Accelerated ageing test to reproduce the degradation of composite material in sea water

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Introduction

Why accelerated ageing test:

Experience shows that there is **degradation of material** with time during the operation due to the **interaction with the surrounding environment**.

→**May lead to the failure of the asset!**

→The **kinetic** of the **degradation** is **slow** (years)

Necessity to get information from the degradation due to natural ageing to intend to design an artificial ageing procedure
→ get similar degradation mechanisms in a shorter time

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Introduction

In the NEMMO project:

Asset → tidal turbine blade

Material: glass fiber reinforced vinyl ester resin composite with a gelcoat surface finishing

Surrounding environment: immersion in sea water

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Identification of mechanisms affecting service-life from a material point of view

Complex interaction between the sea water component and properties and the degradation of the composite material in immersion

- **Water absorption:**
  - Lead to plasticization → diminution of the glass temperature transition, loss of mechanical properties
  - Matrix swelling → create interfacial cracks and fiber/matrix debonding
- **Mechanical load:**
  - the general effect of mechanical loading onto the composite material is to **enhance the moisture-penetration** mechanisms **producing higher rates and maximum levels of moisture penetration**
- **Fouling interaction:**
  - Growth of biofouling on the material → degradation of the material due to the metabolism of the micro and macro-organisms.

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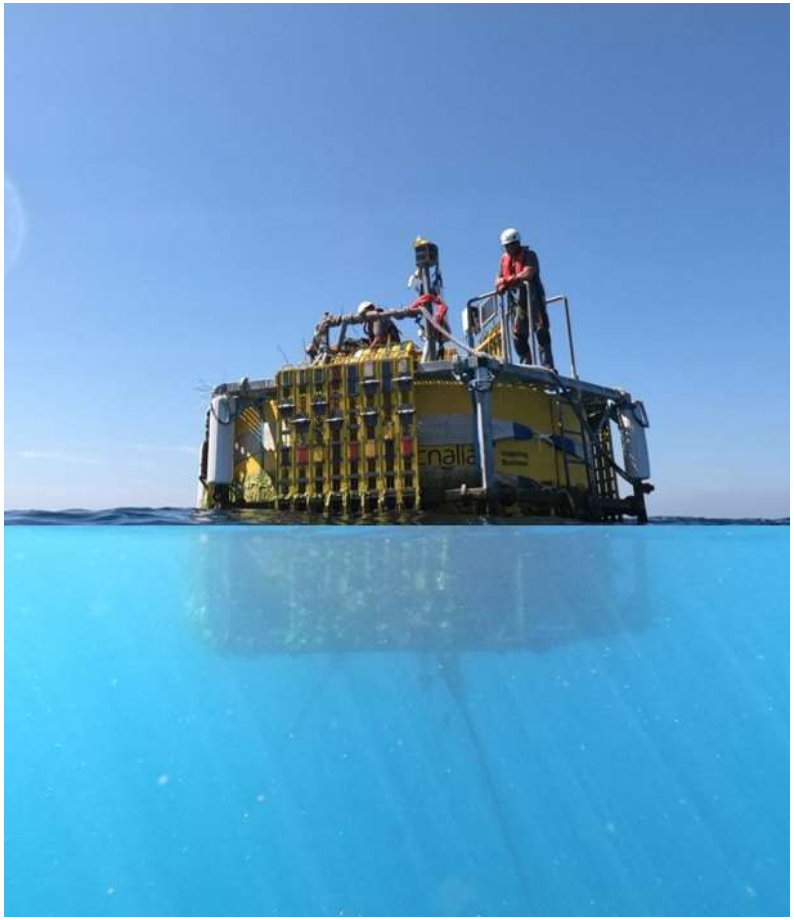
Natural ageing tests

Natural ageing :

- Port of Pasaia: port environment with high fouling development
- Harshlab: offshore facility for material samples exposition

→ **Samples** of the **composite material** will be put in **immersion** for **10 months** in both locations



