TOPIC 9: VIP Characterization

Defects in the envelope barrier of VIP after hygrothermal ageing in severe conditions

F.Dubelley[1,2], E.Planes[1,2] C.Bas[1,2,] B.Yrieix[3] & L.Flandin[1,2]

[1]Univ. Savoie Mont Blanc, LEPMI, F-73000 Chambéry, France

[2]CNRS, LEPMI, F-38000 Grenoble, France

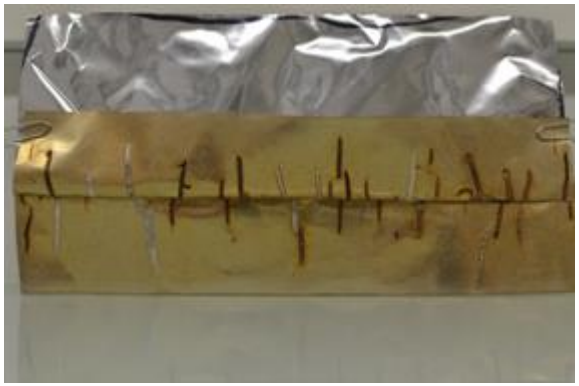
[3]EDF R&D – MMC, 77818 Morêt sur Loing, France

Overview

- Operating conditions in building
  - Depends on applications, climate, face, season, …
  - Severe conditions: **70°C – 90 %HR**

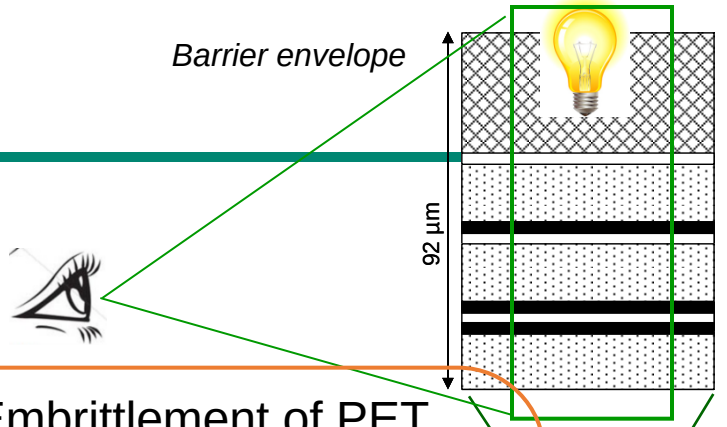
- **Goal:**
  - **Durability:** capacity in time to insulate to the required level under these conditions

→ Example after 3 years of ageing

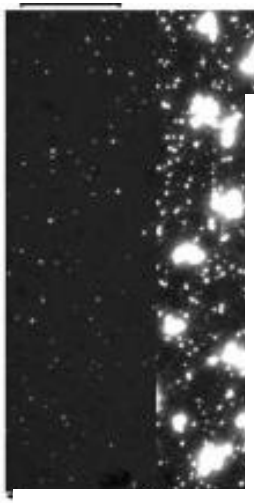
t0 λ = 4 mW/(m.K)	450 days λ = 20 mW/(m.K)	870 days **λ > 20 mW/(m.K)**
		

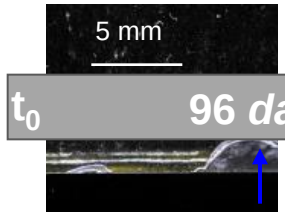
Overview

- After ageing @ *70°C/90%HR*



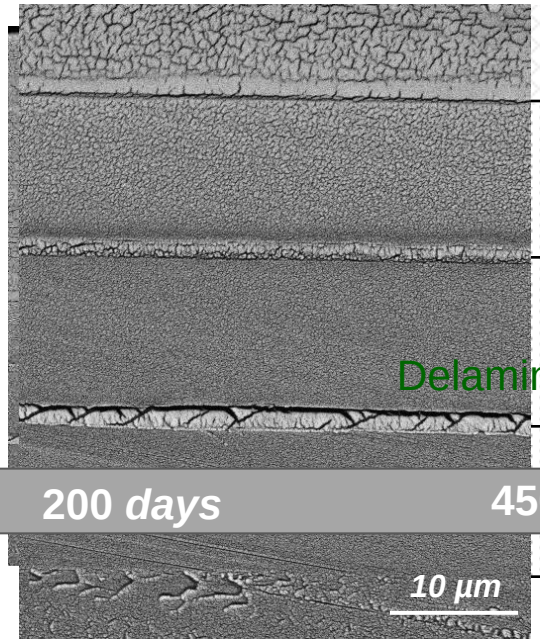
➢ Aluminum loss





Aluminum loss
Delamination PET / PE

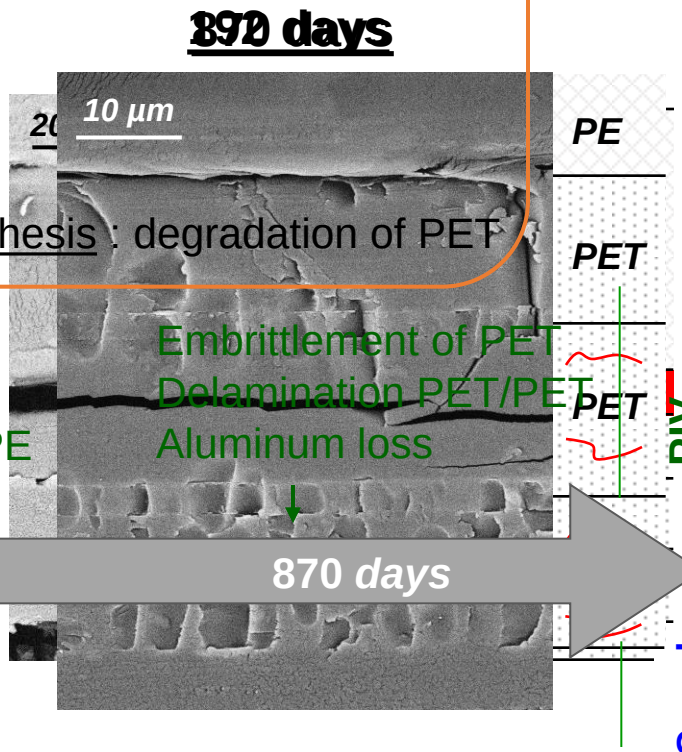
➢ Delamination



➢ Embrittlement of PET

By Scanning Electron Microscopy (SEM)

