



TEXAS TECH UNIVERSITY



Valorisation des fibres de coton de basse qualité: Applications non- traditionnelles

***Noureddine Abidi, Ph.D.
Professor and Director***

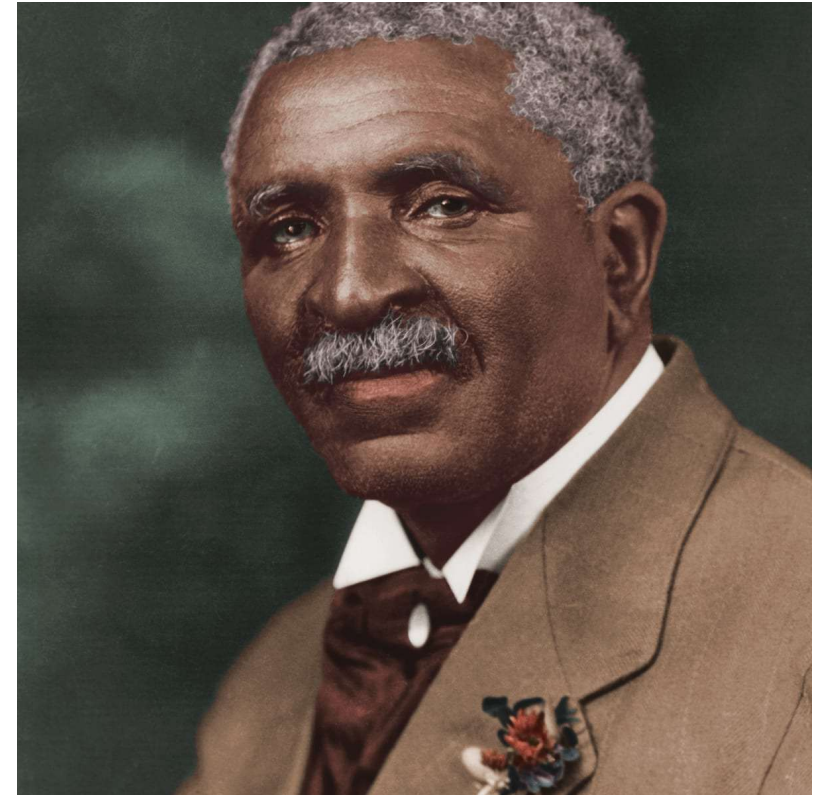


*Fiber and Biopolymer Research Institute
Texas Tech University, Lubbock, TX, USA*



Wastes

“Waste is man-made. Nature produces no wastes; whatever is consumed is returned to the whole in a reusable form. Man fails to utilize appropriately the bounty of nature”



*George Washington Carver,
Botanist and Inventor
(1864-1943)*



Plastic pollution



*Near the Caribbean island of Roatan, off the coast of Honduras)
Photo: Caroline Power, October 2017*

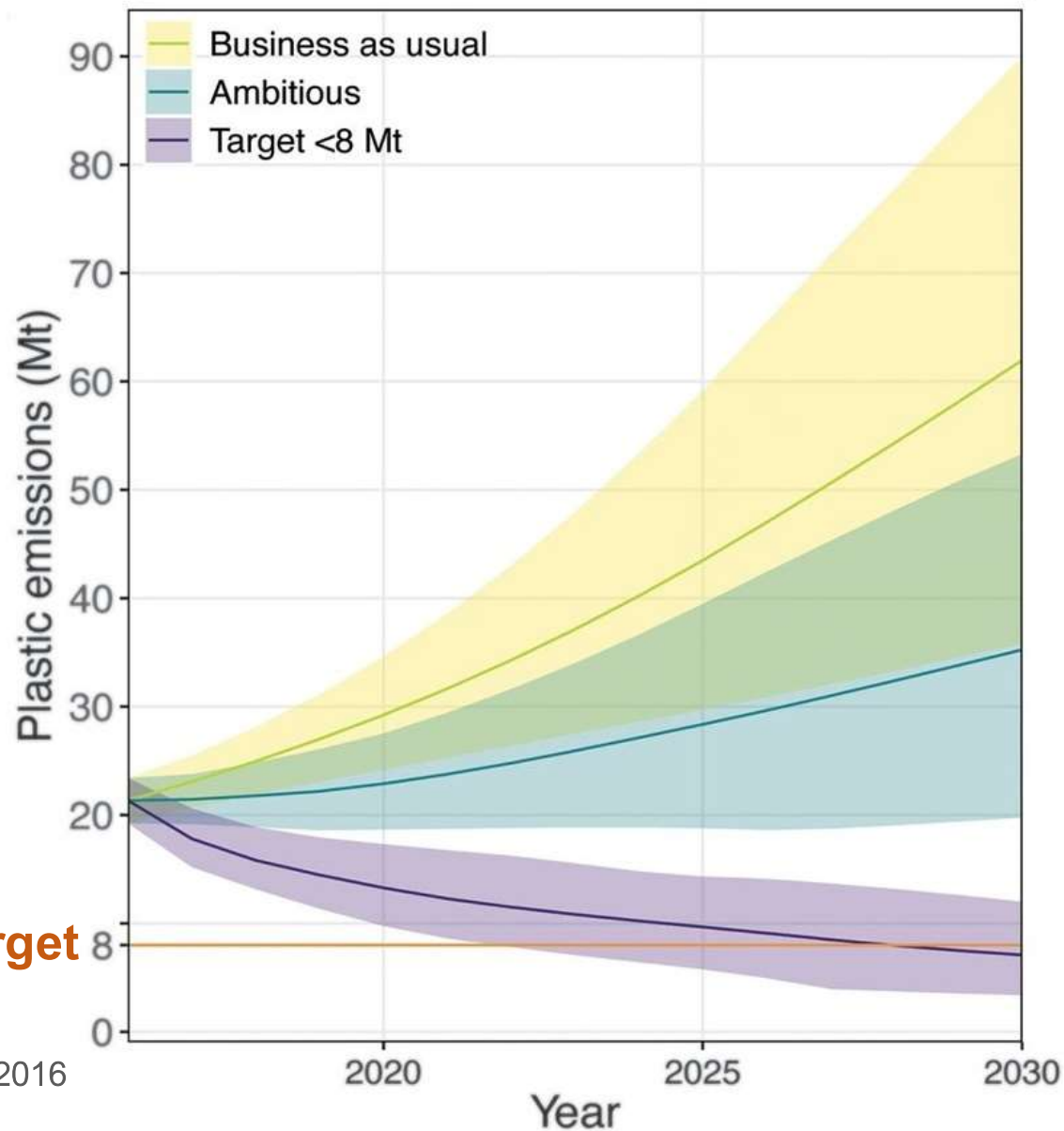


The Ocean will soon have more plastic than fish



Plastic pollution

Science
AAAS



Mt: Million metric tons

Target



Single use plastics



Single use plastics a necessary Evil during the Pandemic

GFP: Insight Drives Innovation, <https://www.gep.com/company>



It takes up to 450 years for a plastic bottle to decompose in landfills



<https://www.savemoneycutcarbon.com/>

6.3 billion tons of plastic since mass production began in the 1950s but only [600 million tons have been recycled.](#) The remaining 4.9 billion tons have been sent to landfills or left in the natural environment

Plastic is broken down by means of photodegradation: UV radiation from the sun breaks down the plastic into smaller pieces overtime.



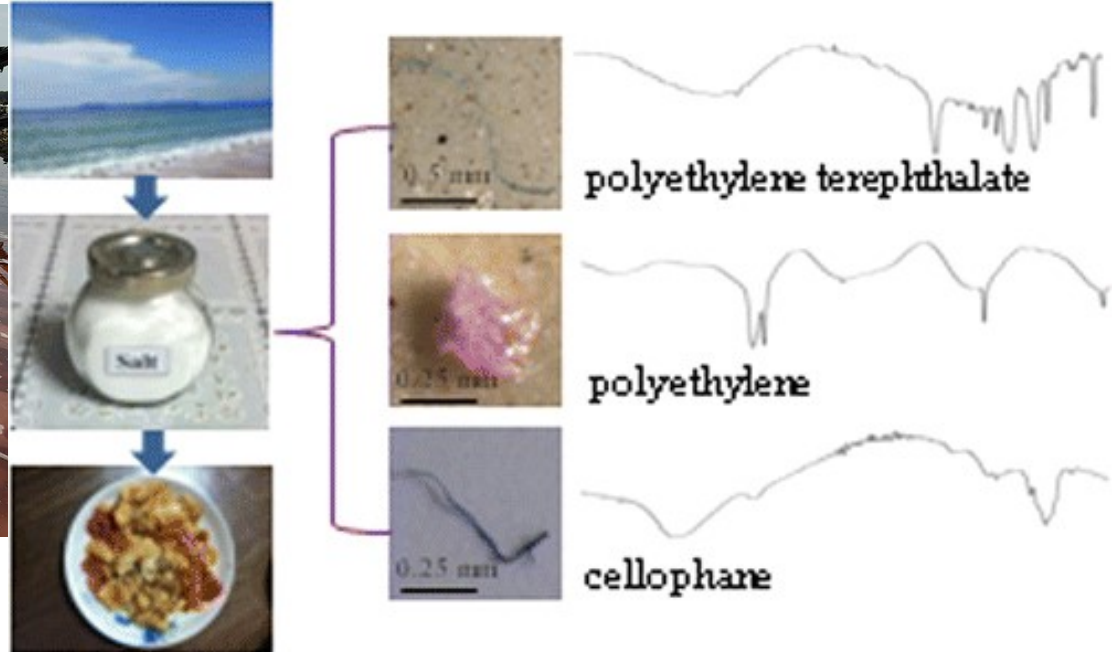
From plastics to microplastics



90% of table salt is contaminated with microplastics

EcoWatch, 2018.

<https://www.ecowatch.com/table-salt-microplastics-2613395969.html>

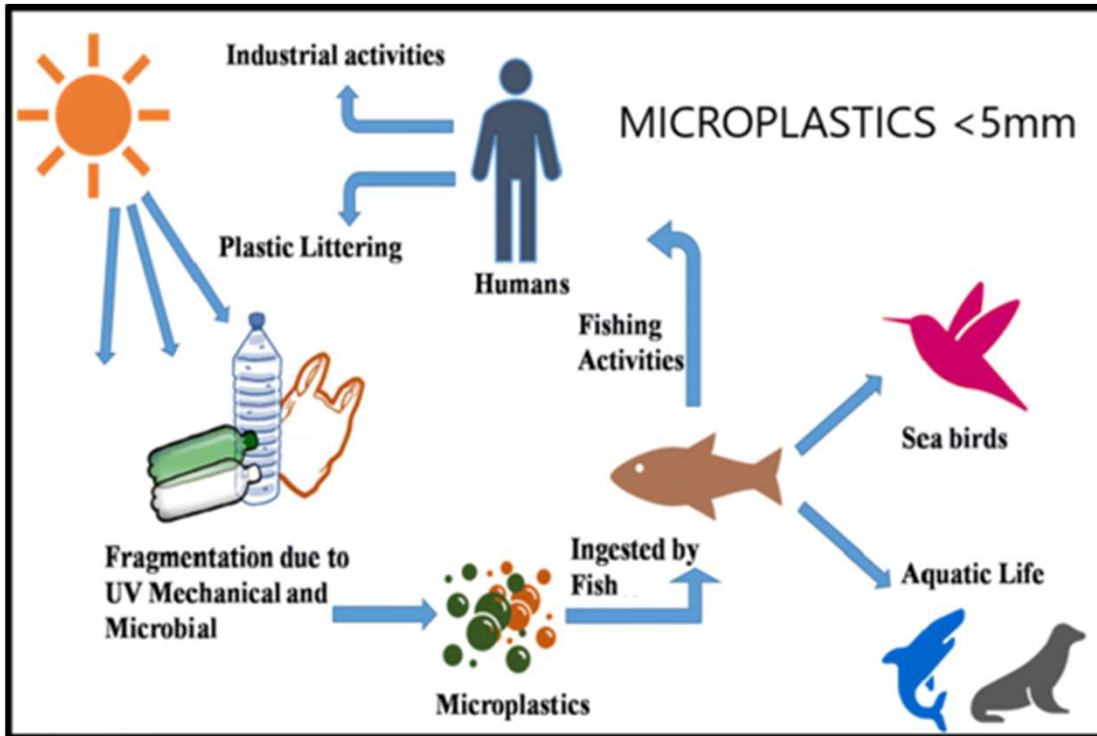


Commercial sea salt samples purchased in China contaminated with tiny microplastics

Phys Org, 2015. <https://phys.org/news/2015-11-commercial-sea-salt-samples-china.html>



From plastics to microplastics



Environ Sci Pollut Res. 28 (2021)19544.



Researchers have found microplastics deep in lungs and in the bloodstream.

