



# ISAET '21

## 7th International Symposium on Asphalt Emulsion Technology

# Polymer Modification Impact on Chip Seal Asphalt Binder Residue Aging and Laboratory Protocols

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# Outline

- Background of some Colas Chip Seal experience
- Curing Vs. Aging
- Existing standards to simulate curing and aging
- Environmentally Aging Testing protocol
- Laboratory Stabilized Vs. Environmentally Aged:
  - Asphalt grade /source, Dosage, Polymer ratio, Emulsions, MSCR and weather
- Conclusions and considerations



# Background: Hot Applied Chip Seal

- 2004 – 2006: Colas Developed Vegeflux<sup>®</sup>, a bio-flux used in Hot Applied Chip Seal but also any other Pavement Preservation technique
- 2006 – 2012: Deployment on the European Market and Return of Experience
- **Safer** for the user, **Sustainable**, and **Eco-Responsible**, **Carbon Sink**
- Stiffening by oxidation/polymerization compare to loss of volatile from petroleum fraction
- Curing and Aging critical !
- Immediate adhesion = High traffic level





# Background: Cold Applied Chip Seal

- Asphalt Emulsion preferred in the Chip Seal techniques in Colas
- ... **Cold Asphalt = COLAS ... 1929 Shell Patent**
- Safer handling, lower energy consumption relative to heat
- Less solvent to none relative to the season
- Better wettability with the aggregates
- Forgiving technique with optimal viscosity
- Viscosity challenges (PMAC, AC base, emulsifiers, etc.)
- Slow Curing the first day = Low to Medium Traffic level
- Hot Vs Cold Applied Chip Seal curing and aging ?



# Hot & Cold Applied Chip Seal

- What are the difference between Curing and Aging for chip Seals?

**Curing:** *necessary time for the binder to reach the performance of the “original” binder after volatilization of petroleum-based solvent or polymerization of bio-solvent for Hot Applied binders or for the water to be expelled and evaporate with its solvent for an emulsion.*

**Aging:** *Hardening above original binder performance, from oxidation with oxygen, UVs, temperatures, weathering, and traffic.*

- What are the risk if Curing is too slow ?
  - A LOT...
- What are the risk if Aging is too fast?
  - NOT a lot...



# Testing Standards

- 1993 – 2010 : **French Standard NF T66-031** - Determination of kinetic of anhydrous and bitumen emulsion binders stabilization
  - *Method at 50 °C for 14 days*
- 2006: **European Standard EN 13074**: Bitumen and bituminous binders - Recovery of binder from bituminous emulsion...
  - *Part 1: Recovery by evaporation for 24 hours at ambient temperature and 24 hours at 50 °C*
  - *Part 2: Stabilization after recovery by evaporation for 24 hours at 85 °C*
- **AASHTO R 78 / ASTM D7497-21**: Recovering Residue from Emulsified Asphalt Using Low-Temperature Evaporative Technique
  - *A - 24 hours at 25 °C and 24 hours at 60 °C , 1 mm residue thickness*
  - *B - 6 hours at 60 °C, 0.25 mm residue thickness*
- **AASHTO R28** - Residue PAV...
- Coming **SPG / EPG Specifications** ...