Hypothesis : degradation of PET

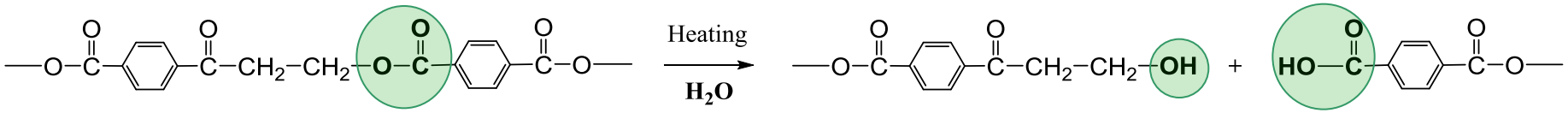


Delamination PET / PE

t_0 — 96 *days* — 200 *days* — 450 *days* — 870 *days*

Degradation of PET

- Well-know mechanism → ***Hydrolysis****



- <u>Goal:</u>

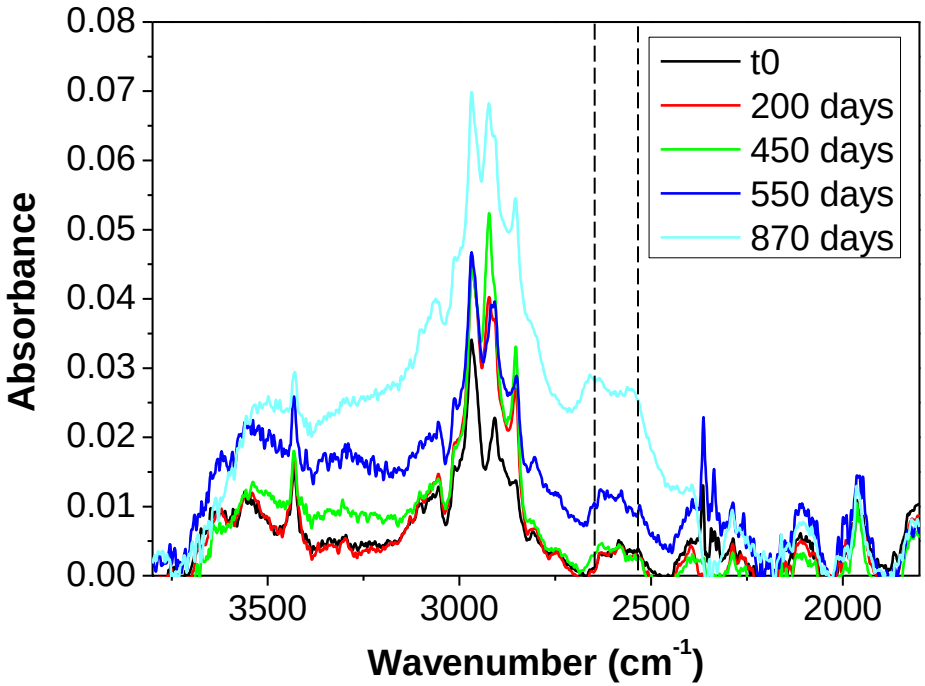
  - Study of the degradation kinetic by IR spectroscopy
  - → **Identification of markers**

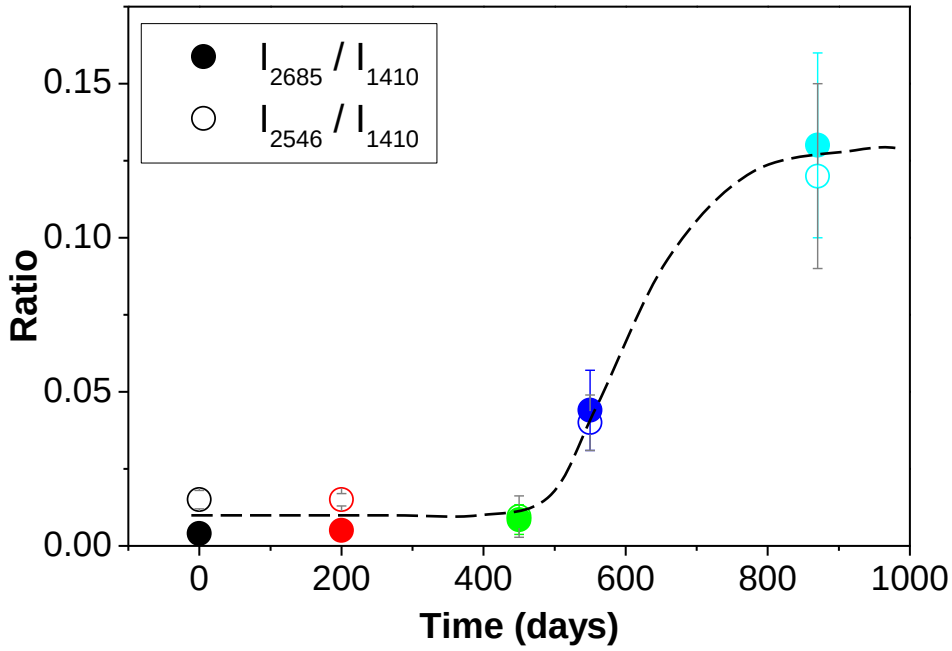
  - Evidence for a degradation gradient in the three PET layers of the barrier envelope by IR microscopy ?

* <u>Adapted from:</u>
T.Wu and Y.Ke, *Polym.Degrad.Stabil.* 91 (2006), 2205-2212
B.J.Holland and J.N.Hay, *Polymer* 46 (2002), 1835-1847

Hydrolysis of PET

- Carboxylic Acids (COOH)
  - *v(OH)*
    - broadband and high intensity between 3500 cm^{-1} and 2500 cm^{-1}
    - two shoulders close to 2600 cm^{-1} and 2500 cm^{-1}