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Natural ageing tests

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Natural ageing tests

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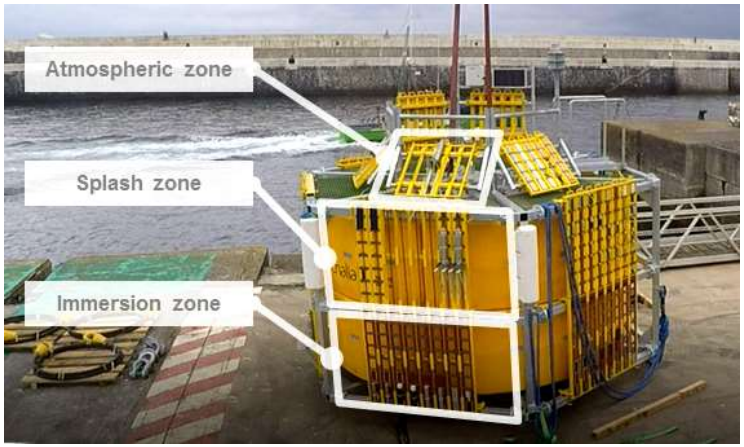

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Design of an adequate accelerated ageing test

The material degradation mechanisms due to the interaction with the environment is are slow phenomenon and high life-time is expected (usual target: 25yrs) → needs of accelerated ageing test as relevant as possible

Design of an accelerated test based on the standard ISO 12944 → used for assessment of protective coating in various natural environment among which the offshore environment is contemplated.

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Design of an adequate accelerated ageing test

The modified ageing cycle proposed:

	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7
							
Aging media	Condensation 24h	Condensation 24h	Immersion seawater 24h	Immersion seawater 24h	Immersion seawater 24h	Immersion seawater 24h	Immersion seawater 24ł
Temp.	T=50°C	T=50°C	T=40 °C	T=40 °C	T=40 °C	T=40 °C	T=-2 °C
Radiation	12h UVA 340, 0,68 W/m2 12 h OFF	12h UVA 340, 0,68 W/m2 12 h OFF					

20 cycles will be performed

The accelerated ageing temperature is set at 40ºC in immersion to accelerate the degradation mechanisms as most of these mechanisms are linked to the Arrhenius law:

$$s = A \exp(-B/T)$$

10ºC increase → x2 acceleration

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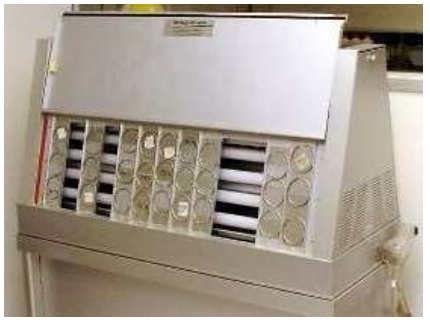
Design of an adequate accelerated ageing test

- **Days 1, 2**: 24 hours water condensation at 50°C combined with 12 hours periods of UV illumination. An irradiance level of 0.68 W/m2 at 340 nm was chosen to match the typical maximum irradiance of summer sunlight at noon.
- **Days 3-6:** Immersion in seawater at 40°C. The elevated temperature, selected to accelerate the degradation process, is low compared to the glass transition temperature of most polymeric matrices. Thus, it can be safely assumed that the degradation mechanisms themselves would not be affected.
- **Day 7:** Immersion in seawater at -2°C. The aim is to simulate low temperature conditions in sea, approaching the lowest possible temperature of the seawater without freezing.



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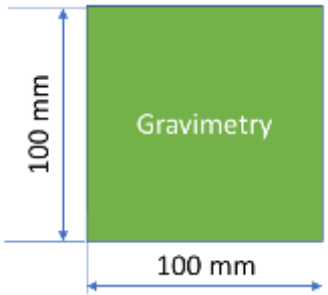
Characterization on the damages

Characterization of the samples:

- Gravimetry (ASTM D5229) → mass variation before and after ageing



$m = (w - w_0)/w_0 \times 100$

- Differential Scanning Calorimetry (DSC) (ISO 11357)

Samples weight should be in the range 5-100 mg
The DSC → from -90ºC to 250ºC at 20ºC/min
Special interest in Tg determination

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Characterization on the damages

Characterization of the samples:

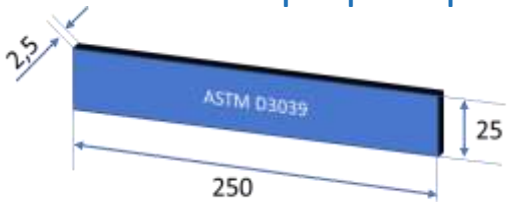
- Fourier Transform Infrared Spectroscopy (FTIR) (ASTM E1252 – 98)

1mg of the samples will be ground into powder with infrared-grade. KBr 100 mg will be used to prepare pellet

Information about the chemical characteristics of the composites polymerization and curing degree) as well as the degradation chemistry (degree of cross linking, scission reactions during aging).

