Photo: viscose filament / SusTexEdu

2.1 / 3 Manufacturing Technologies of Man-made Fibres
SOLUTION SPINNING

This open educational resource has been developed by:

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SusTexEdu

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This learning material was developed in the Erasmus+ funded project Education Partnership of Textile and Clothing Sector Materials & Sustainability (SusTexEdu)

The goal of the project is to research and develop education in the textile and clothing sector related to textile materials, sustainability and circular economy.

The learning material has been prepared for piloting, and students will be asked for voluntary feedback after the course for the further development of the material.

Project coordinator: Metropolia UAS

Partners: Hogent (BE), Mome (HU), Omnia (FI), TTHK (EE), TTK UAS (EE), University of Borås (SE)

Funding: Erasmus+

Project period: 2022-2024

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About this learning unit

CONTENT DESCRIPTION

The learning unit aims to give the student in-depth knowledge of manufacturing technologies of man-made fibres.

LEARNING OUTCOMES

Ability to:

- to characterise the man-made fibres on the basis of the production methods of their diverse properties and reactions towards external influences.
- to name special, innovative fibres for clothing and interior textiles and to identify their end use.
- to identify textile fibres in various textile materials by means of analysis (microscope, solvent)

STUDENT WORKLOAD

2 ECTS, which is equal to 50-60 hours of work:
for example

- Lectures 26-30 h
- Group activities 6-10 h
- Independent study 10-28 h

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Outlines

- Introduction
- Fibre spinning techniques
  - Solution wet spinning
  - Solution dry spinning
  - Gel spinning
- Regenerated fibres
  - Viscose rayon fibres
  - Lyocell fibres
- Application areas

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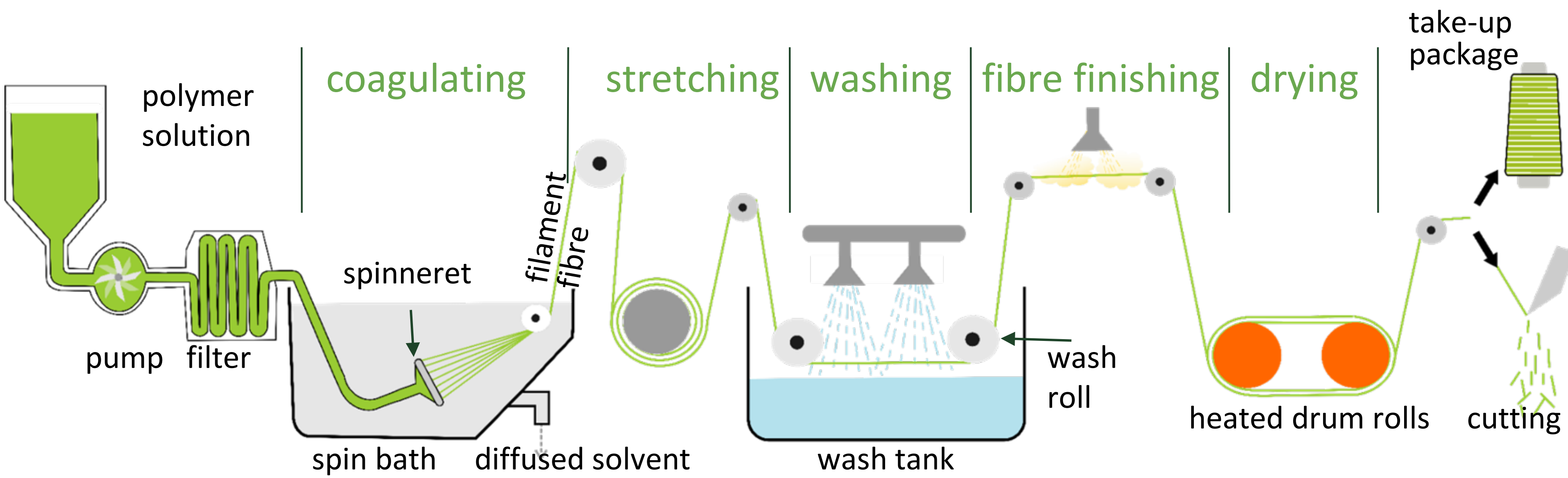
Solution spinning process

- Solution spinning techniques are used for the polymers which don't form ***thermally stable*** and ***viscous melts.***
- In these cases, polymers are dissolved in ***low-molecular*** solvents, form solution or suspension of sufficient viscosity.
- The polymer solution is extruded through spinneret and fibers are formed by the loss of solvent.
- There are two types of solution spinning processes:
  - ***Wet spinning:*** solvent is separated by osmosis or diffusion mechanism
  - ***Dry spinning***: solvent is separated by evaporation

Wet spinning process

- Polymer is dissolved in solvent to form viscous solution called *dope*.
- Dope is extruded in another bath containing second solution called *coagulation bath* or *spin bath*.



Steps involved in wet spinning process. Image by SusTexEdu

- Polymer starts to precipitate by diffusion which coagulates the polymer chains into solid filaments.

VIDEOS to be found with keywords e.g. : Wet spinning of polyacrylonitrile fibers

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… wet spinning process

- The coagulation bath containing low molecular weight substances miscible with solvent but don’t dissolve the polymer.
- To improve the ***homogeneity*** of the spun yarn, the spin bath and spinning solution may contain ***modifying agents*.**
- The polymer precipitates as soon as it exits the spinneret hole in spinning bath and a gel filament formed.
- A surface layer is formed which continue to coagulate towards inside.
- Solvent diffuses from the inner part of the filament to the surface and then into the bath.
- The coagulation rate has strong influence on the gel structure and final properties of produced fibres.