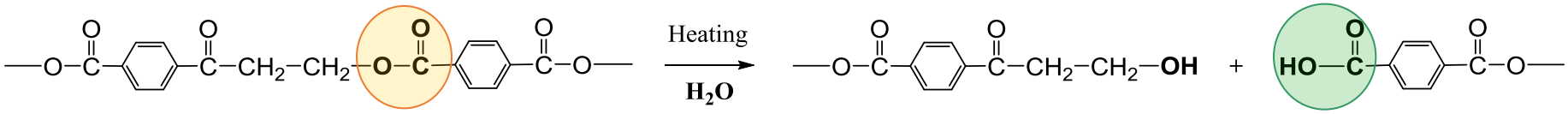


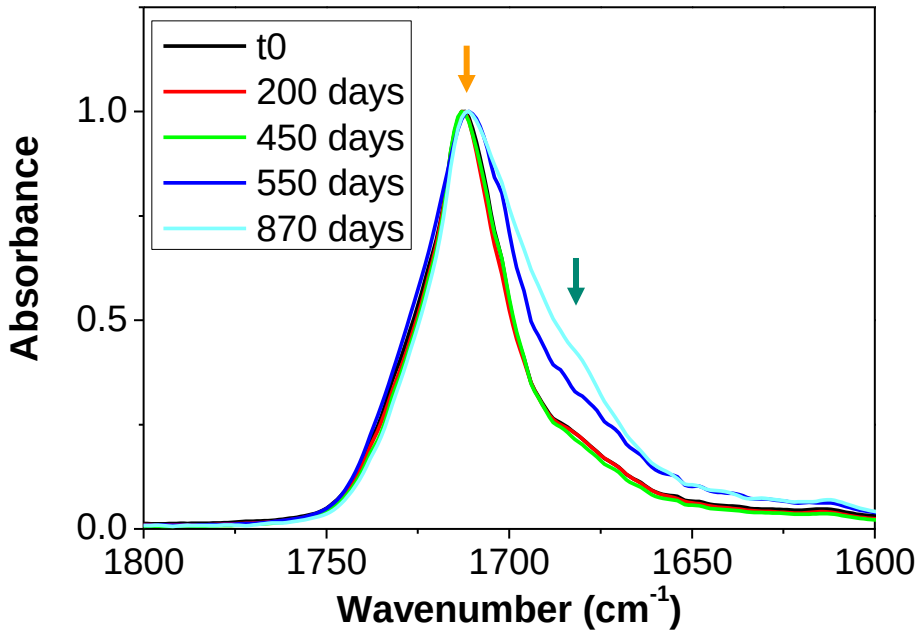
Hydrolysis of PET

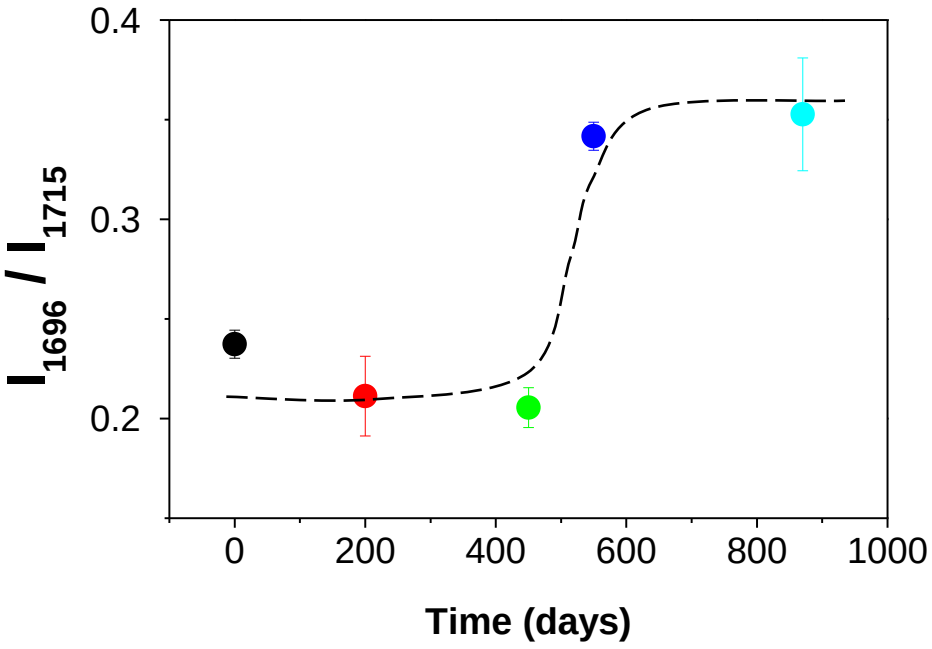
- Carboxylic Acids (COOH)

*J.V Gulmine et al., *Polymer degradation and stability* 79 (2003) 385-397
B.Du et al., *Chinese Journal of Polymer Science* 32 (2014) 230-235

- *v(C=O): 1700 cm^{-1}*
  - Wavenumber modification of band with neighboring links*

