

Wet spinning of cellulose derivatives: biobased, transparent and high-strength fibers

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Context & Objectives

E.U. goals by 2025

- Make plastics circular
- Drive plastics life cycle to net-zero
- Foster the sustainable use of plastics

Need to develop new bio-based and biodegradable materials:
→ from renewable resources
→ with a controlled end-life

Fibers for bio-based and high performance composites

Fibers have a high potential as reinforced materials or reinforcing fillers for innovative composites

Why ?

- High surface area
- Woven/non-woven
- Porous/non-porous
- Release properties
- High mechanical strength
- Flexibility

However

Most fibers are produced by melt processing, a method incompatible with natural and bio-based materials, that usually do not melt

Alternative processing methods compatible with large-scale production, and with a limited environmental impact, are missing

High strength cellulose-based fibers prepared by wet spinning

Objectives

- Use of a cellulose derivative as bio-based and biodegradable alternative material for fibers
- Rapid, scalable and simple processing method of fibers
- High-strength and transparent fibers

Experiments

Materials

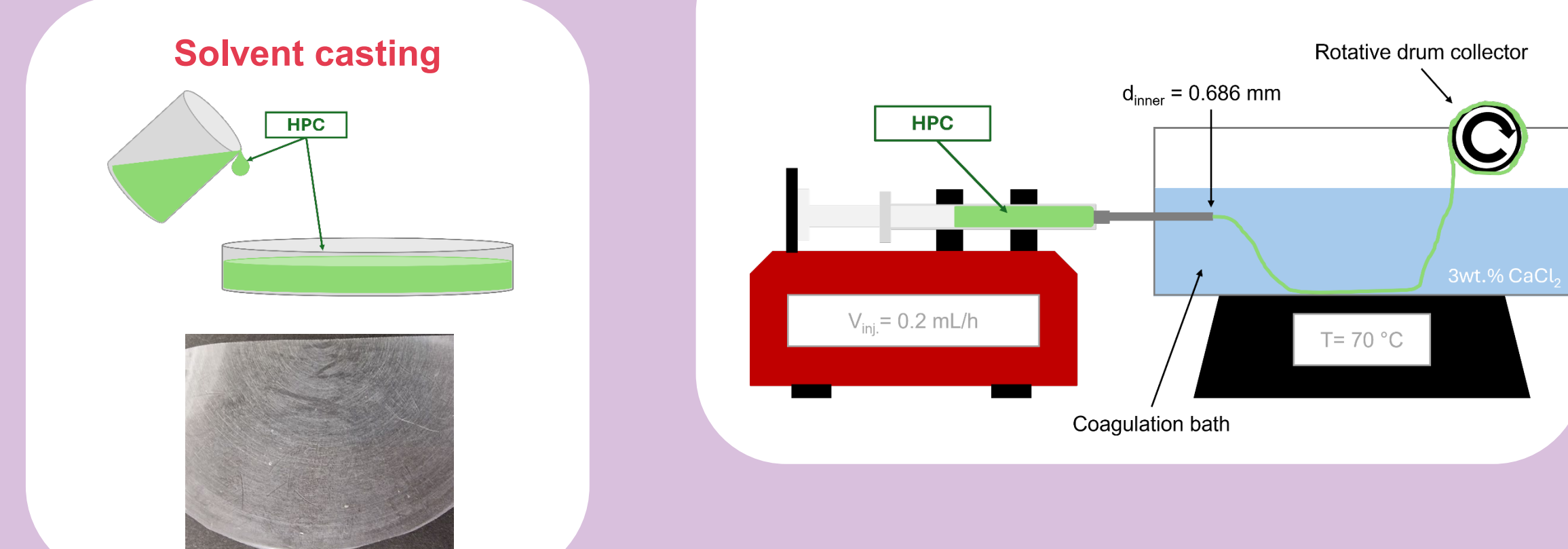
Hydroxypropyl cellulose (HPC) (Mw~10x10⁴, Sigma-Aldrich), calcium chloride (Sigma-Aldrich)

