# Accessing Bitumen Quality and Ageing Behavior – A Journey from the Molecular to the Chemo-Mechanical Level

**Dr. Johannes Mirwald**

**CD Laboratory for Bituminous Materials**

Vienna University of Technology (TU Wien), Austria

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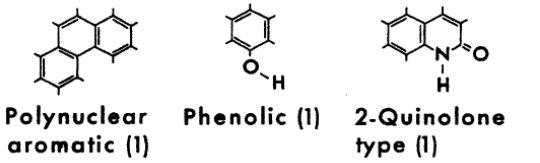
# Overview

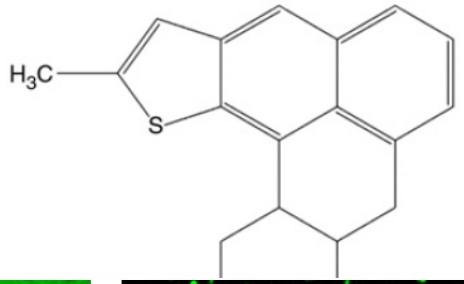
- How do we **understand** bitumen (**quality**) and the changes occurring (**ageing**) during its service life?
  - **Tools** to understand bitumen - **quality**
  - **Factors** involved in the **ageing process** - ageing
- How can we **access** bitumen quality and ageing **effectively** and **time efficient**
  - Access on the **molecular level** by **FTIR Spectroscopy**
  - Ageing Approaches
  - **Correlation** between **mechanical** and **chemical** analysis by combination of **DSR** and **FTIR spectroscopy**

# How do we understand bitumen quality and the changes occurring during its service life?

# How do we understand bitumen?

- **Viscoelastic** material
  - Low temperature: elastic, solid like
  - High temperature: viscos, liquid like
- **Complex** chemical composition
  - Functional gr
  - Polari
- Mysteriou



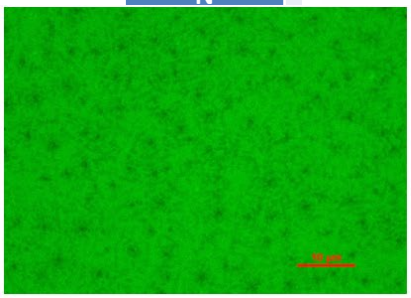



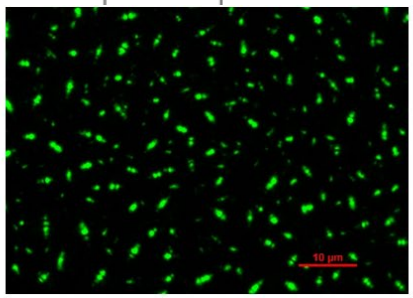

| Element |
|---|
| |
| C |
| H |
| N |

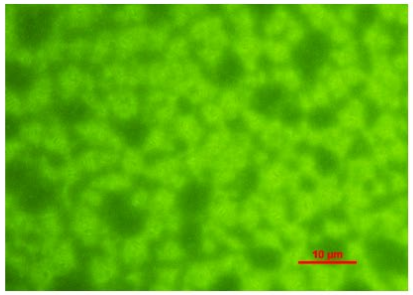
) Sulfoxide (2)

Ketone (2)

