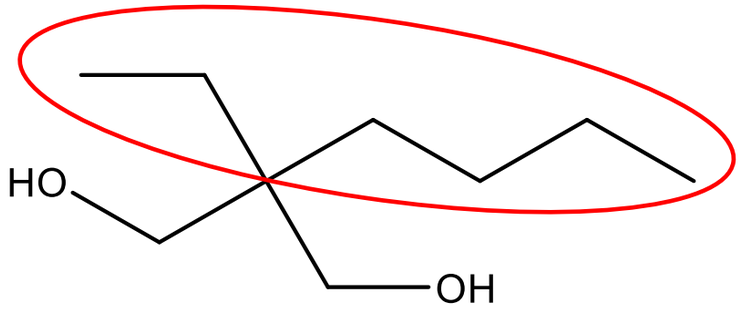
SYNOXOL BEPD™ in PES Powder Coatings

Contents

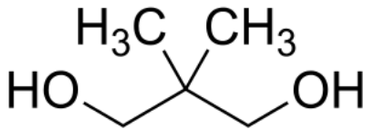
Why Synoxol™ BEPD?

High performance glycol (diol) used in polyester, unsaturated polyester and polyurethane resin production

Structurally analogue to NPG



Synoxol™ BEPD



NPG

Most obvious difference between the two molecules is the increased chain length in the 2 position on the propanediol backbone

•In Powder Coatings key performance attributes of BEPD are:

- Bloom free exterior durable coatings
- Increase in reactivity through adipic acid replacement

