

# Impacts of digestion protocols on chemical and natural fibers

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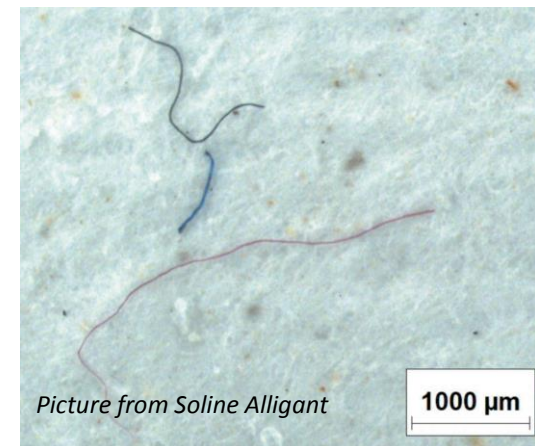
# Introduction

**Digestion protocols** are used to **dissolve organic matter** in environmental samples. In recent works, we observe three important points:

- 1. Fibers** correspond to **the greater fraction** of microplastics in water samples.

	Rain Waters	Washing Machine Effluents	Surface Waters
Fibers (Item/Liter)	20 - 40	9,000 - 35,000	0.03 - 0.05
Fragments (Item/Liter)	Not observed	< 2	0.001 - 0.003

*Table from ASTEE Presentation, Tassin et al., 2018*



- 2. Quality Assurance/Quality Control (QA/QC)** for digestion protocols **are more and more developed for fragments but not for fibers.**

- 3. Impacts of digestion protocols on fibers?**

# Which fibers?

## The most commonly used fibers (Raw fibers)

### Natural Fibers

#### Vegetable



Cotton



Flax

#### Animal

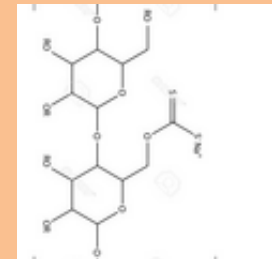


Wool

### Chemical Fibers

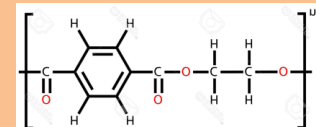
#### Artificial

##### Viscose

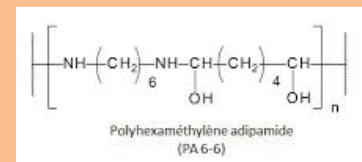


#### Synthetic

##### PET



##### PA 6.6



##### Acrylic

# The selected digestion protocols

Four different digestion protocols have been selected:

- KOH 10%, 40°C, 24h (Dehaut et al., 2016)
- KOH 10%, 60°C, 24h (Karami et al., 2017)
- H<sub>2</sub>O<sub>2</sub> 30%, 50°C, 48h (Stolte et al., 2015)
- NaClO 9%, ambient temperature, one night (Collard et al., 2015)



**After digestion**  
→  
**and filtration**



# Methods

## Fibers Characterisation before and after digestion

### ① Morphological aspects

- Mass variation
- Microscope observation:
  - Optical
  - Scanning Electron Microscope

### ② Mechanical Properties

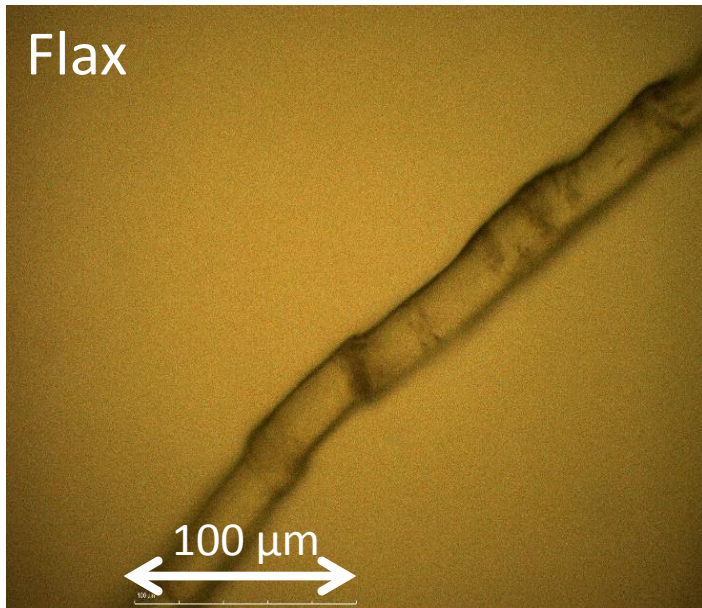
- Linear mass density
- Tenacity = Breaking strain
- Elongation at break

### ③ Thermal and Chemical Properties

- Differential Scanning Calorimetry (DSC)
- Infrared Spectroscopy (FTIR)