- Tensile test (ASTM D3039)

1mg of the samples will be ground into powder with infrared-grade. KBr 100 mg will be used to prepare pellet



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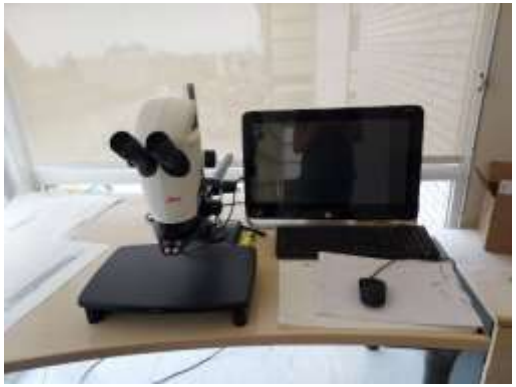
Characterization on the damages

Characterization of the samples:
(same for the 3 ageing)

- Optical: binocular and camera



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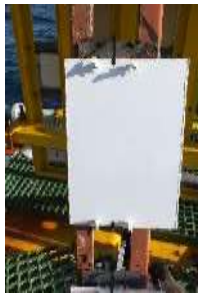
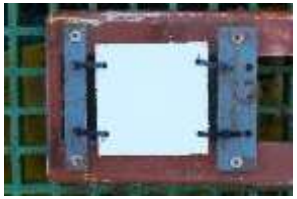
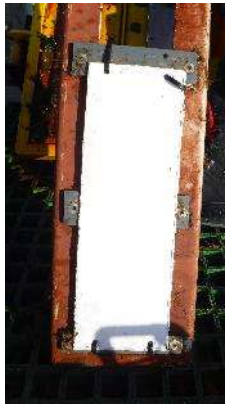
Ageing campaign

- **Samples installed in Harshlab:**

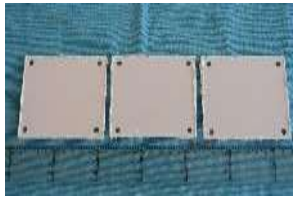


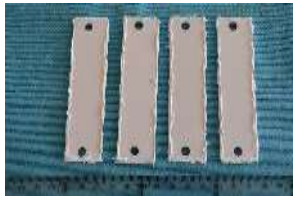


- **Samples installed in Pasaia**







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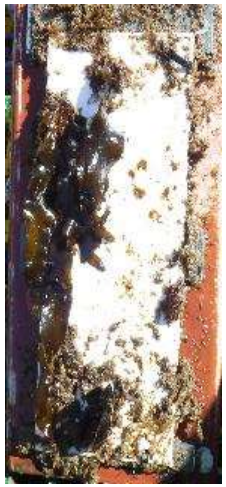
Ageing campaign

HarshLab

3 months



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Ageing campaign

HarshLab

7 months






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Ageing campaign

Pasaia

1 month

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Ageing campaign

Pasaia

3 months






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Ageing campaign

Pasaia

5 months








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Ageing campaign

Pasaia

8 months

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Results

- High presence of fooling → need to remove it → it's a challenge not completely solve to do it adequately

uncleaned

cleaned

10 months in Pasaia port

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Results

- High presence of fooling → need to remove it → it's a challenge not completely solve to do it adequately

uncleaned

10 months in Pasaia port

cleaned

No penetration of the barnacles and other organisms in the material but very strong adherence