SYNOXOL™ BEPD – Powder Coatings

Target applications

Automotive

Architectural

ACE

General Industrial

IT

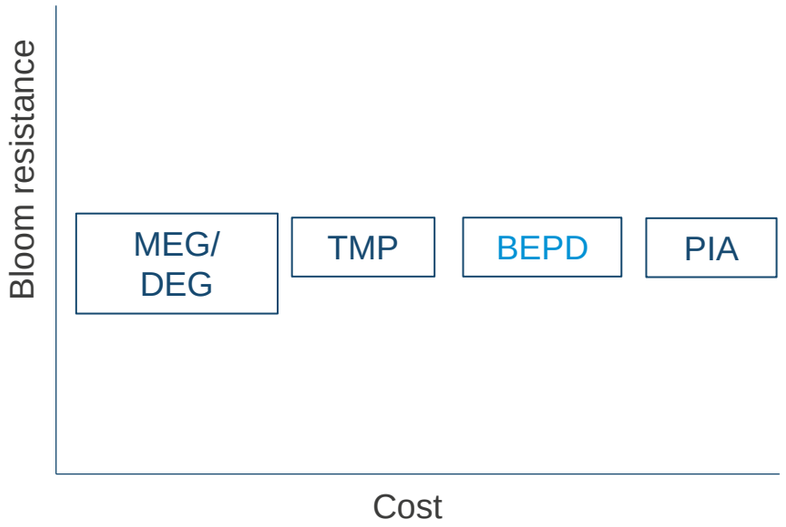
Furniture

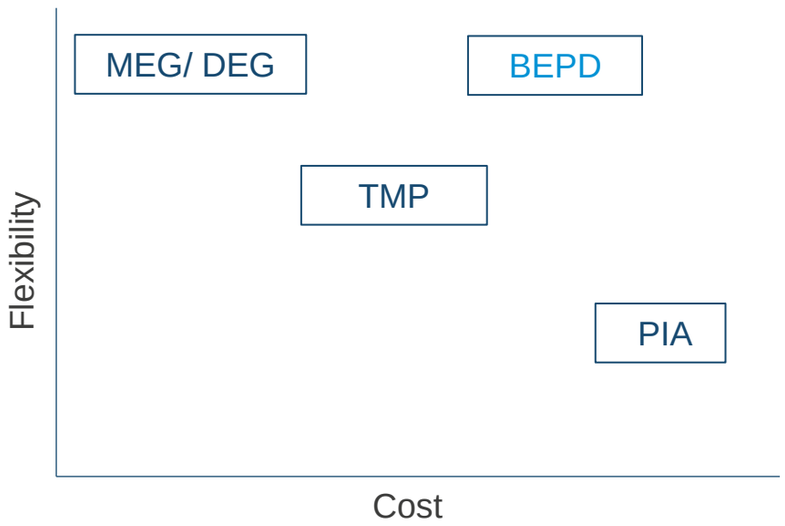
Domestic Appliance

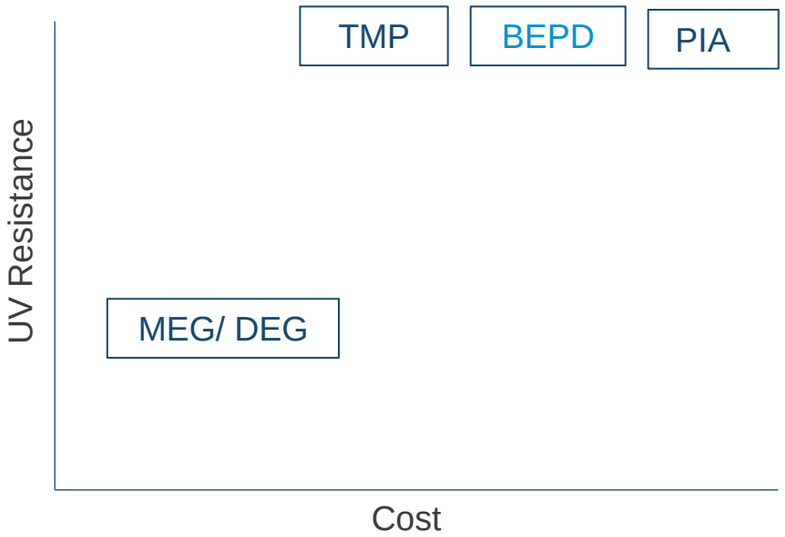
Trade Coaters

Functional

Technology Positioning – Bloom Resistance







Value Propositions

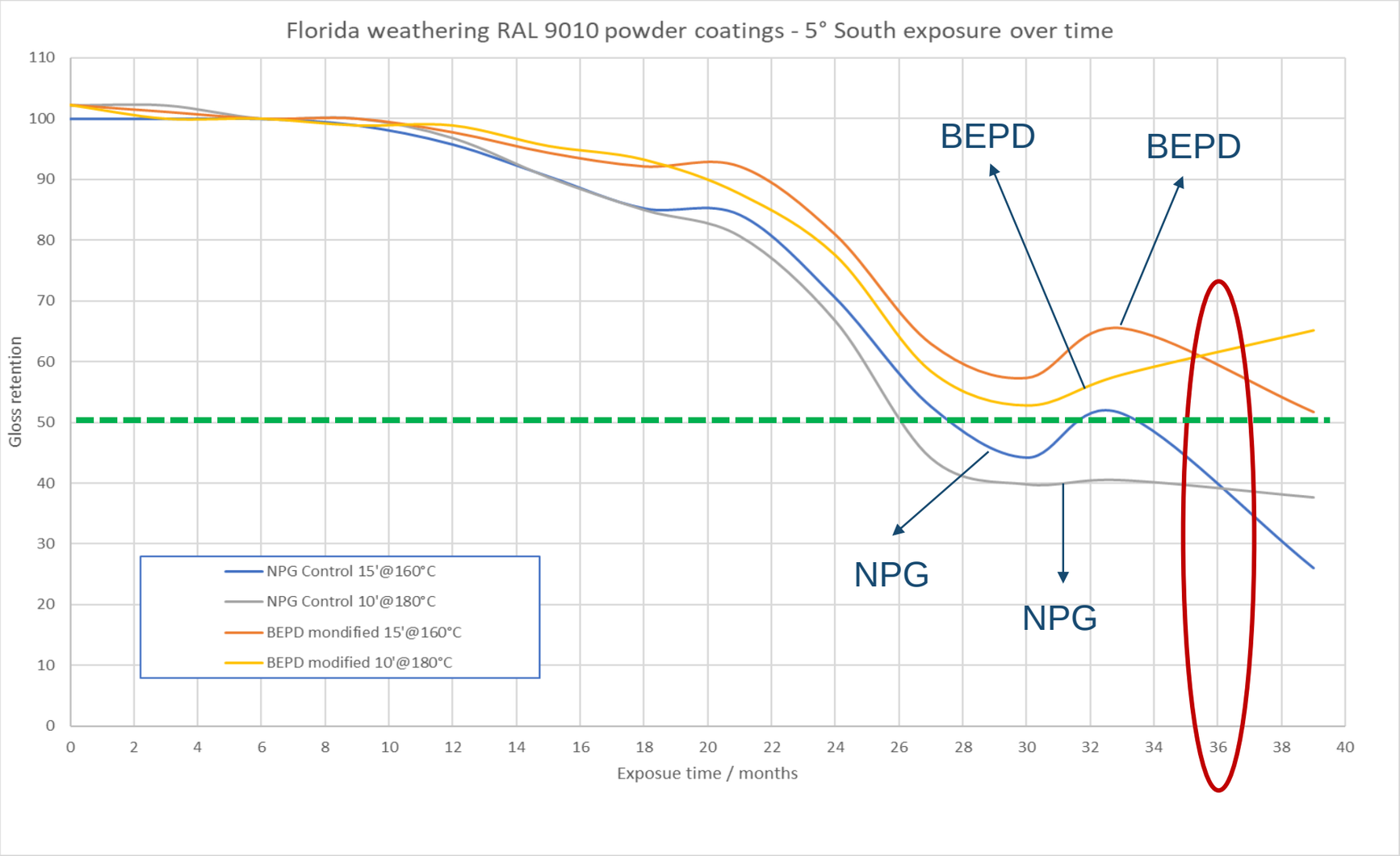
Bloom Resistance

- Suitable for the design of **standard- and super- durable** PES resins & powder coatings for long term UV resistance
  - Architectural / Industrial / Superdurable resin product groups
- Allows formulating **bloom free exterior grade** powder coatings without a compromise on **flexibility**
- Delivers **bloom resistance** resins with **as little as 5% w/w replacement for NPG** – no further resin modification is required
- Implementation **does not require any new polymer registration/ notification** (<2wt% on total formulation) – straight drop in resin formulation

Productivity Increase

- After **5' @ 180°C cure cycle** SYNOXOL™ BEPD can deliver the same performance as super-durable benchmark after **15' @ 180°C** in terms of:
  - MEK double rubs
  - Impact resistance
  - Toluene (chemical) resistance
- Reactivity improvement (higher cross-linking density) would result in up to **3 times faster production rates** (5' vs 15') at 180°C cure temp
- 20% of NPG by weight is replaced with SynoxolTM BEPD along with other acid adjustments to maintain Tg

Super-durable UV Resistance



Results from Florida outdoor testing

93:7 PE:HAA – Super-durable
High Gloss

Qualicoat Class 2 (super-durable) requirement:

- After 12 months: % gloss retention
- After 24 months: % gloss retention
- After 35 months: % gloss retention

BEPD Results

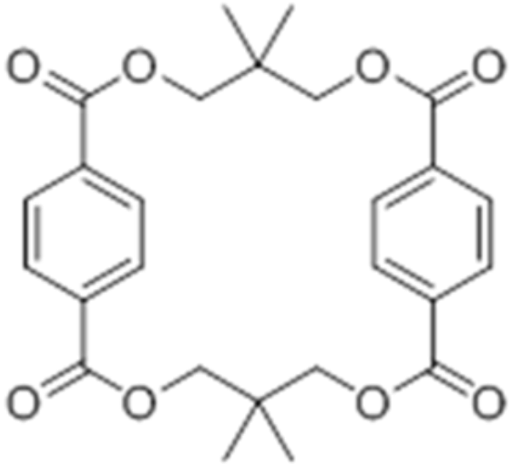
- After 12 months: % gloss retention
- After 24 months: % gloss retention
- After 39 months: % gloss retention

BEPD is suitable for the outdoor applications of Architectural, Industrial & Superdurable PES resins

What is bloom or blooming?

Bloom: Known issue for TGIC & -HAA cured powder coatings at low cure temps

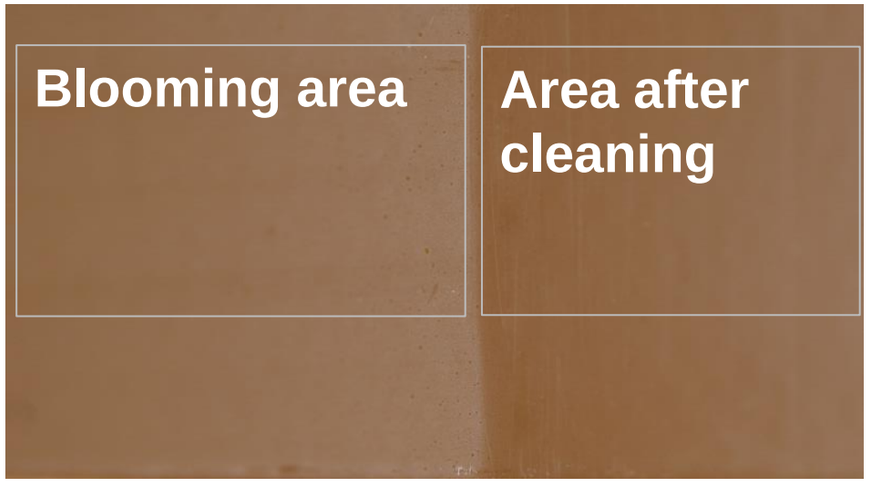
- Proven to be due to presence of cyclomers formed from reaction of NPG with TPA which takes place in most PES resins for powder coatings
- Problem is more prevalent where heavy metal parts are being coating and the peak metal temperature during the cure process is never reached
- Requires down time for parts to be wiped clean
- Clearly relevant for ACE segment. High aesthetic demands of Architectural and Automotive mean bloom is also undesirable in these two market segments as well.



NPG based cyclomer

- Relevant for Americas & China: Already a patent available on TGIC cured powder coatings which shows NPG-cyclomer formation is the root cause of bloom formation
- Relevant for Europe: No work has been done on –HAA to show that NPG-cyclomer formation is the root cause

BEPD reduce the amount of NPG-cyclomers and prevents blooming, consequently



PES resin samples – Bloom Resistance

Resin	D	C	B	A	(Control 1)	(Control 2)
Neopentyl glycol (NPG) / g	316.9	391.3	408.5	421.0	423.5	425.5
SYNOXOL™ BEPD / g	135.8	43.5	19.7	6.6	-	-
Trimethylol propane (TMP) / g	1.1	1.2	1.7	1.7	1.2	3.0
Terephthalic Acid (TPA) / g	576.9	596.1	599.5	602.2	604.1	581.0
Isophthalic acid (PIA) / g	110.0	113.6	114.2	118.2	118.2	104.0
Adipic acid (ADA) / g	-	-	-	-	-	33.0
Total charge / g	1141	1146	1148	1149	1147	1147
Reaction water / g	141	146	148	149	147	147
Theoretical properties at acid value = 35 mgKOH g^{-1}						
Hydroxyl number / mgKOH g^{-1}	9	9	9	9	5	9
Molecular weight (M_n) / Da	2600	2600	2600	2600	2800	3300
Molecular weight (M_w) / Da	5100	5100	5200	5200	5700	5300
Polydispersity	2.0	2.0	2.0	2.0	2.0	2.1
Functionality	1.6	1.6	1.5	1.5	1.8	2.0
Practically measured data						
Acid value / mgKOH g^{-1}	32.3	33.8	32.7	34.7	35.8	35.2
Hydroxyl number / mgKOH g^{-1}	4.2	4.0	8.9	8.6	6.6	6.6
Molecular weight (M_n) / Da	3300	3300	2400	2400	3200	3200
Molecular weight (M_w) / Da	9100	9400	7400	7200	9000	9100
Polydispersity	2.8	2.9	3.1	3.0	2.8	2.8
T_g (onset) / °C	59.8	64.3	63.2	61.7	69.5	63.8
Viscosity (100% solids at 200 °C) / cP	3840	4200	2800	2040	4200	3240