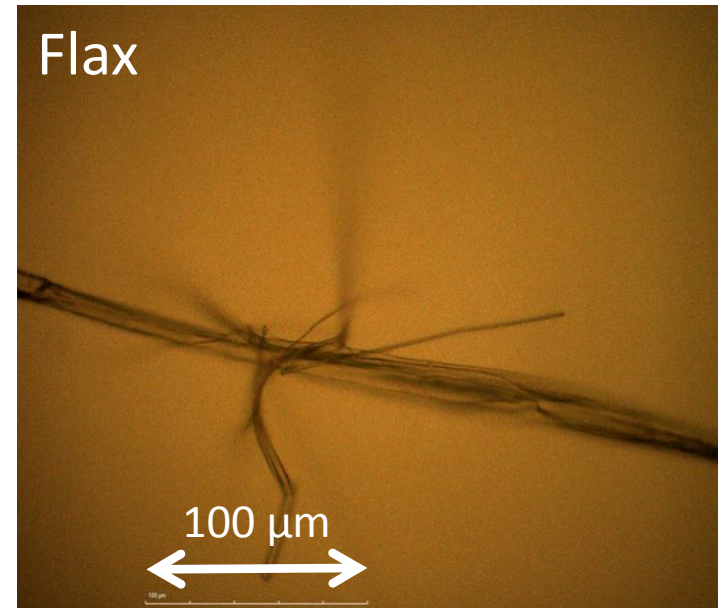
# Results: Morphological study



After digestion

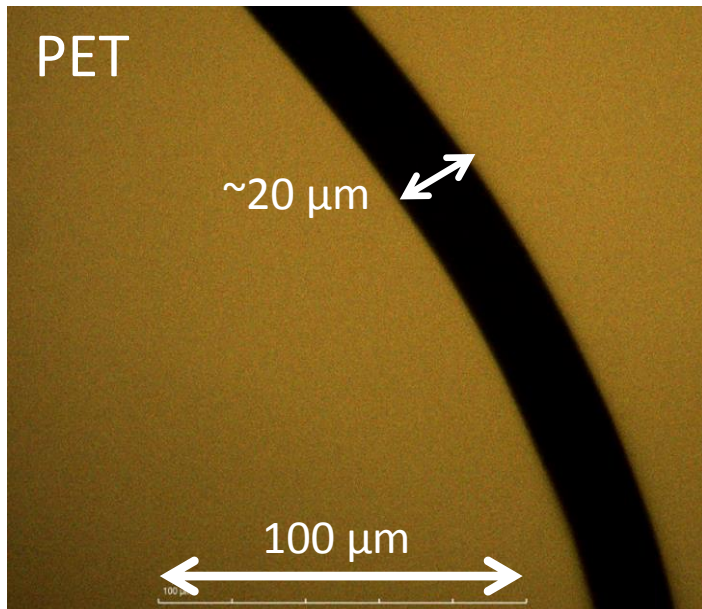


**NaClO 9%,**  
Ambient  
temperature



Flax, Microscope x 40, before and after NaClO digestion

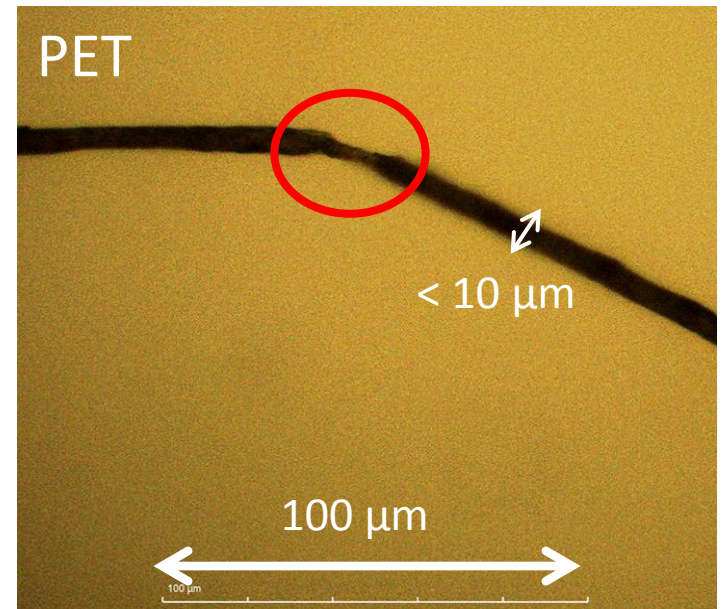
# Results: Morphological study



After digestion

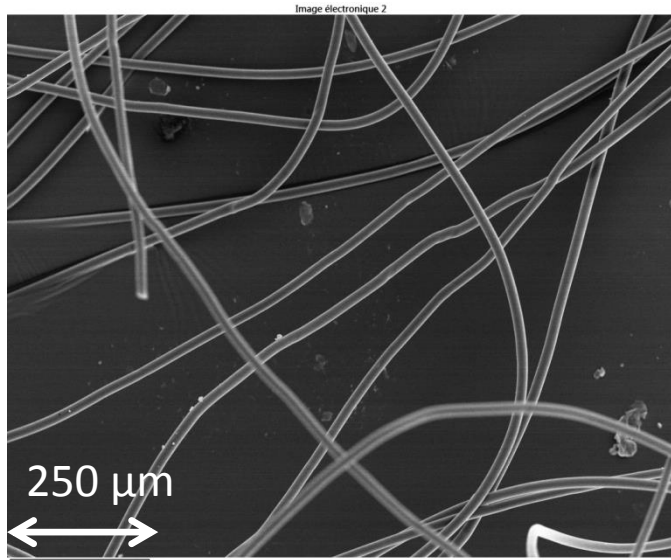


**KOH 10% 60°C**  
**24 hours**



PET, Microscope x 40, before and after KOH 60°C digestion

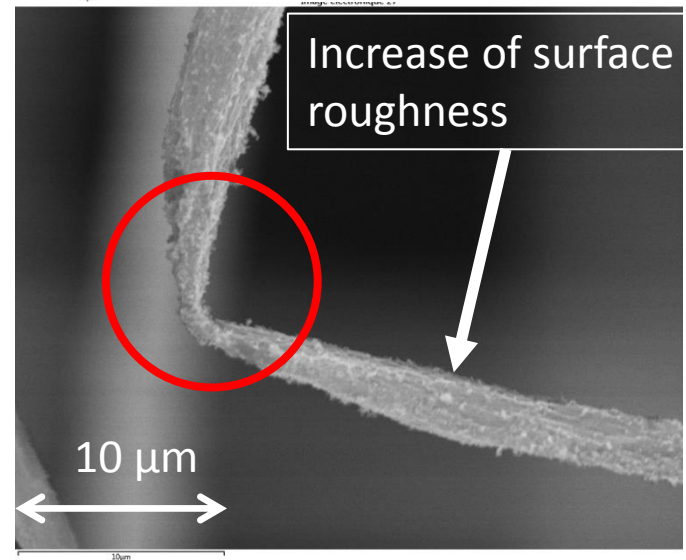
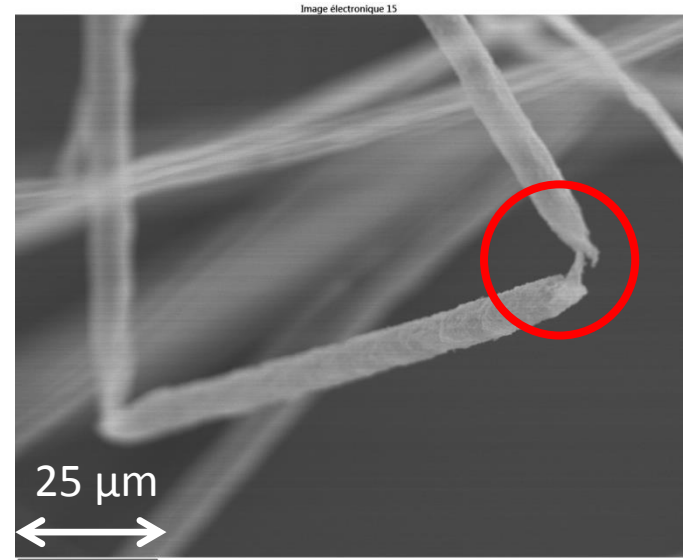
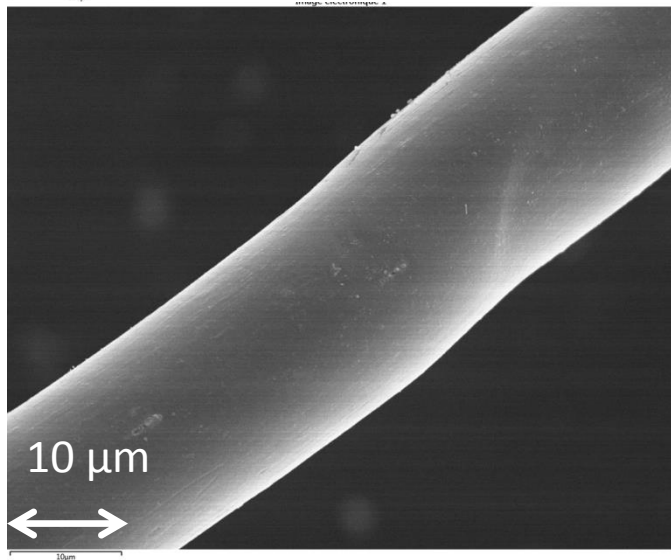
# Results: Scanning Electron Microscope on PET