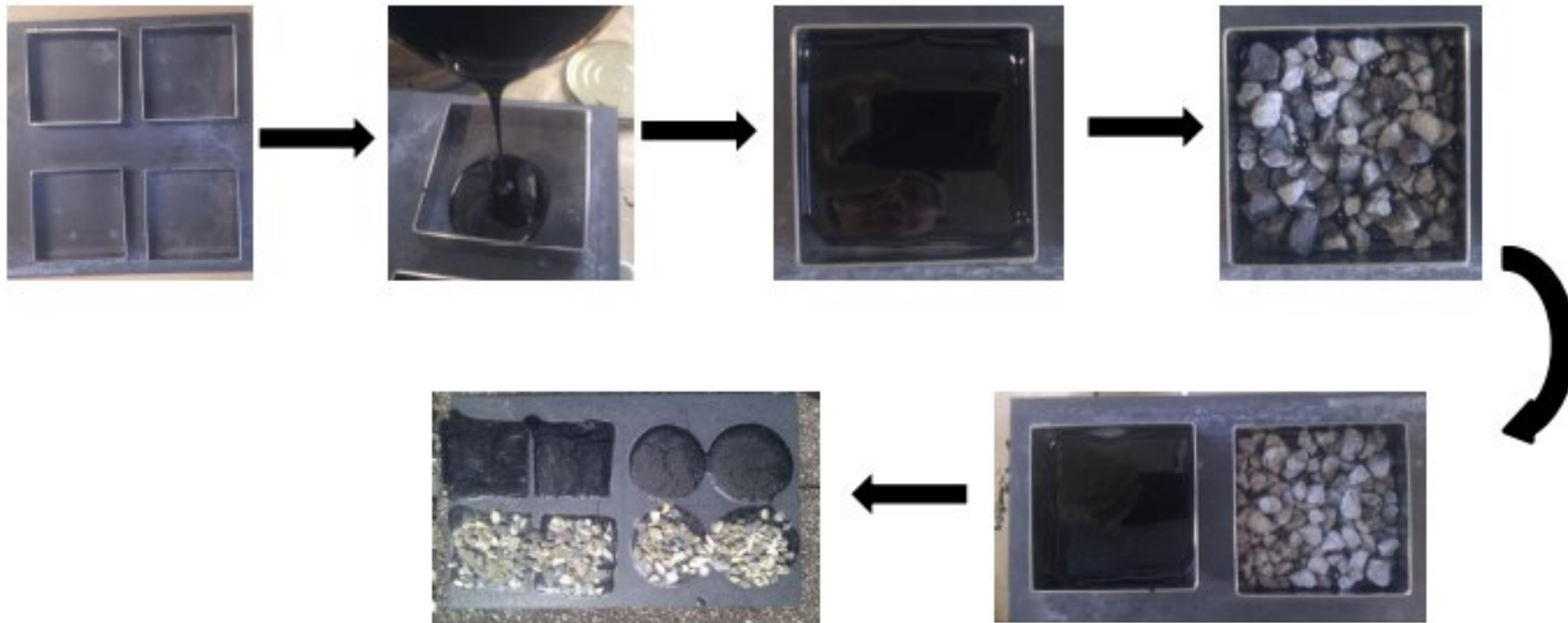
# European Standard EN 13074

- Bitumen and bituminous binders - Recovery of binder from bituminous emulsion or cut-back or fluxed bituminous binders
- Part 1: Recovery by evaporation for **24 hours at ambient** temperature and **24 hours at 50 °C**
  - ⇒ Fresh binder performance (1 to 2 days on the field)
- Part 2: Stabilization after recovery by evaporation for 24 hours **at 85 °C**
  - ⇒ Binder performance after curing (missing time scale)
- Limitations



# Environmentally Aging Test (EAT): Protocol

- Principle: To expose chip seal binder in real weather condition following dosage rate of the EN 13074 of 1 kg/m<sup>2</sup>
- With and without 6/10 mm aggregate





# Environmentally Aging Testing (EAT)- Protocol

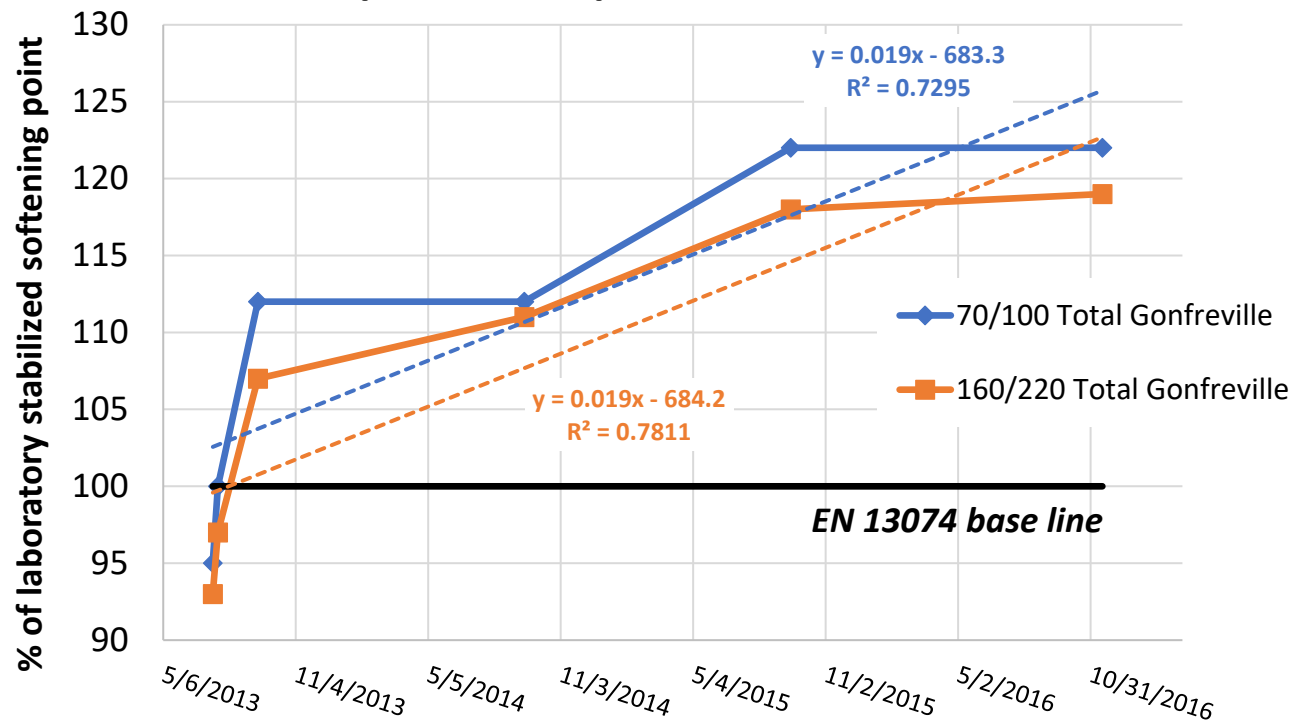
- With a hot spatula , the binder is scraped with and without aggregate at the due date
- Poured into a pen cup with a lid and heat at 120°C for 30 minutes
- The lid helps for retaining the aggregates for pouring into the ring and ball or DSR mold



# EN 130074 Stabilized binder Vs. EAT : Asphalt Grade

- Same asphalt source / different grades / Without Aggregates

**70/100 Vs 160/220 Total Gonfreville**



Binder Total Gonfreville Grade	70/100	160/220
Laboratory Stabilized Binder EN 130074 Softening Point (°C)	49.0	42.8