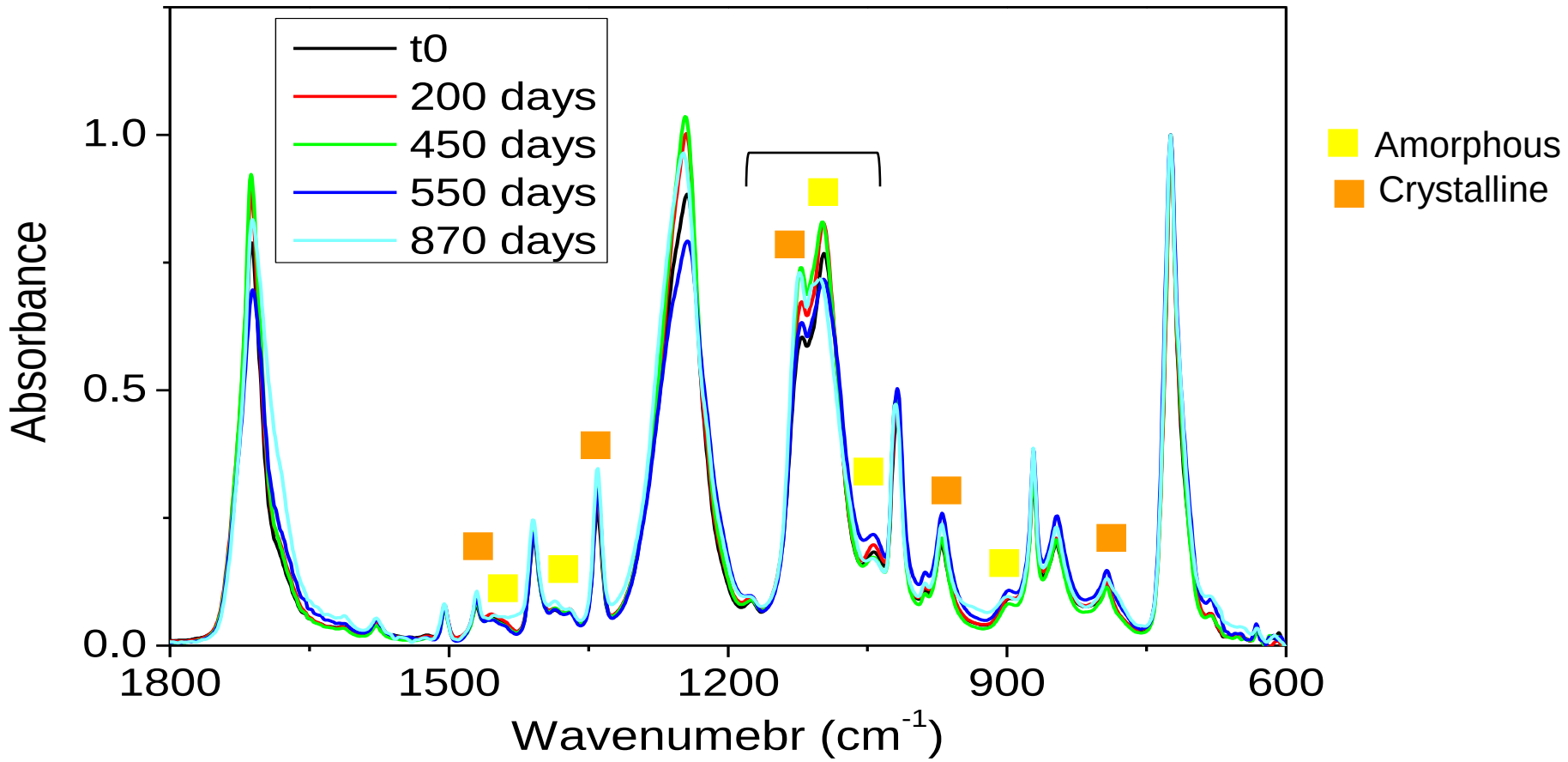






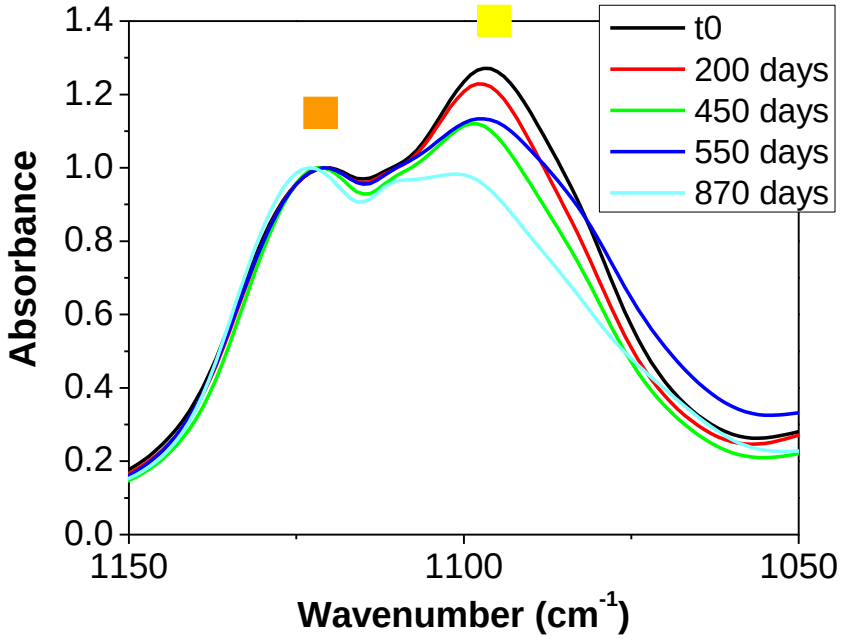
Hydrolysis of PET

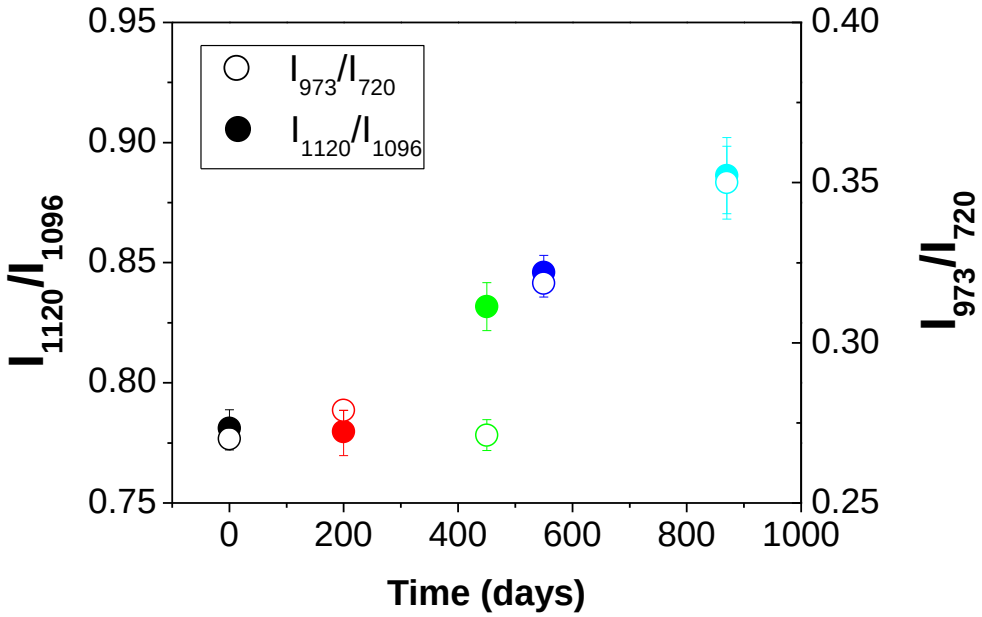
- Chain scission
  - ↘ chain length ⇒ ↗ Amount of small chains ⇒ **Crystallization**