HPC preparation of

Bulk films → Solvent casting
Fibers → Wet spinning

Wet spinning

Step 1: Fiber spinning



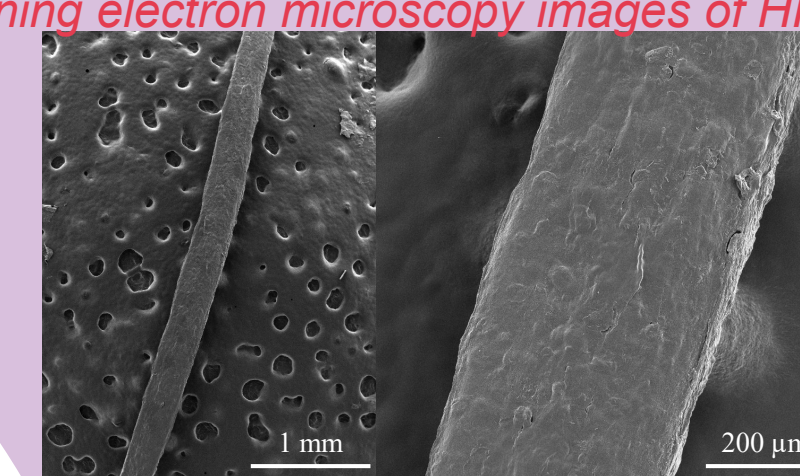
Step 2: Washing
Soak in distilled water

Step 3 : Fiber drying
24h at 50° C

Step 3 : Fiber shaping

Dried fibers on the collector → 2h under ambient conditions to be straightened

Scanning electron microscopy images of HPC fibers



Homogeneous fibers with diameters of 300-400 μm

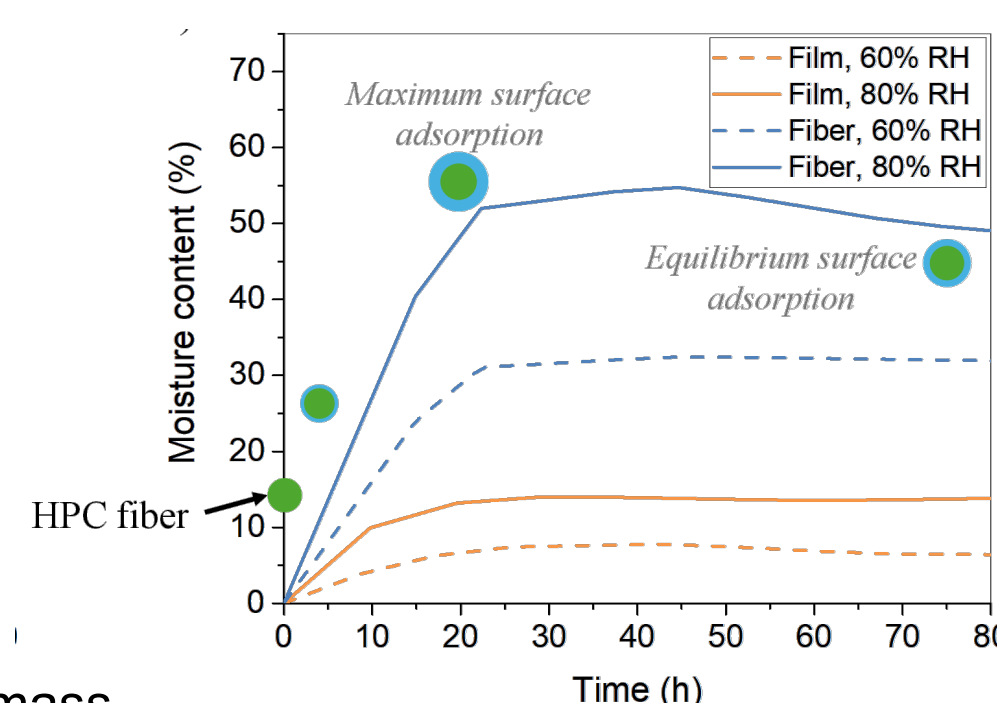
Characterization

Water mass uptake (%)