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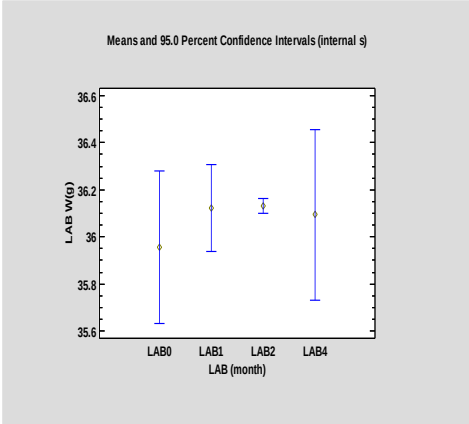
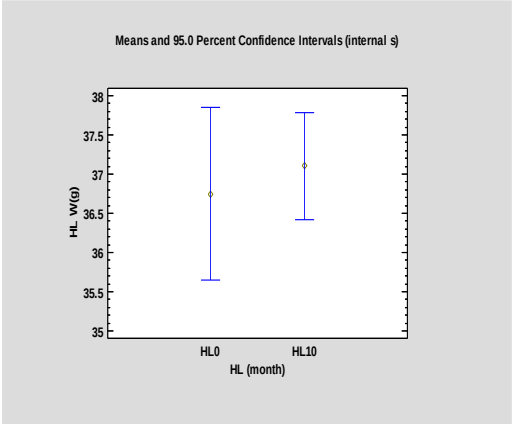
Results

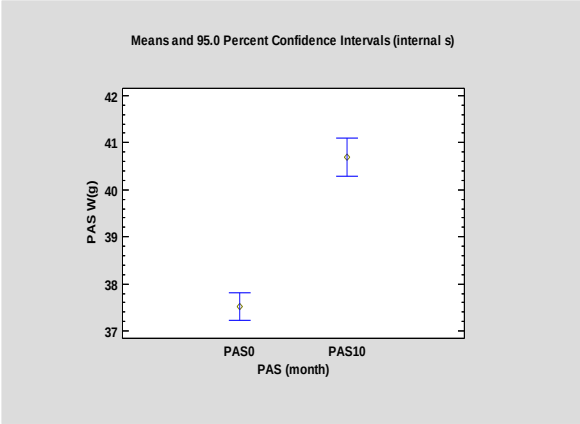
- Gravimetry



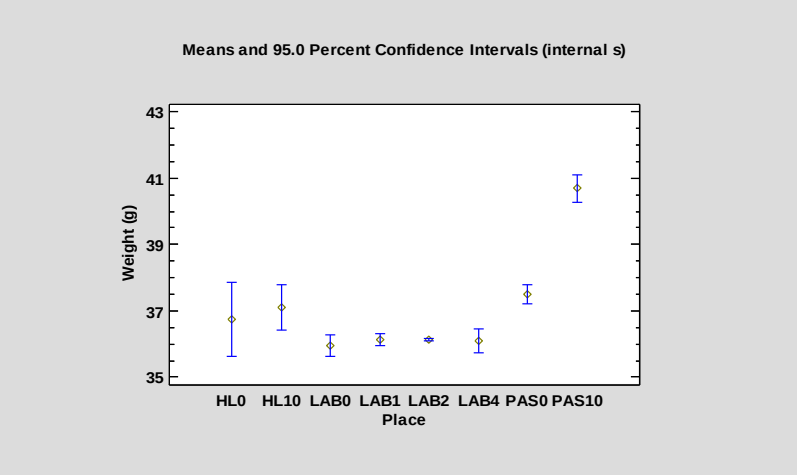
gravimetric measurements on samples taken every month from the accelerated ageing test



gravimetric measurements done before and after ageing in the HarshLab facility



gravimetric measurements done before and after ageing in the port of Pasaia



Strongly affected by the "remaining" of the fouling for the naturally aged samples