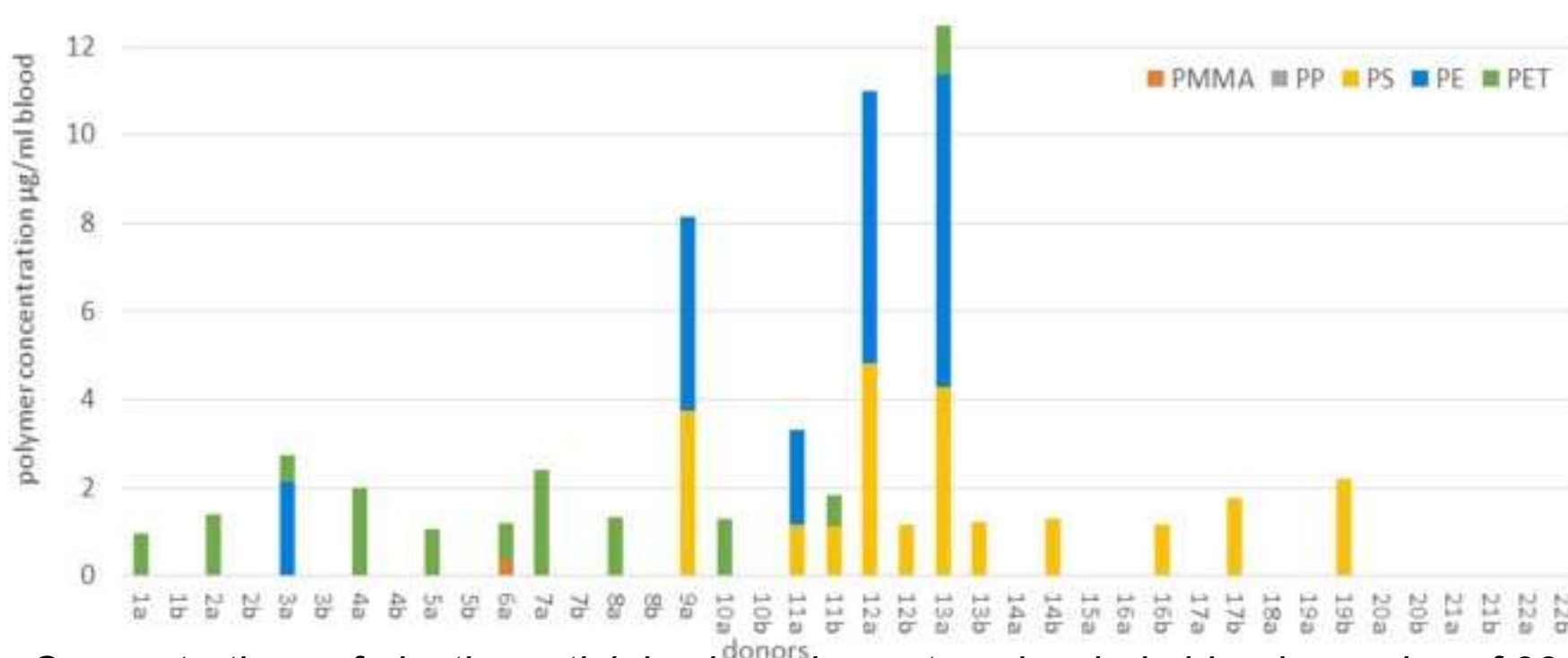
<https://www.nbcnews.com/>



From plastics to microplastics

Four high production volume polymers applied in plastic were identified and quantified for the first time in blood. **Polyethylene terephthalate**, **polyethylene** and **polymers of styrene** were the most widely encountered, followed by **poly (methyl methacrylate)**.

Leslie et al. Environ. Int. 163(2022) 107199



Concentrations of plastic particles by polymer type in whole blood samples of 22 donors. Leslie et al. Environ. Int. 163(2022) 107199



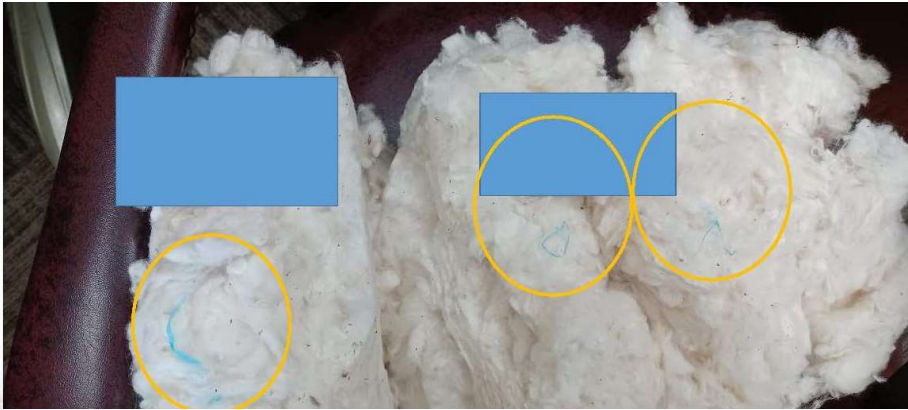
Cotton contamination with plastics



Credit: Dr. John Wanjura, USDA-ARS Lubbock



Cotton contamination with plastics



Credit: National Cotton Council



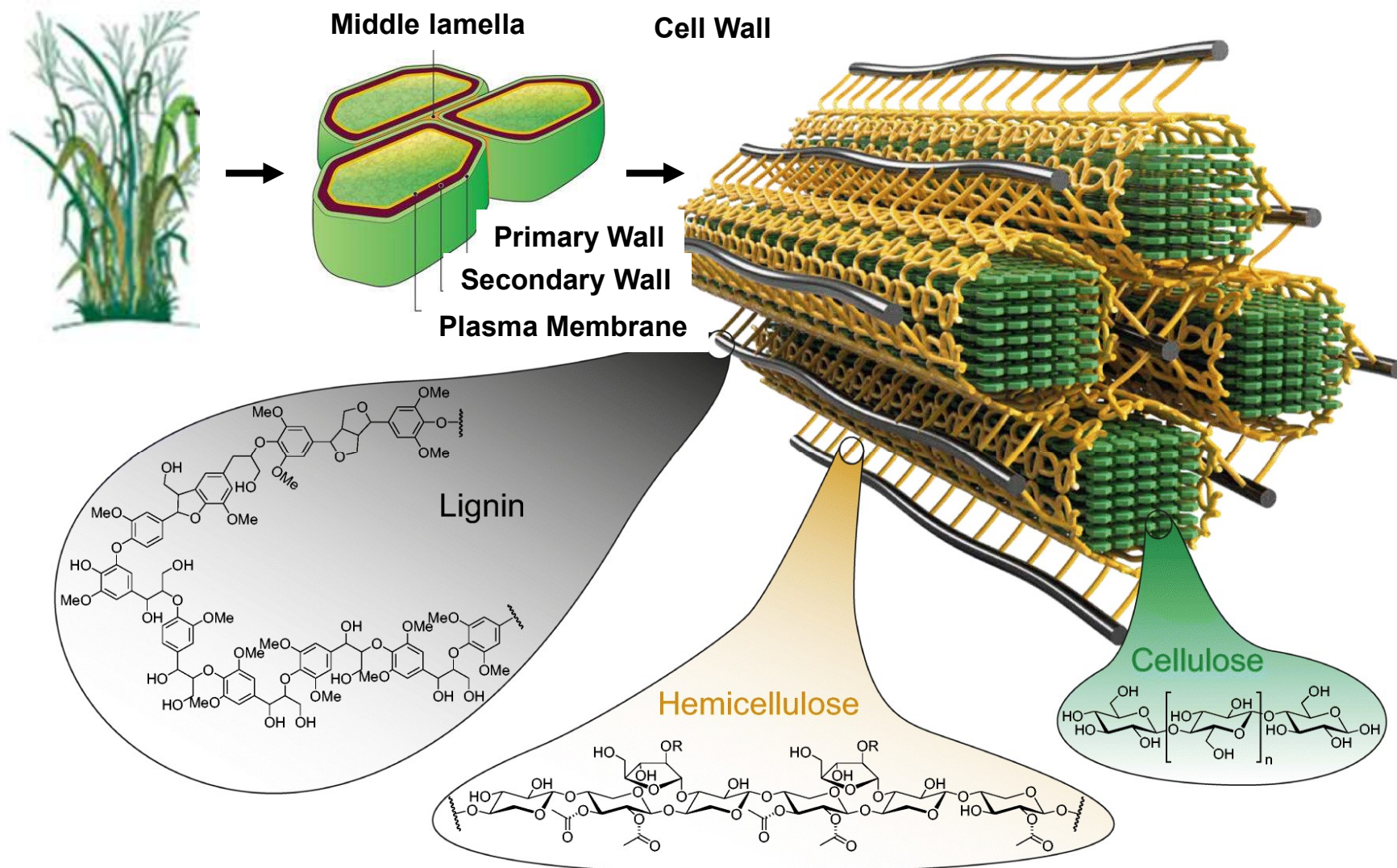
**Could we use discounted
cotton and cotton linter
cellulose to produce bioplastic
films?**



From Cellulose Biopolymer to Bioproducts



Composition of lignocellulosic biomass



Brethauer, S., Shahab, R. L.; Studer, M. H. Impacts of biofilms on the conversion of cellulose. *Appl. Microbiol. Biotechnol.* **2020**, *104*, 5201–5212. <https://doi.org/10.1007/s00253-020-10595-y>



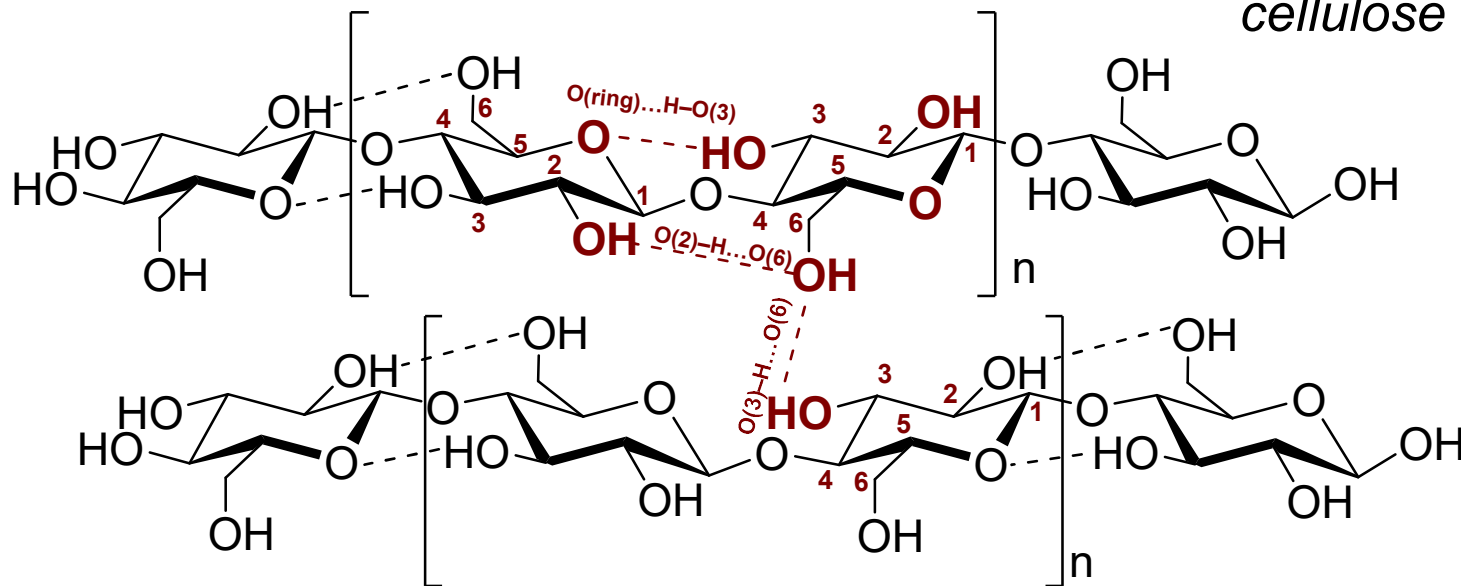
Structural complexity of cellulose

Potential application of cellulose is hindered by:

- ❖ high molecular weight
- ❖ high crystallinity
- ❖ rigidity of the polymeric chain due to extensive hydrogen bonding



Cotton, purest form of plant cellulose





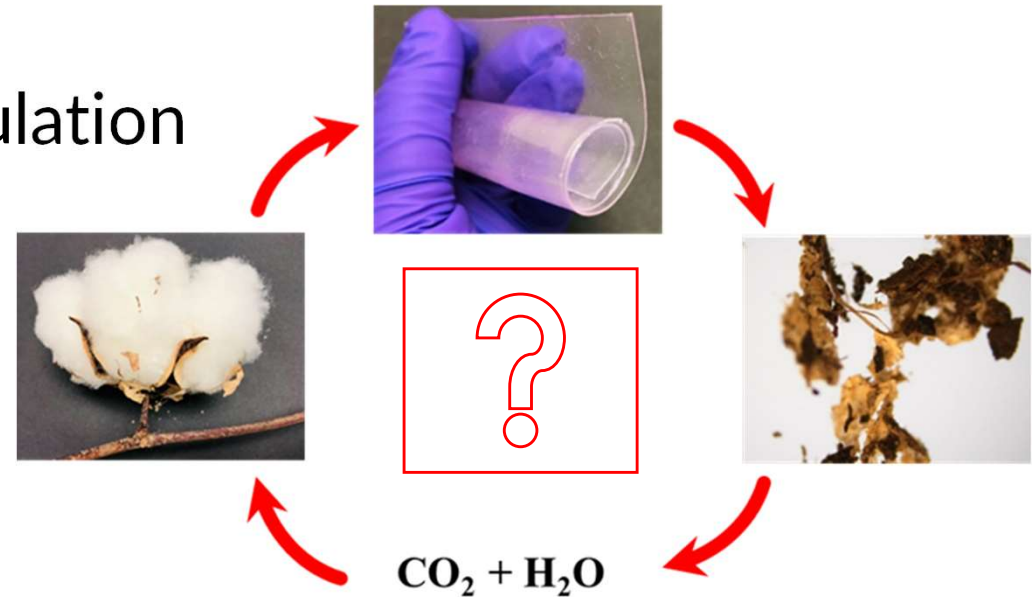
Bioplastic, an ecofriendly alternative

Bioplastics are biobased, and/or biodegradable.