→ Neat asphalt stabilize predictably fast

→ 14 days EAT equivalent => 70/100

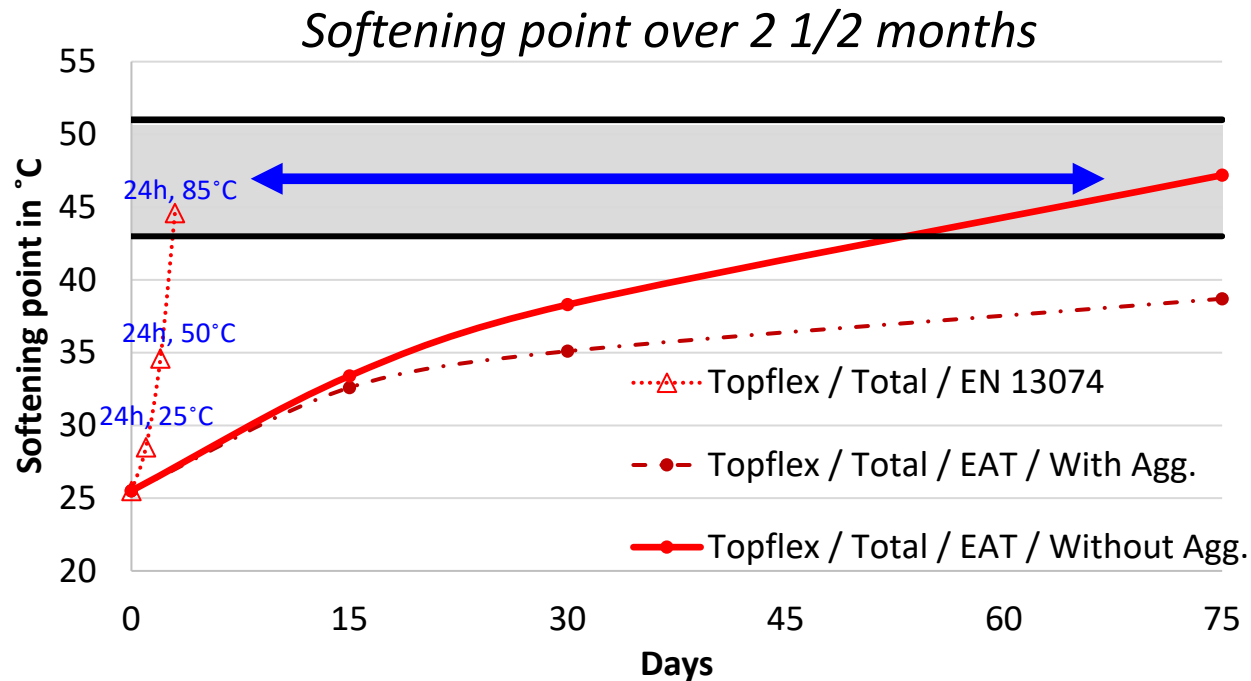
→ 1 month EAT equivalent => 160/220

→ Softer asphalt base, longer stabilization

**Same Asphalt Source, Same Aging Ratio**

# EN 130074 Stabilized binder Vs. EAT : Solvent-Based Binder

- Softening Point of Stabilized Binder by EN 13074 and environmentally aged with and without aggregates



- Penetration grade 70/100
- 43 to 51°C Softening point range
- Solvent based by EN 13074 validated
- Without aggregate
- Quid of the equivalent time lab Vs. field for the binder to cure with the original softening point range
- Longer curing with Aggregate = Post construction risk

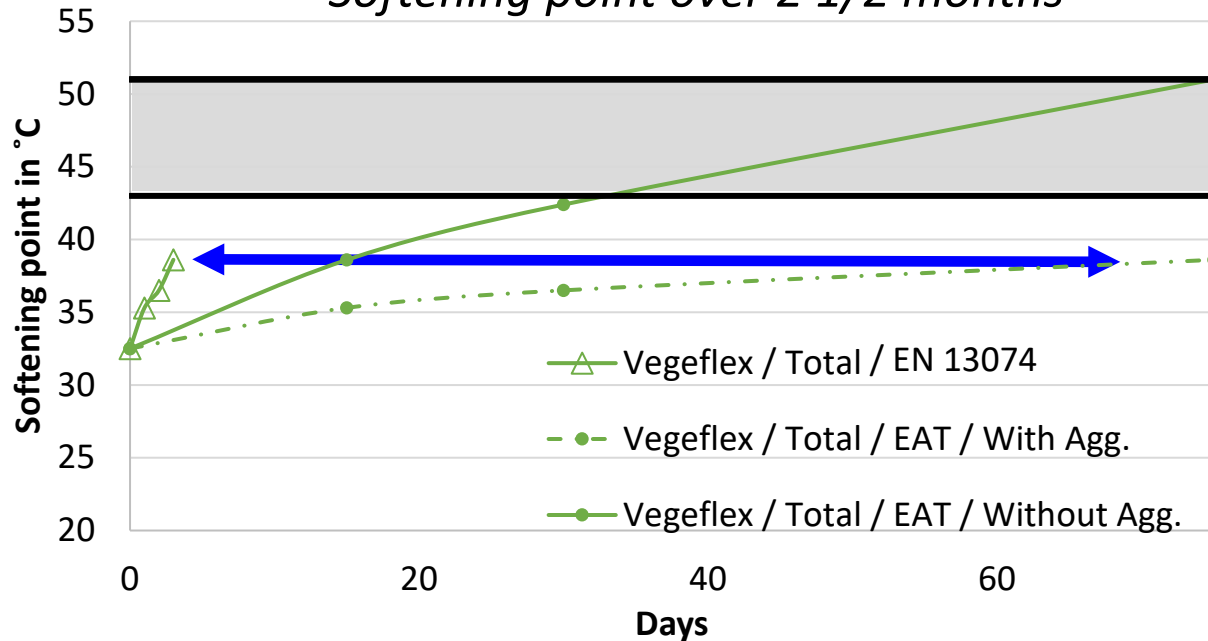


# EN 130074 Stabilized binder Vs. EAT : Bio-Based Binder

- Softening Point of Stabilized Binder by EN 13074 and environmentally aged with and without aggregates