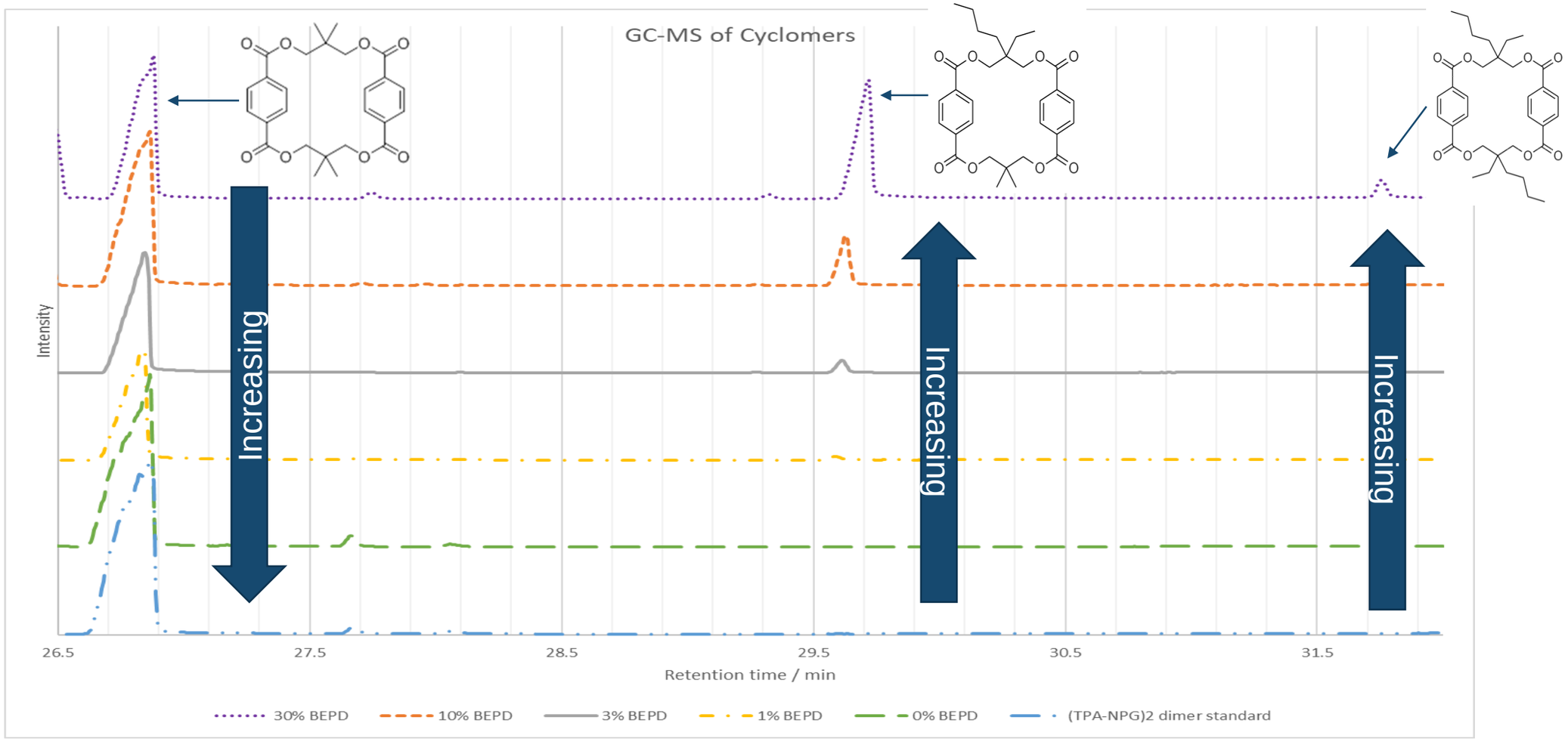
Visual assessment of bloom resistance

- SYNOXOL™ BEPD can be incorporated instead of NPG to reduce blooming
- As amount of SYNOXOL™ BEPD increases; resistance to bloom increases
- 2wt% BEPD addition in resin sufficient to give performance from 200° - 160°C cure temperatures
- This modification does not require polymer re-registration; so can be a straight drop in existing formulations

Gloss Readings at 60º & 20º measurement angles	Control 1	Control 2	Resin A	Resin B	Resin C	Resin D
NPG wt% replacement	0%	0%	30%	10%	5%	2%
10'@200°	92/72	91/72	91/73	91/73	90/67	91/75
15'@180°	93/76	92/75	92/76	93/76	92/72	92/76
30'@160°	77/66	77/68	93/79	94/79	93/76	80/70

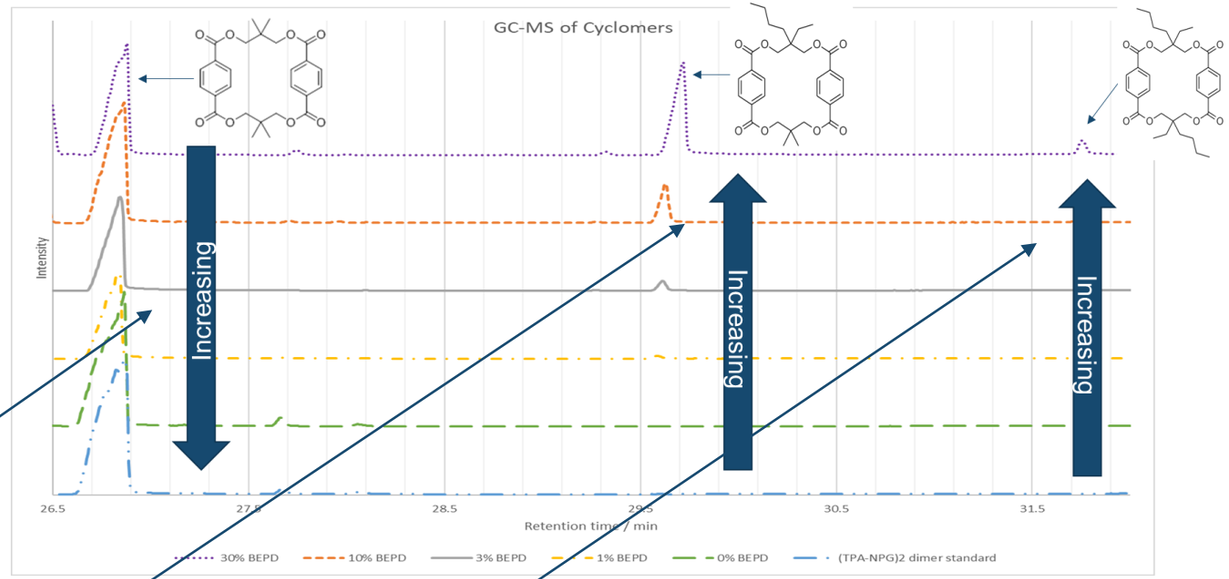
<2wt% BEPD content in PES resin

Analytical assessment of bloom resistance



Analytical assessment of bloom resistance

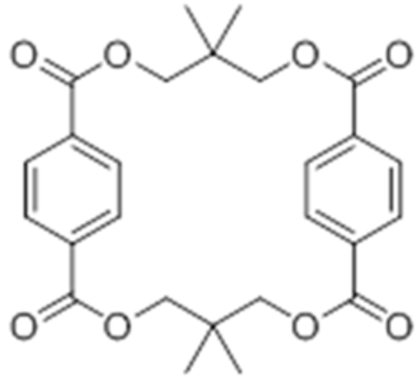
- GC-MS (Gas-Mass chromatography) technique applied
- As the amount of SYNOXOL™ BEPD is increased the % of the TPA-NPG cyclomer decreases to the point where this cyclomer is no longer present in sufficient quantities to cause blooming.
- Crystalline TPA-NPG cyclomer is replaced by amorphous SYNOXOL™ BEPD containing cyclomers which do not form on the metal surface as it cools