ging

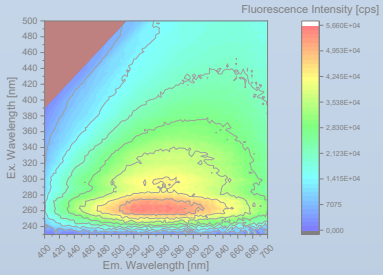




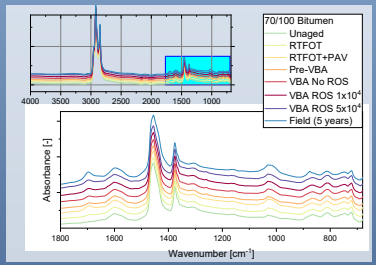


# Tools to understand bitumen

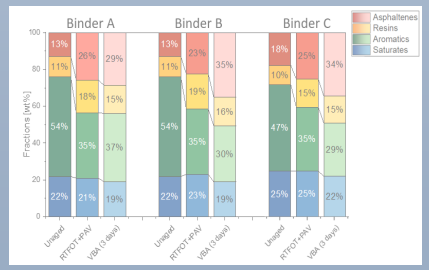
## Fluorescence Spectroscopy




## FTIR Spectroscopy




## SARA Separation

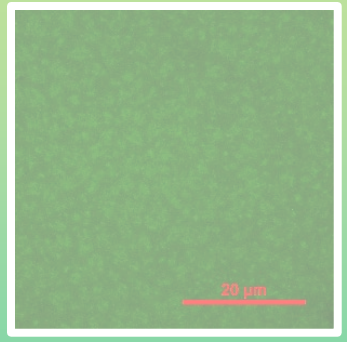


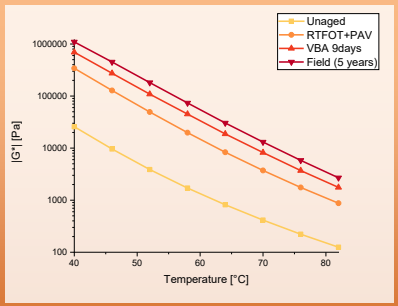

**Microstructure**

**Chemical Composition**

**Mechanical Behavior**

## Optical Microscopy

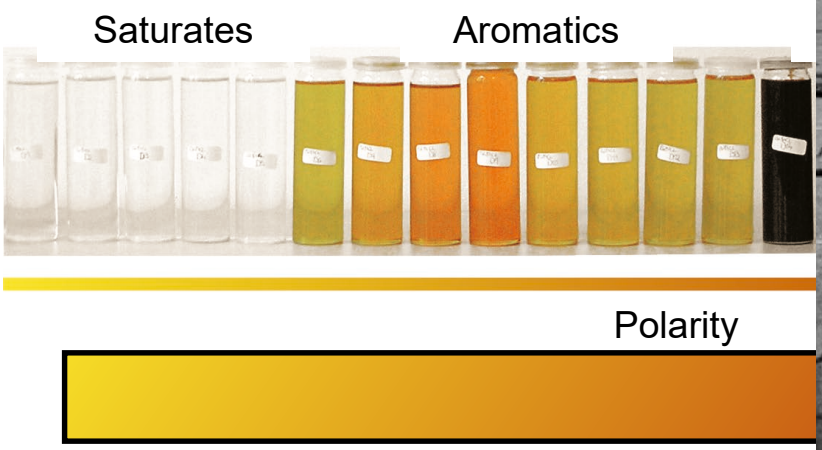


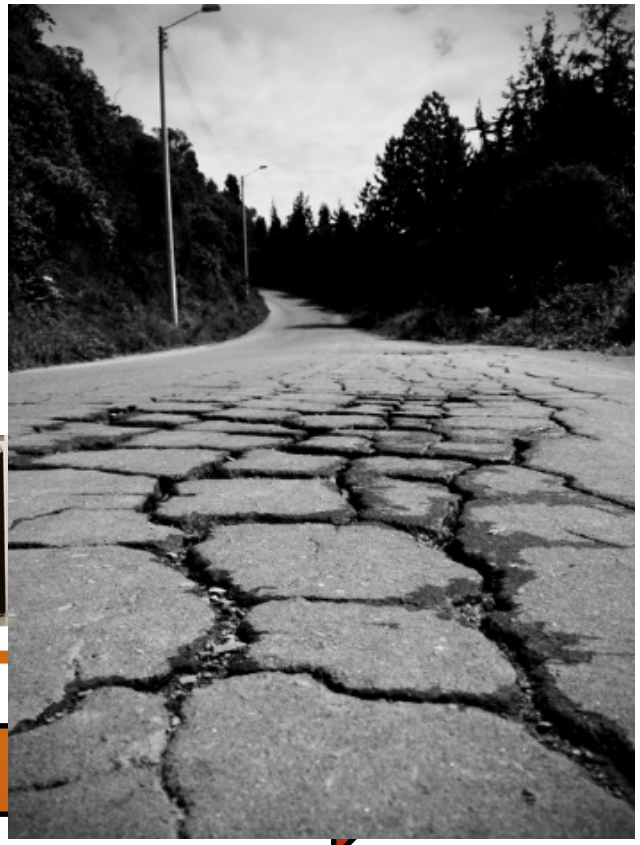




Dynamic Shear Rheometer

# How do we understand ageing?

- How does **bitumen age?**
  - **Incorporation** of **oxygen**
  - Shift of the **polarity gradient**
  - **Embrittlement** and **material failure**



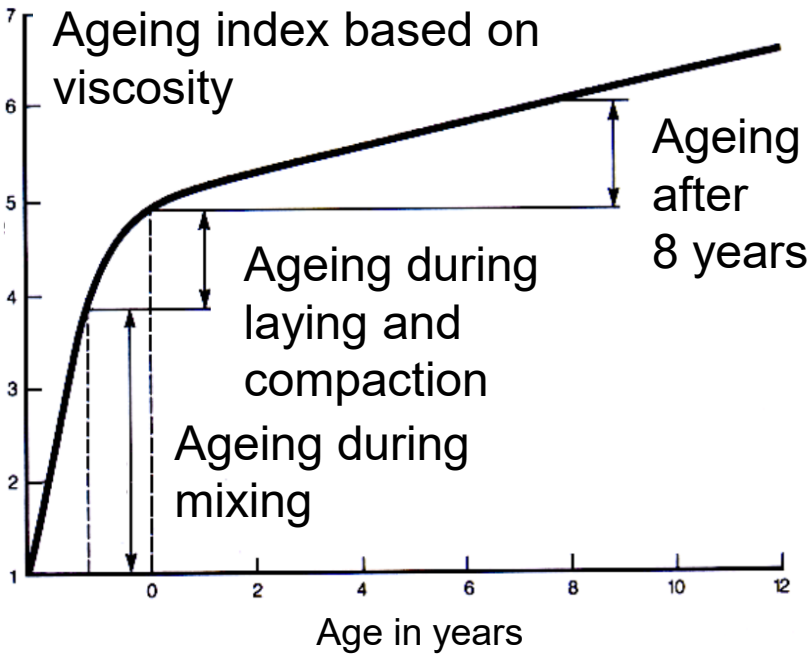
# Ageing

**Short-term ageing**

Evaporation of remaining volatile components and fast oxidation at high temperature

**Long-Term ageing**

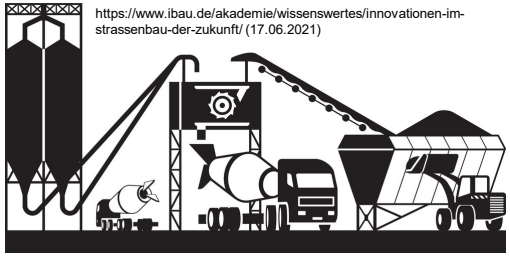
slow oxidation and changes in the colloidal structure

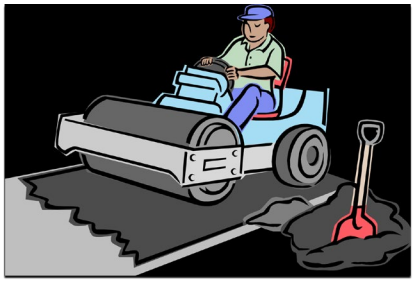


# Ageing

## Short-term ageing of bitumen

### In the field


https://www.ibau.de/akademie/wissenswertes/innovationen-im-strassenbau-der-zukunft/ (17.06.2021)



Ageing inducing factors:

High temperature

Contact with air

### In the lab: RTFOT


http://www.normalab.com/en/produit/rtfot-oven/ (17.06.2021)

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High temperature

Contact with air

# Ageing

## Short-term ageing of bitumen

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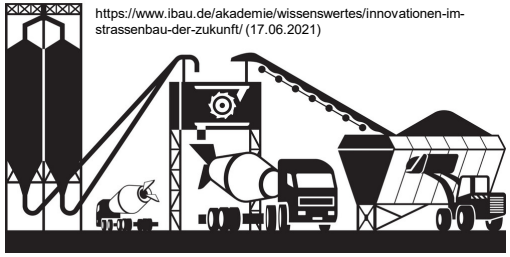
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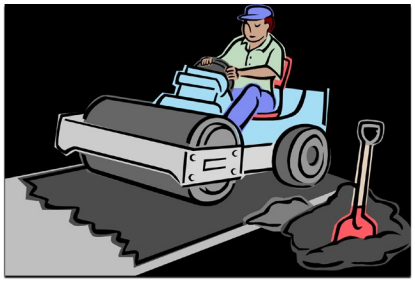

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Ageing inducing factors:

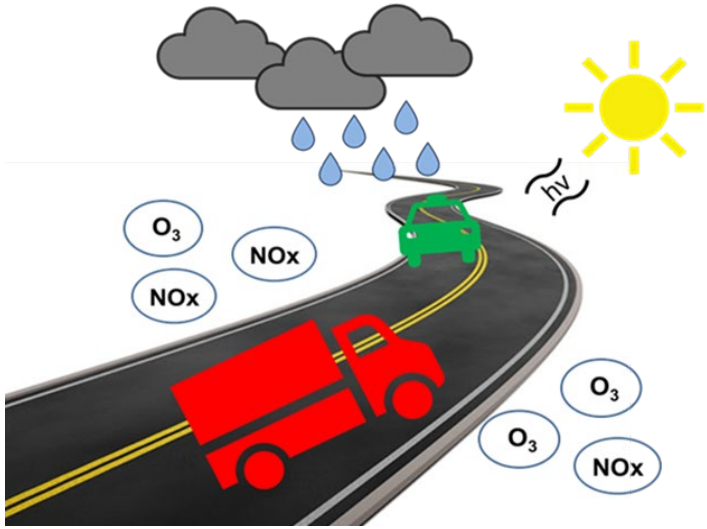
High temperature

Contact with air

# Ageing

## Long-term ageing of bitumen

### In the field




Ageing inducing factors:
Contact with Air, $O_3$, $NO_X$
Moisture
Light
Temperatures ≤80°C
Mechanical stress

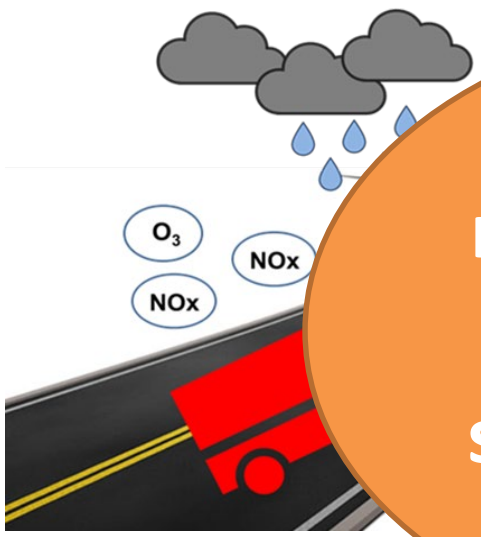
### In the lab: PAV


Ageing inducing factors:
Temperatures ~ 100°C
Contact with Air
2,07 MPa

# Ageing

## Long-term ageing of bitumen

In the field




In the lab: PAV


**What do we need to consider during ageing simulation in the lab?**

Ageing inducing facto

Contact with Air, $O_3$, $NO_X$
Moisture
Light
Temperatures ≤80°C
Mechanical stress

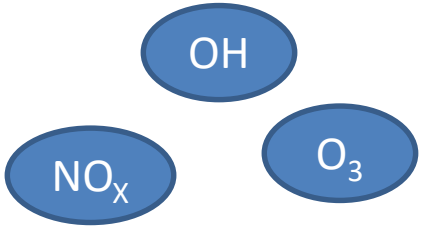
geing inducing factors:

Temperatures ~ 100°C
Contact with Air
2,07 MPa

# Ageing - A realistic lab approach

- Thermal Ageing

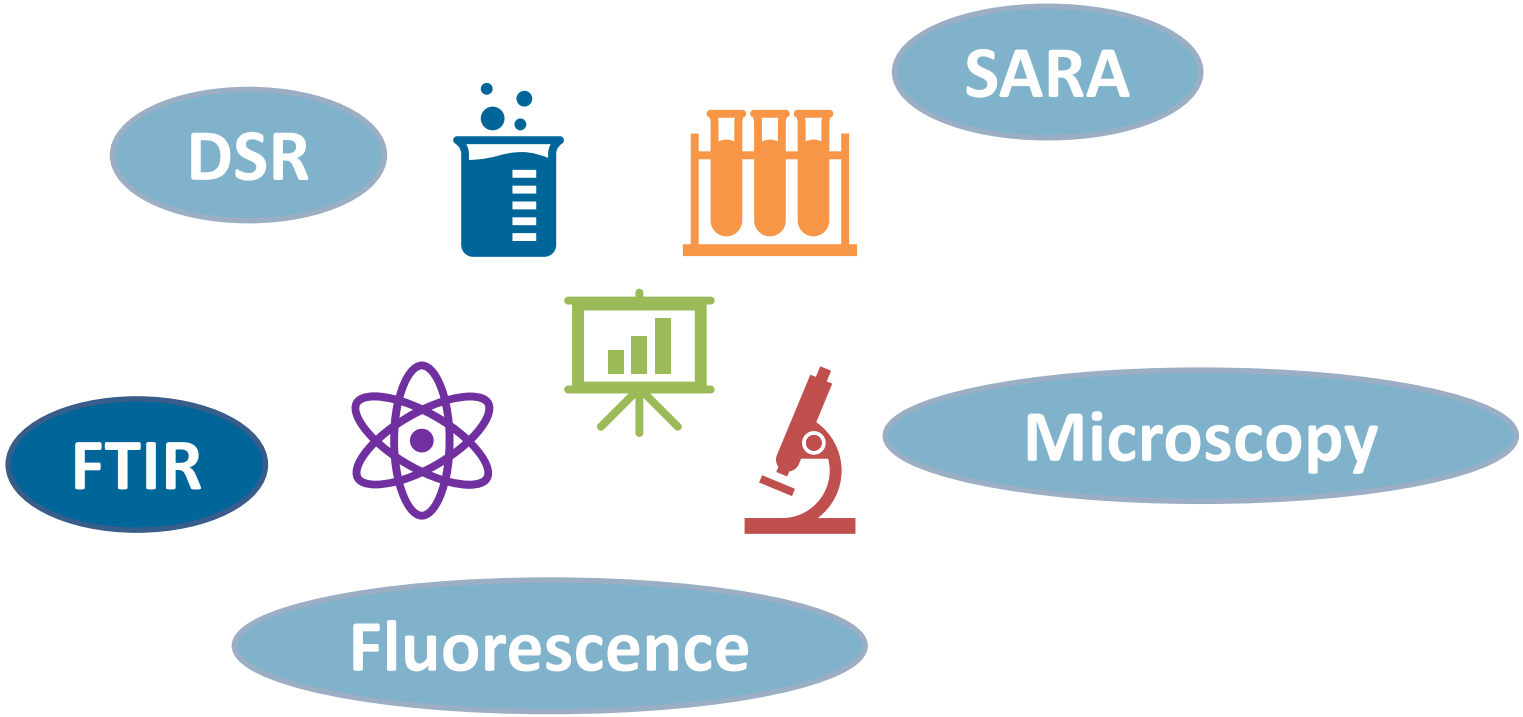
- Chemical Ageing (reactive gasses)




- Photolytic Ageing

# How do we access bitumen quality and the changes occurring during its service life?

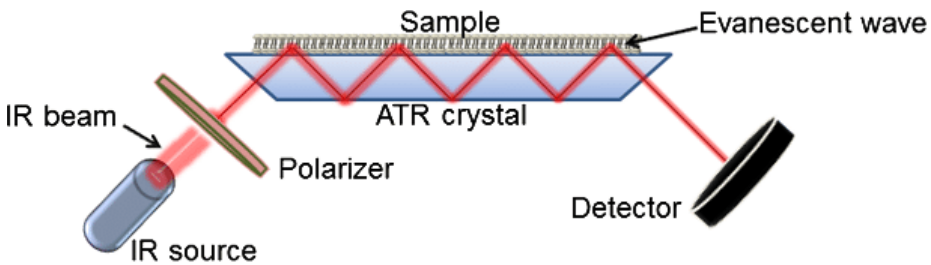
# Access Bitumen Quality



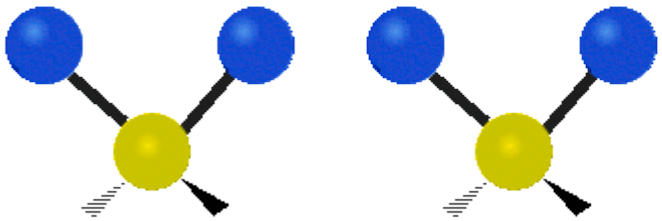
# ATR-FTIR

## Attenuated Total Reflection Fourier Transformation Infrared Spectroscopy

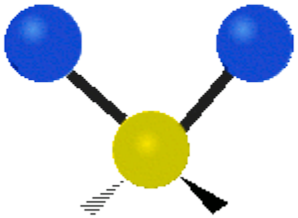
https://www.bruker.com/de/products-and-solutions/infrared-and-raman/ft-ir-routine-spectrometer/alpha-ii-compact-ft-ir-spectrometer.html (23.08.2021)

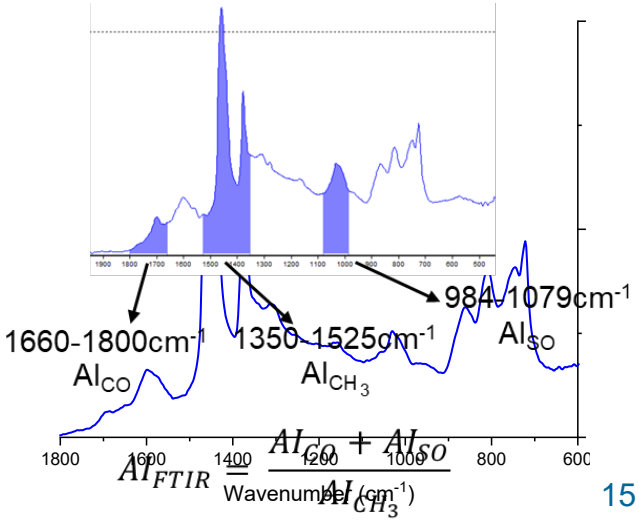



https://www.researchgate.net/figure/Schematic-representation-of-an-ATR-FTIR-system-The-infrared-beam-passes-through-the-ATR_fig1_275021699 (23.08.2021)