- biomass, biocompatible
- limit plastic waste accumulation

Types of bioplastics:

- starch-based plastics
- protein-based plastics
- some aliphatic polyesters

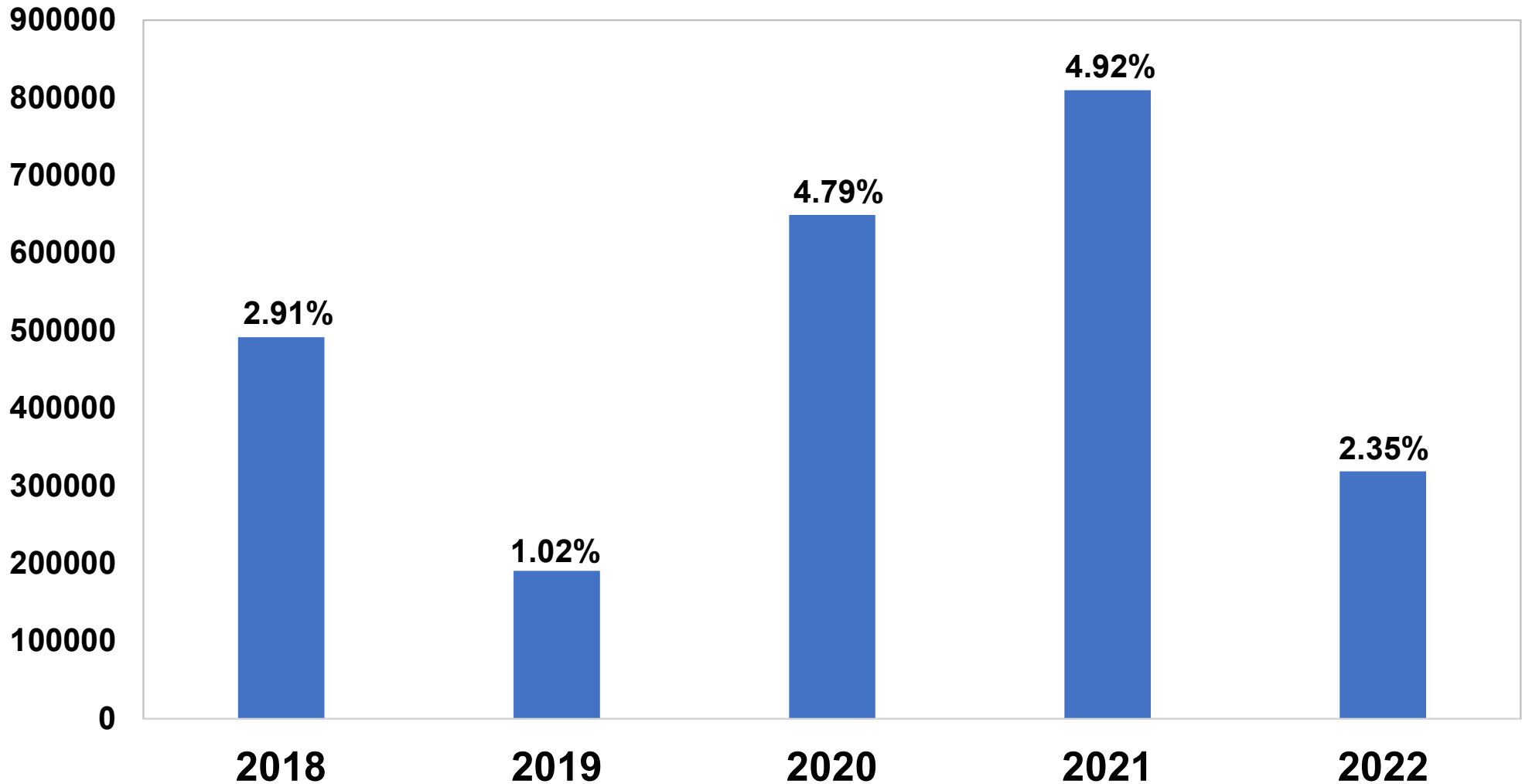


Cellulose is the most abundant biopolymer on earth and could be the source for plastics production



Micronaire 2.0 – 3.2

Number of Bales of Upland Cotton: Micronaire 2.0 – 3.2





Dissolution of cellulose*



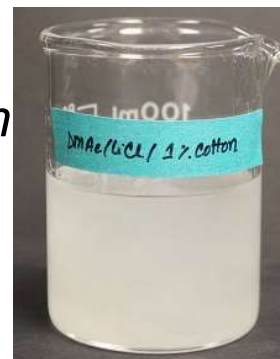
Raw cotton

cleaning



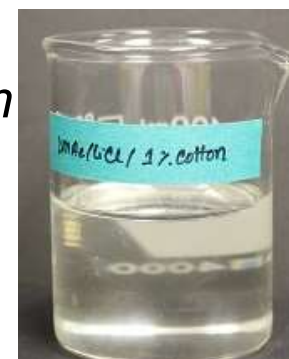
Scoured & bleached cotton

dissolution



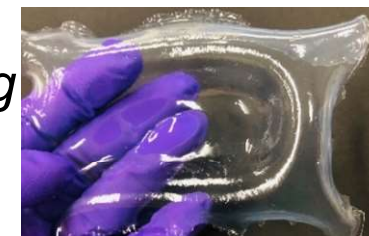
Dissolution of 1% cotton in DMAc/LiCl overnight at 50°C

dissolution



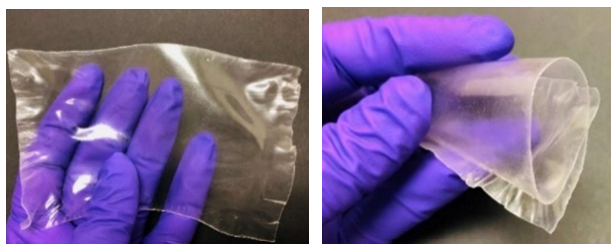
Complete dissolution

gel formation

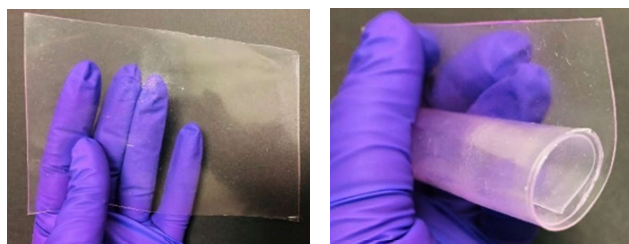


Cellulose hydrogel

Hot-pressing



Cellulose films obtained by hot pressing

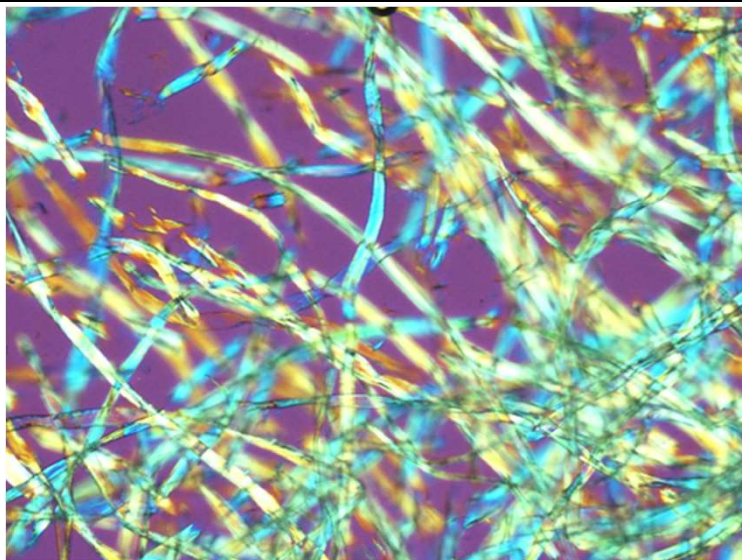


Plasticization with glycerol

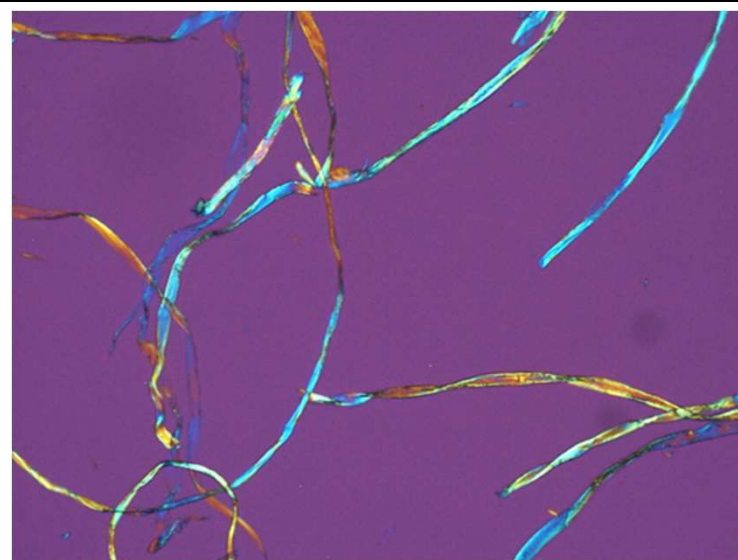
* Abidi, *Patent Pending*



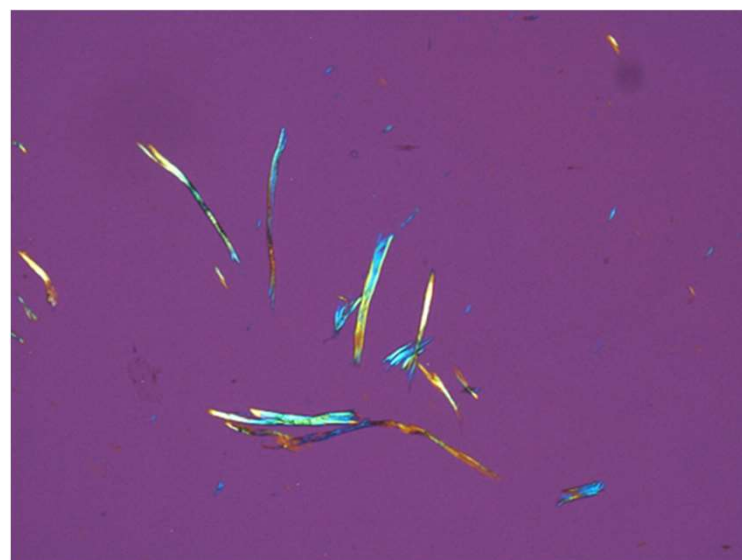
Dissolution of cellulose in DMAc/LiCl



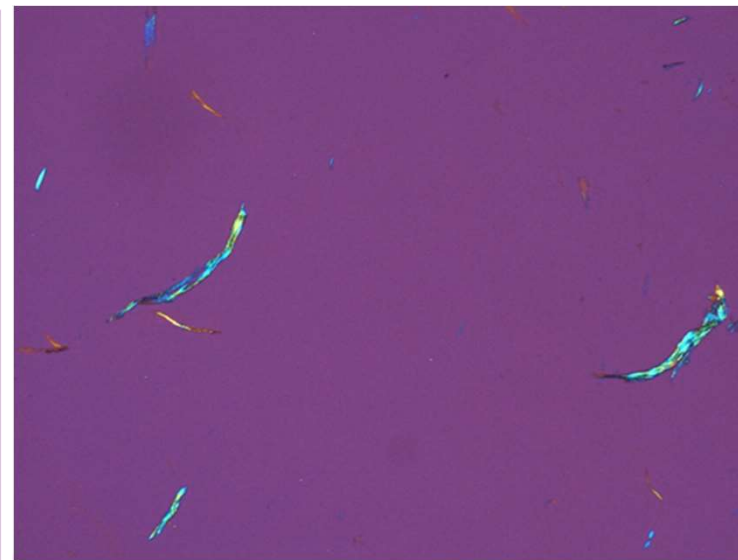
Cotton in DMAc/LiCl at t=0



After 6h at 105°C



After 9h at 105°C

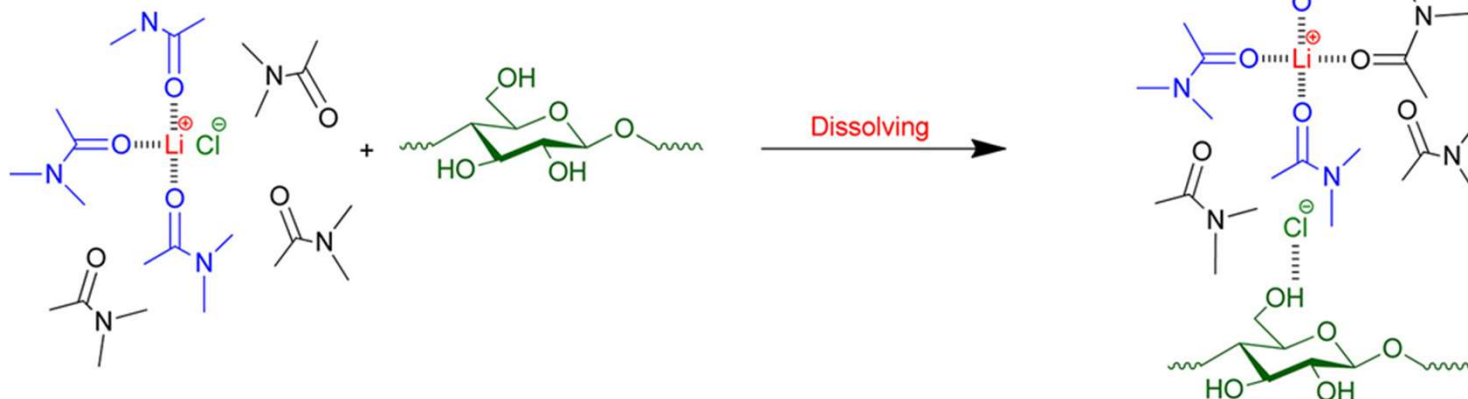


After 12h at 105°C

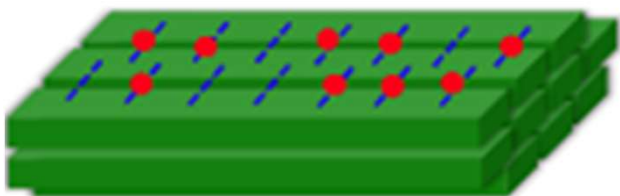


Dissolution of cellulose in DMAc/LiCl

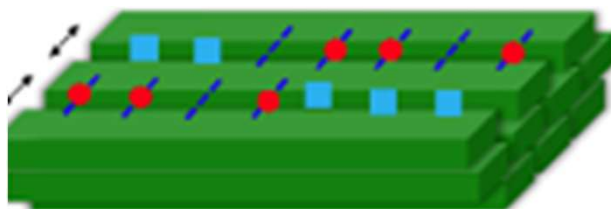
Schematic of the interaction among Li⁺ cation, Cl⁻ anion, and DMAc when cellulose dissolves into the DMAc/LiCl system. Zhang et al. J. Phys. Chem. B. 2014, 118, 31, 6507