Hydrolysis of PET

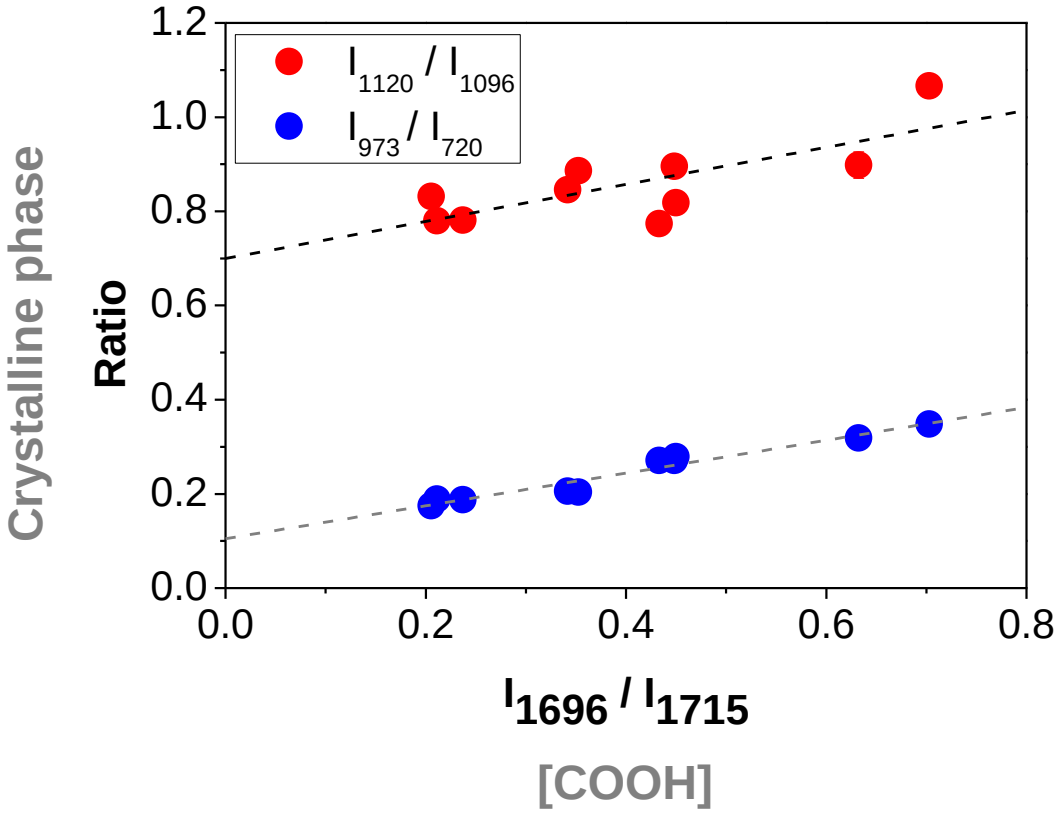
- Crystallinity change
  - ➢ 1120 cm^{-1}: v(C-0-C) in crystalline phase + *trans* conformation of CH_2-CH_2
  - ➢ 1096 cm^{-1}: v(C-0-C) in amorphous phase + *gauche* conformation of CH_2-CH_2
  - ➢ 973 cm^{-1}: v(C-H) in *trans* conformation of CH_2-CH_2





Hydrolysis of PET

- Correlation ***trans* Conformation / [COOH]**



↗ **[COOH]**

↓

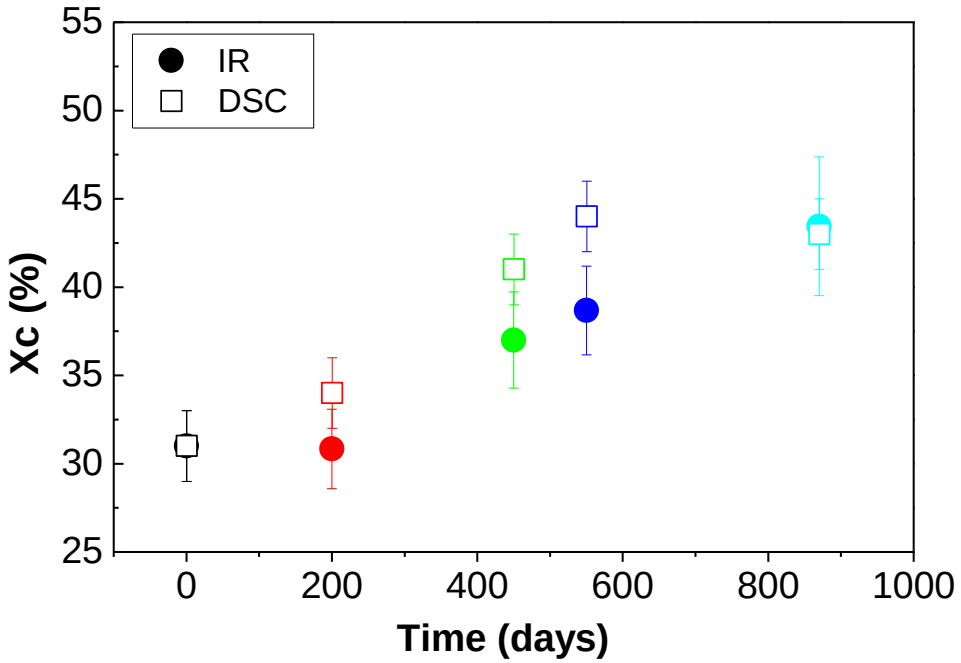
↗ ***trans* conformation**

↓

Confirmation of the PET hydrolysis

Hydrolysis of PET

- Evaluation of crystallinity ratio X_c by IR spectroscopy
- Correlation with DSC (*Differential Scanning Calorimetry*) measurements



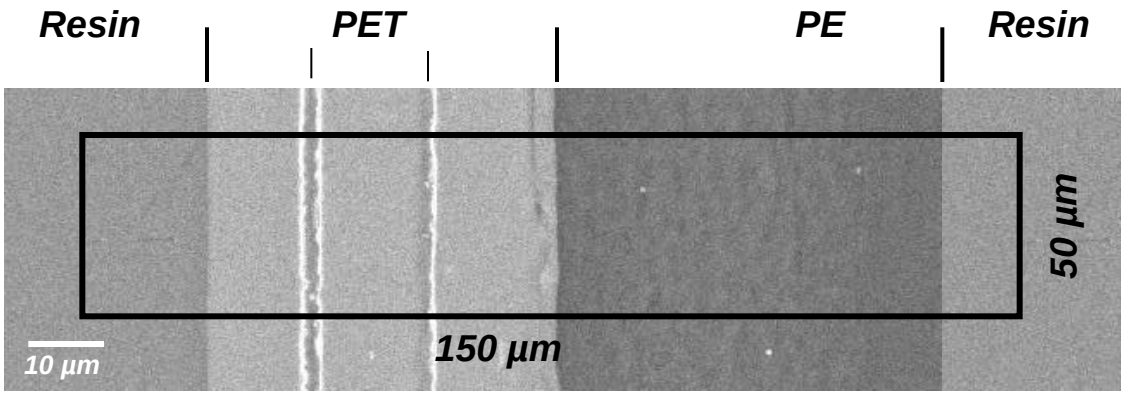
➢DSC measurement:
Hypothesis: uniform degradation of the three PET layers