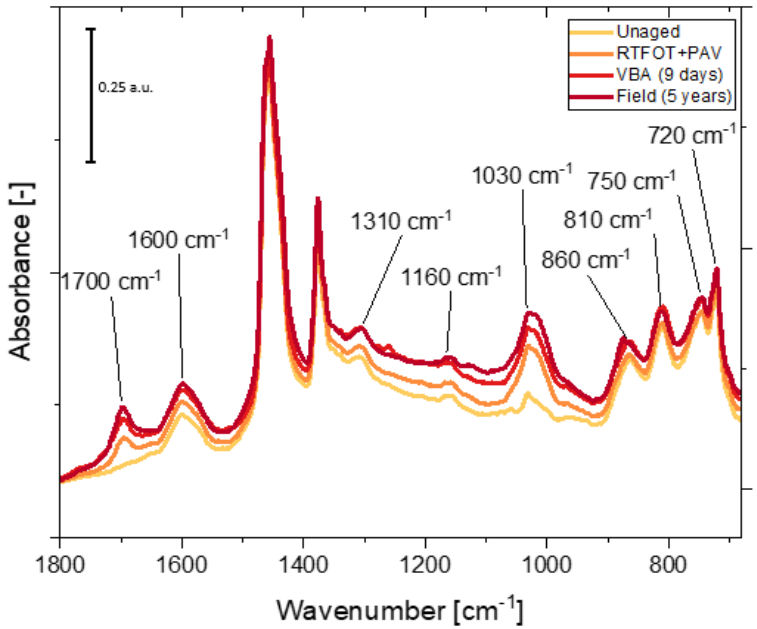
https://de.wikipedia.org/wiki/Deformationsschwingung (23.08.2021)

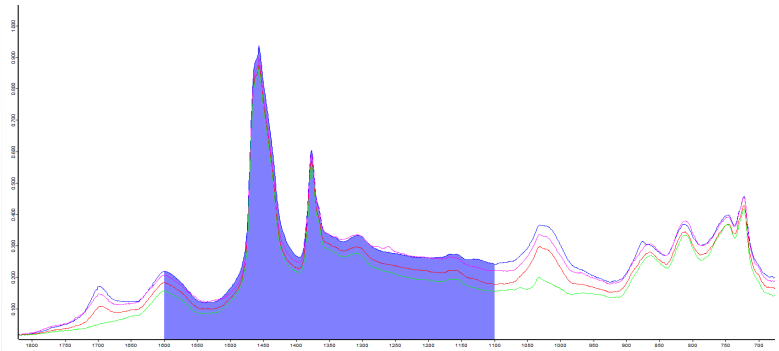





$$AI_{FTIR} = \frac{AI_{CO} + AI_{SO}}{AI_{CH_3}}$$

# FTIR of different crude oil sources



# Access Bitumen Ageing

PAV

Photo-oxidation

VBA

Oven Ageing

# Thermal Ageing – Oven Ageing

- 80 – 135 °C
- 1,013 bar
- 8 h – 3 months
- Air


Disadvantages

- No other parameters than temperature
- Long durations
- High temperatures → The right reactions?
- $O_2$ as the only „reactive“ species → **Pressure Ageing Vessel**

# Access Bitumen Ageing

PAV

Photo-oxidation

VBA

Oven Ageing

# Thermal Ageing - PAV

- 90 – 110 °C
- 20,7 bar
- 20 h
- Compressed Air

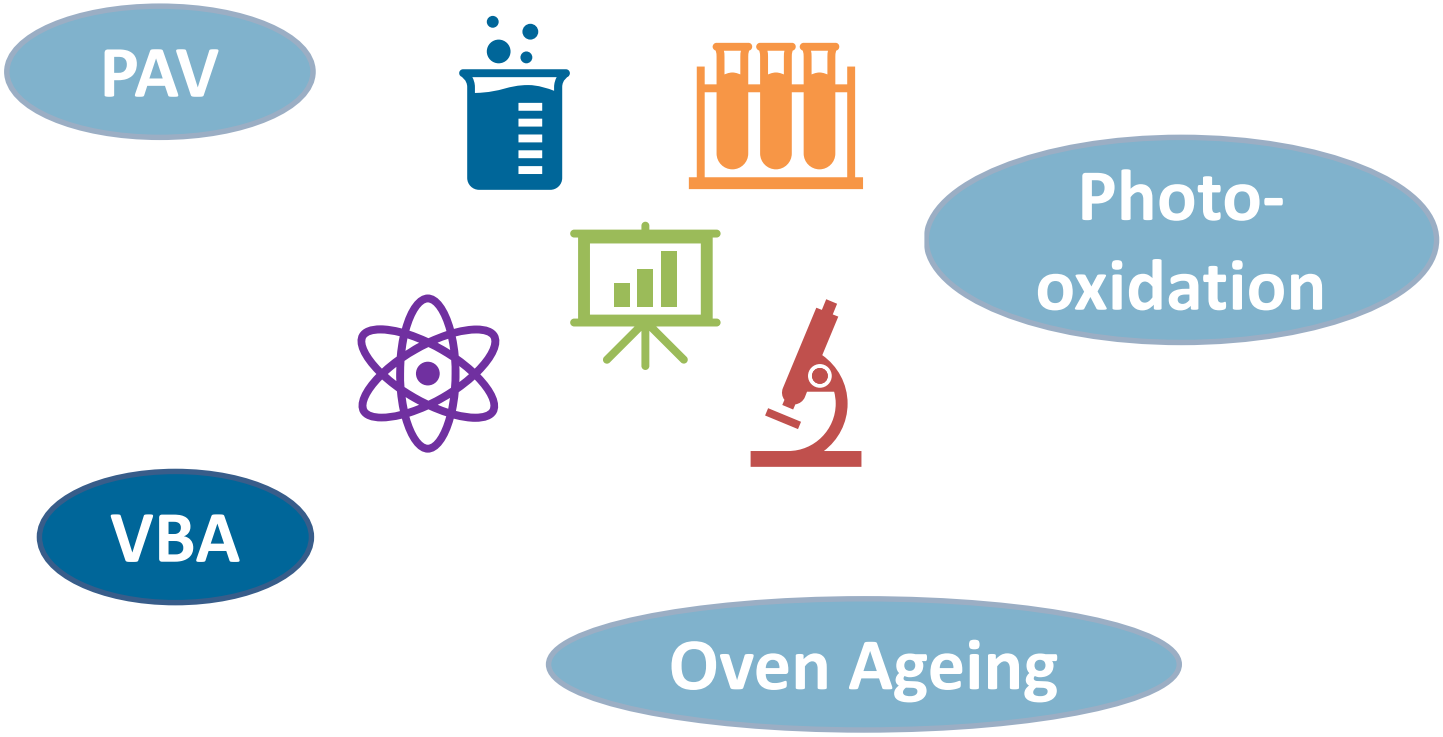
Advantages
- Time efficient
- Standardized Procedure