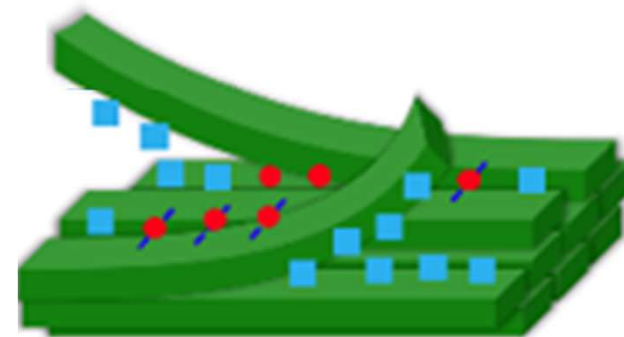
Penetration of anions and cations between cellulose chains



Breaking of hydrogen bonds



Peeling off cellulose chains

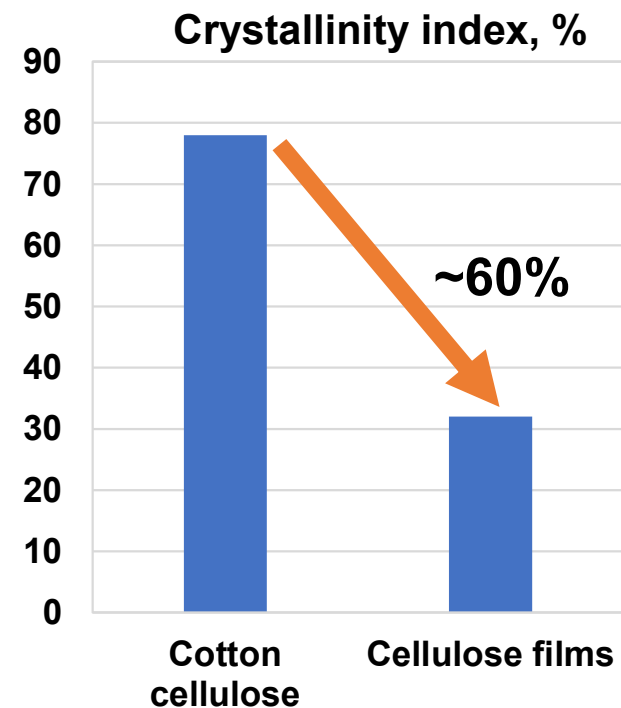
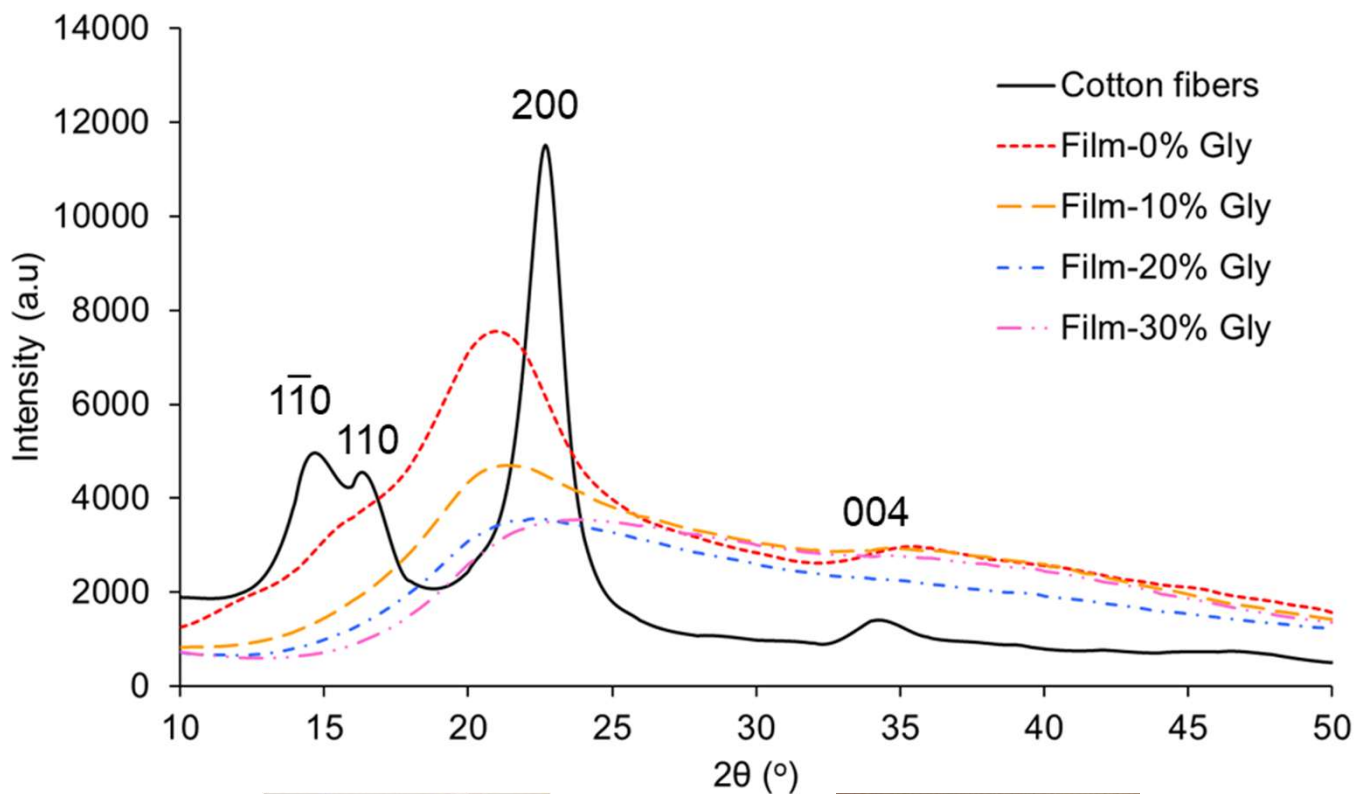


● Anion ■ Cation - - - - - Hydrogen bonds

(Uto et al., 2018)