➢ Hypothesis: degradation gradient

Study of degradation of the inner PET layer by IR microscopy

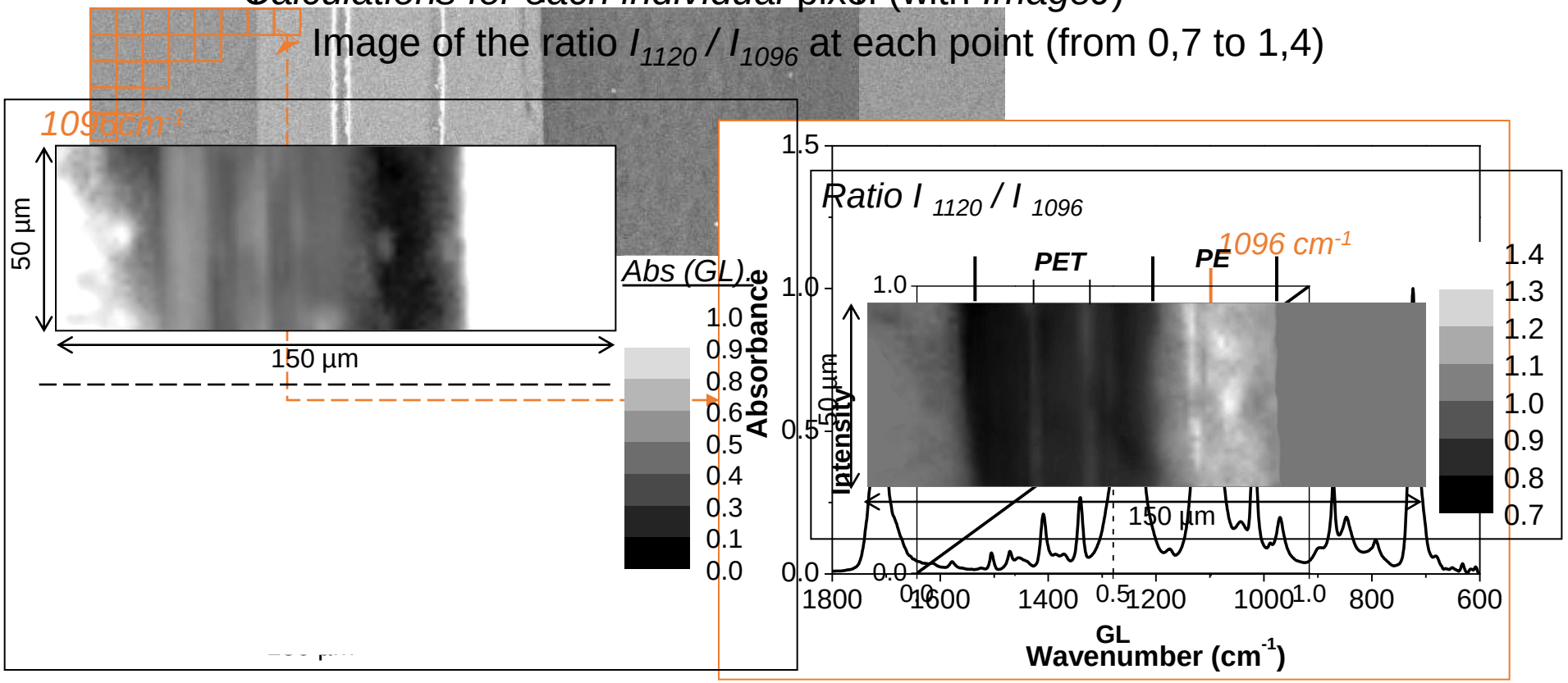
Degradation of the inner PET layers

- IR microscopy
  - Spotlight 400 PerkinElmer
- Sample preparation
  - Hold in a cone
  - Cut with microtome



Degradation of the inner PET layers

- Treatment
  - ~ 3000 spectra / analyzed area
  - Each pixel is a IR spectrum
  - Cartography: absorbance for a given wavenumber
  - *Calculations for each individual* pixel (with *ImageJ*)
    - Image of the ratio I_{1120} / I_{1096} at each point (from 0,7 to 1,4)



Degradation of the inner PET layers

- Application on the barrier envelope reference (t0)

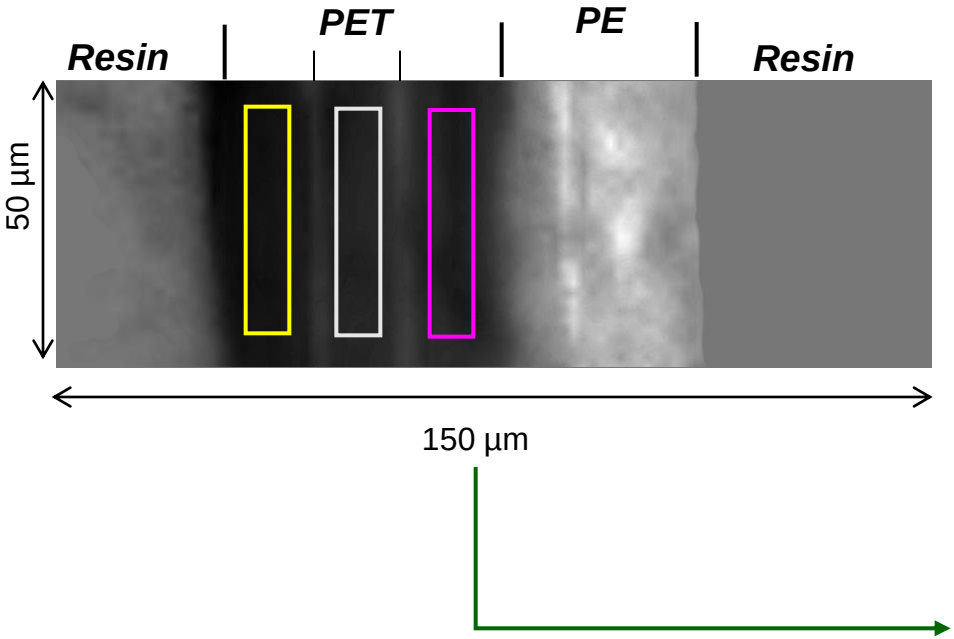
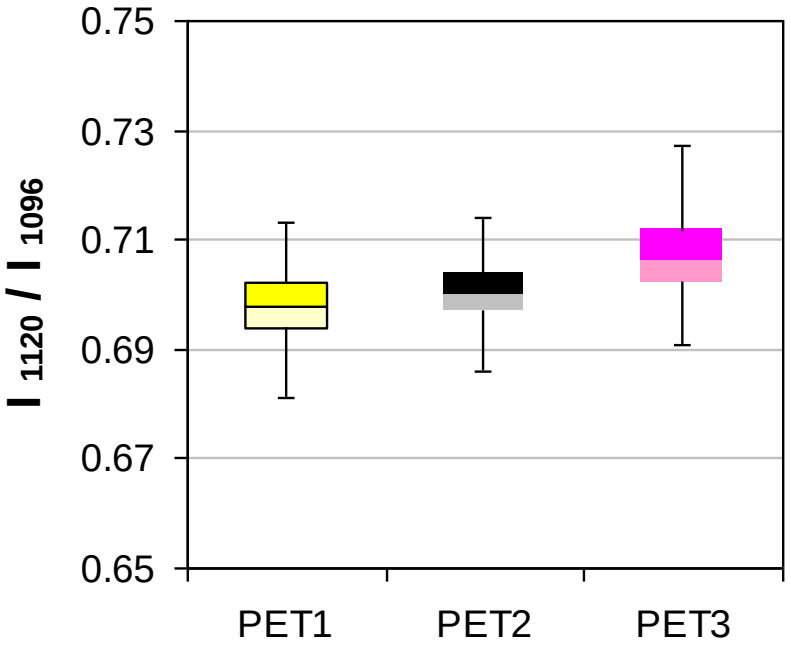