Disadvantages
- No other parameters than temperature
- Far away from field conditions
- Changes in the reaction kinetics?
- $O_2$ as the only „reactive“ species

https://www.eurobitume.eu/de/technik/pruefverfahren/ (17.06.2021)

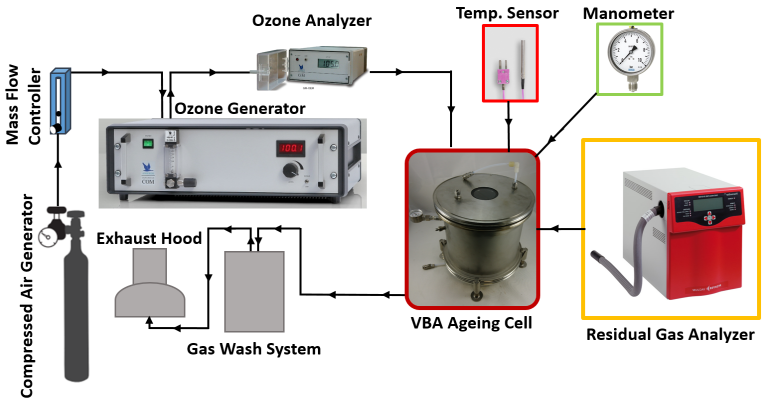
**Chemical Ageing**

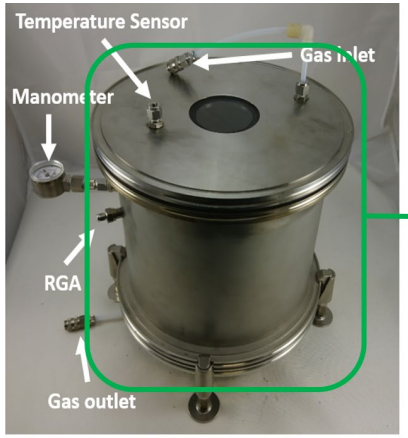
# Access Bitumen Ageing

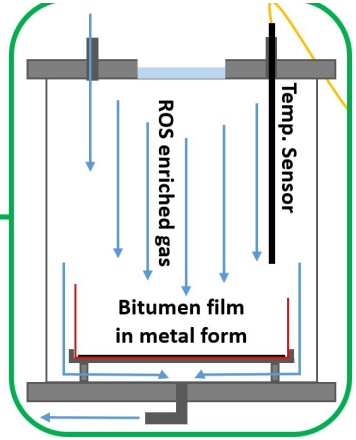


# Chemical Ageing – Viennese Binder Ageing (VBA)

- 80 °C
- 1,013 bar
- 3 – 9 days
- Compressed Air, $O_3$, $NO_X$



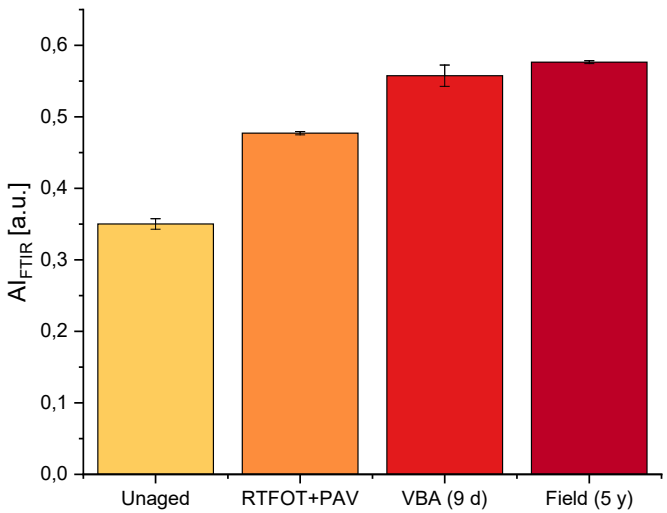




Advantages
- Time efficient (adjustable to needs)
- Thermal and chemical ageing

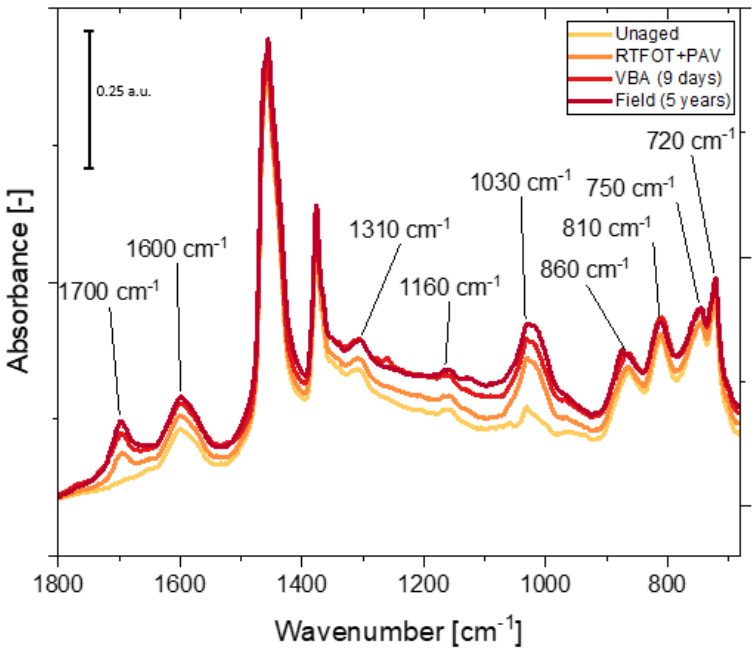
Disadvantages
- Missing OH radicals (moisture)
- Influence of Light