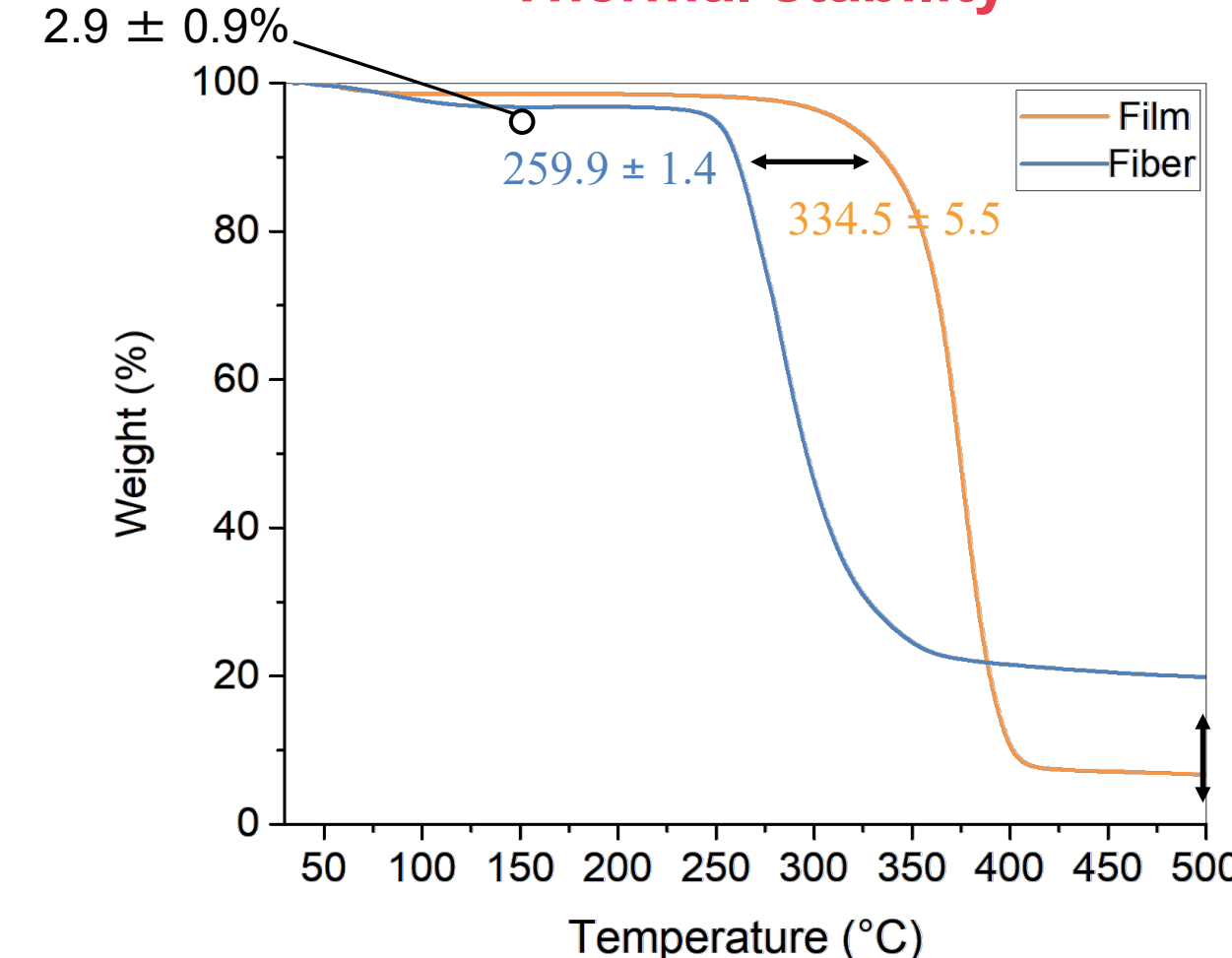
Mass uptake (%)	60% RH	80% RH
HPC fibers	29.6 ± 2.4	38.9 ± 10
HPC films	6.2 ± 1.6	13.1 ± 0.7

Water sorption increases with RH
HPC wet spun fibers have higher water mass uptake than films

Higher surface area of fibers induces faster sorption and increases the number of free sorption sites