*Softening point over 2 1/2 months*



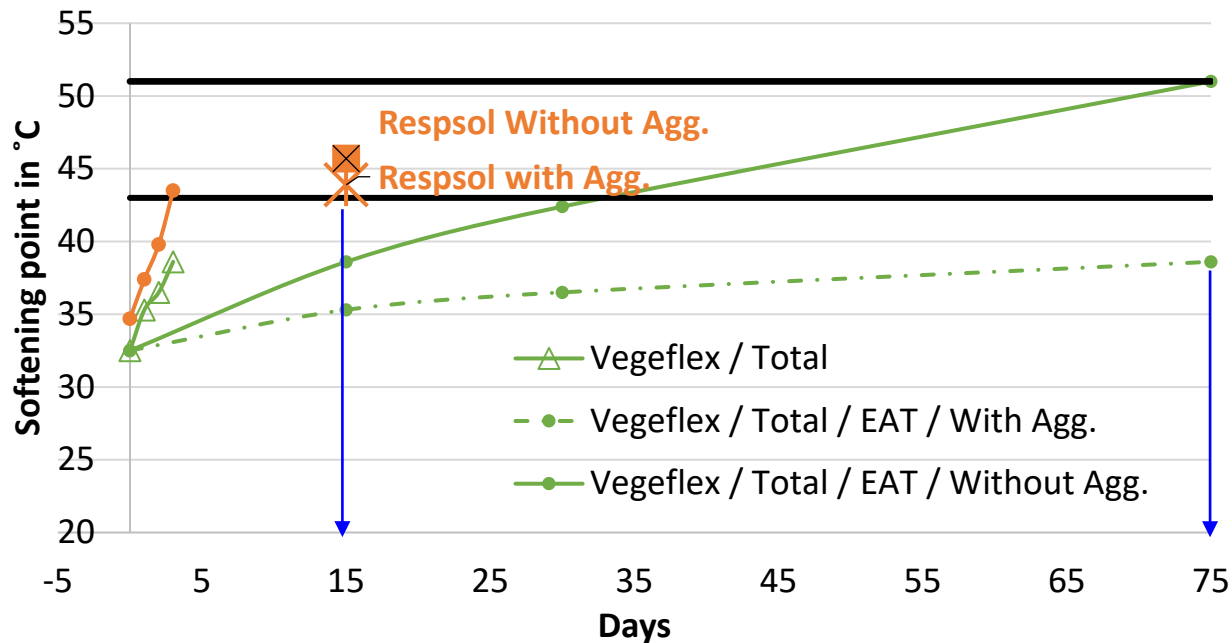
- Penetration grade 70/100
- 43 to 51°C Softening point range
- Bio based by EN 13074 validated
- **With aggregate**
- Lab stabilization needs to be extended with bio-based binder
- Longer curing with Aggregate = Post construction risk

# Laboratory Stabilized Vs. EAT: Asphalt Source

- Softening Point of Stabilized Binder by EN 13074 and environmentally aged with and without aggregates



*70/100 Total Vs Repsol Softening point over 2 1/2 months*



→ Penetration grade 70/100

→ 43 to 51°C Softening point range

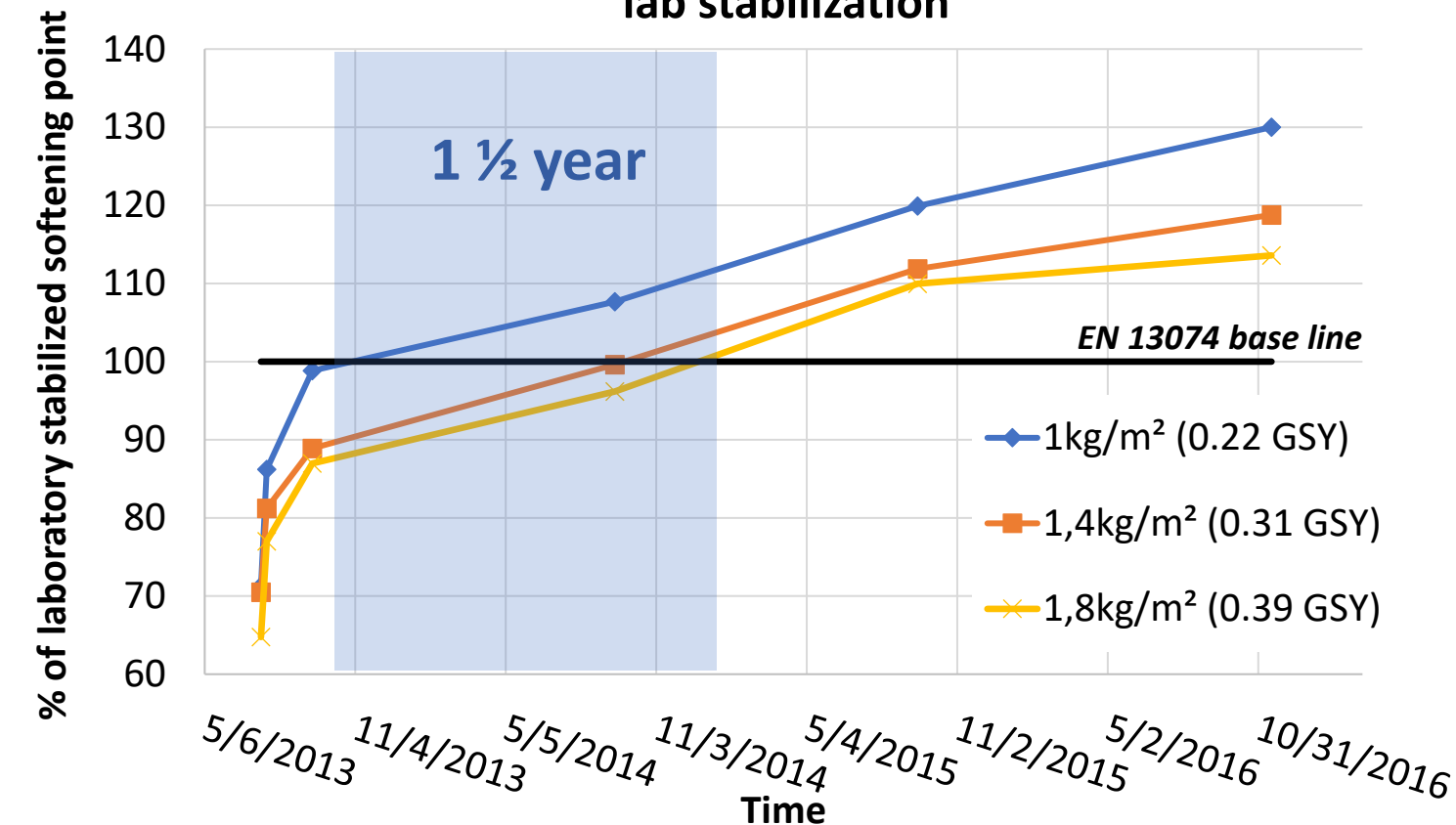
→ Vegeflex curing with Agg. & Total: +3 months