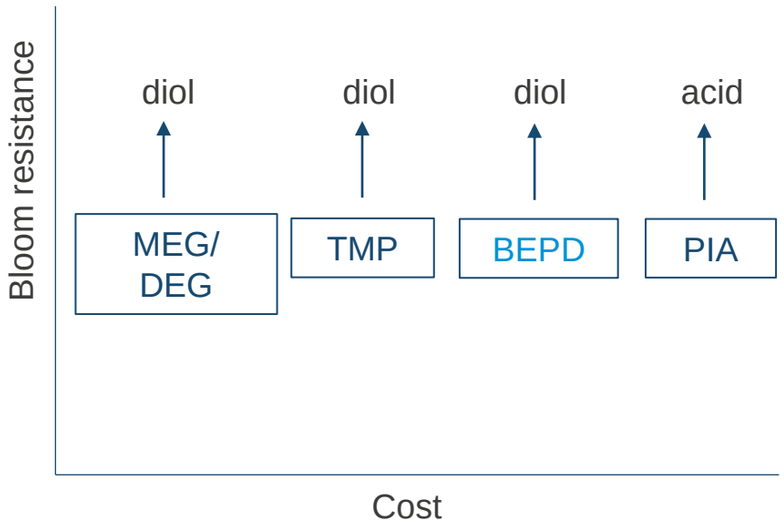


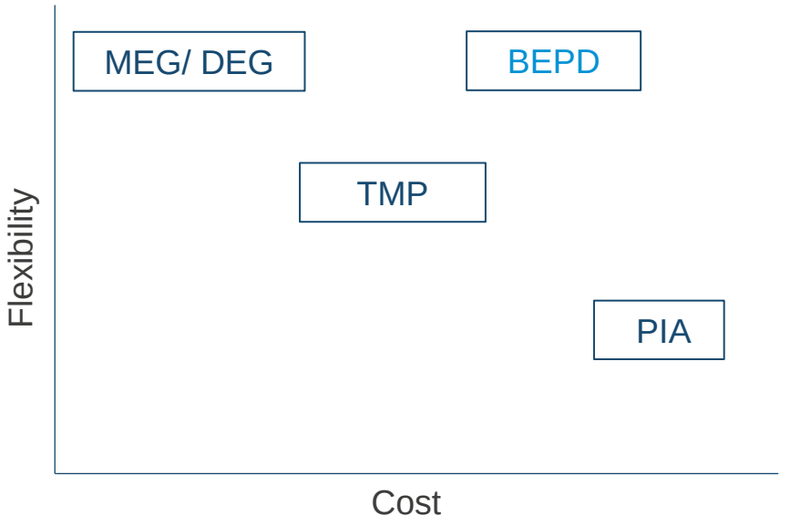
Resin	TPA-NPG cyclomer / wt%	TPA-NPG-SYNOXOL™ BEPD cyclomer / wt%	TPA-SYNOXOL BEPD™ cyclomer / wt%	Bloom at 160 °C	Bloom at 140 °C
A	0.56	0.33	0.03	No	No
B	0.76	0.11	0.00	No	No
C	0.75	0.04	0.00	No	Yes
D	0.80	0.01	0.00	Yes	Yes
Control 1	0.87	0.00	0.00	Yes	Yes
Control 2	0.71	0.00	0.00	Yes	Yes

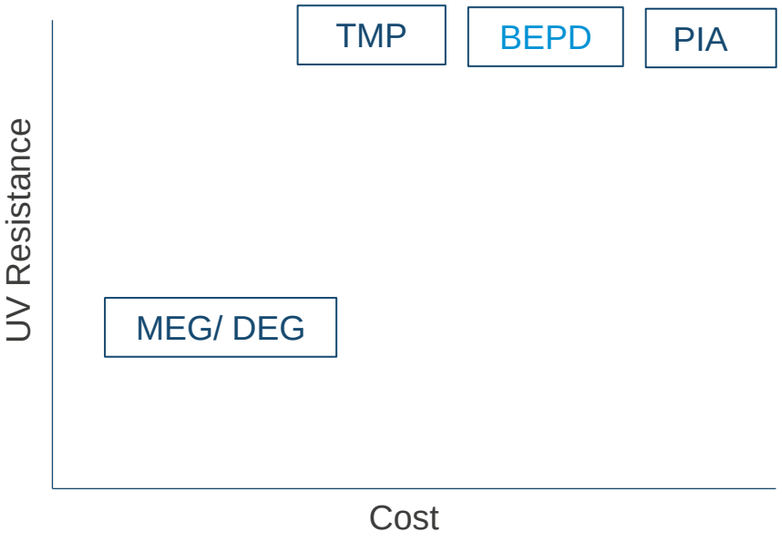
Bloom Resistance Solution

- TPA – NPG cyclomer statistically inevitable
- Need to reduce the amount of TPA-NPG cyclomer present -
  - Either replace some NPG with another diol
  - Or replace TPA with another acid (PIA)
  - Approaches other than BEPD involve compromise on flexibility or UV resistance
  - TMP is a complicated technical solution as it changes resin structure completely
  - BEPD represents a commercially feasible technical solution for exterior grade PES resin









Cost-Benefit Analysis – Bloom Resistance

Replacing NPG with BEPD at low levels can give improved resistance to low temperature blooming:

A 5wt% (based on total glycols) replacement provides resistance at temperatures down to 160°C

- This does not require any new polymer registration/ notification (<2wt% on total formulation)
- It is indistinguishable in the final coating (as measured by FTIR)
- Has no negative impact upon UV durability (unlike other glycols e.g. MEG)
- Has no negative impact upon flexibility (unlike PIA)

Resin	Resin cost/ kg	Coating cost/ Kg	Resin composition	Bloom resistant @
Control	Benchmark	Benchmark	NPG / BEPD / TMP : 99.7/ 0 /0.3 PTA / PIA / ADA : 84 / 16	180ºC
BEPD modified	+3%	+ 2.4% brown + 1.8% white	NPG / BEPD / TMP : 95/ 4.6 / 0.4 PTA / PIA / ADA : 84 / 16 / 0	160ºC
Acid (PIA) modified	+8%	+ 6.4% brown + 4.8% white	NPG / BEPD / TMP : 100 / 0 / 0 PTA / PIA / ADA : 70/ 24.5 / 5.5	160ºC

Assumption: Brown coating formulation = 80% binder; White coating formulation = 60% binder

Value Propositions

Bloom Resistance

- Suitable for the design of **standard- and super- durable** PES resins & powder coatings for long term UV resistance
  - Architectural / Industrial / Superdurable resin product groups
- Allows formulating **bloom free exterior grade** powder coatings without a compromise on **flexibility**
- Delivers **bloom resistance** resins with **as little as 5% w/w replacement for NPG** – no further resin modification is required
- Implementation **does not require any new polymer registration/ notification** (<2wt% on total formulation) – straight drop in resin formulation

Productivity Increase

- After **5' @ 180°C cure cycle** SYNOXOL™ BEPD can deliver the same performance as super-durable benchmark after **15' @ 180°C** in terms of:
  - MEK double rubs
  - Impact resistance
  - Toluene (chemical) resistance
- Reactivity improvement (higher cross-linking density) would result in up to **3 times faster production rates** (5' vs 15') at 180°C cure temp
- 20% of NPG by weight is replaced with Synoxol™ BEPD along with other acid adjustments to maintain Tg

Routes to high resin reactivity = high productivity

If I replace all ADA in my resin with PIA will I get a more reactive resin?