Thermal stability

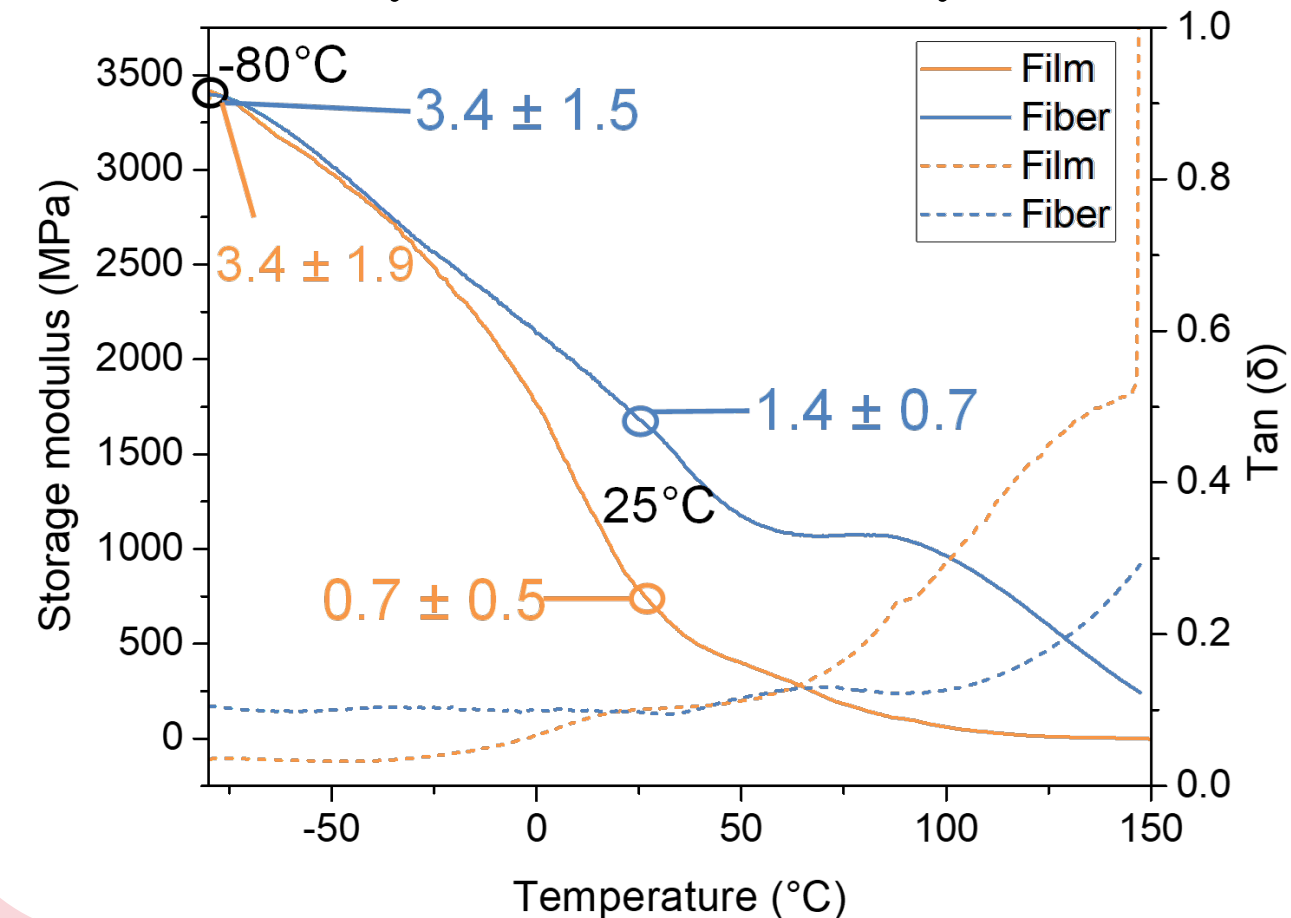


HPC wet spun fibers have a lower degradation temperature than bulk films (difference of ~75° C)