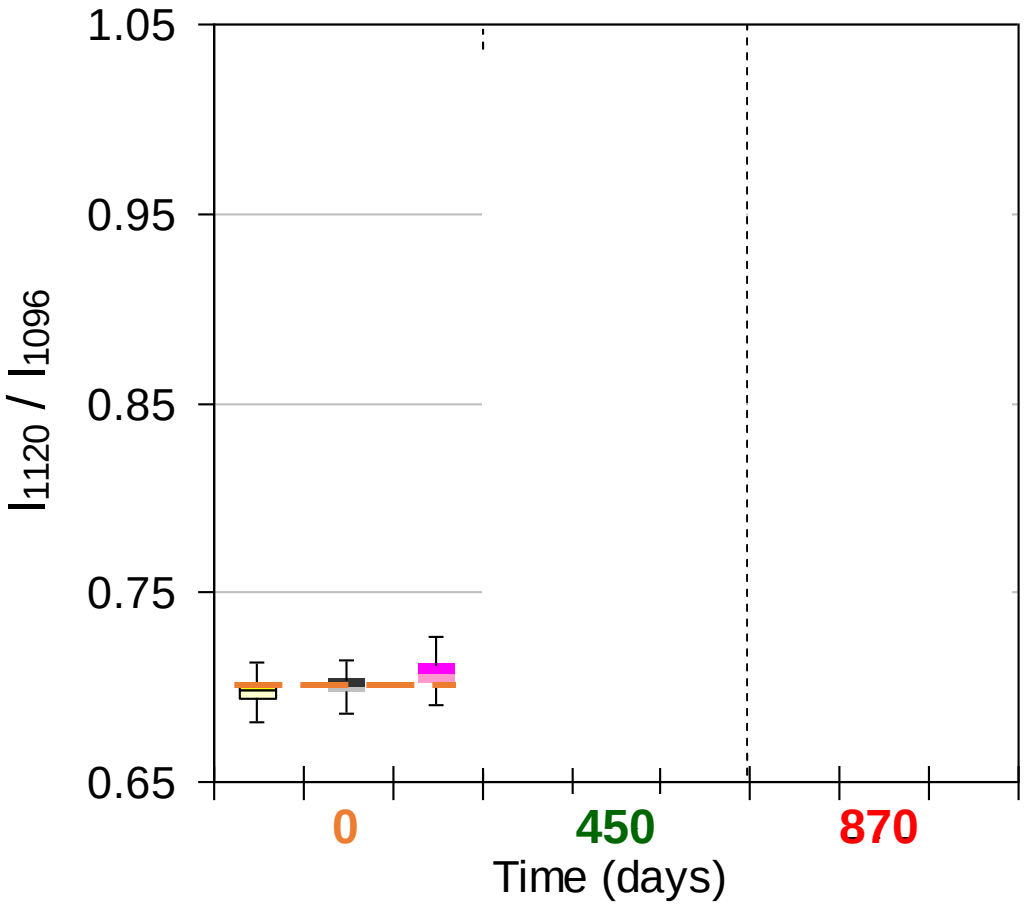
Image treatment

Pixel value = I_{1120} / I_{1096}



Degradation of the inner PET layer

- Results I_{1120} / I_{1096}



Variation of degradation
PET1 < PET2 < PET3

⬇

Not in agreement with gas transfer*

⬇

Other origin
Hypothesis: Hydrolysis of PU adhesive

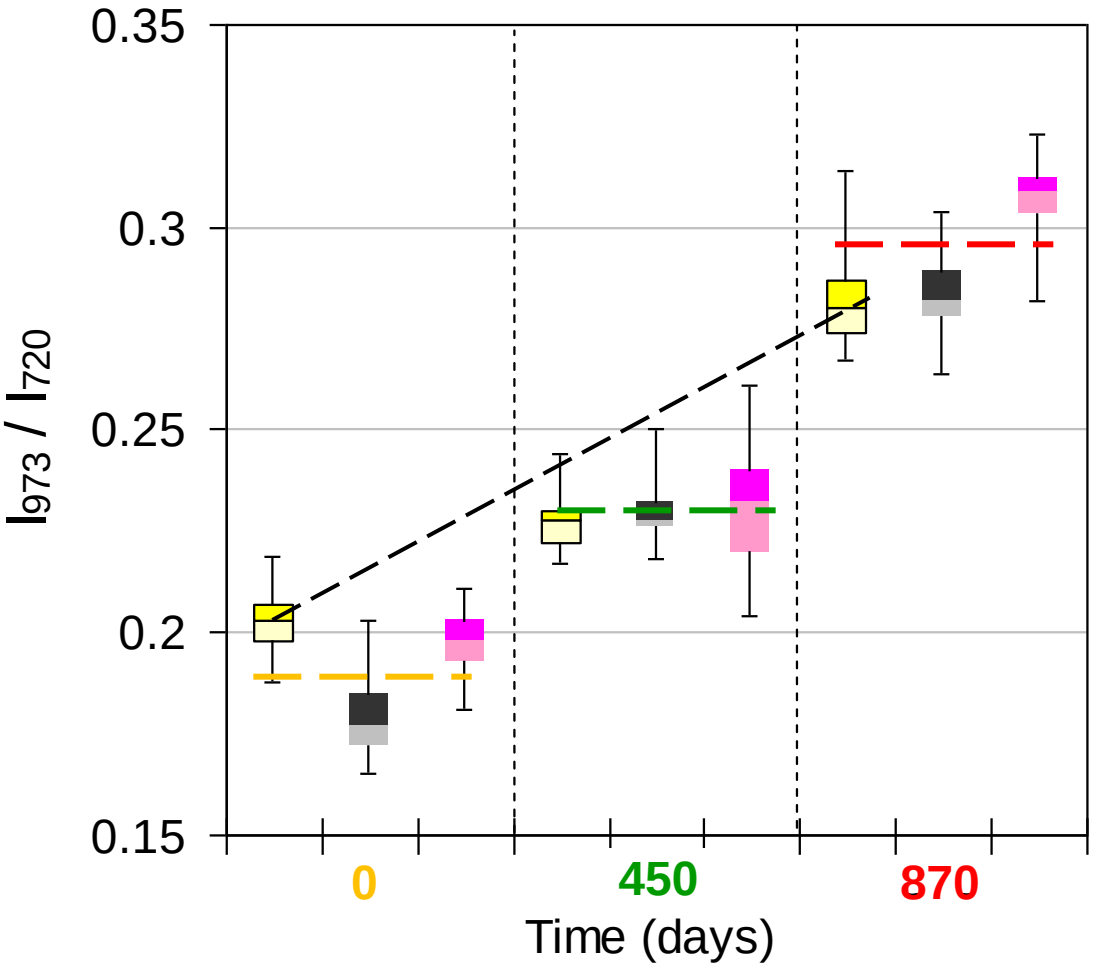
⬇

Study of the second IR ratio I_{973}/I_{720}

* E.Pons et al., *Energ.Buildings* 85 (2014) 604-616

Degradation of the inner PET layers

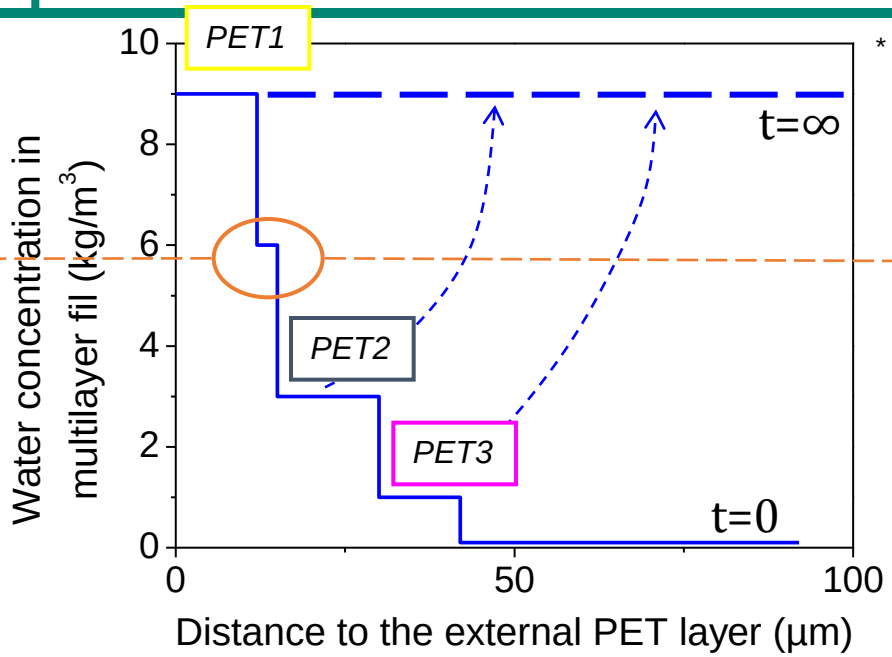
- Results I_{973} / I_{720}



- PET1: Measure in agreement with the macroscopic study
  - No degradation gradient : PET1 = PET2 = PET3

Conclusion

- Hypothesis

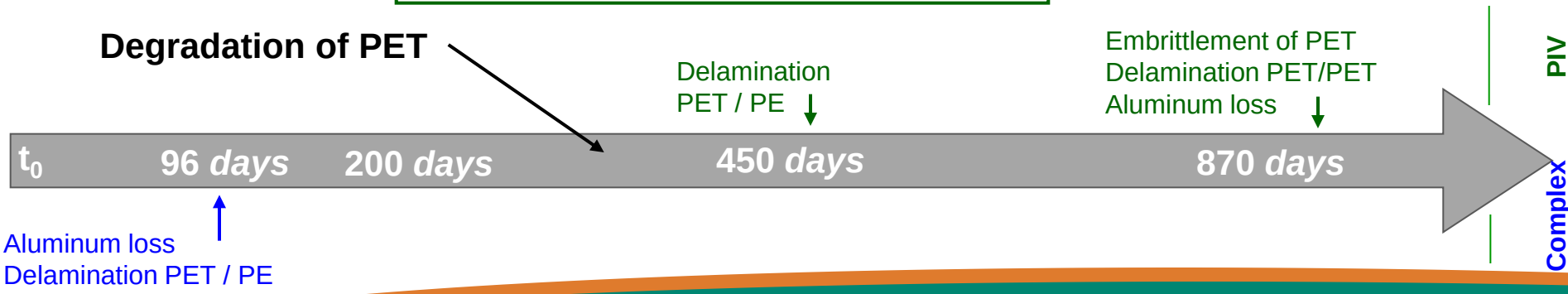


* E.Pons et al., *Energ.Buildings* 85 (2014) 604-616

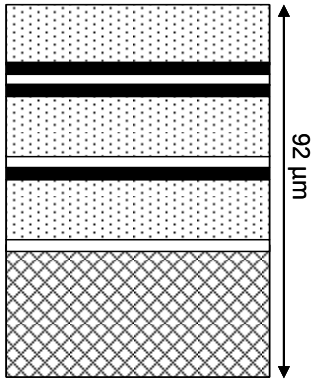