Dissolution of cellulose in DMAc/LiCl





Thermal stability of cellulose films

Polyvinyl Chloride: 200-300°C

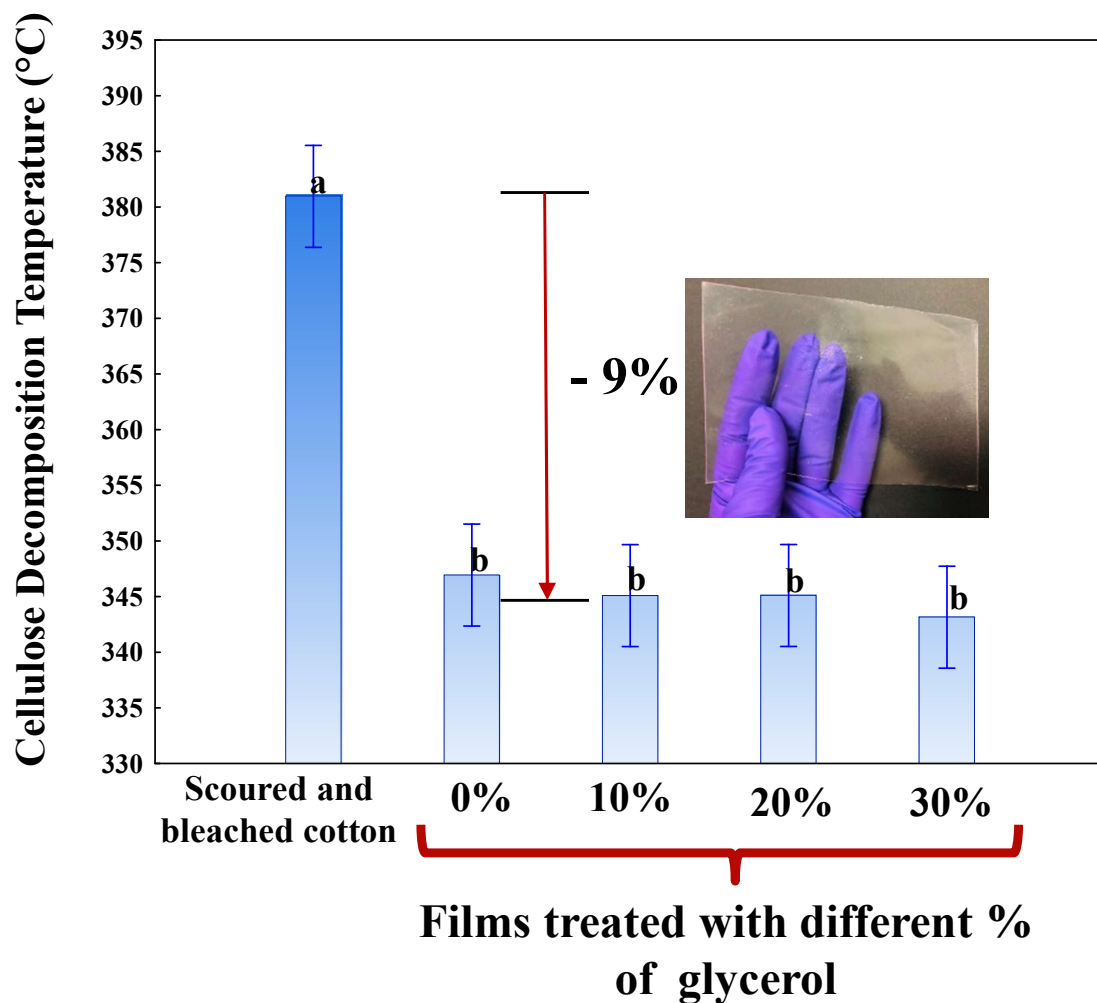
Polyethylene terephthalate: 283-306°C

Polyethylene: 335-450°C

Polypropylene: 328-410°C

Polystyrene: 300-400°C

Cellulose films: 340-355°C

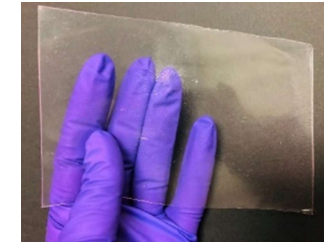
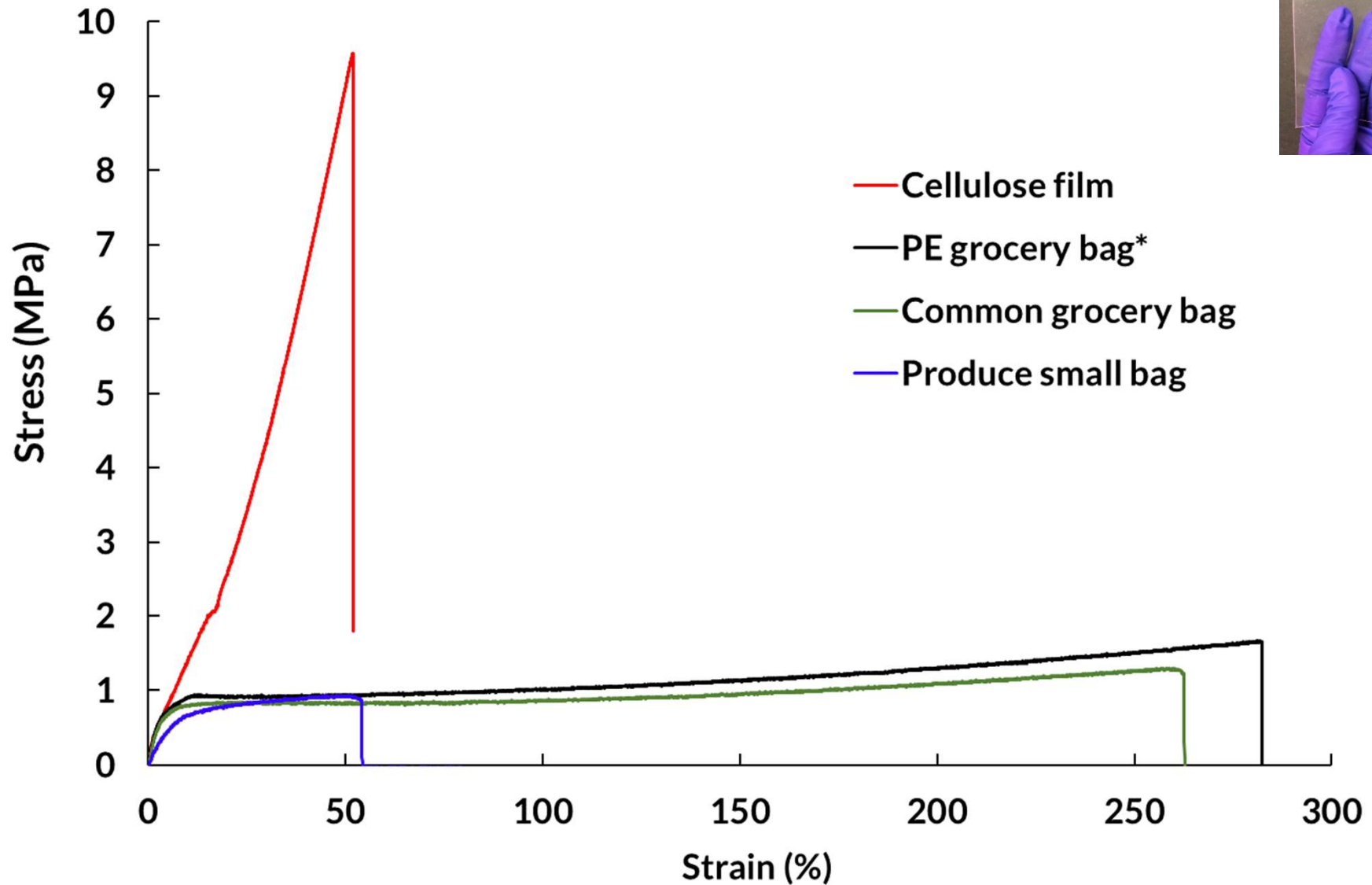


video





Tensile properties of cellulose films





Biodegradation of cellulose films



*Preconditioned soil beds
($12 \pm 2\%$ moisture)*



Soil burial of cellulose films



*High tunnel and sample trays placed inside
a high tunnel*



*Soil moisture measurement
using digital soil moisture meter*



Biodegradation of cellulose films

Start: 11 Aug. 2021



Day 0



Day 7



Day 14



Day 21



Day 28



Day 35



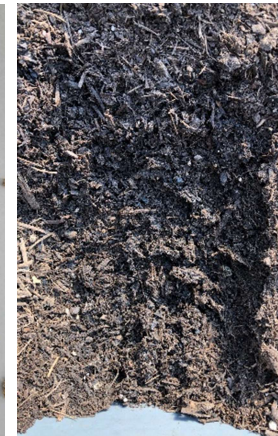
Day 42



Day 49



Day 56



Day 63

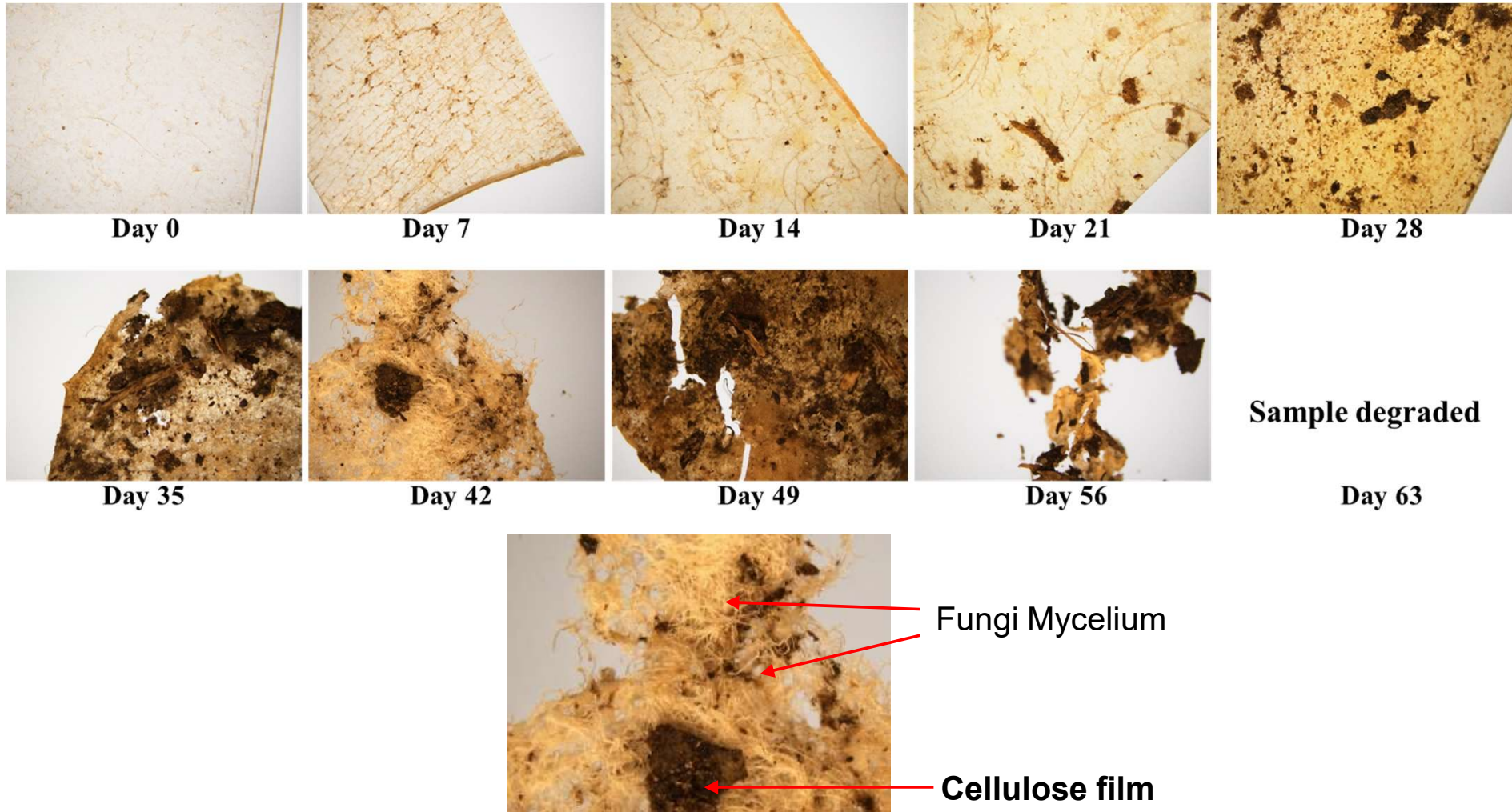
End: 13 Oct. 2021

(Samples were degraded)

Major changes: discoloration, shrinkage, adhesion of soil particles, appearances of whitish substance, holes, cracks, and fragmentation.



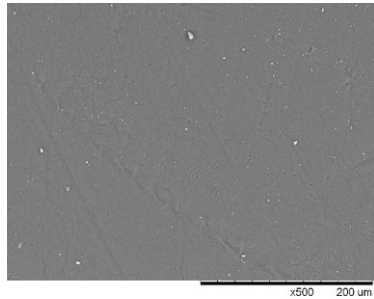
Biodegradation of cellulose films



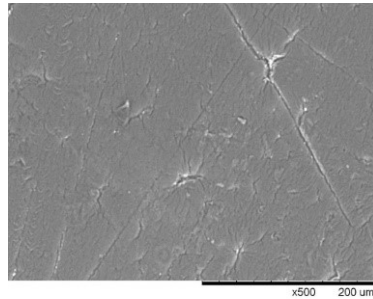
Major changes: discoloration, adhesion of soil particles, and appearances of holes, cracks, and fungi mycelium.



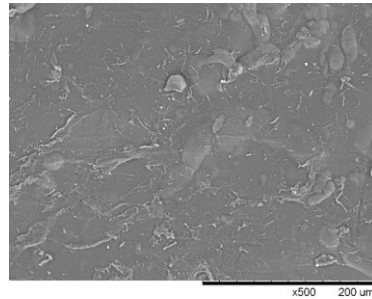
Biodegradation of cellulose films



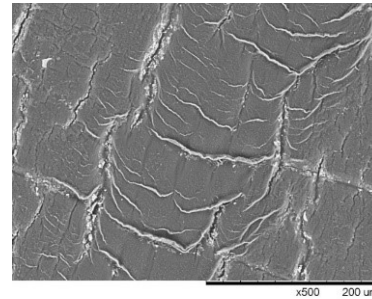
Day 0



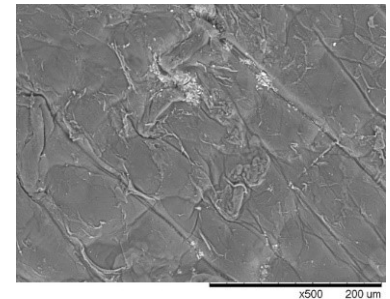
Day 7



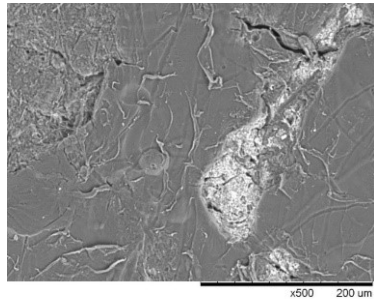
Day 14



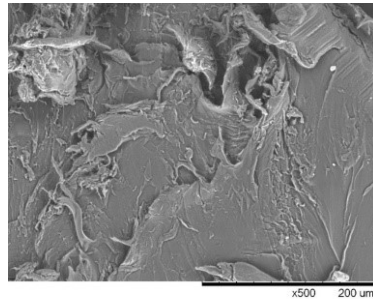
Day 21



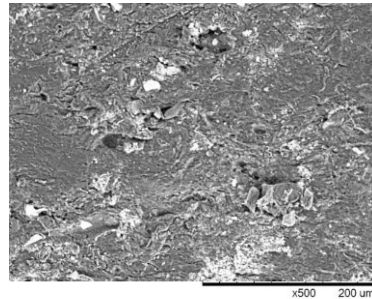
Day 28



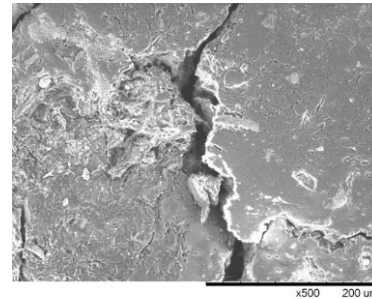
Day 35



Day 42



Day 49



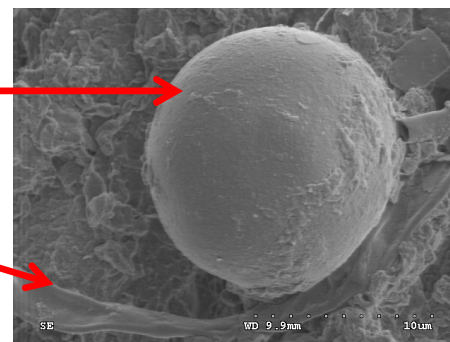
Day 56



x3k

Conidium
(spore of a fungus)

