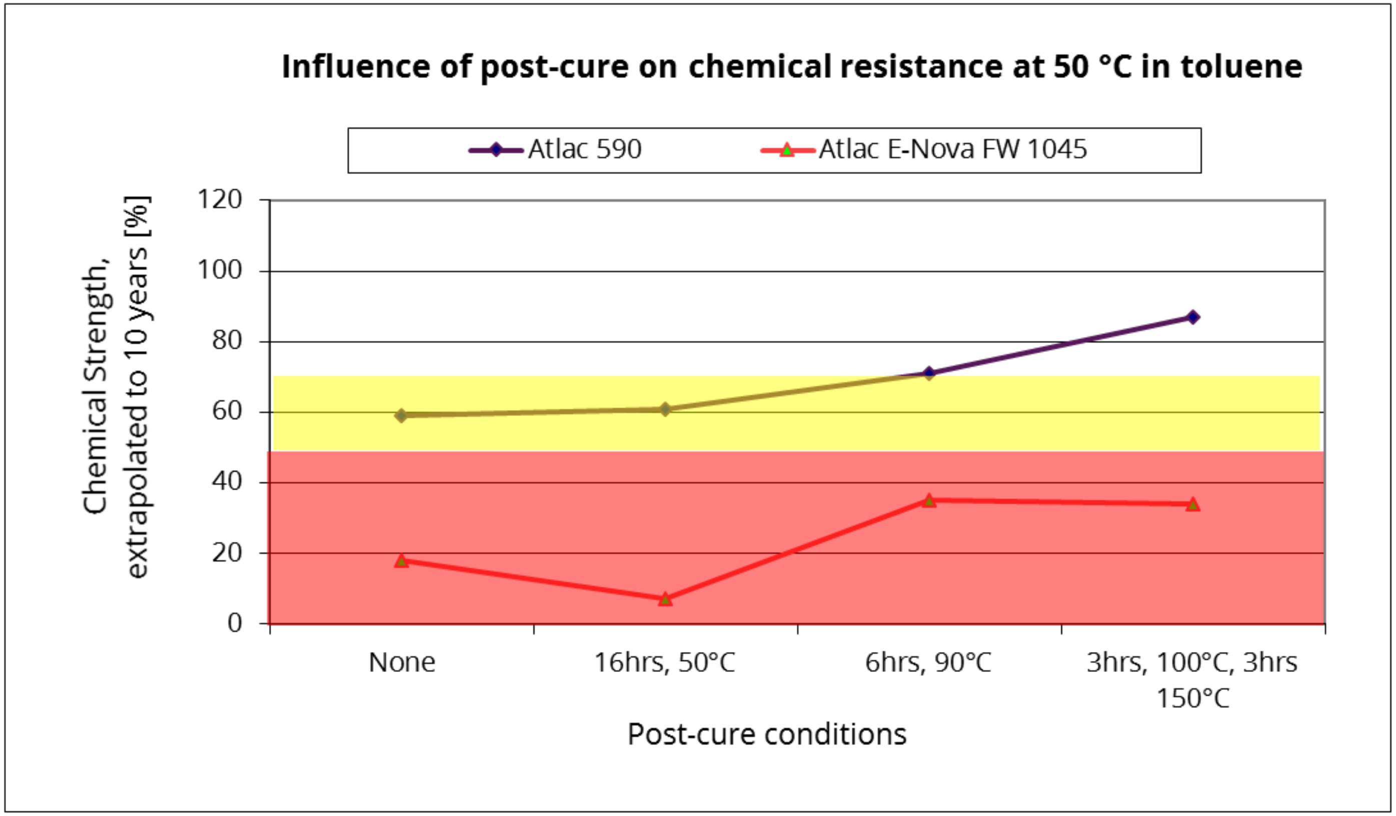
EFFECT POST-CURE MORE PRONOUNCED AT HIGHER EXPOSURE TEMPERATURES



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CONCLUSIONS ON POSTCURE

- HDT of ambient cured composites will stay around 50-60°C and it will not reach maximum HDT value
- No post-cure and mild post-cure results in same degree of chemical resistance
- In order to obtain highest possible heat resistance and chemical resistance, composite equipment has to be post-cured at a temperature around maximum HDT/Tg value
  - Will result in highest heat resistance
  - Will result in highest chemical resistance
- For less demanding circumstances follow the standards

GENERAL RECOMMENDATIONS

- Postcure is strongly recommended in BPO/amine cure system, and should be done within 2 weeks after construction
- Postcuring temperature should also reach the inner part of the tank
- Ambient temperature cured composite components will resist aqueous acid and salt solutions at ambient conditions
- Ambient temperature cured composite components will resist aqueous solutions also at elevated temperature.
  - Post curing process progresses faster then diffusion process