… wet spinning process

- Different technical designs are possible for wet spinning process
  - *Horizontal or vertical spinning bath*
  - *Addition of more baths*
- Distance between the holes of the spinneret should be very small, for commercial applications there are about ***2000 holes*** in spinneret.
- The maximum take-up velocities are limited **(*100-150 m/min*)**
- The **slowest** spinning process
- Two types of wet spinning: coagulation and regeneration
  - Filament precipitation with chemical reaction
  - Fibrous polymer regenerated from a soluble derivate
- Examples are acrylic, aramid, elastane, PVC, viscose and cupro

… wet spinning process

Factors affecting fibre properties

- Materials properties
- Type of solvent
- Concentration of polymer
- Temperature of spinning solution
- Composition, concentration and temperature of spin bath
- Stretching
- Heat setting

Viscose rayon production

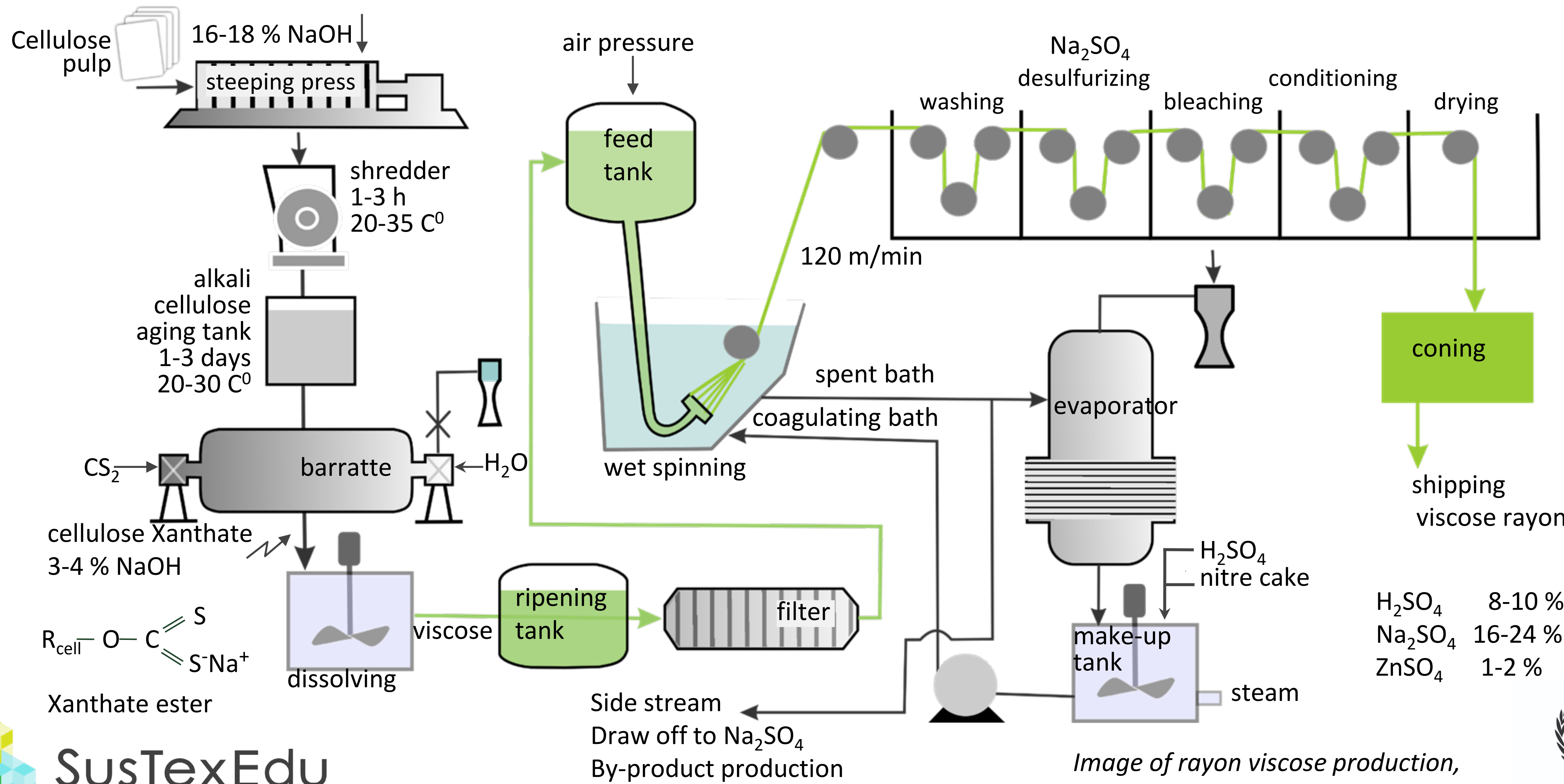

Image of rayon viscose production, presented by SusTexEdu

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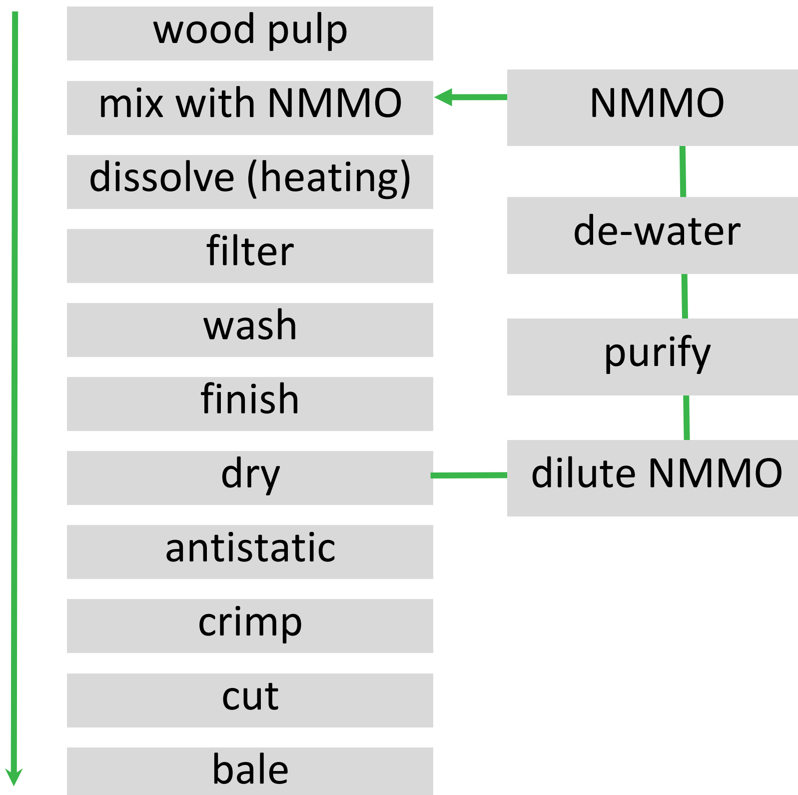
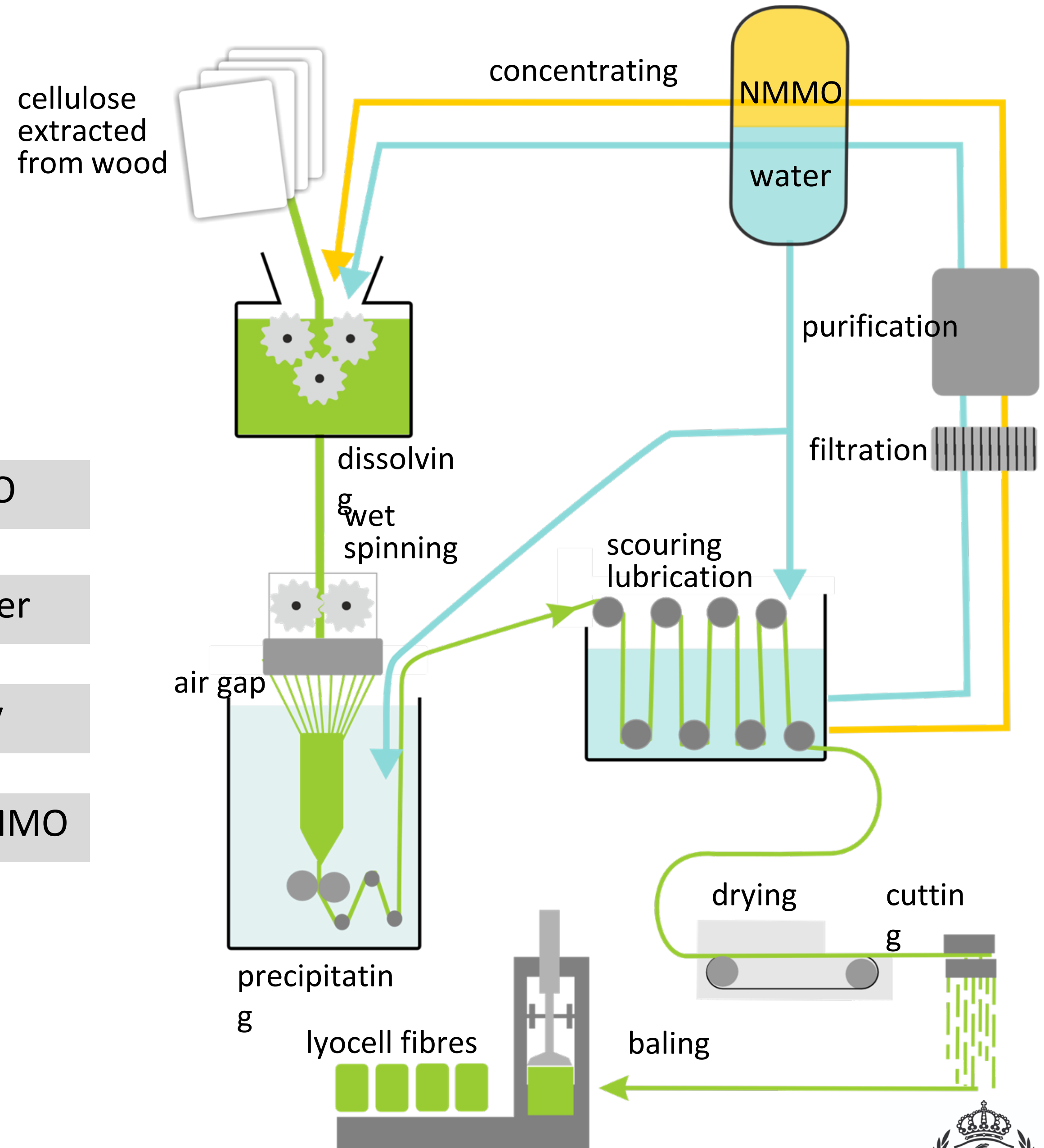
Lyocell fibres

Lyocell Rayon Production





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TAB, *2015-09-23. Image of Lyocell production and recycling of water and NMMO, presented by SusTexEdu*

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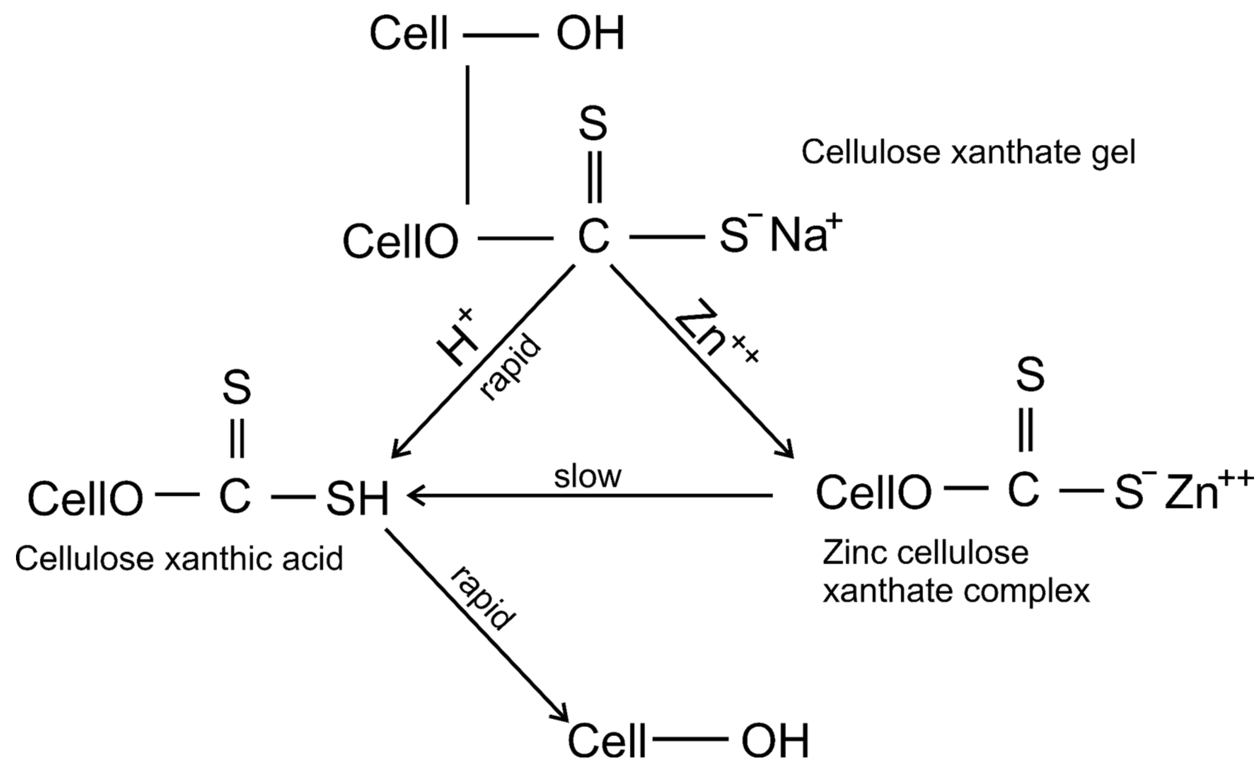
… viscose rayon production