# Comparing PAV, VBA and Field

## DSR

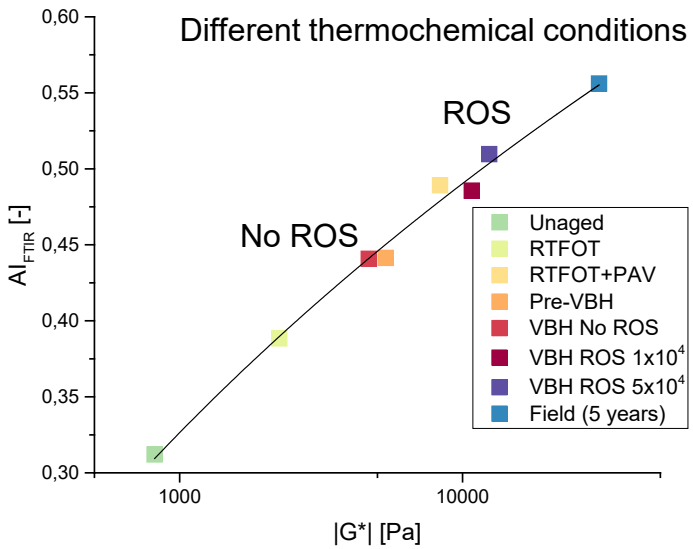



## ATR-FTIR

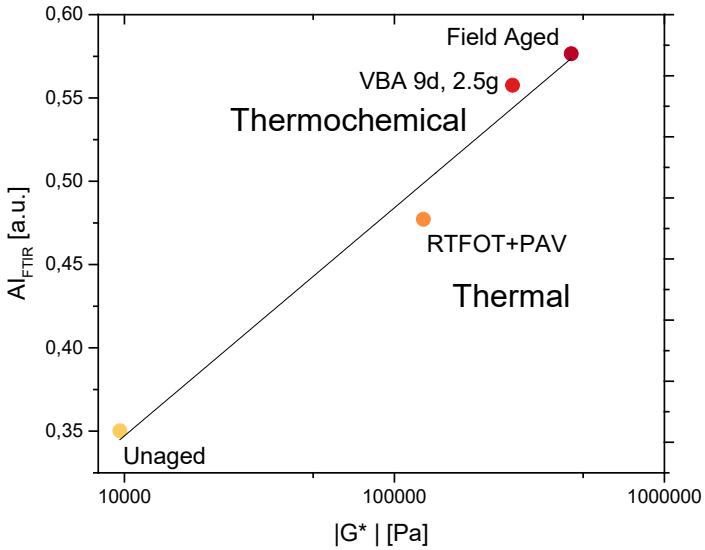


# Chemo-Mechanical Correlation

- Incorporation of oxygen via formation of functional groups
- Increase in stiffness (@ f=1.59Hz | 46°C)
- Link between **chemical surface** and **mechanical bulk methods**