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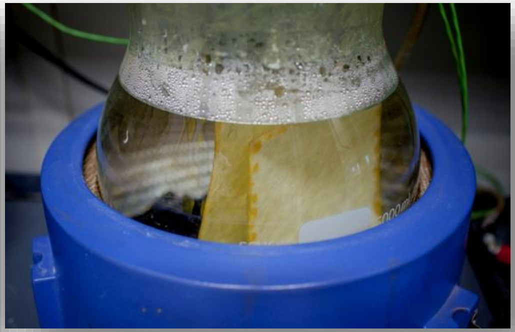
GENERAL RECOMMENDATIONS

- Barcol of finished part should be at least 80% of the value quoted by the resin manufacturer
  - To be measured on sections cut out after post cure
  - If not possible to take out sections, it is required to connect a test laminate to the tank representative for the entire process
  - It is not allowed to measure Barcol hardness on the tank wall (part interior), as this may damage the surface and reduce tank lifetime.

- The maximum service temperature given in the chemical resistance guide is based on fully postcured material in combination with the correct reinforcement material and curing system

PROVIDING EXPERTISE FOR OUR CUSTOMERS

- Excellent track record of use Atlac resins in Industrial markets
- Extensive experience in chemical resistance testing, laminate build-up for maximizing chemical resistance
- “Chemical Resistance Advice” as official document for your reference
- Experienced Technical Service team to support you in troubleshooting and continuous process improvement

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ALIANCYS CAN HELP IN MAKING THE BEST RESIN SELECTION FOR YOUR APPLICATION

- To make accurate recommendations we need to know:
  - Chemical environment, composition, concentrations, pH values, storage conditions
  - Service temperature, temperature profiles, maximum temperatures
  - Mechanical exposure, pressure, static and cyclic loading
  - Type of composite material/ build-up used (fiber volume, chemical resistance layer)
  - Equipment and process
- Available in 5 languages
- chemical.resistance@aliancys.com

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Technical advice

To	ABCD	From	Ronald Uitterdijk
For the attention of	S. Cole	Phone	+31 38 4569241
Fax		E-mail	Ronald.Uitterdijk@dsm.com
Your Reference		Our reference	1200145
E-mail	scole@abc.com	Date	2012/04/13

Dear Mr. Cole,

Find hereby our advice regarding your question about resin suitability for Nitric acid.

Kind regards,
Ronald

Information

Working temperature 40 °C

Chemical(s)	Information	Concentration
Nitric Acid		15 %

Advice

Based on the above data and on the information currently available, on behalf of DSM Composite Resins AG, we suggest the following product(s) for the mentioned application

ATLAC 382
ATLAC 4010
ATLAC 430
ATLAC 580
ATLAC 590
Atlac E-Nova FW 1045
Atlac E-Nova FW 2045
PALATAL A 410-01

A prerequisite of this recommendation is that the product must be well fabricated, well designed and properly cured. The recommendation is derived from data generated from the testing of fully post-cured composite materials; further information on post-curing is available in the product data sheet or by contacting your local technical service.