YES

What will happen as a result?
- Tg will increase
- Viscosity will increase

What does that mean?
- Resin is likely to be difficult to process in plant
- Coating may well not have good aesthetics

Therefore I have to use ADA and cannot have a more reactive resin

FALSE

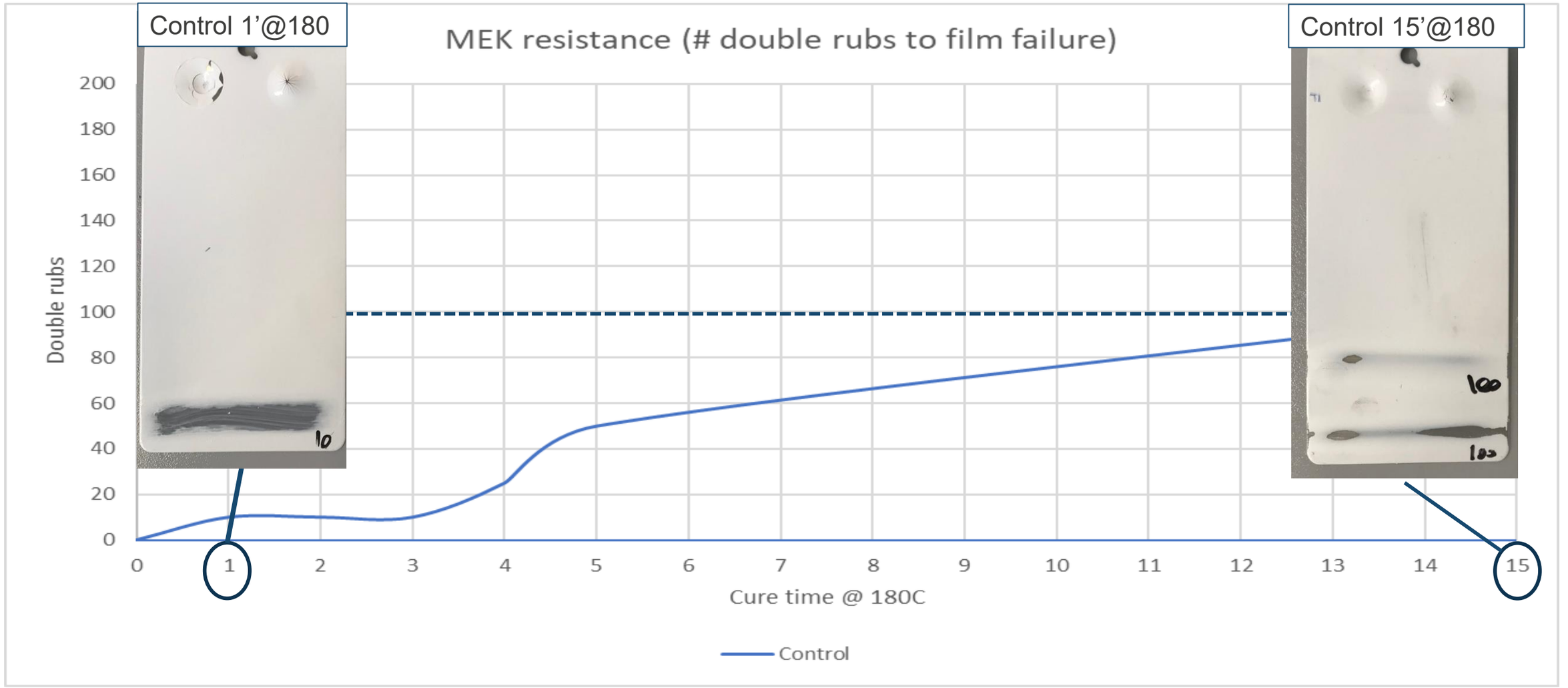
It is possible to replace ADA with PIA if you replace NPG with SYNOXOL™ BEPD and use SYNOXOL™ BEPD as a route to increase reactivity without increasing viscosity

PES resin samples – Reactivity Increase

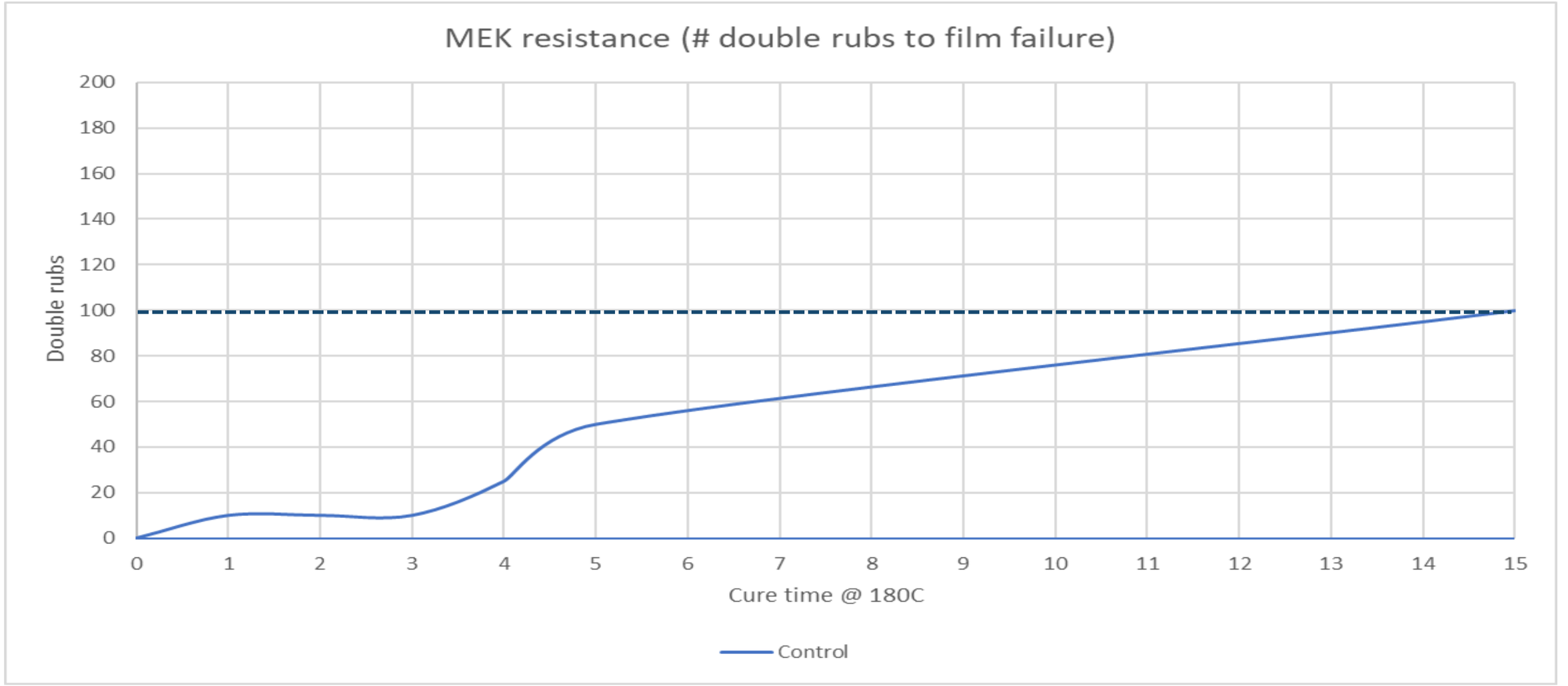
Table 9 – Superdurable Polyester resin formulations