→ Vegeflex curing with Agg. & Repsol: 15 days

**Curing is Asphalt Source Relative**

# EN 130074 Stabilized binder Vs. EAT: Dosages

Binder at different dosage rates environmentally aged vs.  
lab stabilization



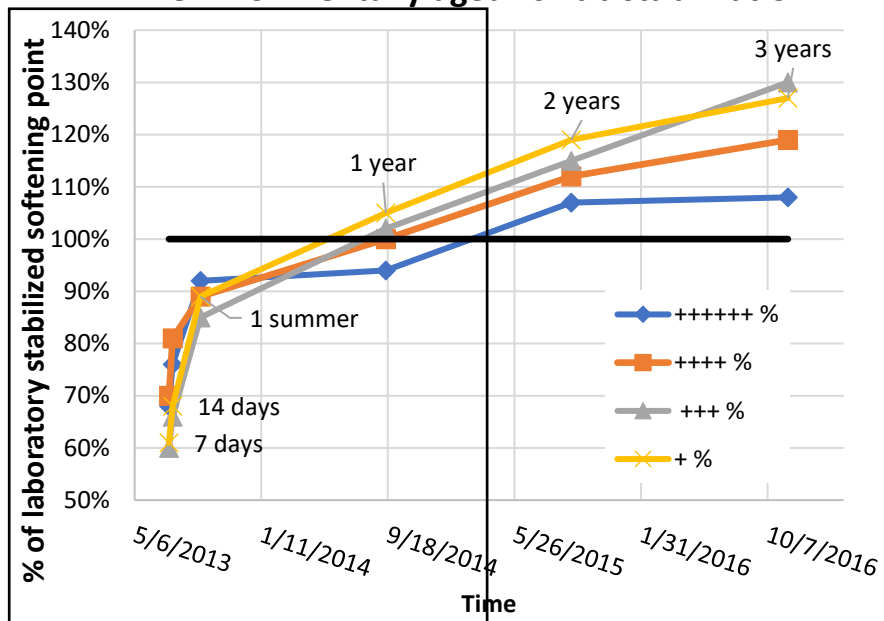
- Solvent-based Binder + Agg.
- $1 \text{ kg/m}^2 = 1 \text{ mm} \Rightarrow$  Testing condition
- $1.4 \text{ to } 1.8 \text{ kg/m}^2 \Rightarrow$  Field condition
- 6 months to 1 ½ year difference
- Higher film thickness, Longer stabilization