After digestion

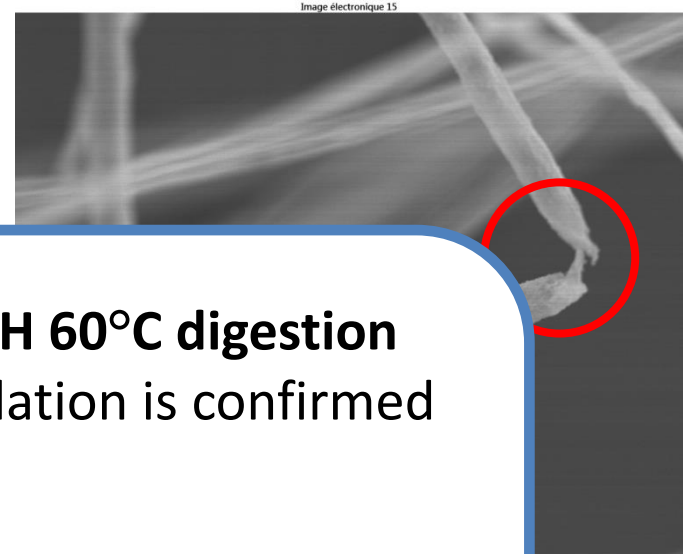
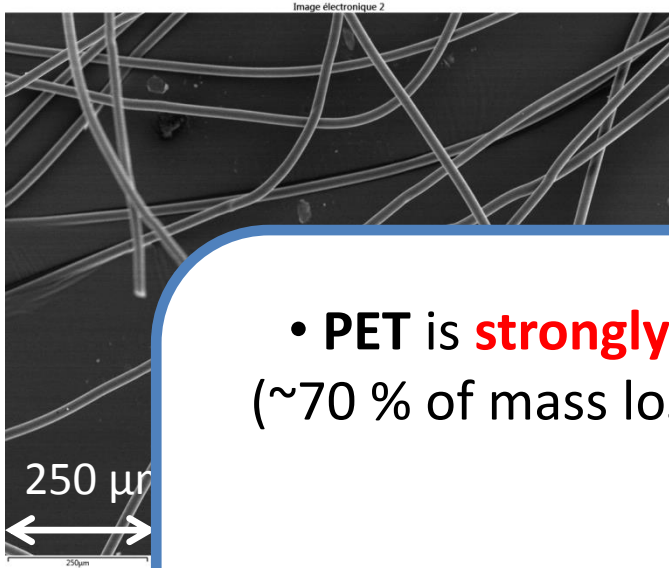


**KOH 10% 60°C**  
**24 hours**





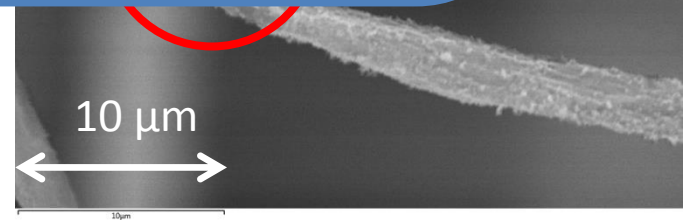
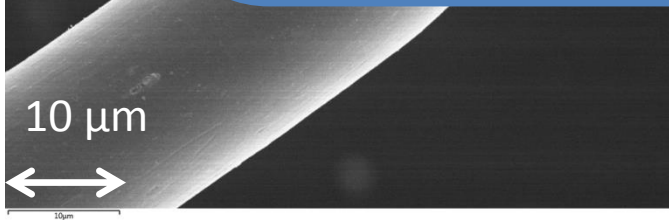
# Results: Scanning Electron Microscope on PET



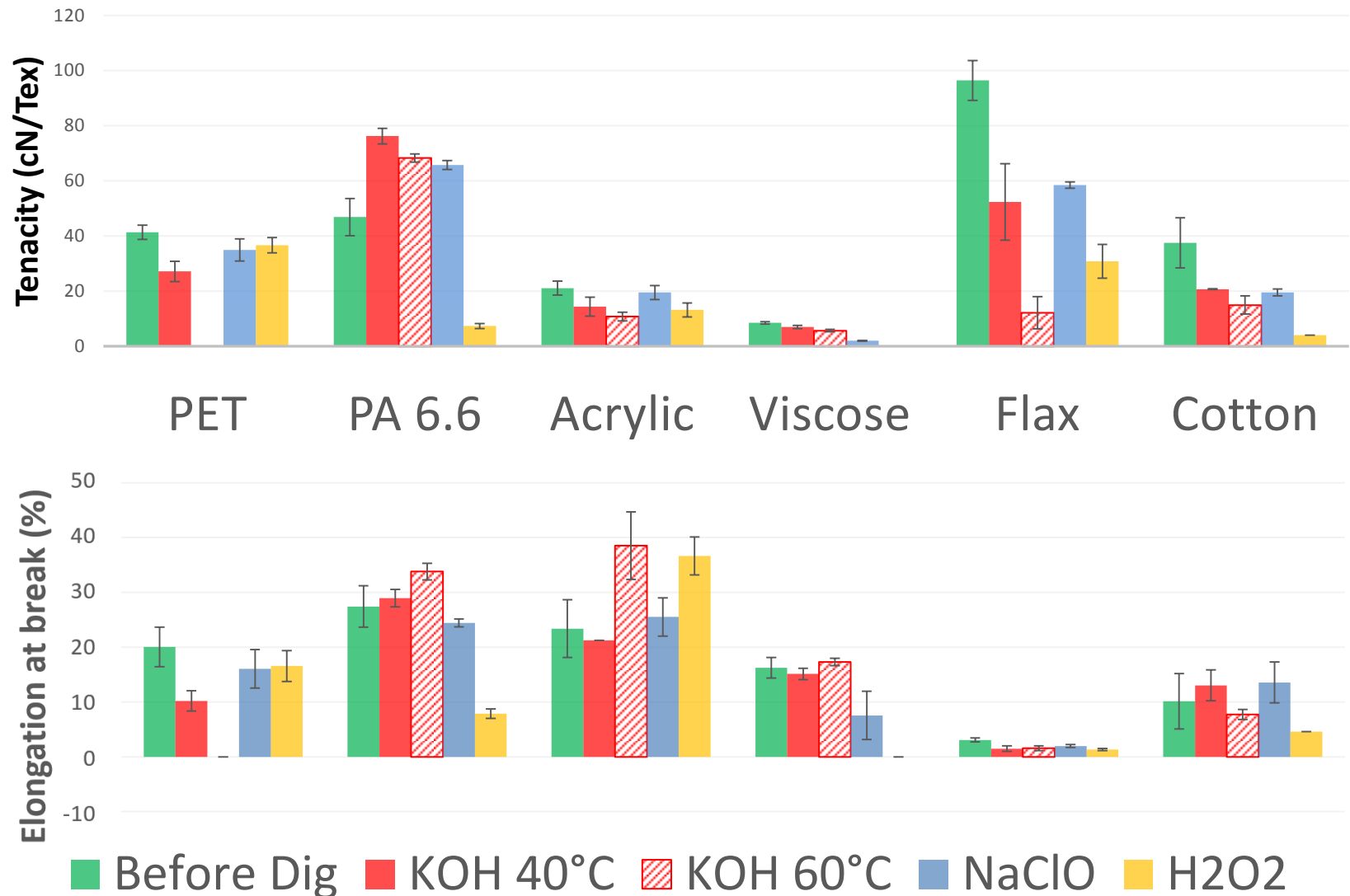
- PET is **strongly** affected by **KOH 60°C digestion** (~70 % of mass loss), high degradation is confirmed with SEM.