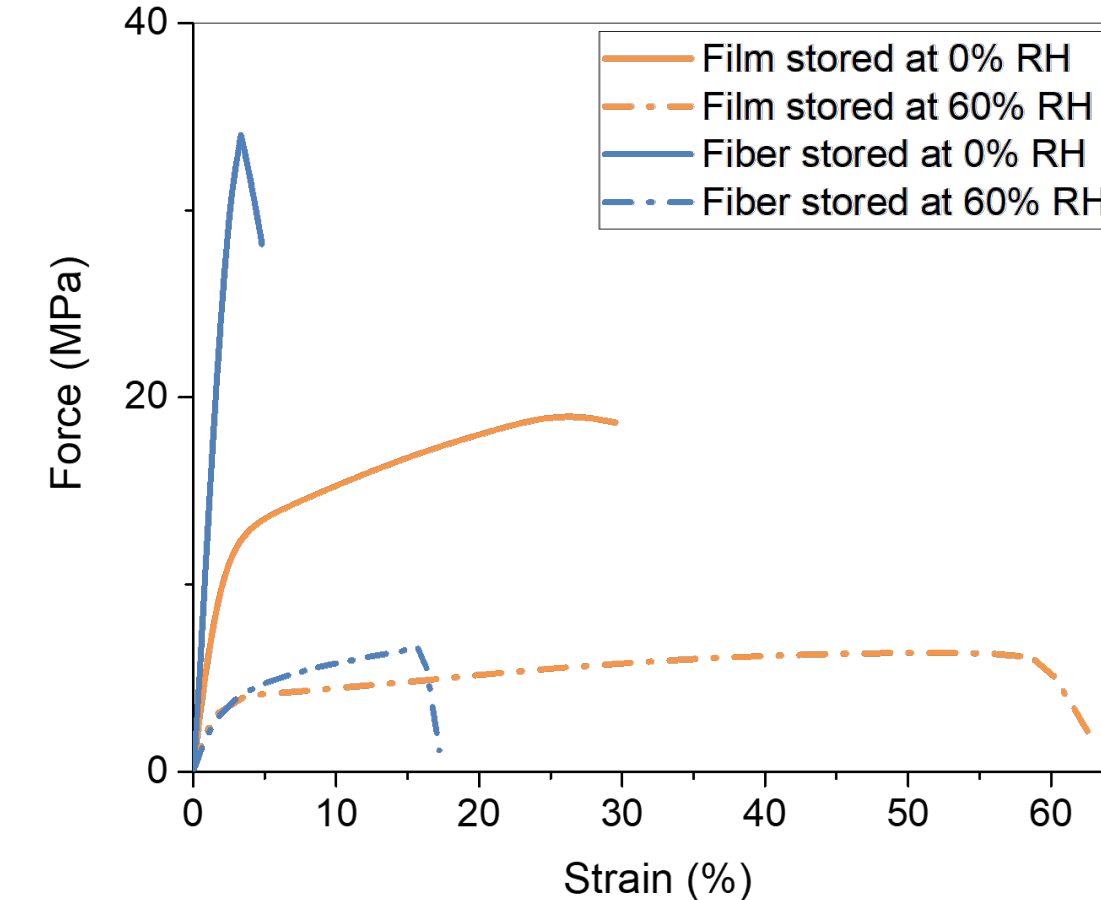
Effect of coagulation bath and/or presence of porosities¹