Spinning with modifiers

- The viscose fibre properties can be changed by controlling reaction conditions and other parameters.
- The most important modifier is zinc which is used in spinning bath.
- 95% of the rayon produced today is spun with zinc in spin bath.



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… viscose rayon production

- With modifiers high strength viscose yarns such as, Tyre yarn, High wet-modulus (HWM) yarn and High tenacity viscose yarn can be produced.
- Without or low concentration of modifier Polynosic viscose yarn can be produced.

Tyre Yarn

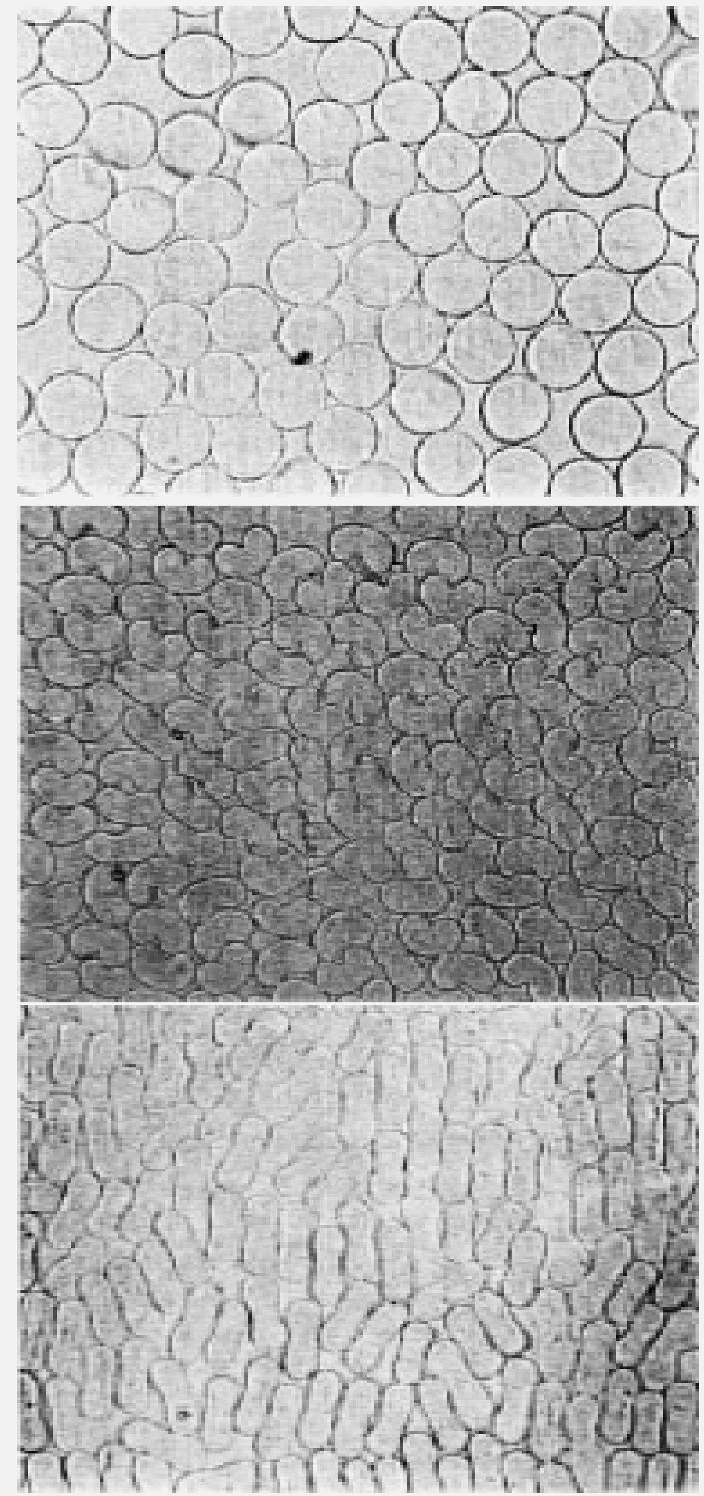
- A viscose solution of viscosity 100 poise containing modifiers 1-3% by weight of cellulose and with a CS2 content of 40% is spun into spinning bath containing:
  - H_2SO_4 8-10%
  - Na_2SO_4 16-24%
  - $ZnSO_4$ 6%
- The spin bath temperature is kept around 55 ^{0}C and the spinning speed is between 40 m and 60 m/min. The stretch applied is 75-125%.

Cross-sectional shape

Round: wet spun and slow coagulation

Bean: wet spun and fast coagulation

Dog-bone shaped: dry spun



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Dry spinning process

What is dry spinning?

“Dry spinning is the fiber formation process that transforms a high vapor pressure polymer solution to a solid fiber by controlled solvent evaporation in the spin line.“
(Imura Y., Hogan R.M.C. & Jaffe M. 2014)

What types of fibres are produced by dry spinning?

Dry spinning is used to produce fibres from polymers such as cellulose acetate, cellulose triacetate, polyvinyl chloride (PVC) polymers and copolymers of vinyl chloride (VC), and acrylonitrile etc. acrylics, spandex, PBI fibres also produced
by this process.

Why would we use dry spinning?

Dry spinning is used when the polymer is vulnerable to thermal degradation, and can not form thermally stable or viscous melts, or when specific surface characteristics of the filament are required.