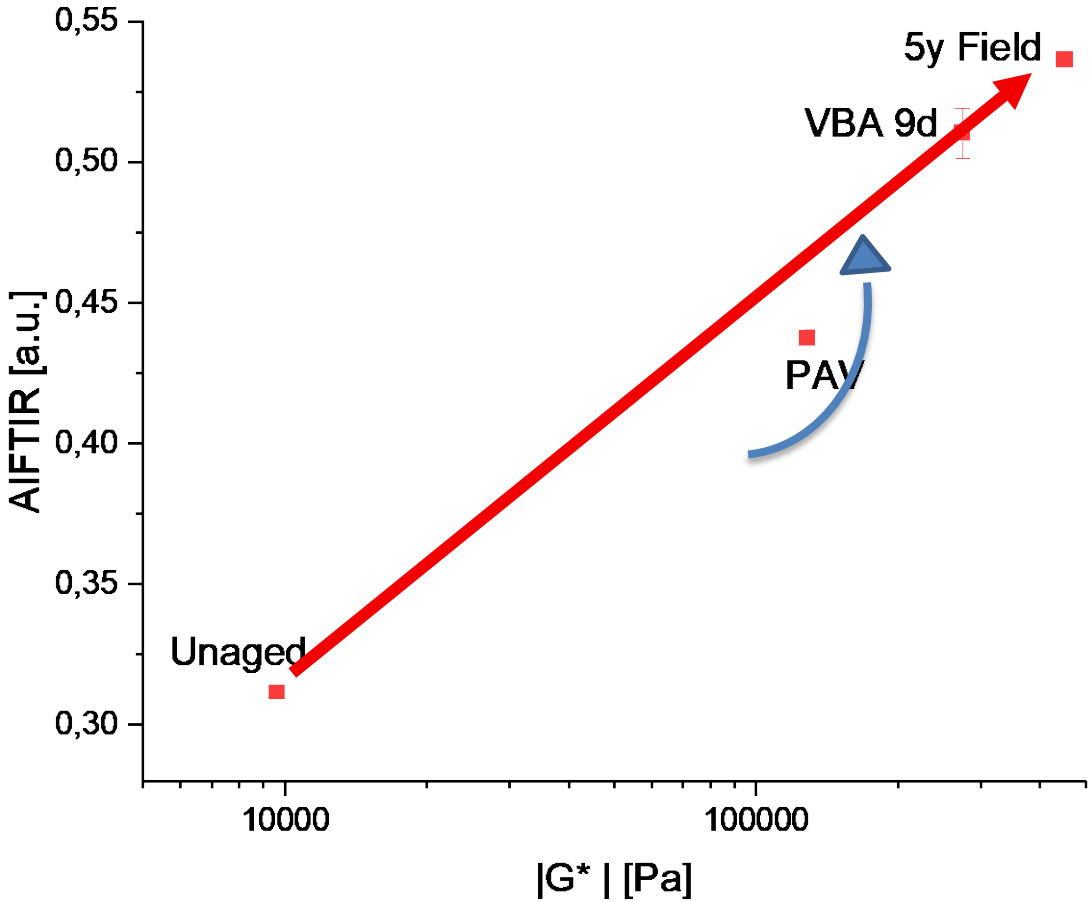

J. Mirwald, D. Maschauer, B. Hofko, H. Grothe: "*Impact of Reactive Oxygen Species on Bitumen Aging – The Viennese Binder Aging Method*"; Construction and Building Materials, **270** (2020), 119495



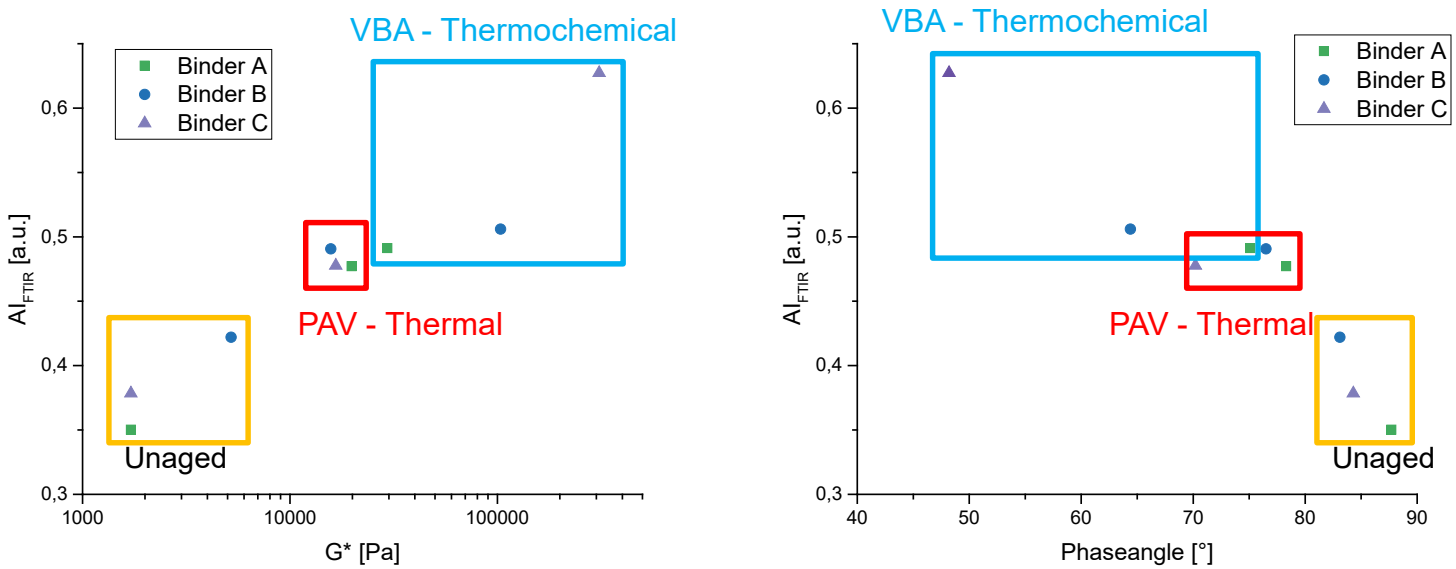

J. Mirwald, S. Werkovits, I. Camargo, D. Maschauer, B. Hofko, H. Grothe: "*Understanding Bitumen Ageing by Investigation of its Polarity Fractions*"; Construction and Building Materials, **250** (2020), 118809

# Chemo-Mechanical Correlation

- Differences between pure thermal and thermochemical ageing



# Comparison of Different Crude Oil Sources but same Specification Class




J. Mirwald, S. Werkovits, I. Camargo, D. Maschauer, B. Hofko, H. Grothe: "*Investigating Bitumen Long-Term-Ageing In the Laboratory by Spectroscopic Analysis of the SARA Fractions*"; Construction and Building Materials, **258** (2020), 119577

# Access Bitumen Ageing

PAV

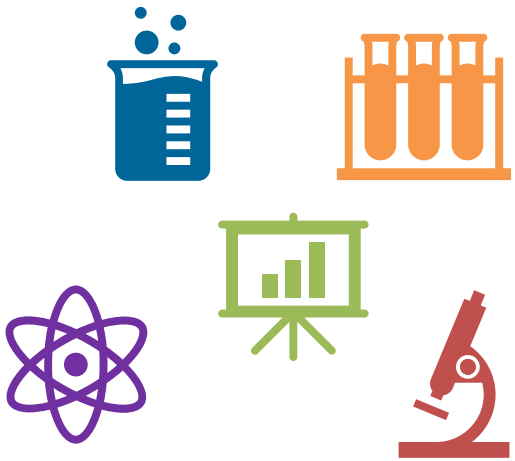


Photo-oxidation

VBA

Oven Ageing

# Take Home Messages

- **FTIR Spectroscopy** is the most promising candidate for chemical analysis of bituminous materials
  - Factors need to b[illegible] **pre-standardization**
- **Chemo-**[illegible]
  compre[illegible]ing
  behaviour o[illegible]
  - Links oxygen incorporation to increase in stiffness
- **Different ageing mechanism** can contribute to long-term ageing and need to be considered for accessing bitumen quality
  - **Thermal**
  - **Chemical**
  - **Photolytic**

Questions?

johannes.mirwald@tuwien.ac.at