Lab Stabilization not representative of “field curing with Aggregates” condition

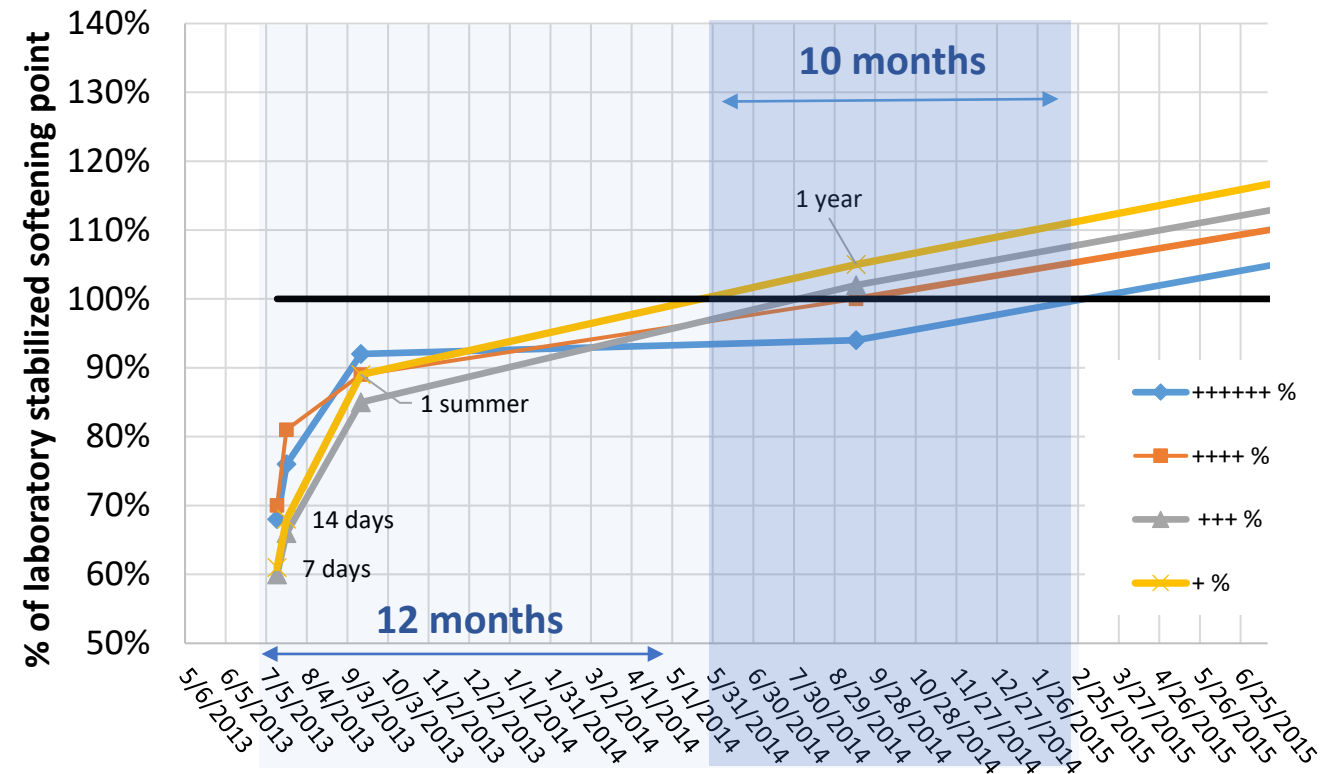


# EN 130074 Stabilized binder Vs. EAT: Polymer %

Topflex binder at different polymer %  
environmentally aged vs. lab stabilization



Topflex binder at different polymer %  
environmentally aged vs. lab stabilization



- Hot Applied, 1,4kg/m<sup>2</sup> (0.31 GSY)
- Solvent-based / Aggregates

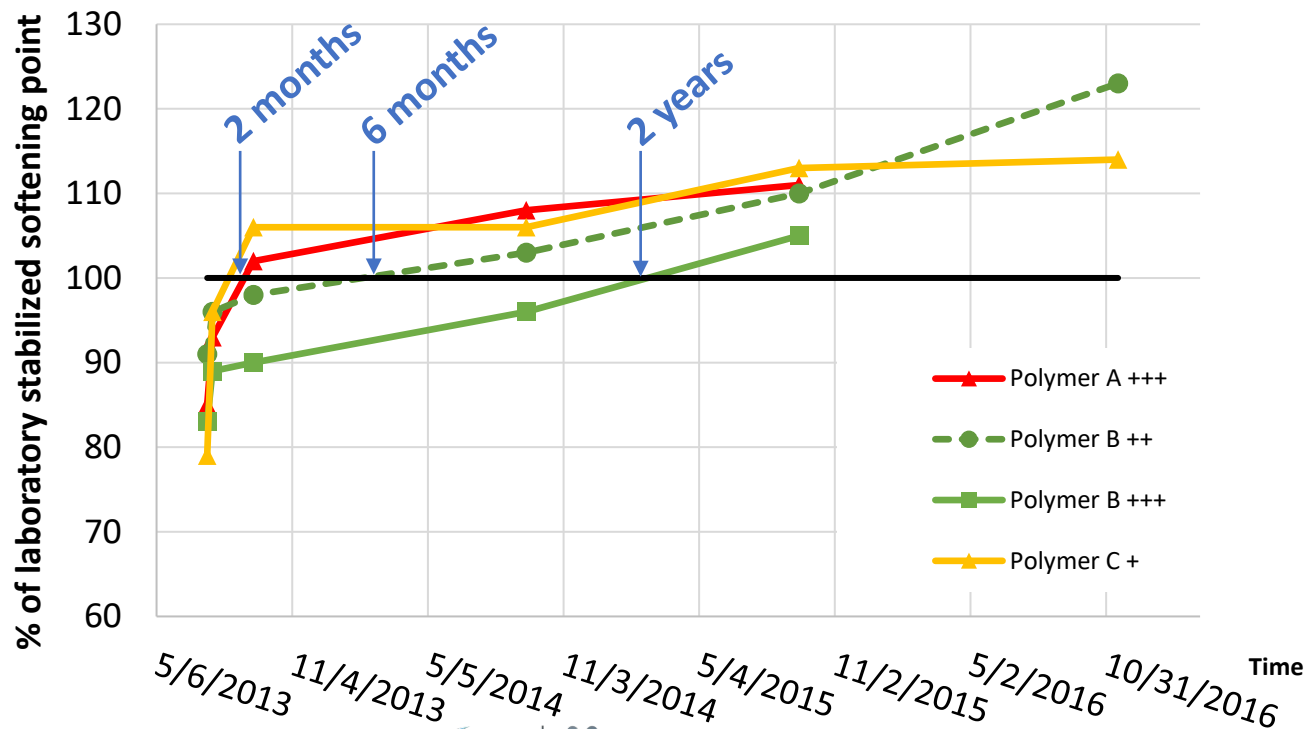
→ 10 to 22 months to reach 100% lab stabilized performance

Curing relative to % Polymer

# EN 130074 Stabilized binder Vs. EAT : Asphalt Polymer Modified Emulsion

- Polymer Type / Polymer Dosage/ Emulsion dosage of 1.8 kg/m<sup>2</sup>

NEOCOL polymer modified emulsion binder residue with  
different polymers environmentally aged vs. lab stabilization