Resin	A (Control)	B
Neopentyl glycol (NPG) / g	435.6	362.3
SYNOXOL™ BEPD) / g	-	90.5
Ethylene Glycol (MEG) / g	1.8	1.8
Terephthalic Acid (TPA) / g	34.6	33.2
Isophthalic acid (PIA) / g	651.0	658.9
Adipic acid (ADA) / g	25.9	-
Total charge / g	1149	1147
Reaction water / g	149	147
Theoretical properties at acid value = 20 mgKOH g-1		
Hydroxyl number / mgKOH g^{-1}	9.6	9.6
Acid excess / %	2.2	2.3
Molecular weight (M_n) / Da	3800	3800
Molecular weight (M_w) / Da	7500	7500
Polydispersity	2.0	2.0
Functionality	1.4	1.4
Practically measured data		
Acid value / mgKOH g^{-1}	20	21
Hydroxyl number / mgKOH g^{-1}	5.0	6.6
Molecular weight (M_n) / Da	3800	6200
Molecular weight (M_w) / Da	7400	11900
Polydispersity	2.0	2.9
T_g (onset) / °C	56.4	56.2
Viscosity (100% solids at 200 °C) / cP	2400	2880

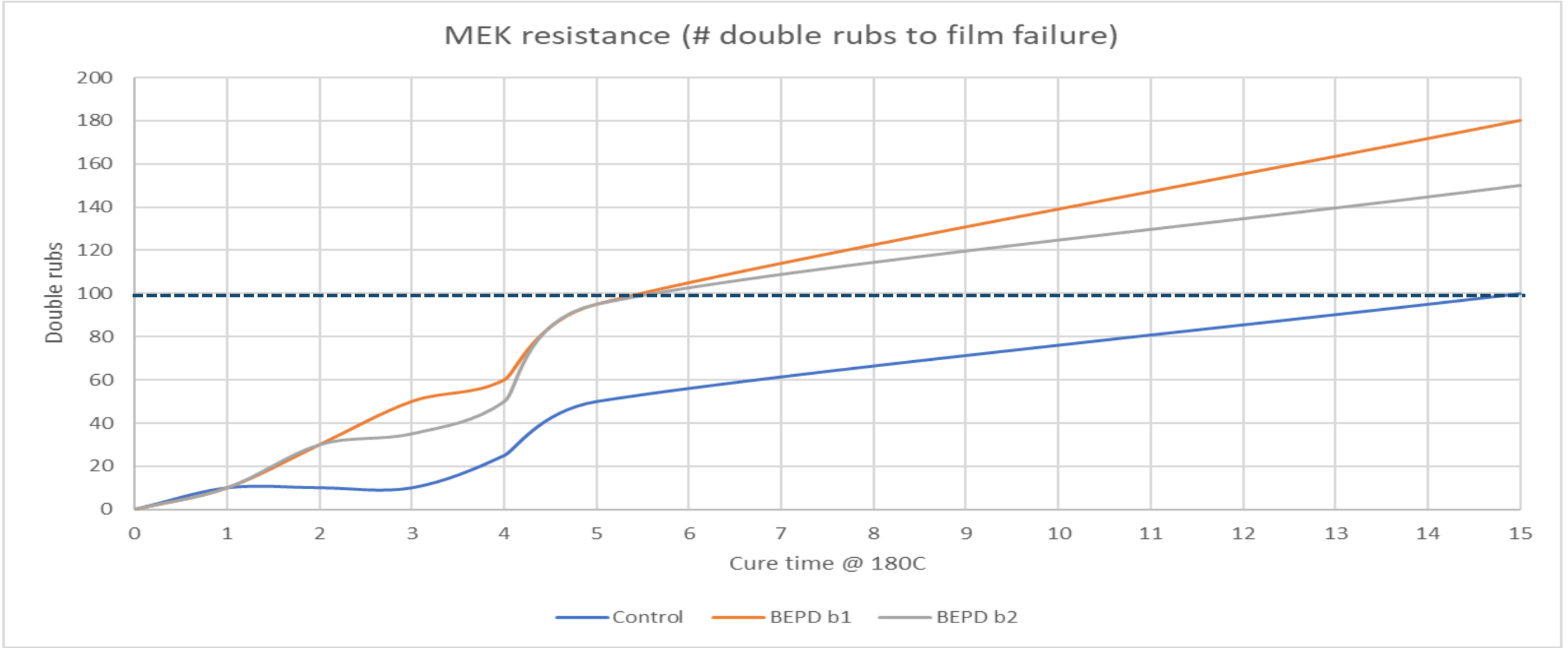
Cure profile



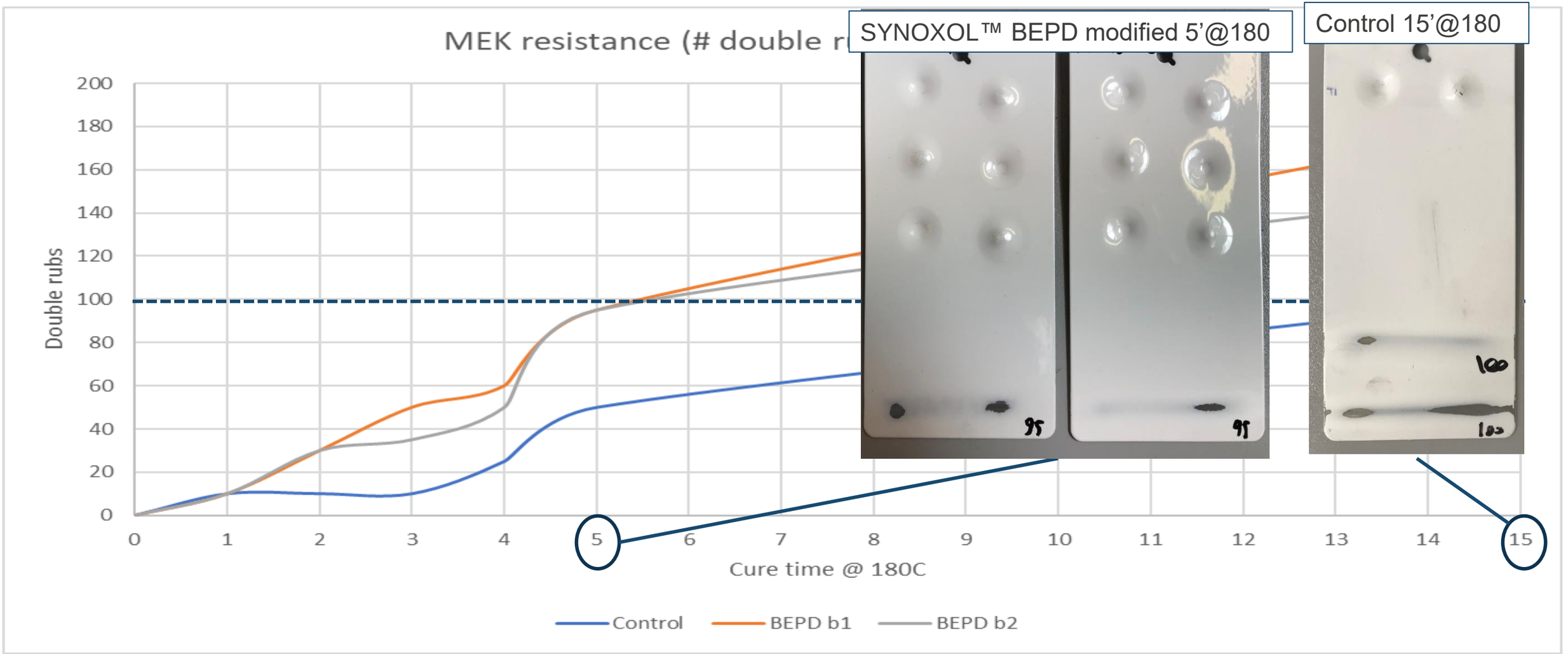
Cure profile



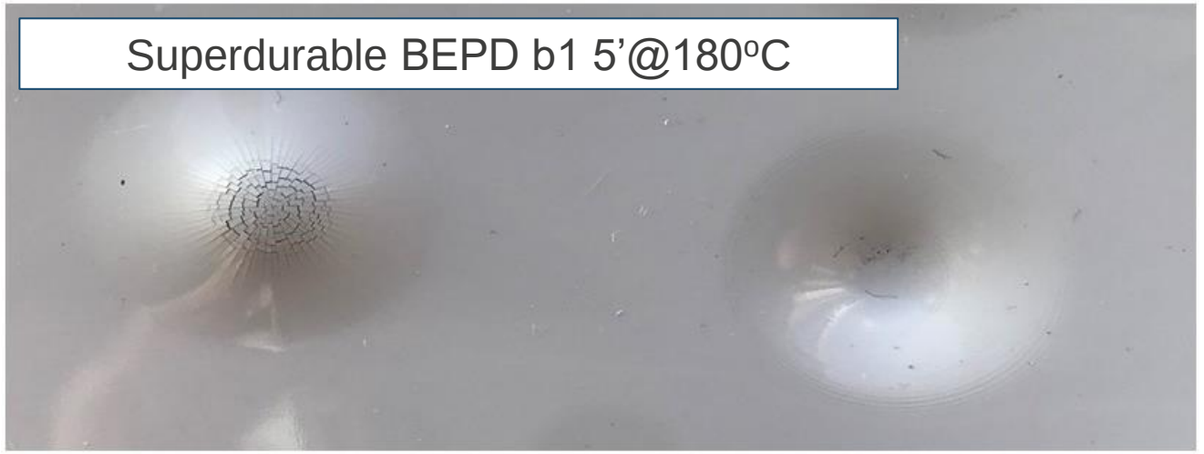
Cure profile

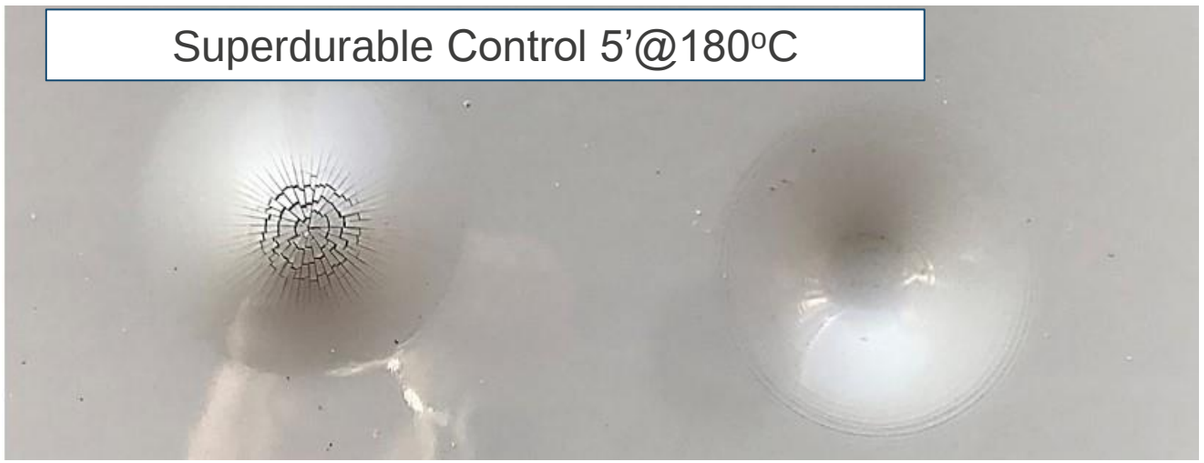


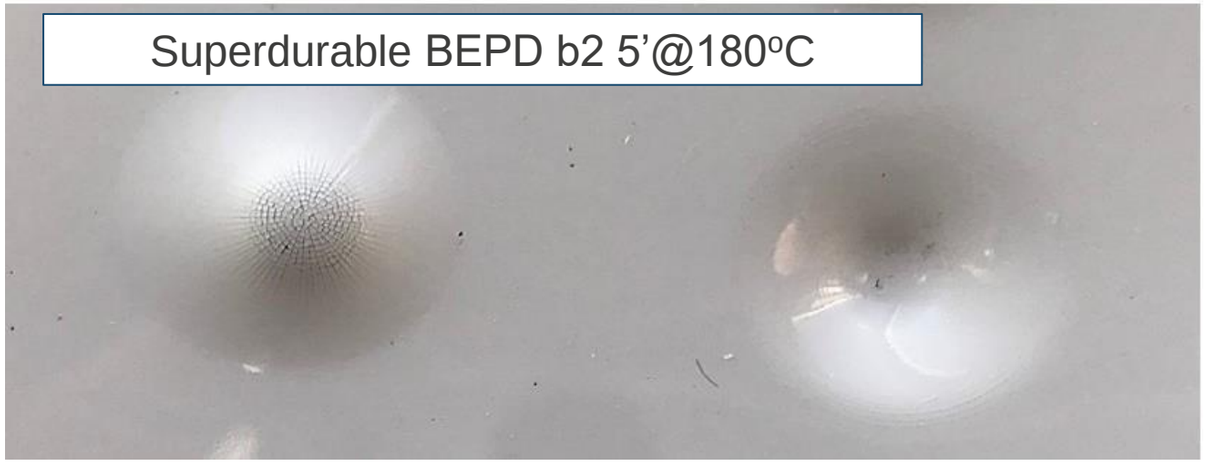
Cure profile