Additional notes

Acid resistant glass should be used in chemical resistant barrier

Signed by	Ronald Uitterdijk
Date	2012/04/13

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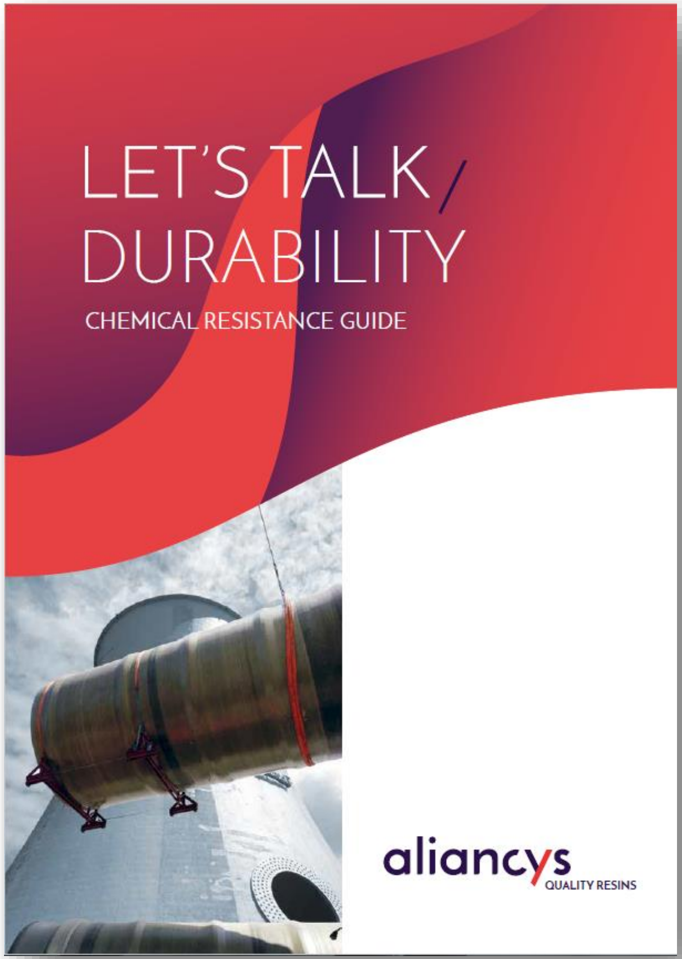
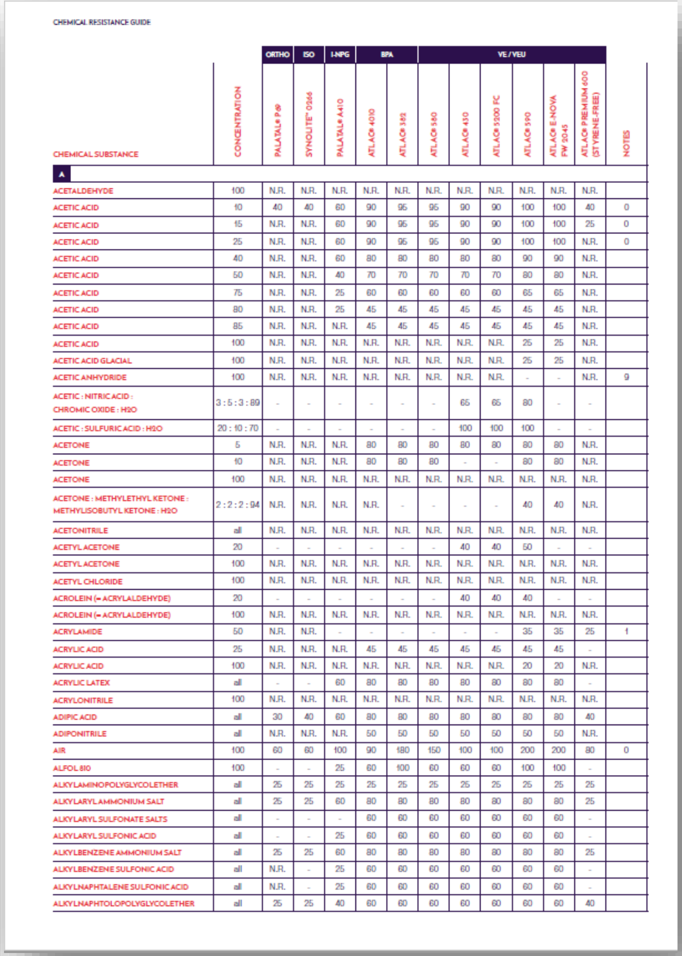
CHEMICAL RESISTANCE GUIDE