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...dry spinning process

The basic steps

1. Dissolving polymer in a volatile solvent, which is blended with different additives to form a dope.
2. Dope is filtered and pre-heated, to achieve correct consistency.
3. Dope is then extruded through the spinneret into the evaporating cabinet which contains pre-heated gas, air (acetate) or an inert gas, for example nitrogen (polyurethane).
4. The solvent is evaporating by the pre-heated gases which makes the polymer solidified in the form of filament. Solvent is recovered and reused.
5. The solidified filaments are drawn through drawing rollers to align the polymer chains to get better tenacity.
6. The filaments are wound onto a bobbin or cut into staple fibres.

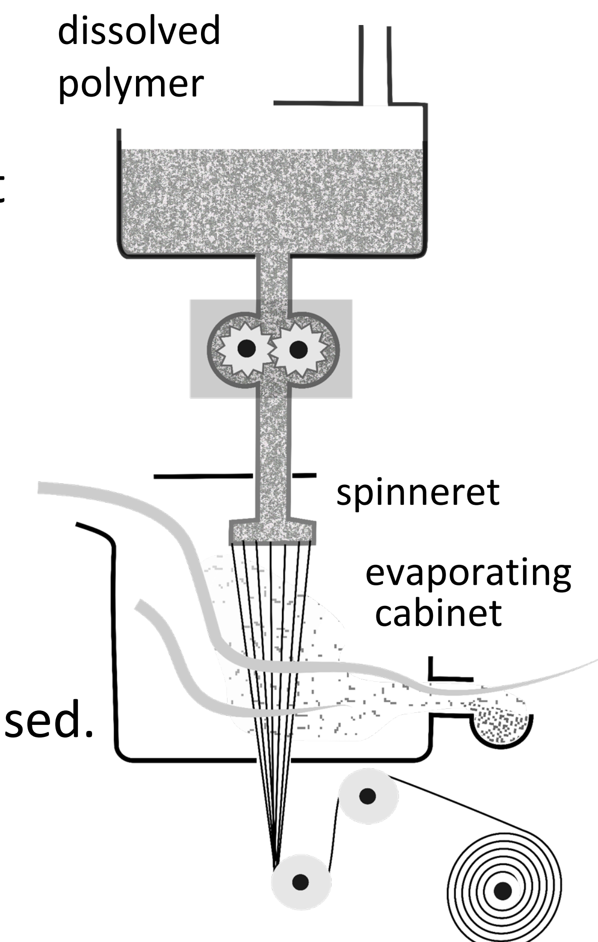

Image of dry spinning: SusTexEdu

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…dry spinning process

- The typical height of the spinning cabinet is about ***3 to 1 m*.**
- The spinning cabinet is flushed with ***pre-heated gas*** (nitrogen, air, carbon dioxide, superheated steam or solvent vapor)
- Fiber surface hardens first, while solvent still diffusing out from the interior.
- ***Irregular cross-sections*** are generally obtained with dry spinning??
- About ***1-50% residual solvent*** is used in solvent cabinet which acts as plasticizer and removed by washing.
- ***Environmental*** issues concerning solvent handling not only limits its uses but also increase the cost of the process.
- PVC, PAN, cellulose acetate, elastane etc.

…dry spinning process

Spinning parameters

- Temperature
- Mass fraction of solvent
- Mass flow rate of solution
- Diameter of spinneret
- Velocity of quench air
- Temperature of quench air
- Length of spinline
- Initial imidization degree of polymer
- Glass transition of polymer
- Heat capacity of polymer
- Heat capacity of solvent
- Take-up velocity
- Density of polymer
- Density of solvent
- Total pressure

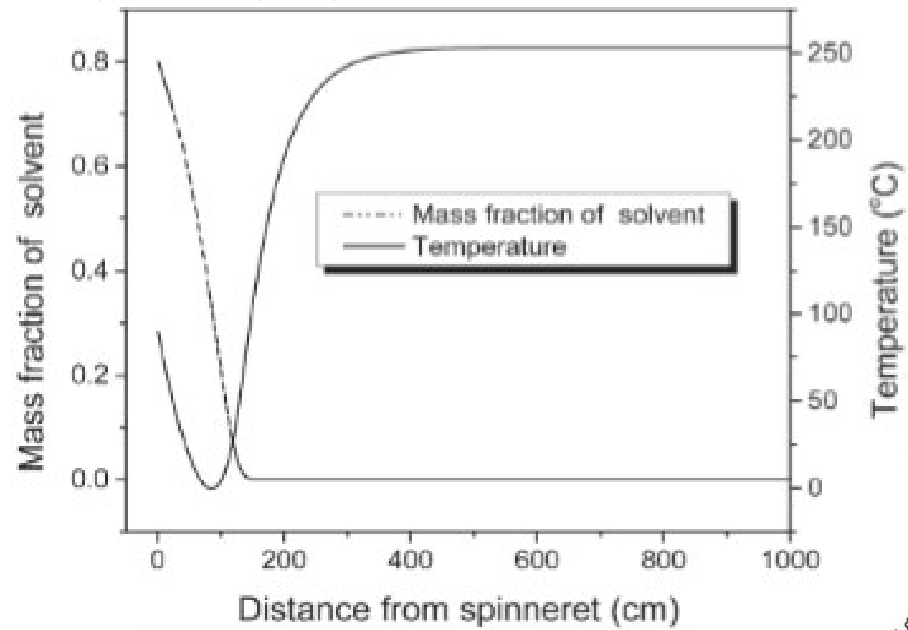
…dry spinning process

Optimum settings for dry spinning

- Solution concentration: 15% - 40%
- Viscosities: 300 - 5000 P (g/cm/s)
- Temperature of the inlet gas:
  100 ° C - 250 ° C
- Spinning velocities: 100 - 1000 m/min
- Production rates: 100 - 800 m/min

Variation in process parameters

Variation of solvent mass fraction and temperature along the spin line for a specific polymer.