Hypha



x5k

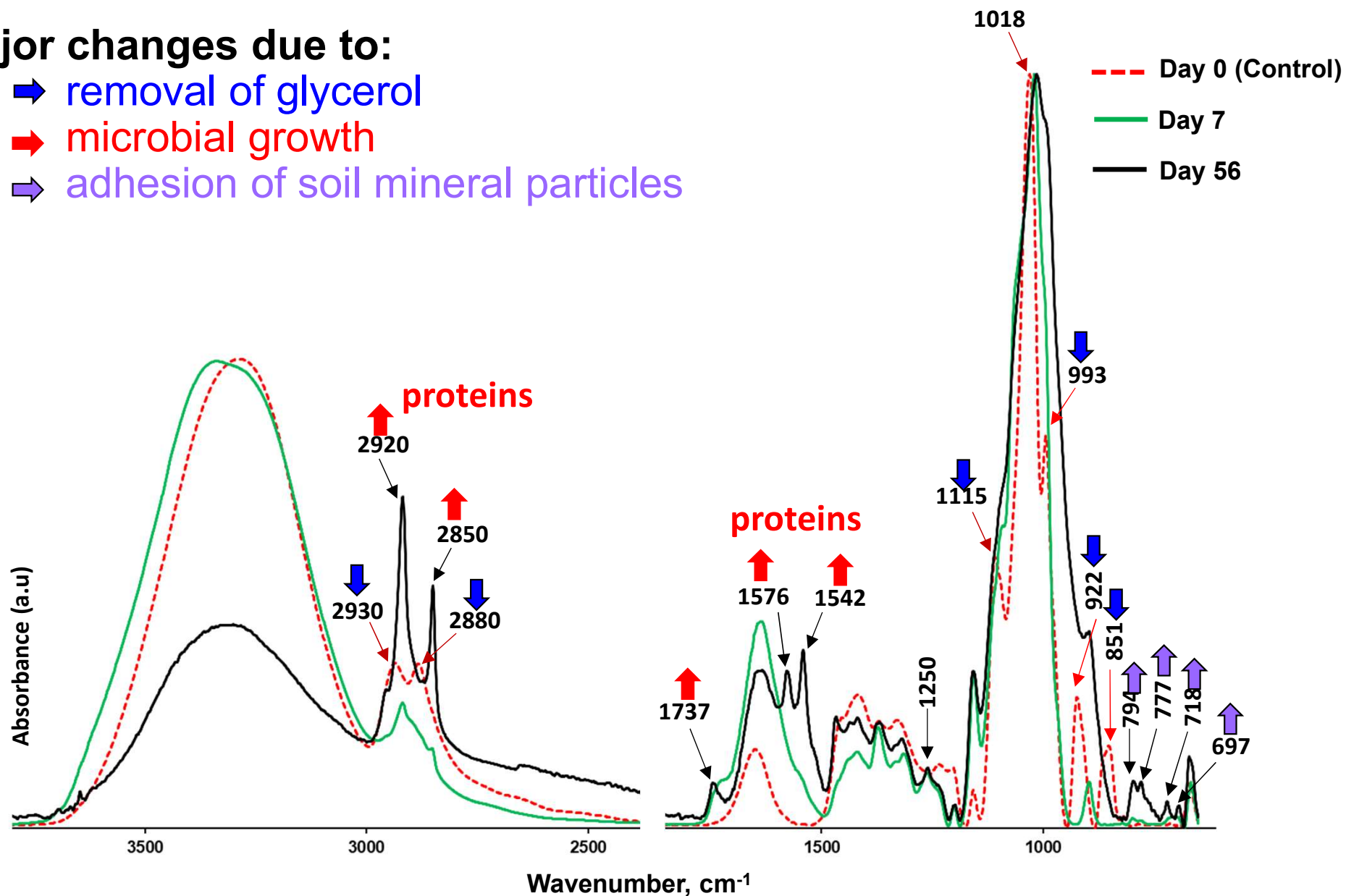
Conidium, a type of asexual reproductive spore of fungi usually produced at the tip or side of hyphae (filaments that make up the body of a typical fungus) or on special spore-producing structures called conidiophores. The spores detach when mature.



FTIR of cellulose films

Major changes due to:

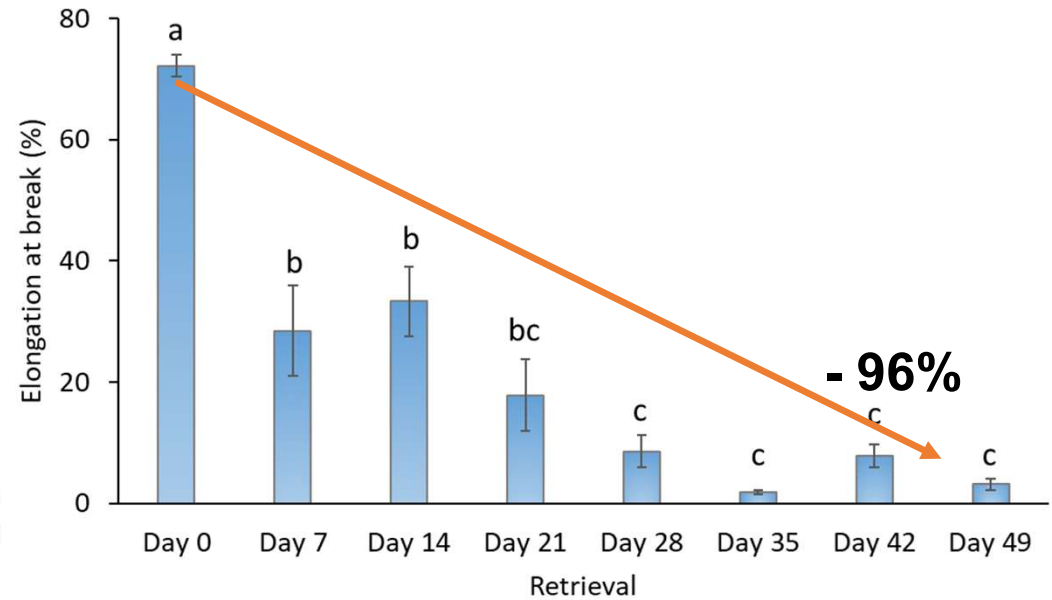
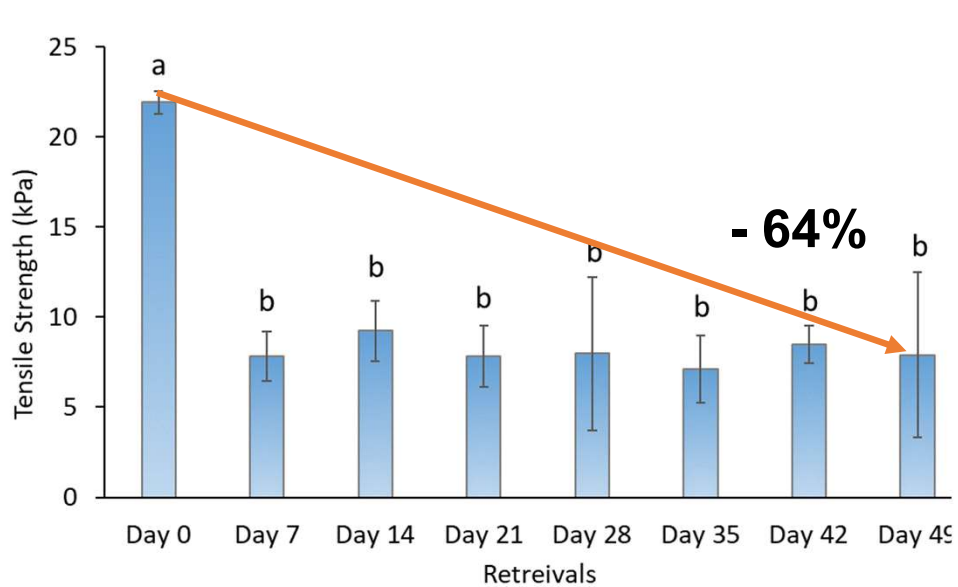
- ➡ removal of glycerol
- ➡ microbial growth
- ➡ adhesion of soil mineral particles





Tensile properties of films as a function of time

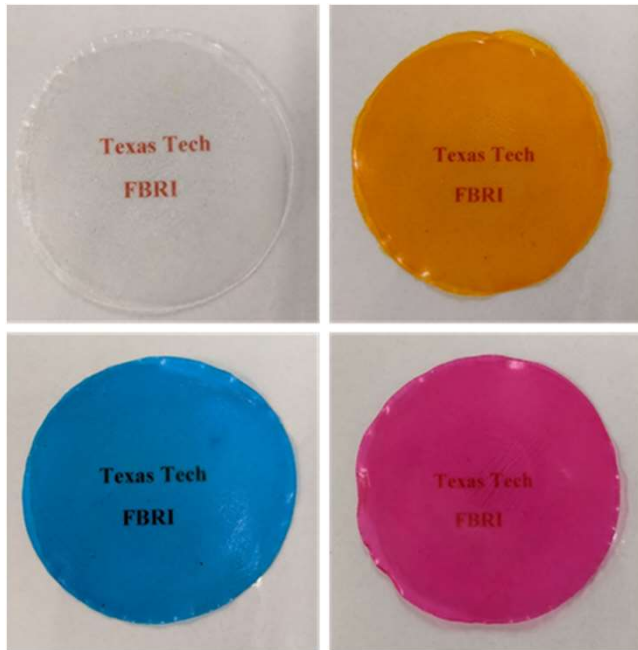
Cellulose films buried in the soil



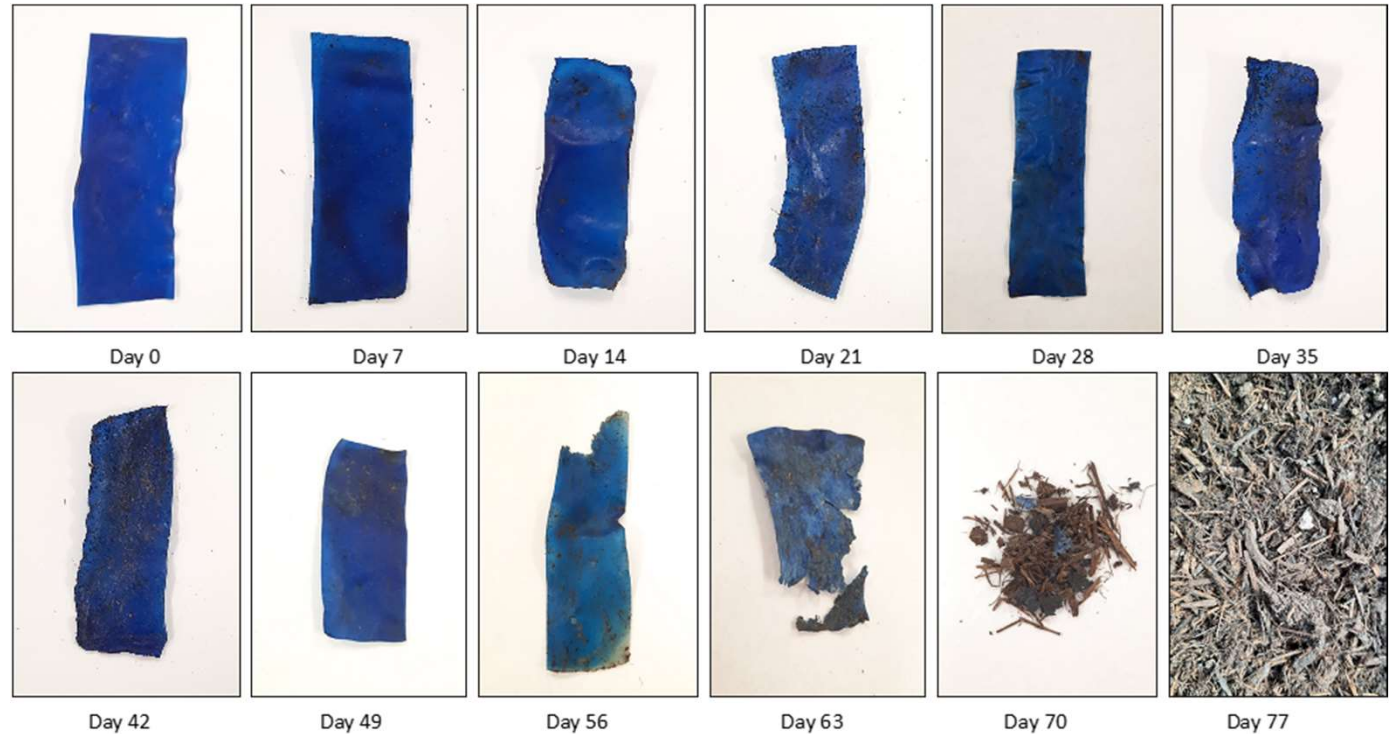


Dyed cotton cellulose-based films

Cellulose-based films can be dyed using cotton dyes



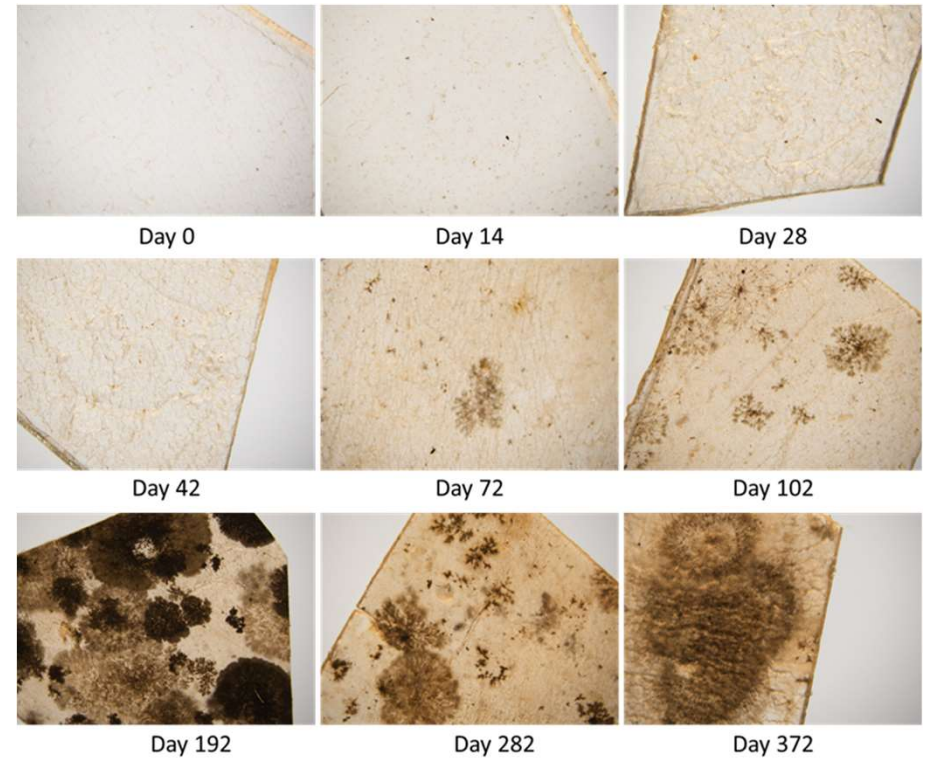
Dye molecules do not inhibit the biodegradability of films