CHEMICAL RESISTANCE GUIDE

		ORTHO	ISO	I-NPG	BPA		VE / VEU						
CHEMICAL SUBSTANCE	CONCENTRATION	PALATAL® P69	SYNOLITE™ 0266	PALATAL® A410	ATLAC® 4010	ATLAC® 382	ATLAC® 580	ATLAC® 430	ATLAC® 5200 FC	ATLAC® 590	ATLAC® E-NOVA FW 2045	ATLAC® PREMIUM 600 (STYRENE-FREE)	NOTES
A													
ACETALDEHYDE	100	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	
ACETIC ACID	10	40	40	60	90	95	95	90	90	100	100	40	0
ACETIC ACID	15	N.R.	N.R.	60	90	95	95	90	90	100	100	25	0
ACETIC ACID	25	N.R.	N.R.	60	90	95	95	90	90	100	100	N.R.	0
ACETIC ACID	40	N.R.	N.R.	60	80	80	80	80	80	90	90	N.R.	
ACETIC ACID	50	N.R.	N.R.	40	70	70	70	70	70	80	80	N.R.	
ACETIC ACID	75	N.R.	N.R.	25	60	60	60	60	60	65	65	N.R.	
ACETIC ACID	80	N.R.	N.R.	25	45	45	45	45	45	45	45	N.R.	
ACETIC ACID	85	N.R.	N.R.	N.R.	45	45	45	45	45	45	45	N.R.	
ACETIC ACID	100	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	25	25	N.R.	
ACETIC ACID GLACIAL	100	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	25	25	N.R.	
ACETIC ANHYDRIDE	100	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	-	-	N.R.	9
ACETIC : NITRIC ACID : CHROMIC OXIDE : H2O	3 : 5 : 3 : 89	-	-	-	-	-	-	65	65	80	-	-	
ACETIC : SULFURIC ACID : H2O	20 : 10 : 70	-	-	-	-	-	-	100	100	100	-	-	
ACETONE	5	N.R.	N.R.	N.R.	80	80	80	80	80	80	80	N.R.	
ACETONE	10	N.R.	N.R.	N.R.	80	80	80	-	-	80	80	N.R.	
ACETONE	100	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	
ACETONE : METHYLETHYL KETONE : METHYLISOBUTYL KETONE : H2O	2 : 2 : 2 : 94	N.R.	N.R.	N.R.	N.R.	-	-	-	-	40	40	N.R.	
ACETONITRILE	all	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	
ACETYL ACETONE	20	-	-	-	-	-	-	40	40	50	-	-	
ACETYL ACETONE	100	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	
ACETYL CHLORIDE	100	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	
ACROLEIN (= ACRYLALDEHYDE)	20	-	-	-	-	-	-	40	40	40	-	-	
ACROLEIN (= ACRYLALDEHYDE)	100	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	
ACRYLAMIDE	50	N.R.	N.R.	-	-	-	-	-	-	35	35	25	1
ACRYLIC ACID	25	N.R.	N.R.	N.R.	45	45	45	45	45	45	45	-	
ACRYLIC ACID	100	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	20	20	N.R.	
ACRYLIC LATEX	all	-	-	60	80	80	80	80	80	80	80	-	
ACRYLONITRILE	100	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	
ADIPIC ACID	all	30	40	60	80	80	80	80	80	80	80	40	
ADIPONITRILE	all	N.R.	N.R.	N.R.	50	50	50	50	50	50	50	N.R.	
AIR	100	60	60	100	90	180	150	100	100	200	200	80	0
ALFOL 810	100	-	-	25	60	100	60	60	60	100	100	-	
ALKYLAMINOPOLYGLYCOLETHER	all	25	25	25	25	25	25	25	25	25	25	25	
ALKYLARYL AMMONIUM SALT	all	25	25	60	80	80	80	80	80	80	80	25	
ALKYLARYL SULFONATE SALTS	all	-	-	-	60	60	60	60	60	60	60	-	
ALKYLARYL SULFONIC ACID	all	-	-	25	60	60	60	60	60	60	60	-	
ALKYLBENZENE AMMONIUM SALT	all	25	25	60	80	80	80	80	80	80	80	25	
ALKYLBENZENE SULFONIC ACID	all	N.R.	-	25	60	60	60	60	60	60	60	-	
ALKYLNAPHTALENE SULFONIC ACID	all	N.R.	-	25	60	60	60	60	60	60	60	-	
ALKYLNAPHTOLOPOLYGLYCOLETHER	all	25	25	40	60	60	60	60	60	60	60	40	

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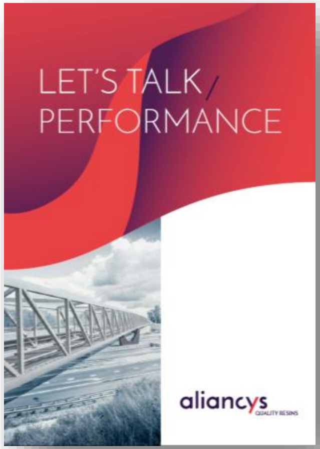
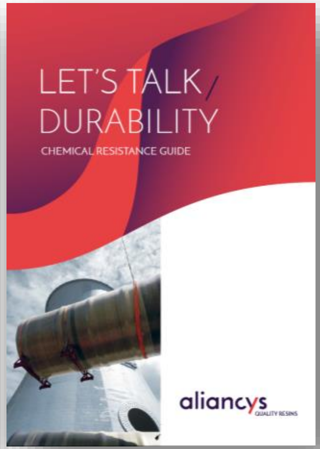
MORE INFORMATION

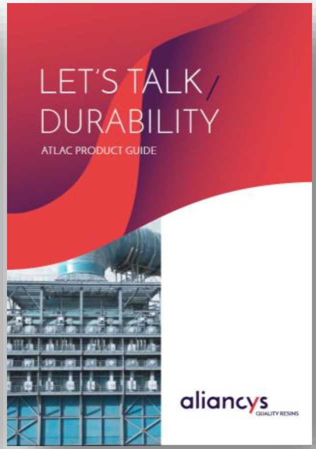
- Product and case study information on www.aliancys.com
- Please contact your Aliancys Technical Service representative for more detailed information and for our Chemical Resistance information service



Product Selector Guide



Chemical Resistance Guide



Atlac Product Guide

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