- Flax hull is shattered by **NaClO digestion**. SEM analysis will complete this observation.

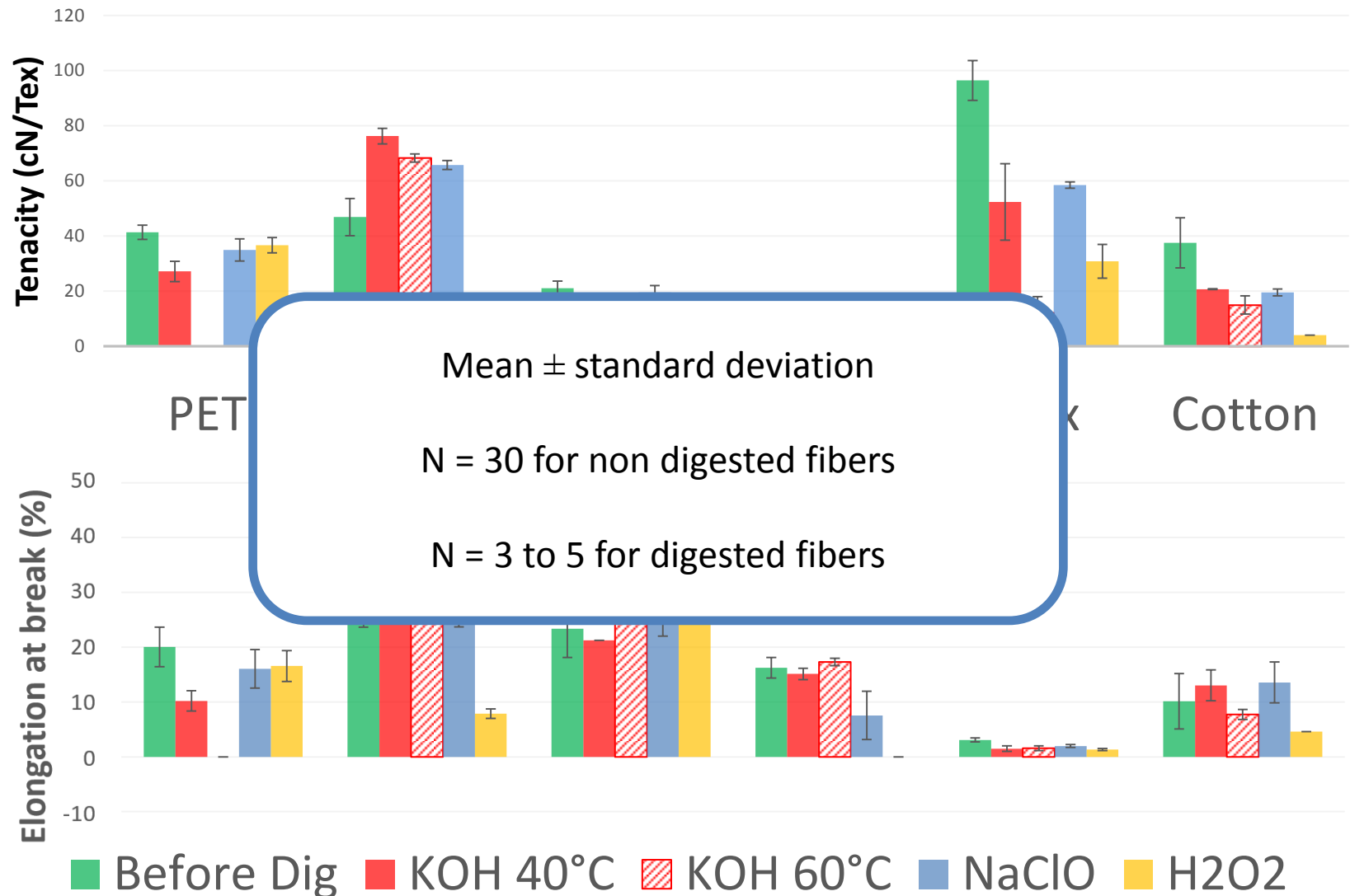
- Wool is **totally** dissolved, except for  $H_2O_2$ .