CaCl_2

Dynamic mechanical analysis



Tensile test



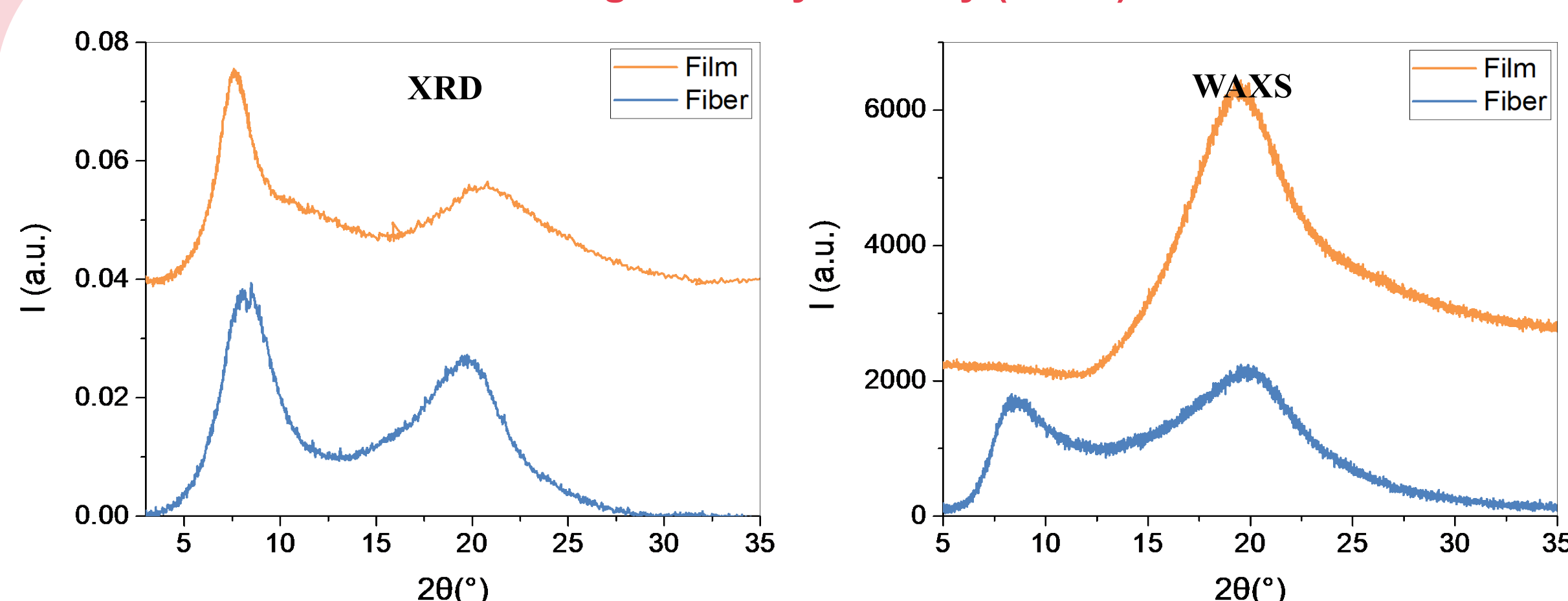
Mechanical tests

Storage	0% RH	60%RH
Young modulus (GPa)		
HPC fibers	1346 ± 148	258 ± 76
HPC films	657 ± 44	272 ± 20
Tensile strength (MPa)		
HPC fibers	35.7 ± 6.0	8.0 ± 1.4
HPC films	19.1 ± 1.1	4.2 ± 0.6
Strain (%)		
HPC fibers	3.5 ± 0.7	18.4 ± 5.3
HPC films	26.8 ± 1.6	62.3 ± 13.5

Water plasticization

Wet spun fibers present higher stiffness and strength due to more ordered structures^{3,4}
Shear force applied during wet spinning induces alignment of HPC chains

Degree of crystallinity (D.o.C)



Lack of resolution with XRD for fibers

D.o.C (%)	XRD	WAXS
HPC fibers	36.1 ± 3.6	44.3 ± 0.7
HPC films	42.7 ± 2.2	43.4 ± 0.9

Crystalline peaks of HPC: 2 θ ~8° and ~20°^{1,2}
Wet spinning does not affect crystallinity of HPC

Conclusion & Perspectives

Conclusion

- For the first time, wet spun HPC fibers were prepared
- Wet spinning enhances the alignment of HPC chains
- The degradation temperature of HPC fibers is compatible with most processing conditions and applications
- HPC fibers have higher mechanical resistance than films
- Despite a higher water sorption, fibers present better mechanical strength than film at 60% RH

Perspectives

- Reinforcement of fibers with the addition of fillers
- Investigation of the biodegradability of fibers in soil and water
- Investigation of release properties of fibers containing active compounds

1. Byun and Kang, *Carbohydrate Polymers*, 2023
2. El-Wakil et al., *Industrial Crops and Products*, 2016
3. Kim et al., *International Journal of Precision Engineering and Manufacturing-Green Technology*, 2019
4. Iwamoto et al., *Biomacromolecules*, 2011