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Courtesy: S.M. Patway & H. Ljungholm

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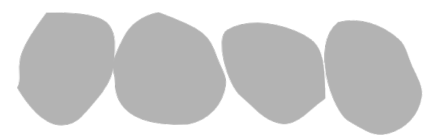
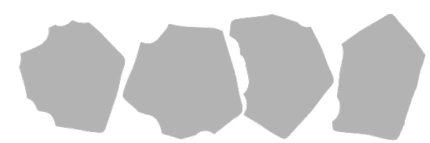
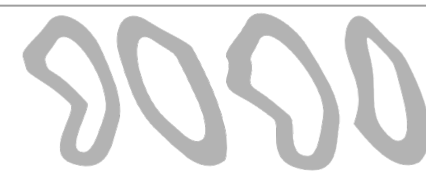
…dry spinning process

General properties of dry spun fibres

- High bulk-to-weight ratio
- Better cover
- Insulating properties in the resulting fabric
- Good dimensional stability with uniform breaking elongation
- Good tenacity and resilience
- Reduced modulus
- Better tensile properties than melt- and wet spinning

Irregular cross-sections are generally obtained with dry spinning?

Fibre cross sectional shape:	Ratio: evaporation rate / diffusion rate:
	- 1
	>1
	>>1

SusTexEdu Courtesy: S.M. Patwary & H. Ljungholm

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Gel spinning process

❖ Gel spinning also known as *semi-melt spinning*, is used to get high-strength and high elastic fibres in the gel state.

❖ A polymer solution or plasticized gel is extruded through the spinneret and cooled in solvent/water and stretched.

❖ Spinning process is divided into ***four steps:***

1. ***Dissolution:*** Dissolution of polymer in solvent can *disentangle* the polymer chain entanglements which aid the gel fibre stretching.
2. ***Spinning and formation:*** Spinning through spinneret, cooling by air or water lead the crystal formation and retain the disentangled state of polymers.

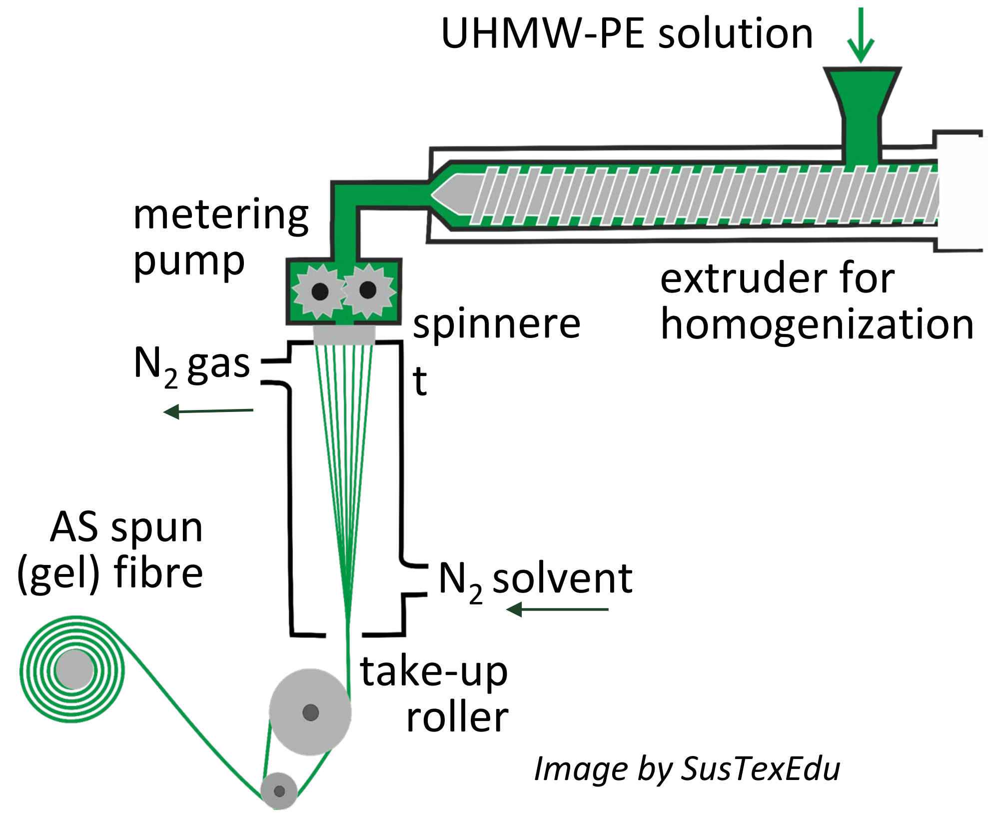


Image by SusTexEdu

1. ***Solvent removal:*** Natural drying method and use of extractant are more commonly used method.
2. ***Stretching***: *High magnification stretching* can change the folded polymer chain to straight chains, improve the crystallinity and orientation. This stretching enhances the fibre performance.

… gel spinning process

Comparison with wet and dry spinning

- Raw materials of high molecular weight

  Polymer of high mol. wt can withstand at higher stretching conditions which also reduce the defects in the fiber structures.