- RH
- PET hydrolysis
- Formation
  Carboxylic acid function
  Ethylene glycol
- Change of pH

→ Aluminum Corrosion
→ Loss of barrier properties
→ Inner PET layer degradation

Degradation of PET

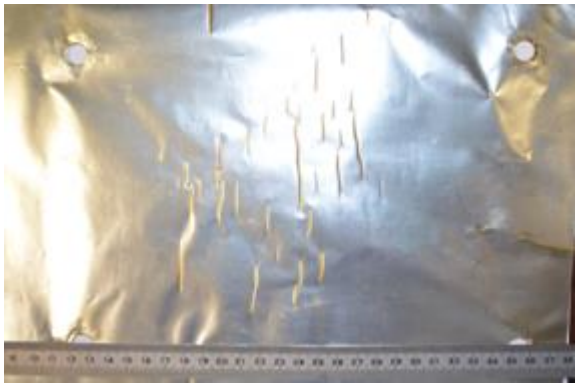


Conclusion



- Optimized Structure
- Components optimization

→ Ageing at 70 °C – 90 %HR – **200 days**

Envelope A (*Ref*)	Envelope B (*New*)
	

Acknowledgments

- ANR (French National Research Agency) for their financial support of EMMA-PIV projects n°12-VBDU-0004

- Special thanks to industrial partners:
  EDF *(E.Pons, EnerBat department)*

  REXOR *(P.Rousset, E.Perrin, B.Larrieu)*

- ADEME :French Agency for Environment and Energy Management *(S.Kherrouf)*

BACKUP

Degradation of inner PET layers

- Influence of the polyurethane adhesive