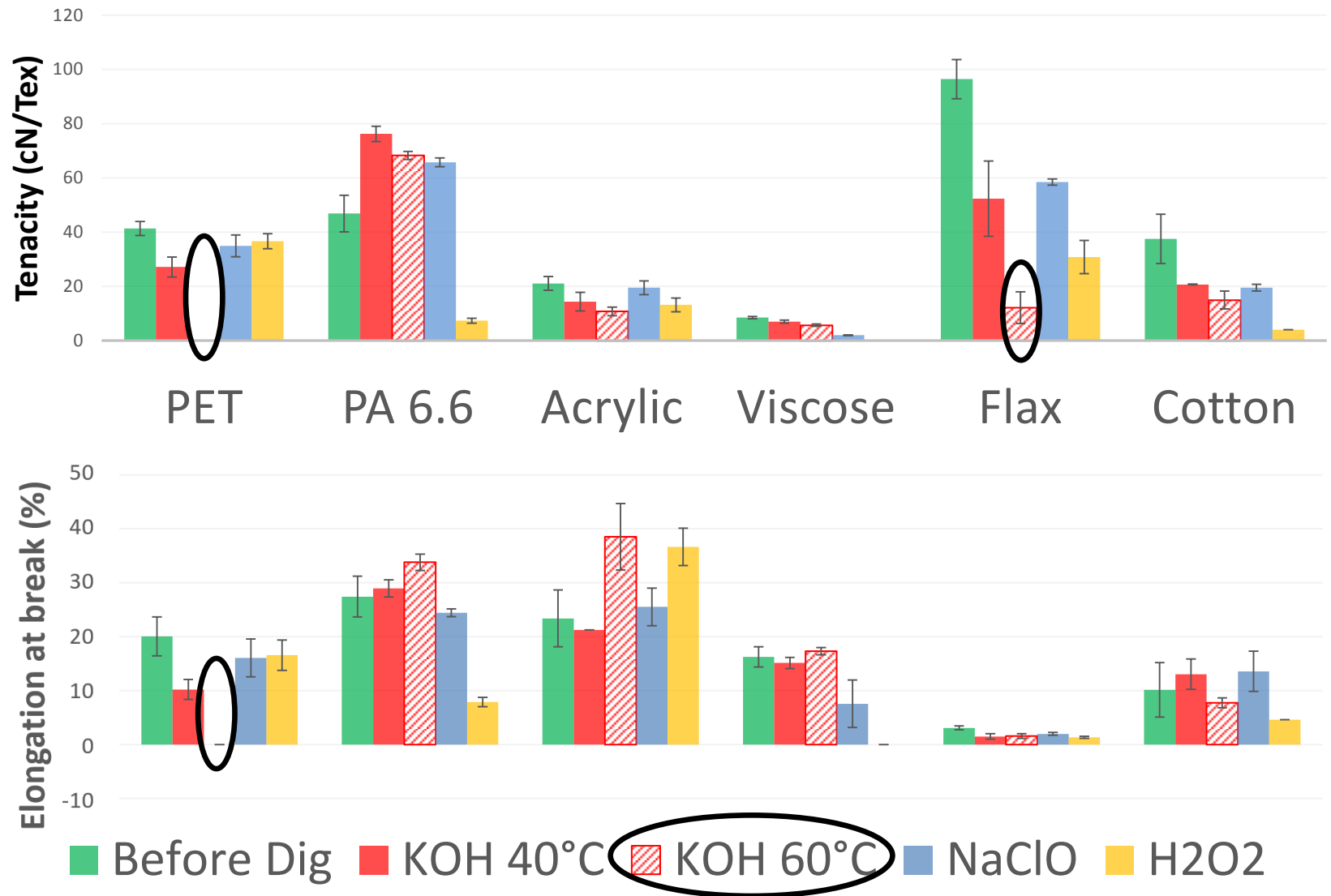
# Results: Mechanical Properties



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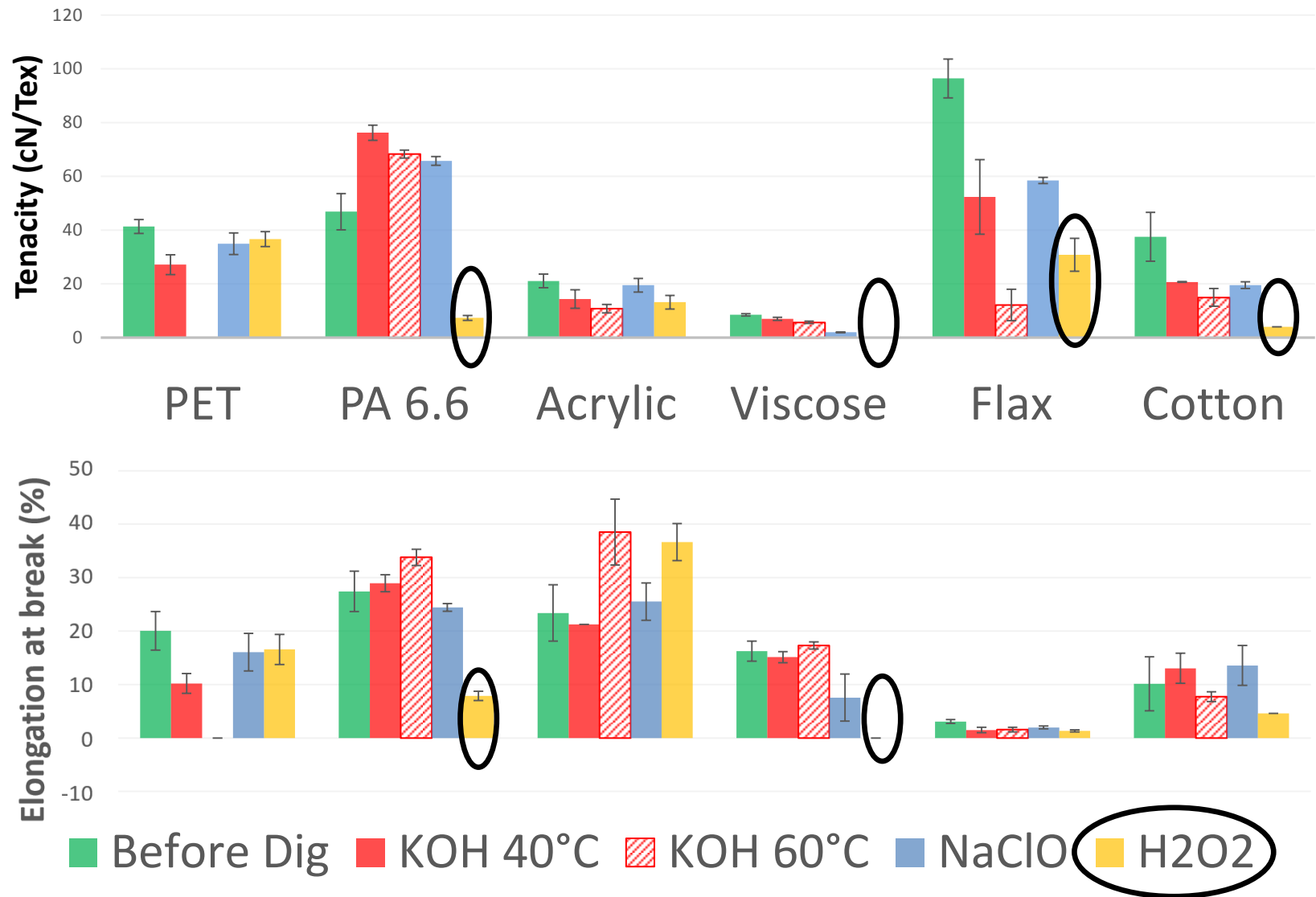


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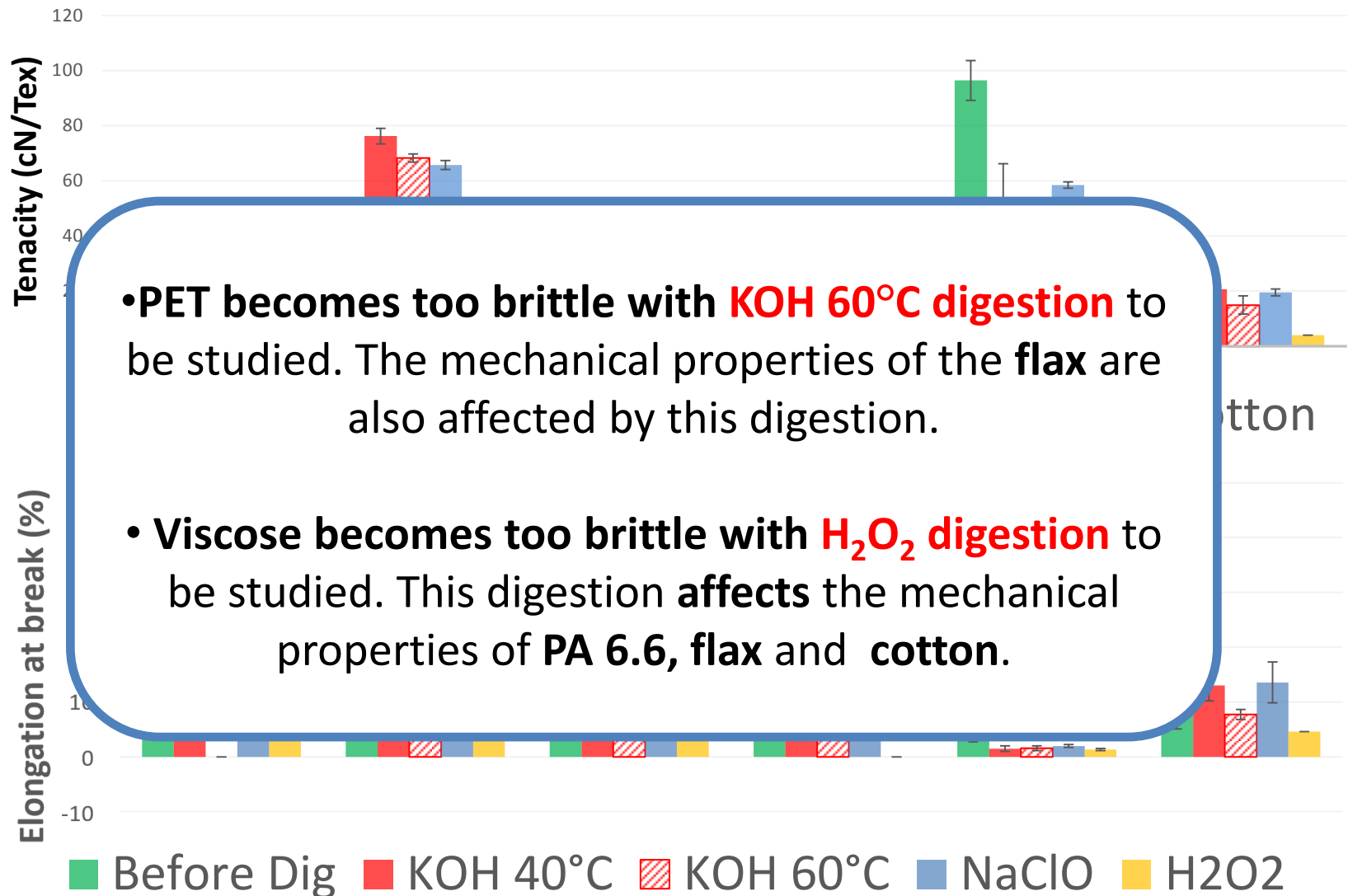