- Dilute solution

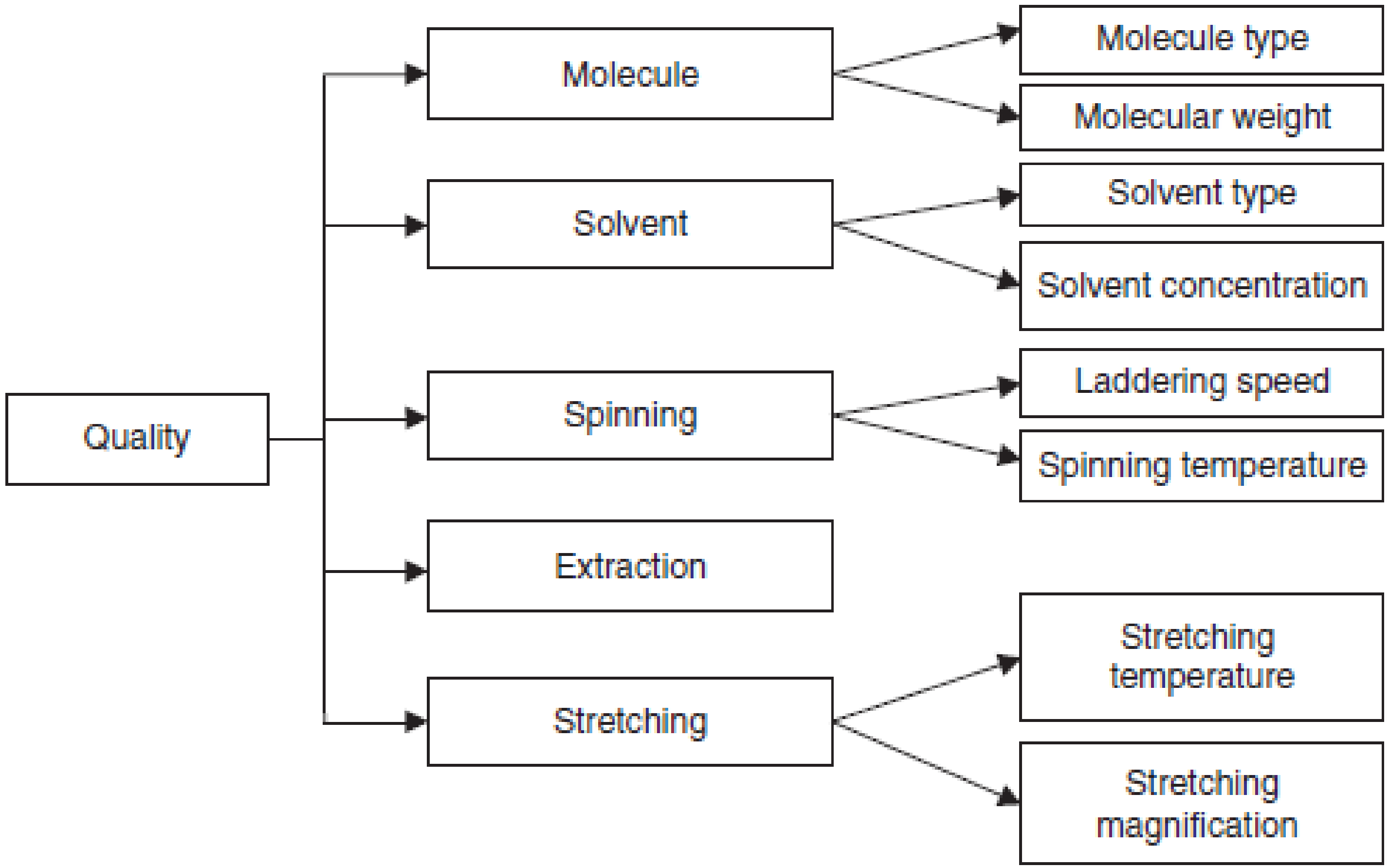
  Dilute solution reduces the gel viscosity, improve gel fiber spinning, and enhance the disentanglement of high polymer chains.

- High stretching

  Its unique feature of gel spinning which can change the folded polymer molecular chains to straight chains and introduce highest level of mechanical properties as compared to other spinning methods.

… gel spinning process

Factors affecting on gel spinning



 Gel spinning of synthetic polymer fibres. J.Kuo, W.I.Lan (2014)

Stretching extension effects

Low

- Crystals gradually change into *microfibrillar*.
- Orientation, gradually rising strength and crystallinity.
- Orientation and crystal *growth slow down.*

Half

- *Spot defects* occur on the surface.
- Molecular chains become gradually intense.
- Fibre internal crystallization starts to change.
- Make the arrangement of *microfibrillar denser.*
- Increase of number of molecular chains in the unit area.
- *Fibre strength and modulus* will be enhanced to the limit

Max.

- Crystallization is damaged and will *break the fibre.*

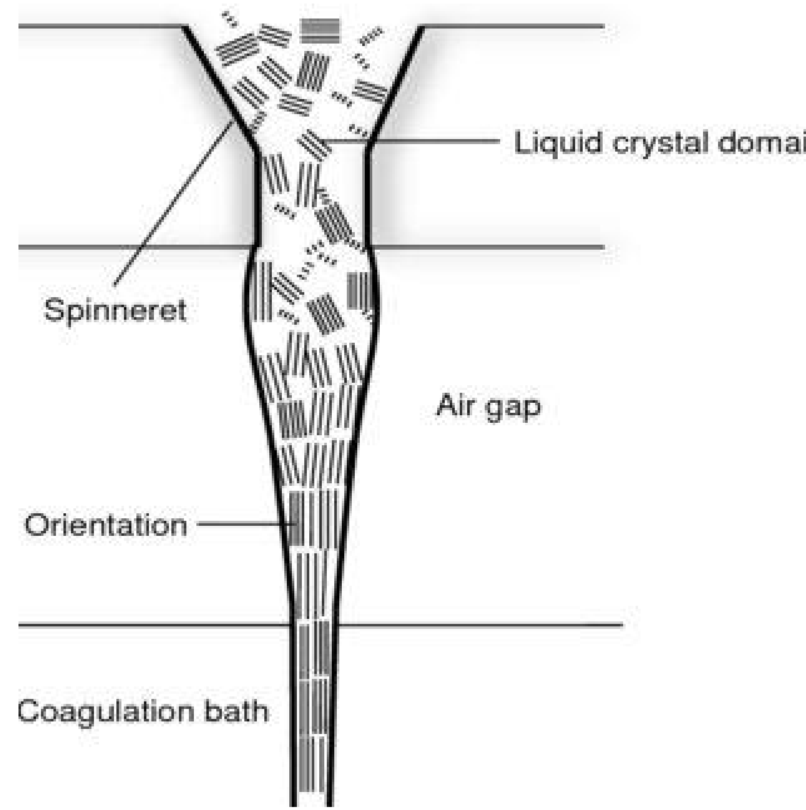


Image from publication: High Strength and High Modulus Electrospun Nanofibers by J. Yao, C.W.M. Bastiaansen and T. Peijs (2014)

SusTexEdu Gel spinning of synthetic polymer fibres. J.Kuo, W.I.Lan (2014)