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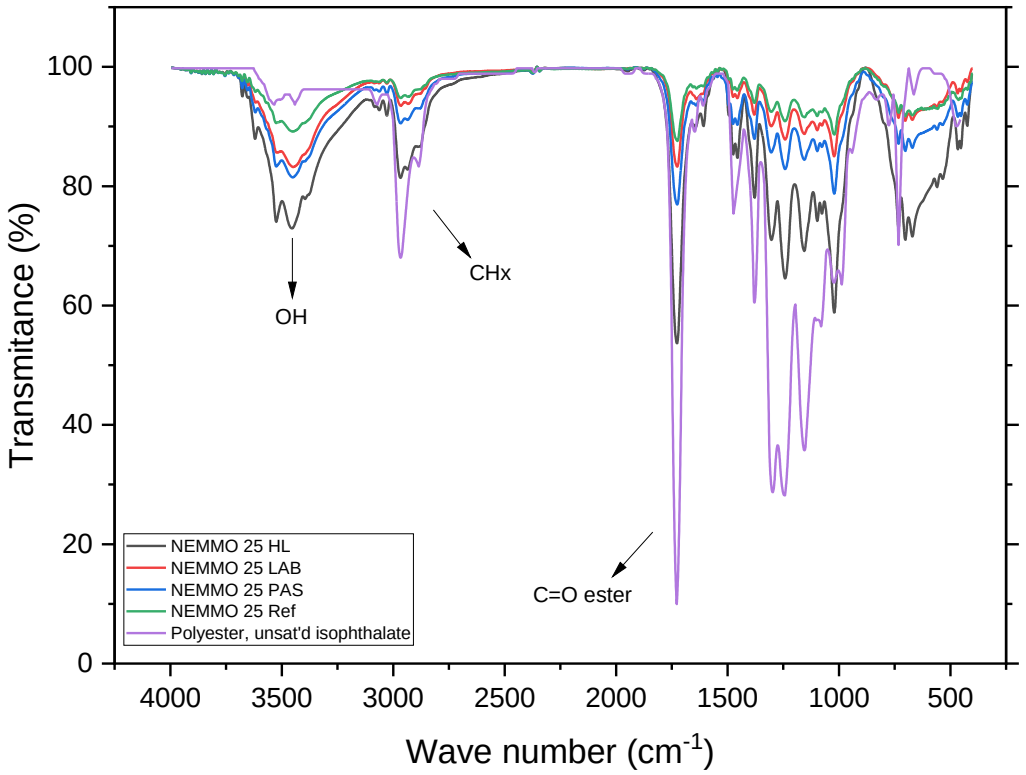
Results

- FTIR



No chemical degradation of the material (no effect of UV)

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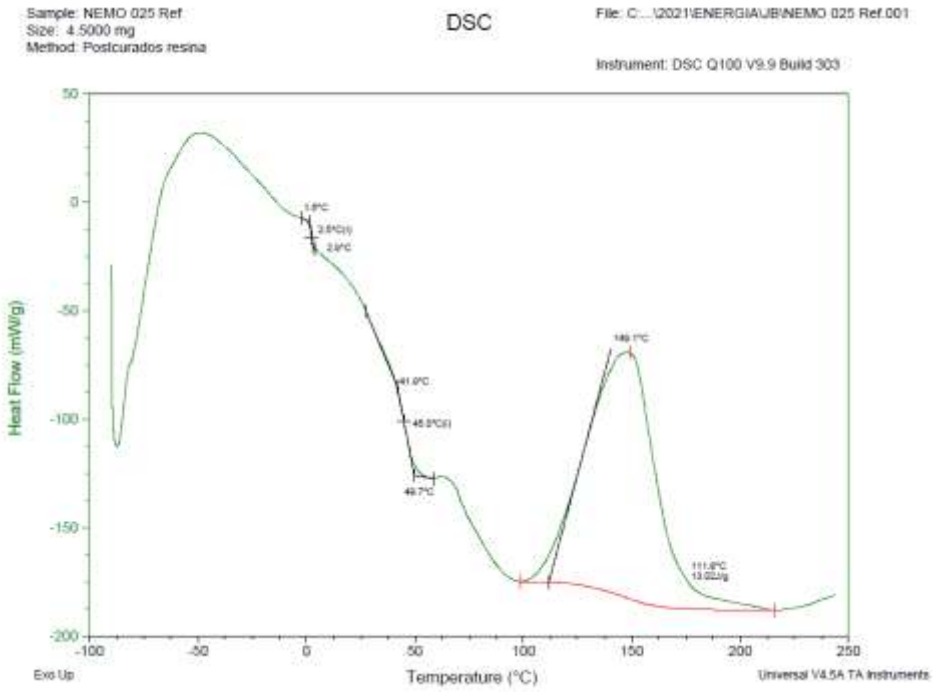
Results