Degradation of cellulose-based films

Cellulose films as foil cover





Degradation of petroleum-based films

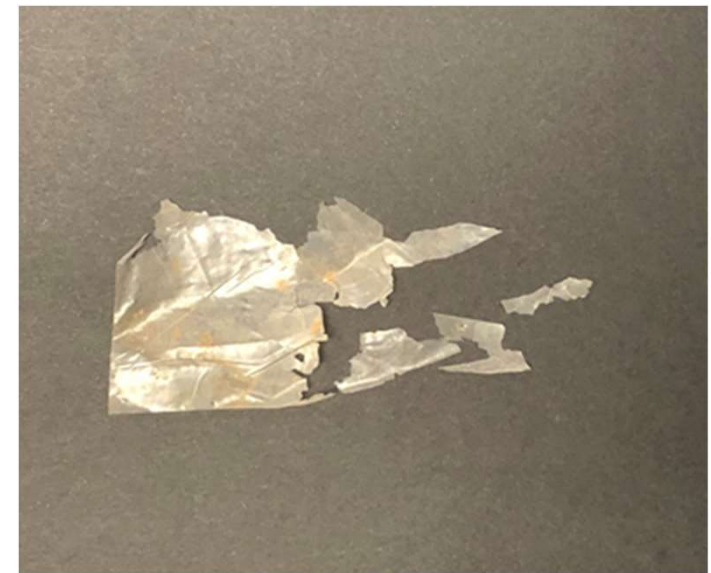
Regular plastic buried in the soil



Day 0

After 2 years

Regular plastic shopping bag under the action of sunlight

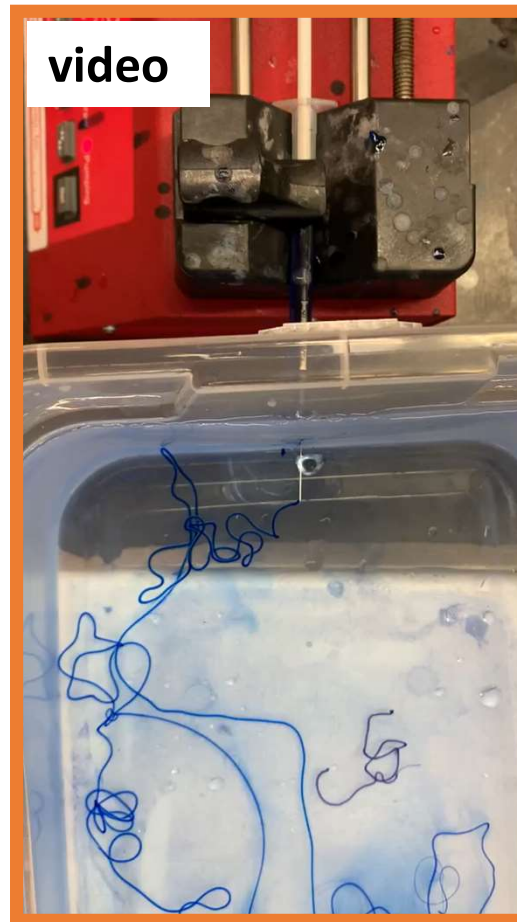
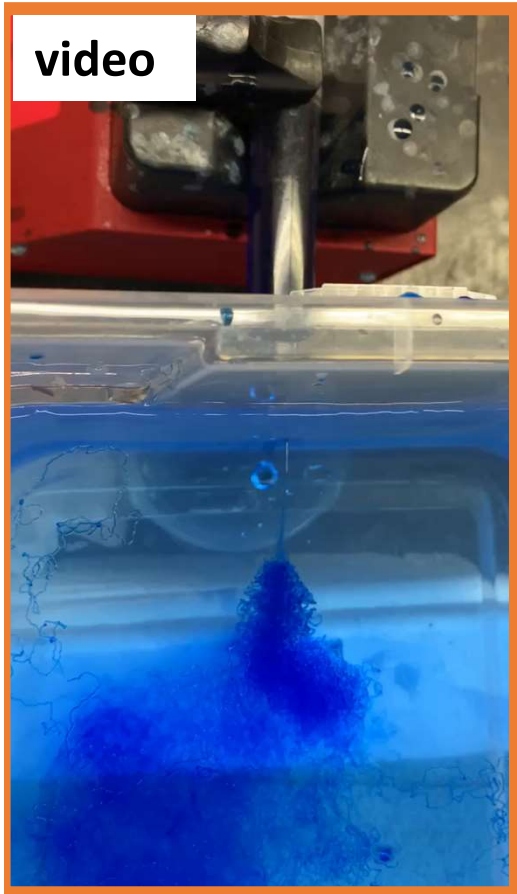


After 2 years



Wet spinning from cotton cellulose solution

Wet fibers



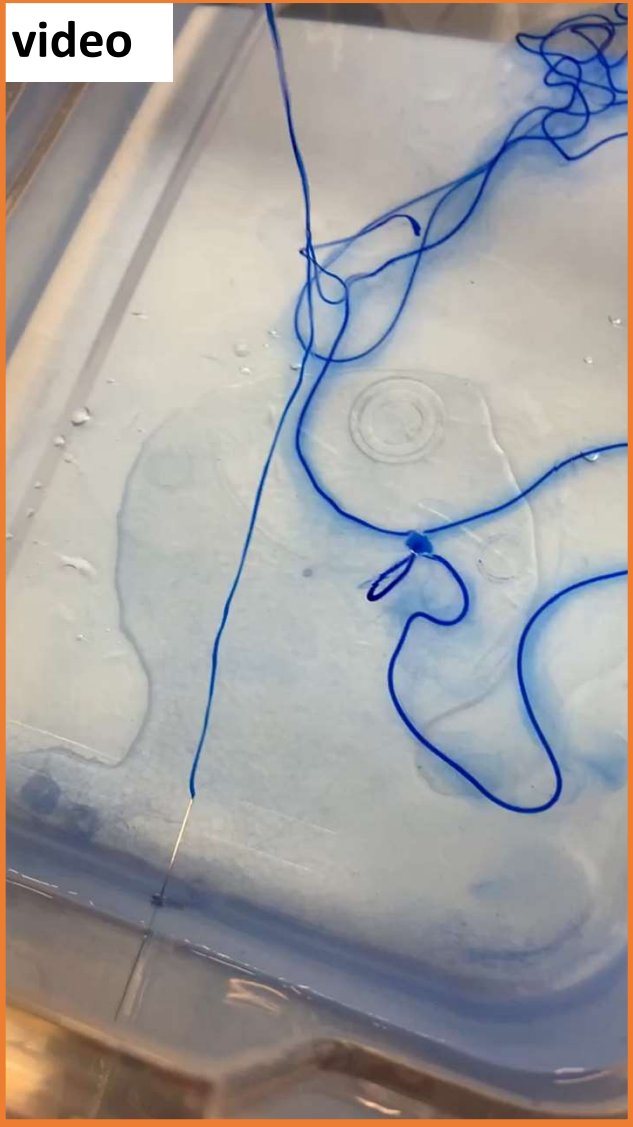
Dried fibers





Wet spinning from cotton cellulose solution

video



Wet fibers



Dried fibers

video



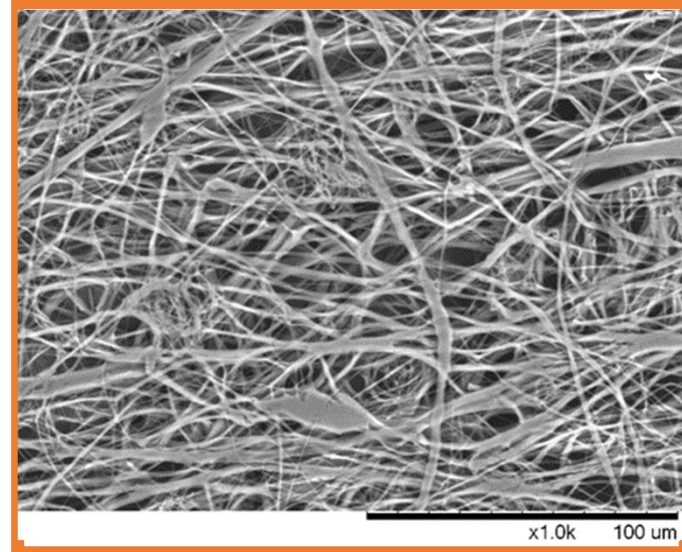
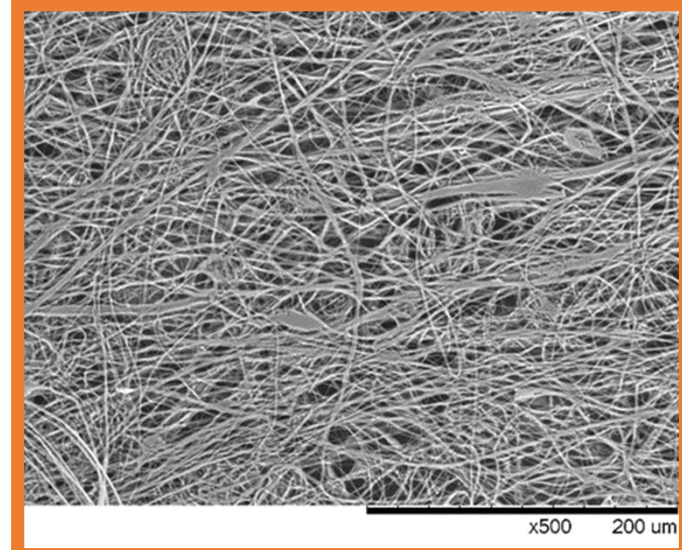


Electrospinning from cotton cellulose solution

video



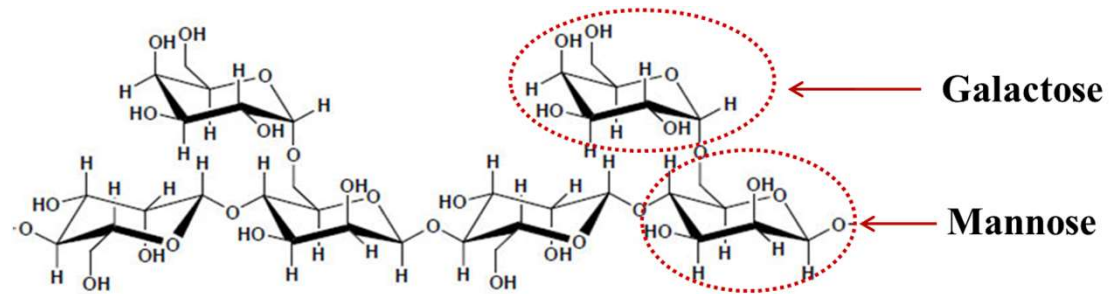
Electrospun fibers





What is guar gum?

- A galactomannan extracted from guar or cluster bean (*Cyamopsis tetragonolobus L.*).
- Emerging as one of the most versatile and low-cost water-soluble biopolymers with unique fascinating properties.

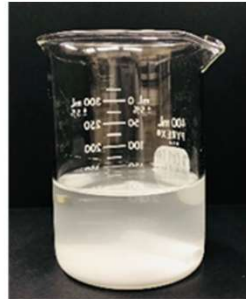


Uses of guar gum

- Additive to pharmaceuticals, food, cosmetics, and several consumer products
- Hydraulic fracturing in oil well drilling to harvest shale gas and oil



Cellulose-guar composite solution



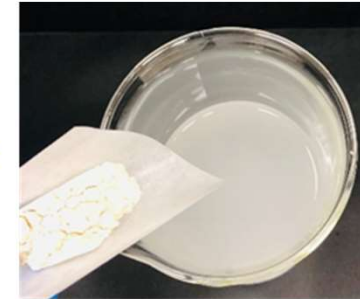
**MCC soaked
in water**

Sonication

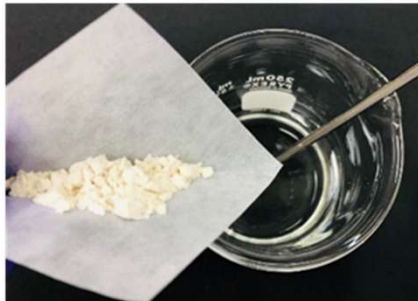


**MCC
suspension**

*Dispersion
of guar gum*



Overnight magnetic stirring



**Dispersion of guar gum in
water/CNC solution**

*Overnight
magnetic
stirring*



**Viscous guar or
guar/cellulose solution**



**Cast-drying
Wet spinning
Freeze-drying**



Cellulose-guar composite materials

Films



Guar/guar-cellulose aqueous solution

Casting



Guar gum solution cast in Petri dishes

Drying



Dried guar-based films

Filaments



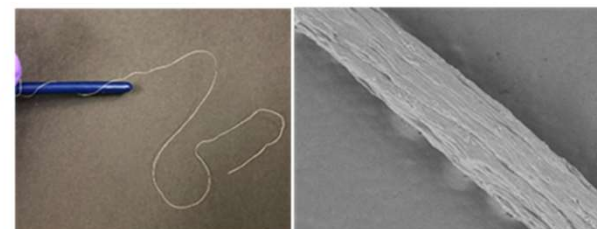
Guar/guar-cellulose aqueous solution

Wet spinning



Alcohol-based coagulation bath

Air-drying

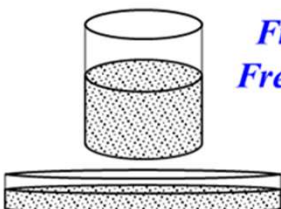


Guar-based filament

Aerogels

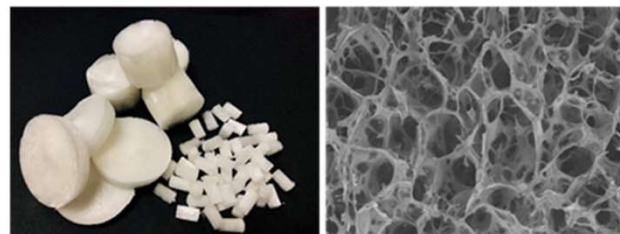


Guar/guar-cellulose aqueous solution



Guar-based solution in small containers

Freezing & Freeze-drying



Dried guar-based aerogels



Cellulose-guar composite materials

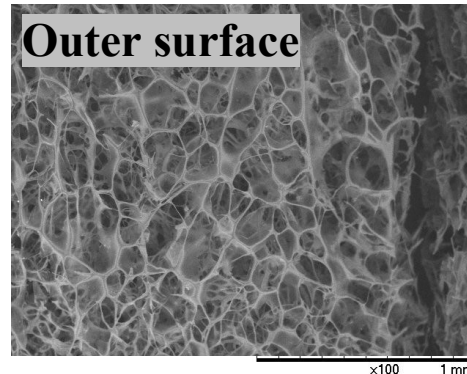
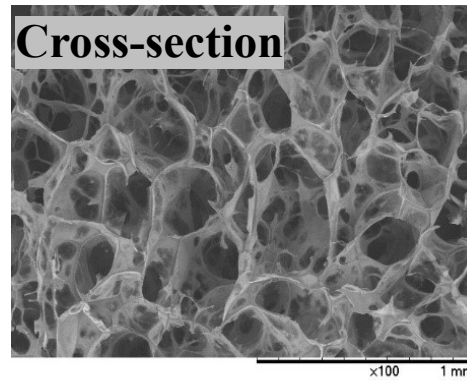