Impact resistance 5’ (min) vs 15’ (min) std



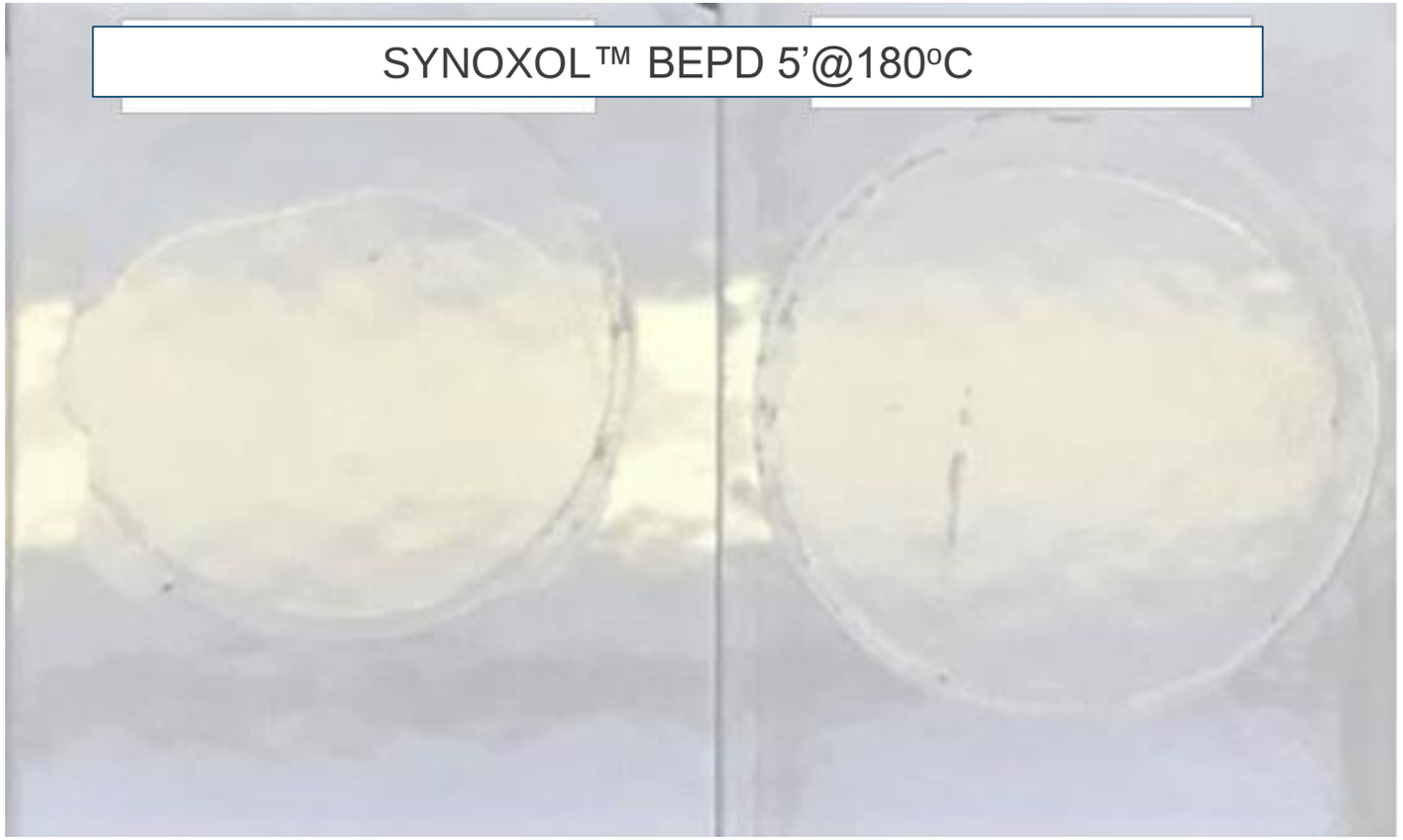




SynoxolTM BEPD gives on par – if not better impact resistance when cured at 180ºC for 5 minutes; in comparison to the benchmark cured at 180ºC for 15 minutes

Toluene stain resistance after 30’ min exposure

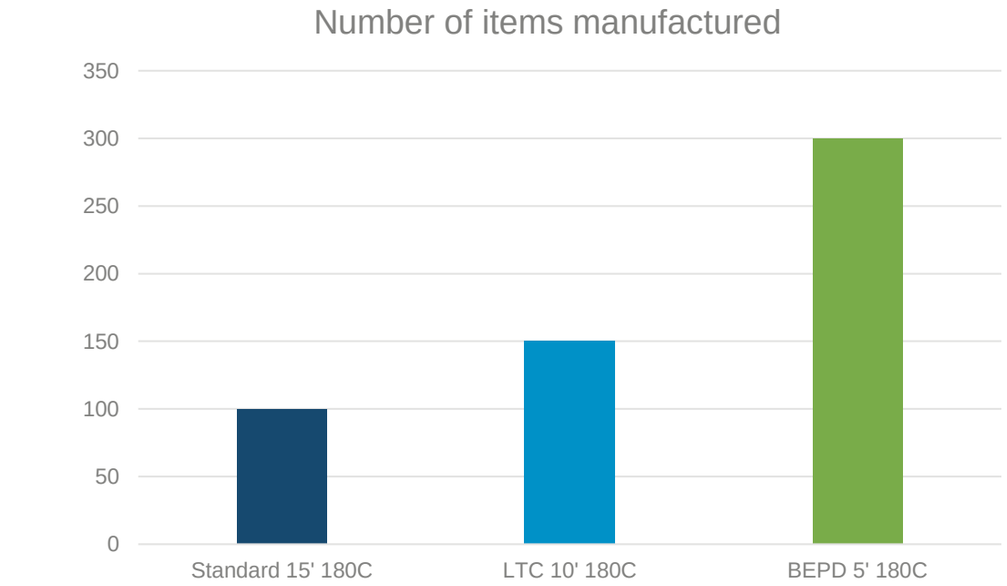
Loss of gloss & colour becomes dull

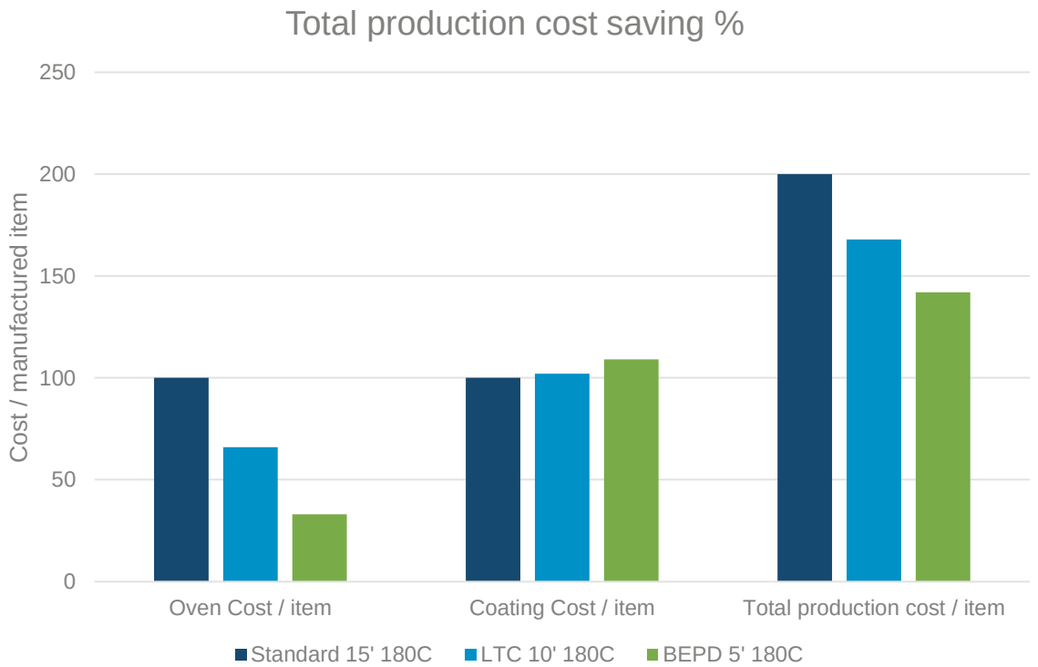


No gloss or colour change with BEPD samples

Cost-Benefit Analysis – Total Production Cost Saving

Resin type	Cure time @ 180°C	Number of Items coated	Oven Cost per item	Coating cost per item	Total production cost per item	Total production cost saving per item
Superdurable PES Control resin	15	100	100 / 100 = 1 (100)	(100)	100+100 = 200	100
Superdurable PES Low Temp Cure resin	10’	150	100 / 150 = 0.66 (66)	(102)	66+102 = 168	84 → 15%
Superdurable PES BEPD modified (20wt% NPG replaced with BEPD)	5’	300	100 / 300 = 0.33 (33)	(109)	33+109 =142	71 → **30%**





Summary

Two unique value propositions for PES powder coating resins:

1) Bloom resistance for exterior grade resins

- Standard durable and Superdurable PES resins for outdoor applications – *Architectural* / *Industrial* / *Superdurable product groups*
- Bloom free powder coating application down to 160°C core metal temp when 5wt% of NPG is replaced with Synoxol™ BEPD
- No compromise on flexibility to achieve bloom resistance for exterior grade powder coatings
- Very small amount of Synoxol™ BEPD in resin formulation is enough to solve the blooming issue
- Synoxol™ BEPD addition to PES resin backbone does not affect CAS No or Resin classification
- Simple and “Straight drop in” technical solution to solve blooming issue for PES powder coatings

2) Faster cure and productivity increase for cure temps of 180°C-200°C

- 5 minute cure time, instead of 15 minutes, at 180°C to achieve acceptable cross-linking density
- Increase production speed of powder coating lines by up to 3 times compared to the benchmark
- Deliver upto 30% total production cost saving per powder coated item
- 20wt% of NPG is replaced with Synoxol™ BEPD along with further acid adjustments to maintain Tg