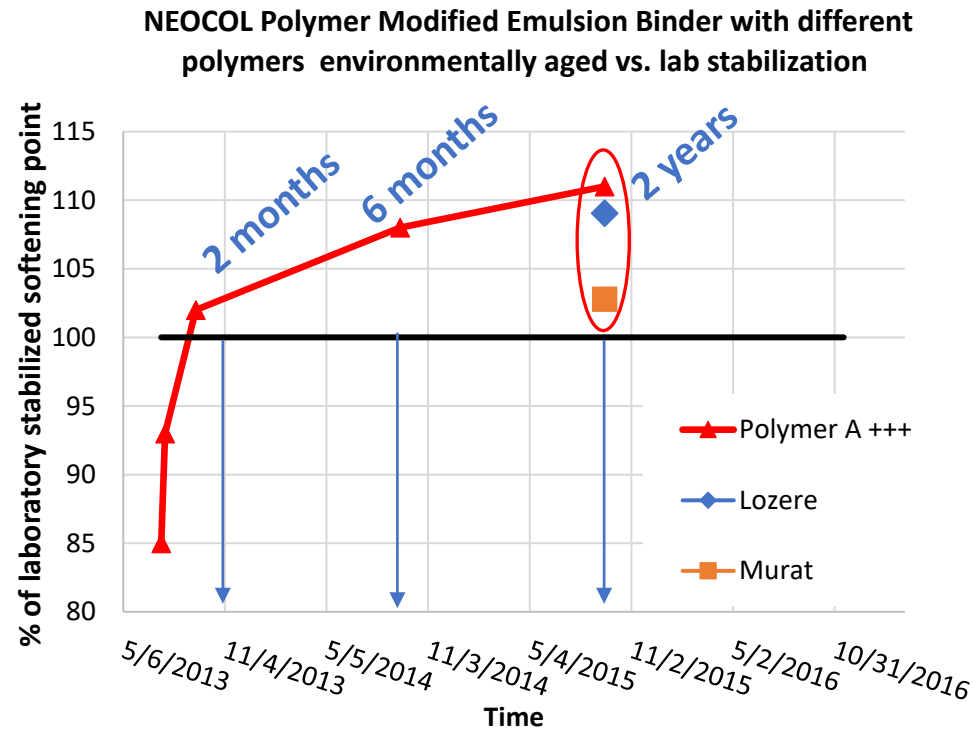
→ 1.8 kg/m<sup>2</sup> Emulsion => 1.2 kg/m<sup>2</sup> binder residue

→ 2 months to 2 years curing depending polymer type and polymer ratio

→ Laboratory Stabilization not representative of “field curing” condition

# EN 130074 Stabilized binder Vs. EAT : Field comparison

- Polymer Type / Polymer Dosage/ Emulsion dosage of 1.8 kg/m<sup>2</sup>



→ Sampling from 2 years old jobsite

→ Same emulsion formula with A +++

→ Lozere Softening Point (SP) of 62.6°C

→ Murat SP of 59.0°C

→ EN 13074 stabilization SP of 57.4°C

Environmentally Aged sample at the laboratory is representative of field curing condition



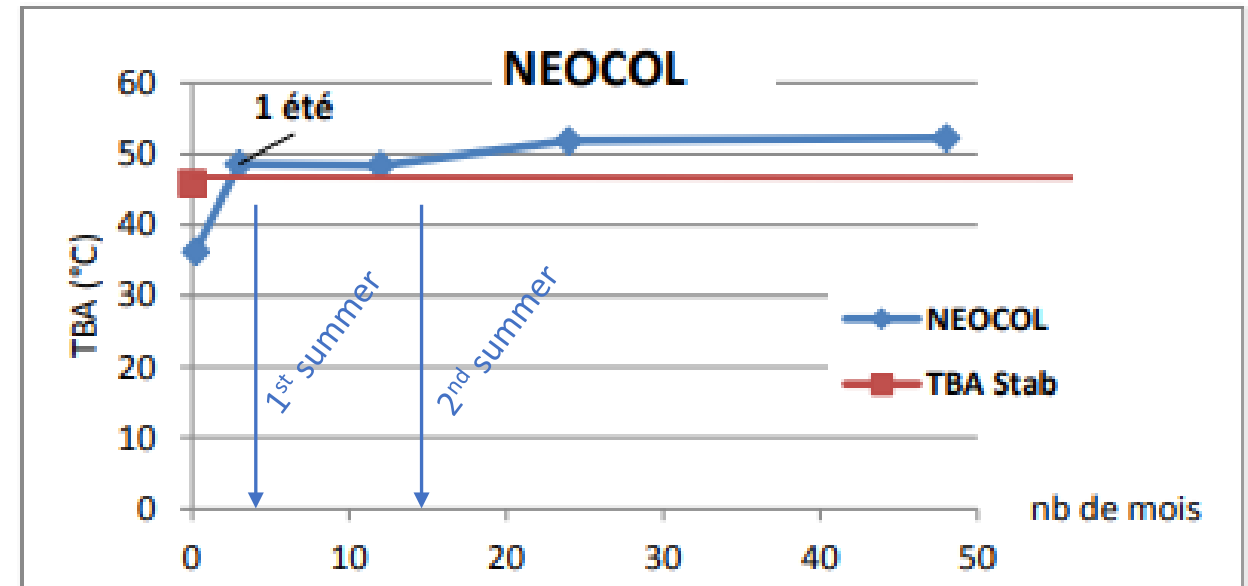
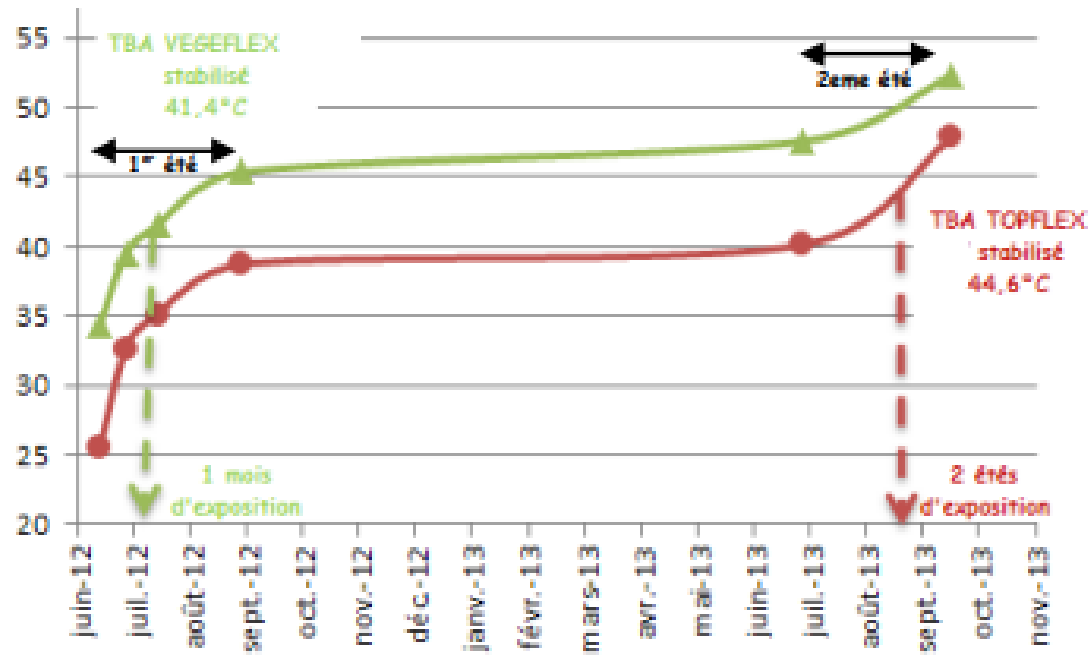
# EN 130074 Stabilized binder Vs. EAT : : MSCR at 64°C