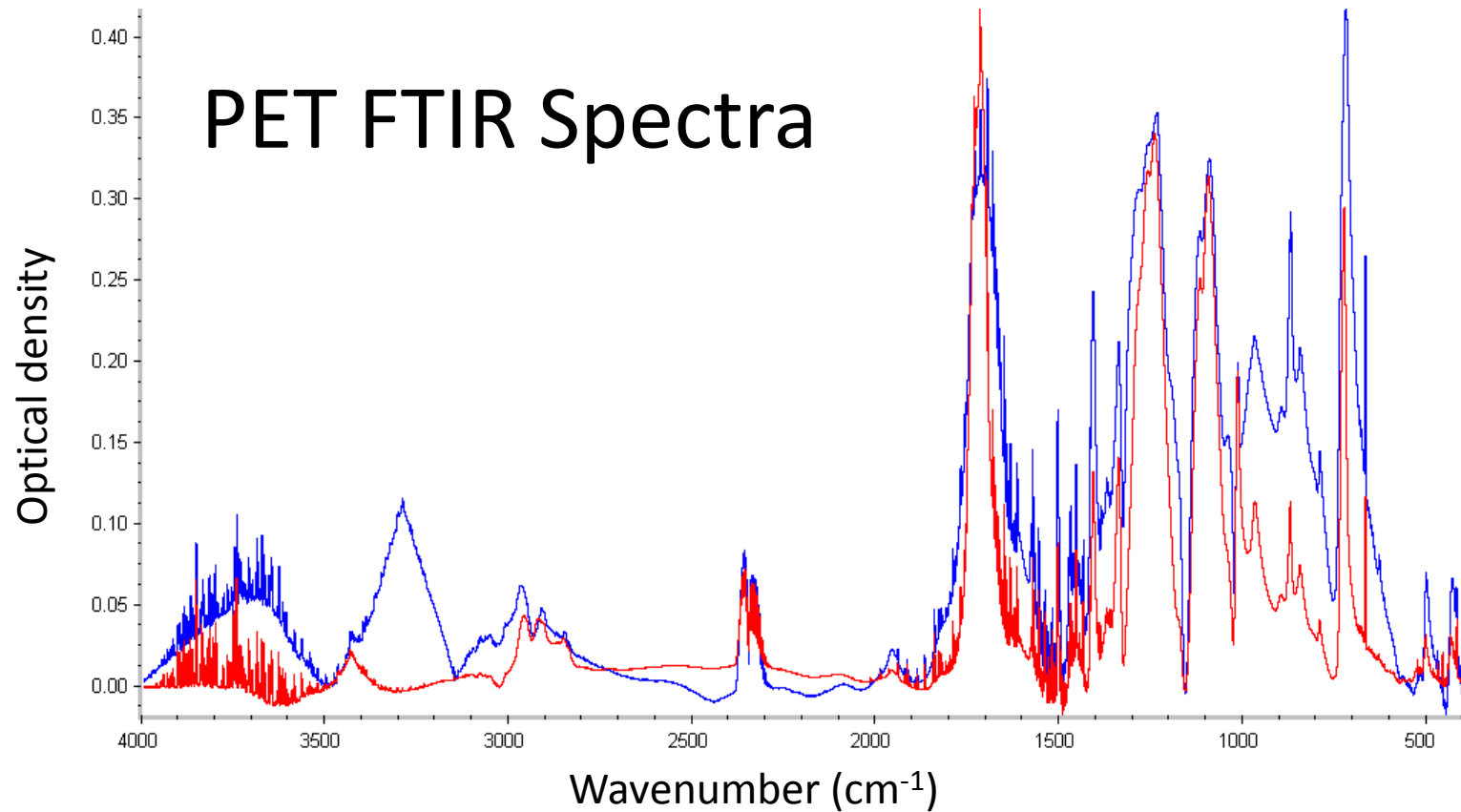
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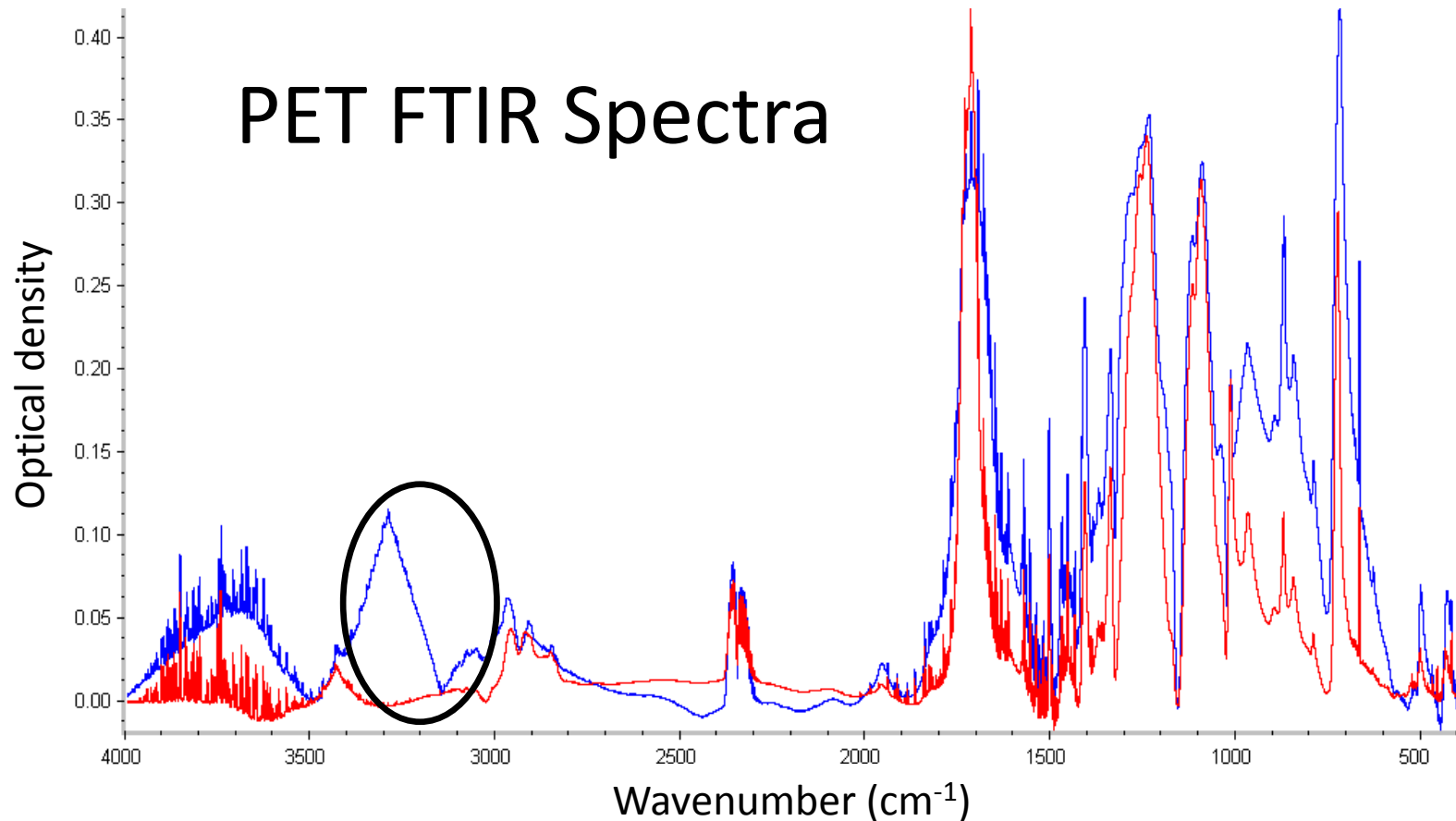
# Results: Thermal and Chemical Properties