- DSC



The DSC measurement did not allow to get exploitable spectra to extract Tg

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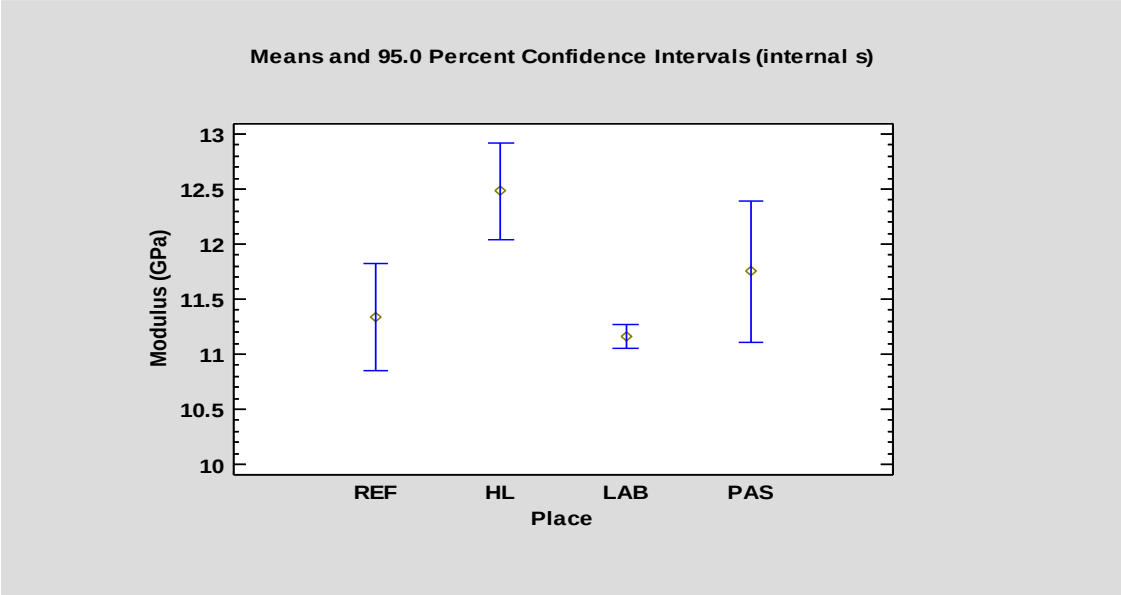
Results

- Tensile tests

Place	Count	Average	Standard deviation	Coeff. of variation	Minimum	Maximum
REF	5	11.34	0.391152	3.44931%	10.9	11.8
HL	5	12.48	0.349285	2.79876%	12.1	12.9
LAB	5	11.16	0.0894427	0.801458%	11.1	11.3
PAS	4	11.75	0.404145	3.43953%	11.4	12.3
Total	19	11.6789	0.61335	5.25176%	10.9	12.9



No significant variation after between the samples after the 3 ageings

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Summary of the results

The characterization tests shows:

- The 3 ageing did not affect the IR and DSC spectra

→ No chemical changes → Low effect of UV

- The 3 ageing did not lead in significant difference in mechanical properties (no plasticization due to water uptake)
- Gravimetry show a slight water uptake in the artificial ageing. In Harshlab and Pasaia, there is a clear increase of weight, but it is due to both water uptake and fouling remaining (hard to clean) → no conclusion can be taken for water uptake

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Conclusion

- The material chosen by Magallanes is well suitable for the environment (immersion in sea water)
- The UV have a very poor effect so it can be removed from the artificial ageing and substituted by more immersion time
- The new artificial ageing procedure proposed is:

	Day 1	***Day 2***	***Day 3***	***Day 4***	***Day 5***	***Day 6***	***Day 7***
							
Aging media	Immersion seawater 24h	Immersion seawater 24h	Immersion seawater 24h	Immersion seawater 24h	Immersion seawater 24h	Immersion seawater 24h	Immersion seawater 24h
Temp.	T=30 °C	T=30 °C	T=30 °C	T=30 °C	T=30 °C	T=30 °C	T=-2 °C

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Ongoing…

- Ageing of the reference and enhance material in natural environment

- Accelerated artificial ageing with the new procedure on the reference and enhanced materials

	Day 1	***Day 2***	***Day 3***	***Day 4***	***Day 5***	***Day 6***	***Day 7***
							
Aging media	Immersion seawater 24h	Immersion seawater 24h	Immersion seawater 24h	Immersion seawater 24h	Immersion seawater 24h	Immersion seawater 24h	Immersion seawater 24h
Temp.	T=30 °C	T=30 °C	T=30 °C	T=30 °C	T=30 °C	T=30 °C	T=-2 °C

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Thank you for your attention!

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