Jnr 3.2 kPa (kPa <sup>-1</sup> ) at 64 °C										
	Topflex Polymer Modified Binder						Neocol - Polymer Modified Emulsion			
Dosage rate (kg/m <sup>2</sup> )	1.4	1.4	1	1.4	1.8	1.4	1.2	1.2	1.2	1.2
Polymer Level	+ Low	+++ Med.	++++ High	++++ High	++++ High	++++++ Very High	A +++ High	B ++ Med.	B +++ High	C + Low
1 years	5.9	5.3	0.5	3.2	4.2	3.2	2.4	5.7	5.6	6.8
3 years	0.8	0.9	Too Stiff to recover	0.6	1	Too Stiff to recover	1.1	2.8	2.8	6.2

SPG at Temp. Maximum Jnr @3.2 kPa	Traffic Level
< 8 kPa <sup>-1</sup>	Low (0-500 ADT)
< 5.5 kPa <sup>-1</sup>	Medium ( 500-2500 ADT)
< 3.5 kPa <sup>-1</sup>	High( > 2500ADT)

- Comparing to recent SPG Jnr specifications, residue are formulated for low and high ADTs
- Medium ADTs with intermediate polymer dosages will be challenging as curing seems dependent of Polymer Nature, type, Lab aging ?

# EN 130074 Stabilized binder Vs. EAT : Climatic variations



- Hot Applied required up to 2 summers
- Emulsion only 1 summer with the proper polymer

Summer curing is critical for durable chip seal

# Conclusion

- Focus on post-construction curing, most critical stage for durable chip seal
- European Stabilization EN 13074 (24hours @ 25°C, 24hours @ 50°C, 24hours @ 85°C) is considered to simulate curing on the field
- The environmentally Aging Test demonstrates that curing is relative to :
  - **Hot applied Solvent/Bio-Based or Cold Applied Emulsion**
  - **Binder dosage:** Heavier, slower the curing
  - **Asphalt Source and Grade:** 2 weeks to 2 months curing delay
  - **Polymer type:** 2 months to 2 years curing delay
  - **Polymer / binder ratio:** 2 months to 2 years delay
  - **Summer Curing** : 1 months of hot temperature to secure the job through the winter
- European Stabilization EN 13074 is not equally “CURING TIME” representative for different binders (15 days to 2 years depending hot or cold applied, formulation and application rate)
- Environmentally aged samples seems to be representative of field curing over 2 years
- Polymer Modified Binder curing behaviors needs to be further investigated before to create specifications



# Considerations

- This study conducted from 2012 to 2016 and did not consider the SPG and EPG specifications at that time unfortunately
- EPG and SPG working group are discussing the Long-Term Aging
- Long Term Aging after curing is interesting but not necessary as binder residues stiffness reach quickly a plateau
- Chip seal generally also fail within the first year (dirty agg., bonding issues, too cold temperature, dosage, too stiff binder to start, etc, ) and mostly after the first winter exacerbating the problems
- Chip seal rarely fail after 3 years because the AC became too stiff. Even with polymerization of bio-based binders.
- Chip Seal durability is highly dependent of the job preparation, Aggregates cleanliness, crew competency, etc.
- Many time it is just not the right technique because the owner has been waiting too long...



# COLAS



## *Any Questions ?*

## *Be safe, Thank you*



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