Freezing followed by freeze-drying

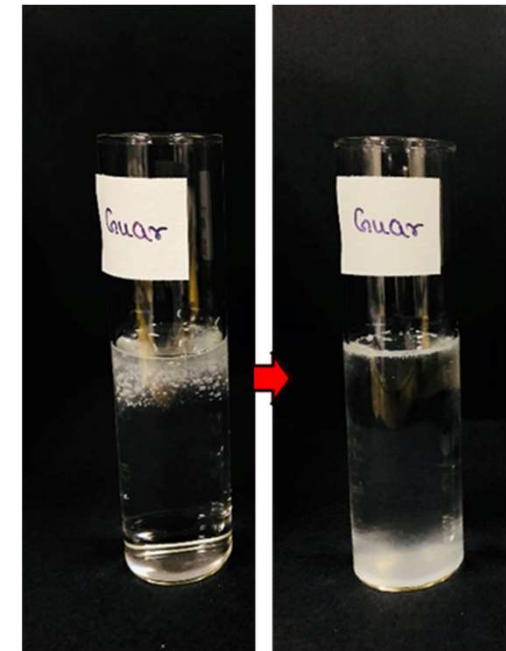


- Shape and size can be changed using different molds
- Can be used as a carrier for active ingredients

Porous structure



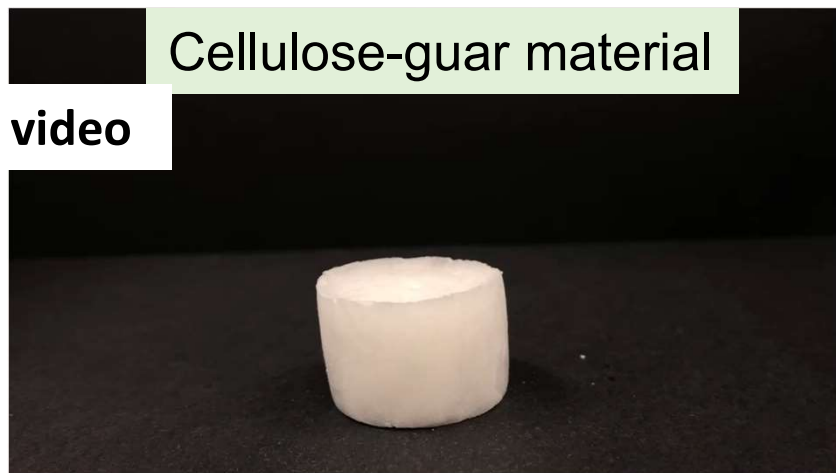
*Water soluble
(~2-3 min with stirring)*



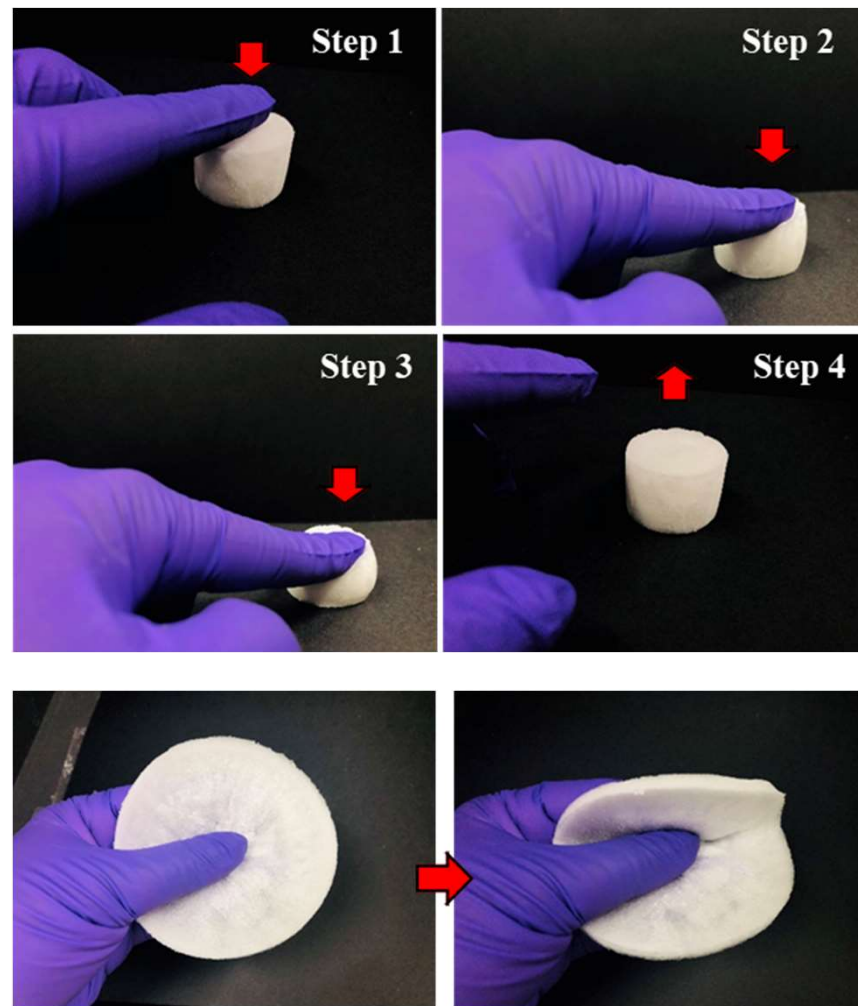


Cellulose-guar composite materials

- Easily compressible or foldable



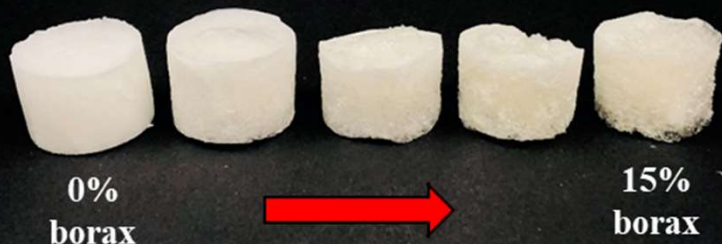
Polystyrene packaging



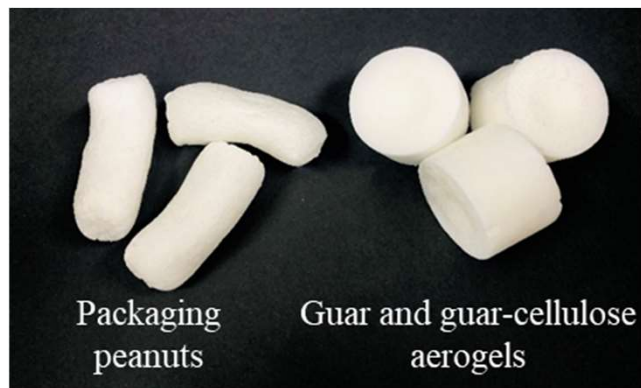


Cellulose-guar composite materials

Crosslinked with different concentrations of borax

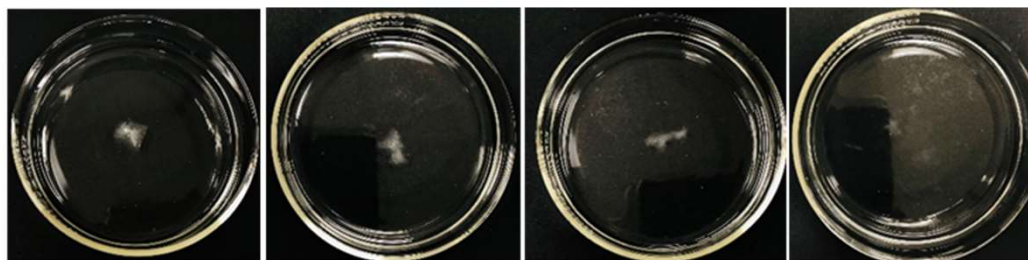


video



- Water solubility can be customized by crosslinking

No crosslinking



Crosslinked



0 min

15 min

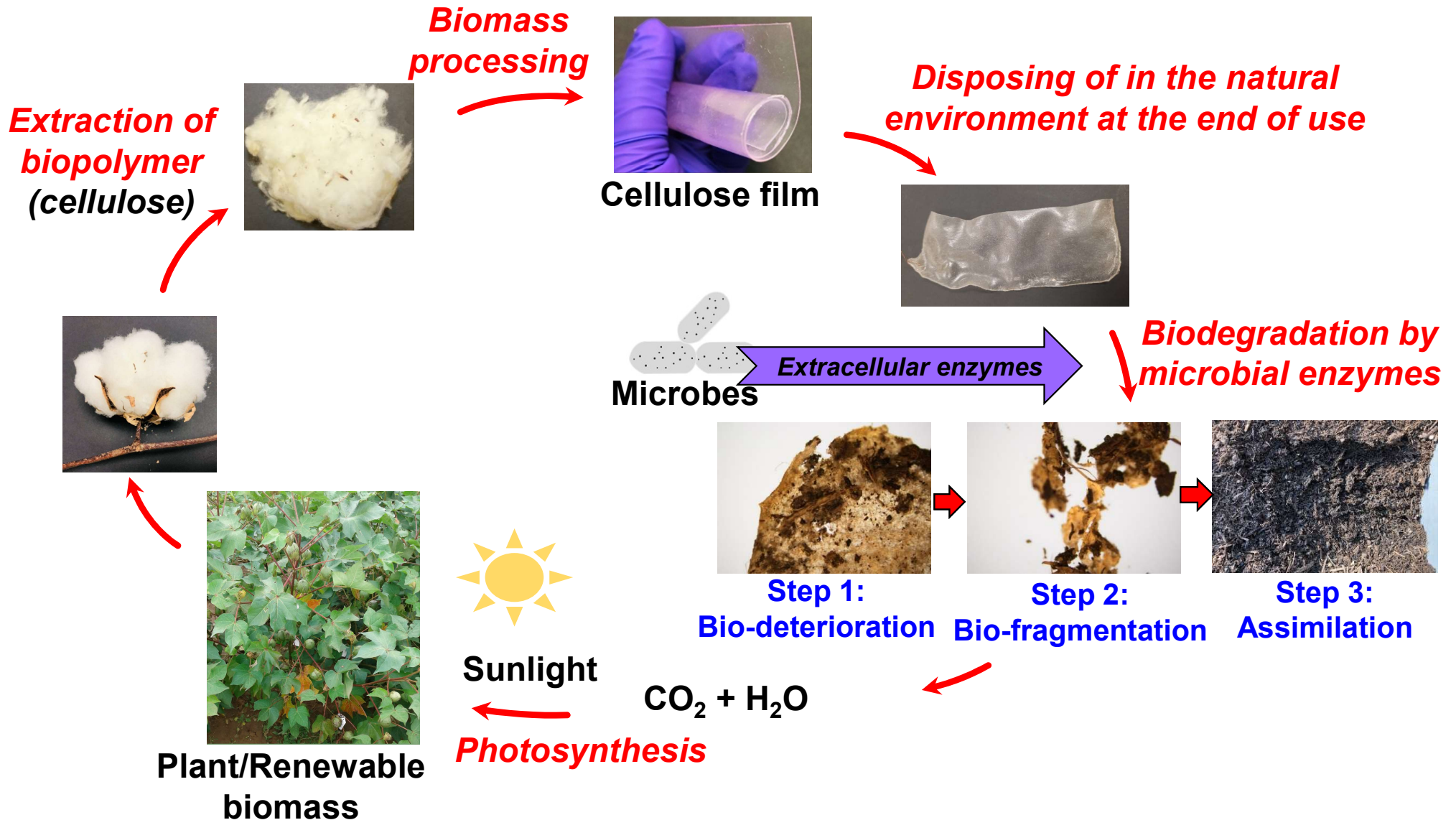
1 h

3 h

- Guar-based aerogels resemble packaging peanuts



Carbon cycle of cellulose films





Thank you