Blue curve : before KOH 10 % 60°C digestion

Red curve : after KOH 10% 60°C digestion

# Results: Thermal and Chemical Properties



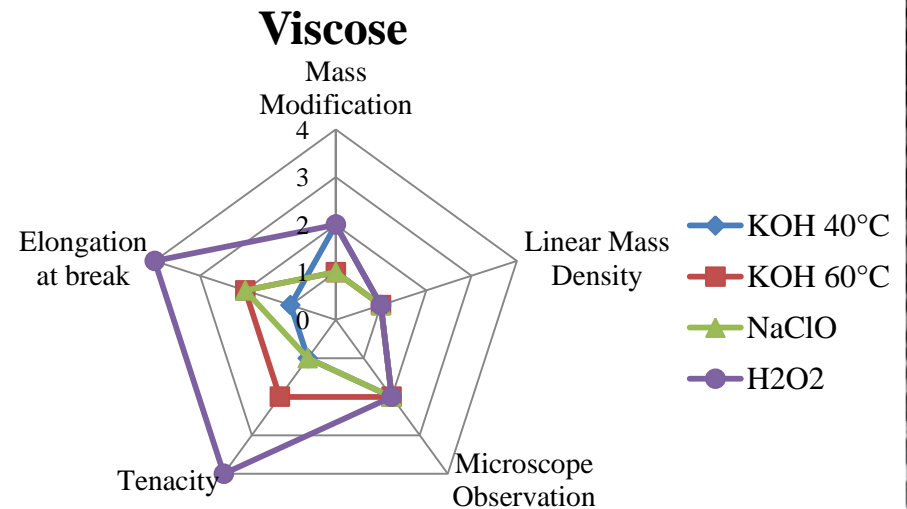
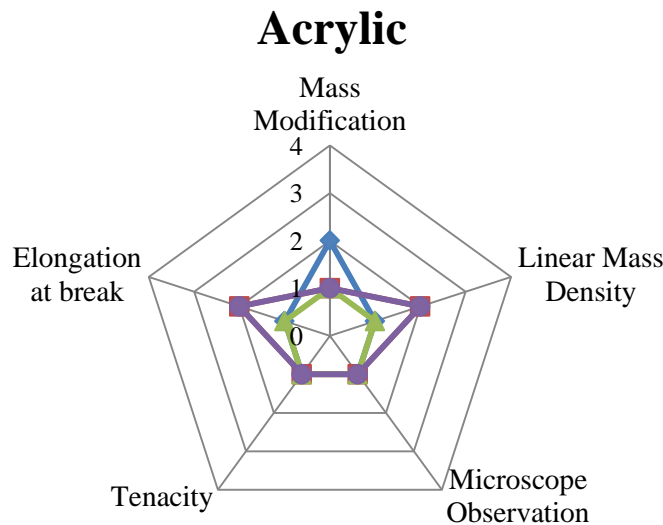
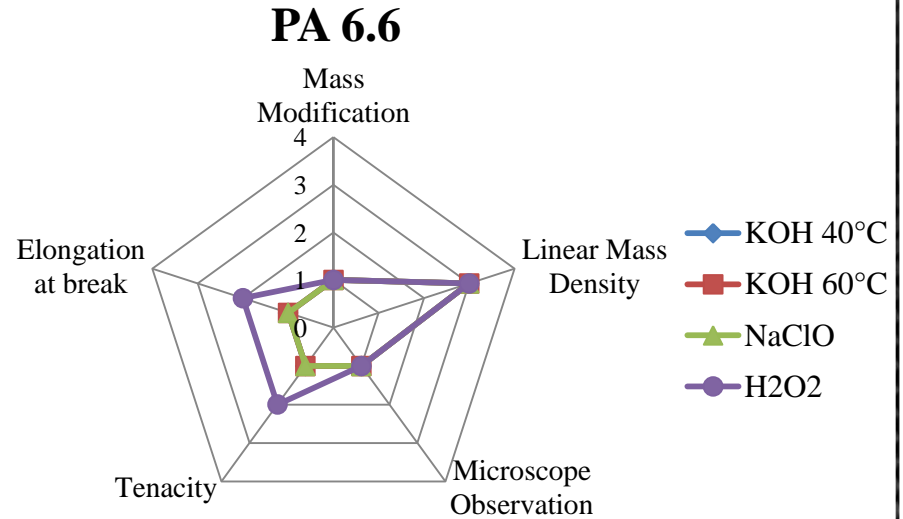
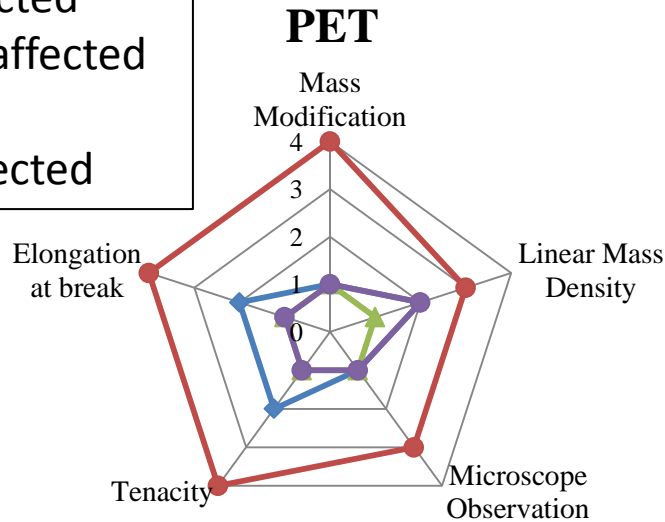
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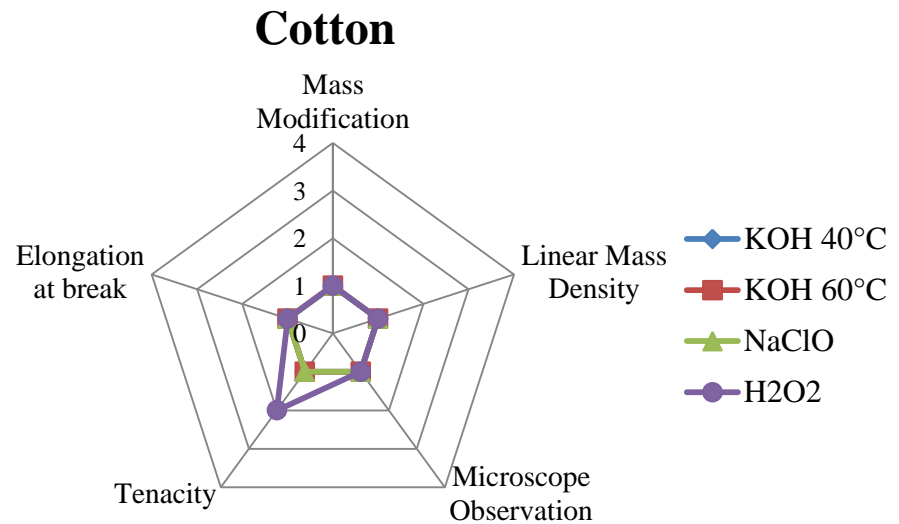
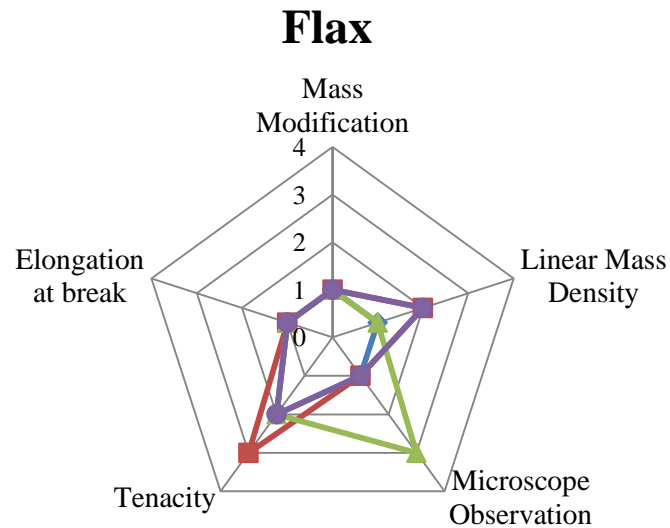


# Conclusion for chemical fibers

1 = Not affected  
2 = Slightly affected  
3 = Affected  
4 = Very affected



# Conclusion for natural fibers



Number of criteria > 3

	PET	PA 6.6	Acrylic	Viscose	Wool	Flax	Cotton
KOH 40°C	0	1	0	0	5	0	0
KOH 60°C	5	1	0	0	5	1	0
NaClO	0	1	0	0	5	1	0
H2O2	0	1	0	2	5	0	0

# Conclusion

## Natural Fibers

Wool → **Totally Dissolved,**  
except with **H<sub>2</sub>O<sub>2</sub>**

Flax → Ok except with  
**NaClO**

## Chemical Fibers

PET → **Strongly affected by**  
**KOH 10% 24h 60°C**

Viscose → Affected by **H<sub>2</sub>O<sub>2</sub>**  
but **30% 48h 50°C**

PA → Slightly Affected by  
all digestion  
protocols

# Conclusion

**KOH 40°C digestion** seems more relevant for 6 types of fibers.

**T > 60°C → Not recommended**

**Which Digestion ? → Which Fibers ?!**

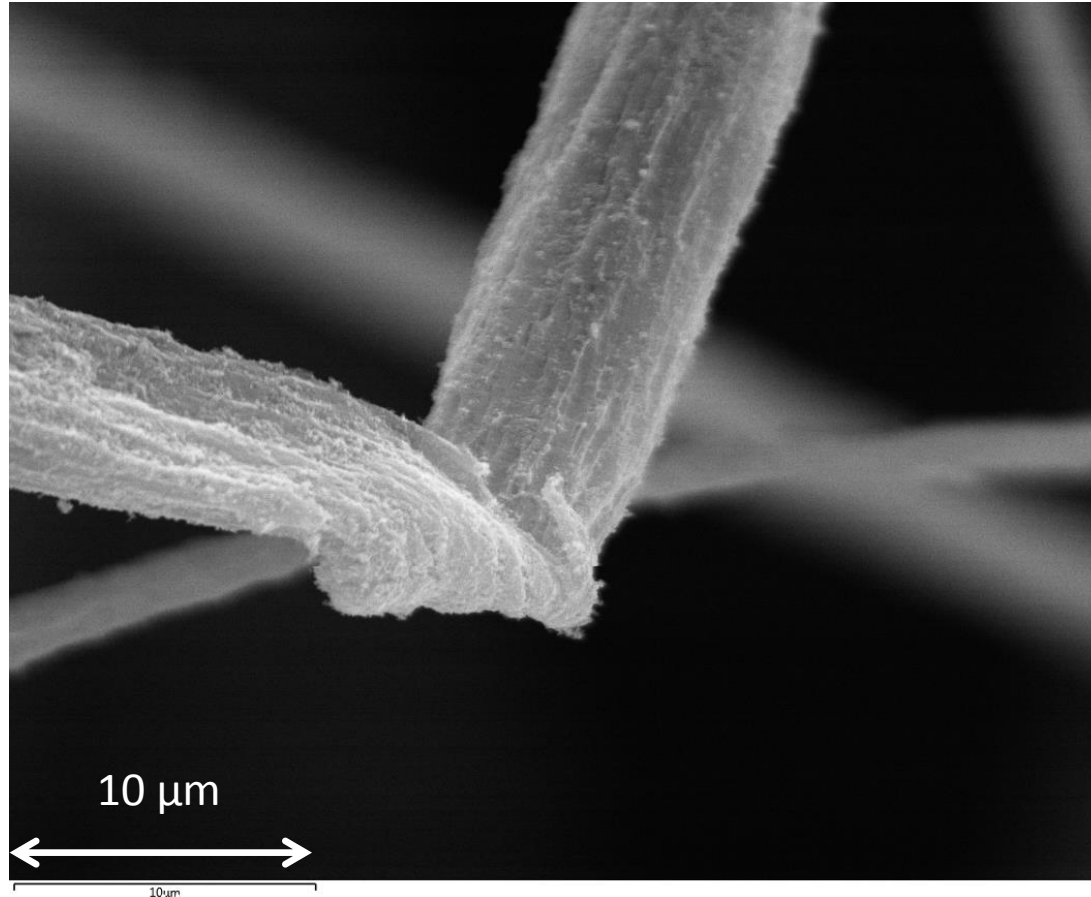
*Global Conclusion*

- More observations with the SEM
- **Fenton's reagent ?  
Enzymatic digestion ?**
- What will happen with **environmental fibers ?**

*Outlook ?*



# Thank you for your attention!



## Questions ?