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Electrospinning process

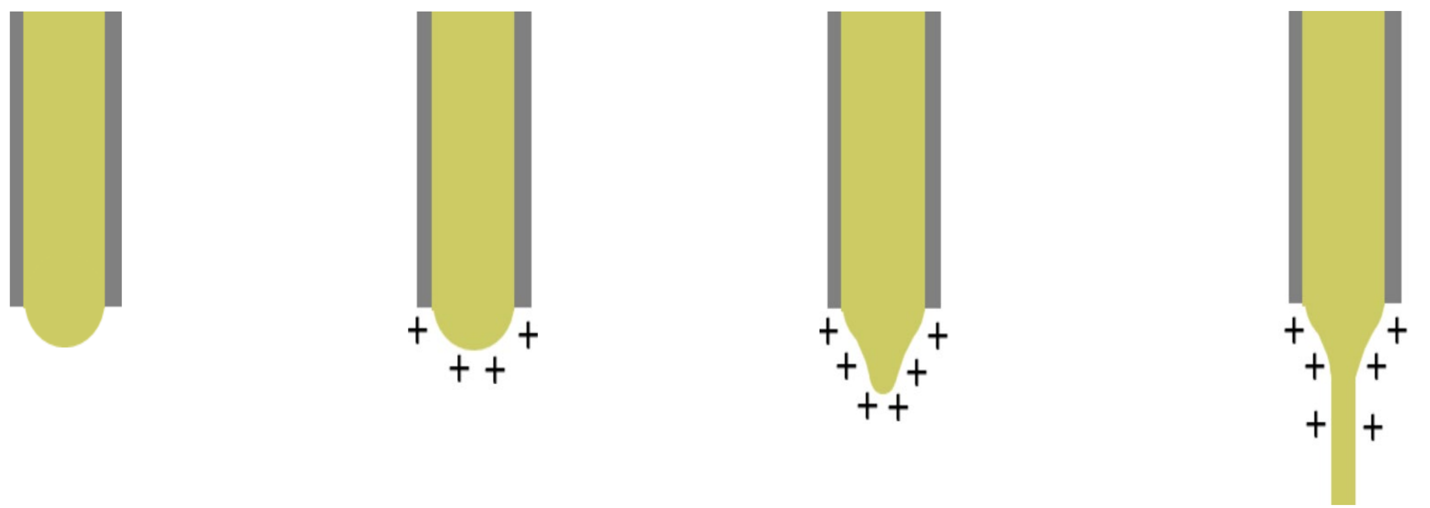
- Electrospinning uses electrostatic forces to produce fine fibres from polymer solutions or melts.
- Characteristic for this method is that the fibres produced have a *thinner diameter* and a *larger surface area* than those obtained from conventional spinning processes.
- Basic Principle

  "Strong mutual electrical repulsive forces overcome weaker forces of surface tension in the charged polymer liquid".

VIDEOS to be found with e.g. keywords: *Nanoah Electrospinning*

… electrospinning process

Basic principles – Jet initiation



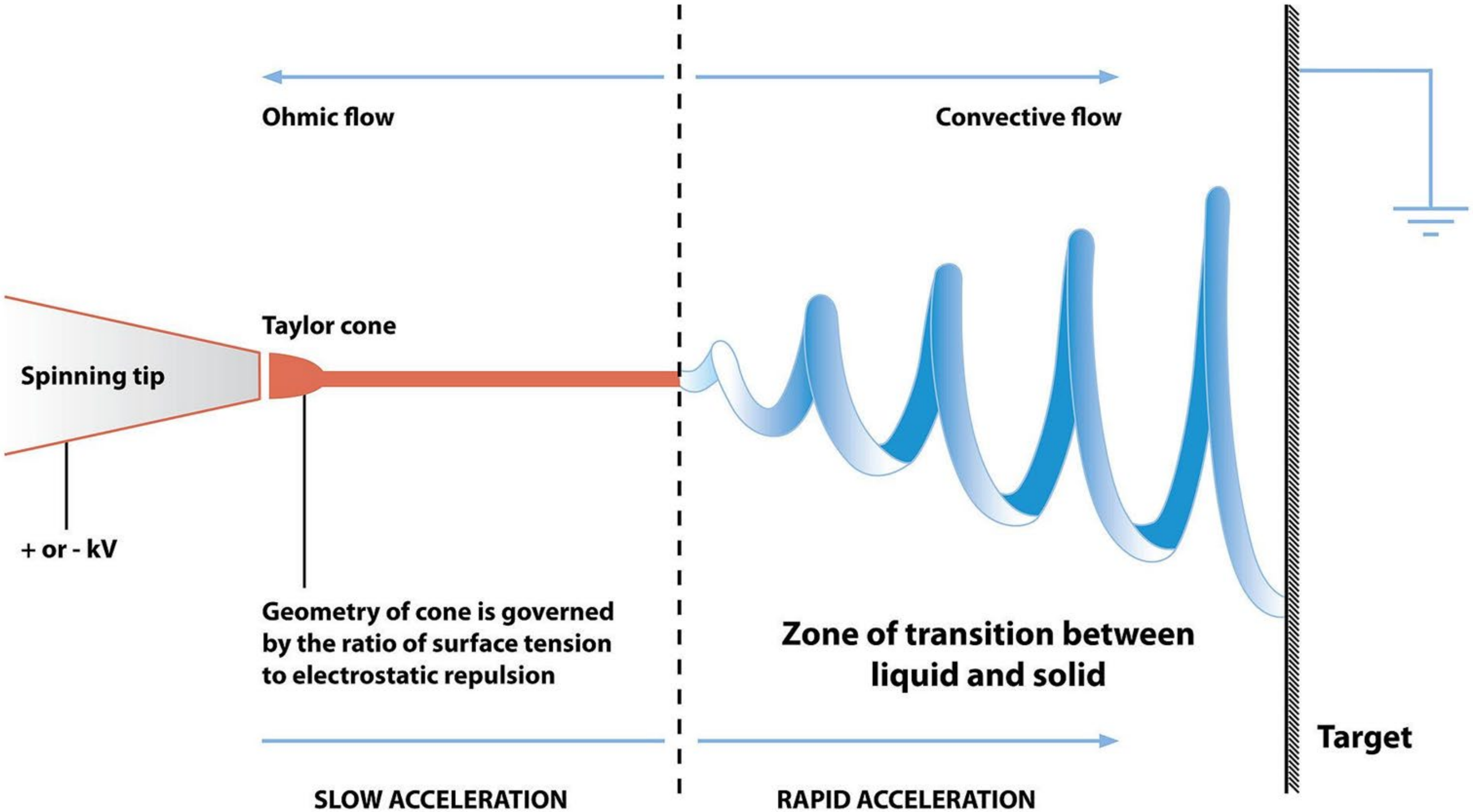
No charges

High voltage

Increasing voltage

Critical voltage
Taylor cone

… electrospinning process



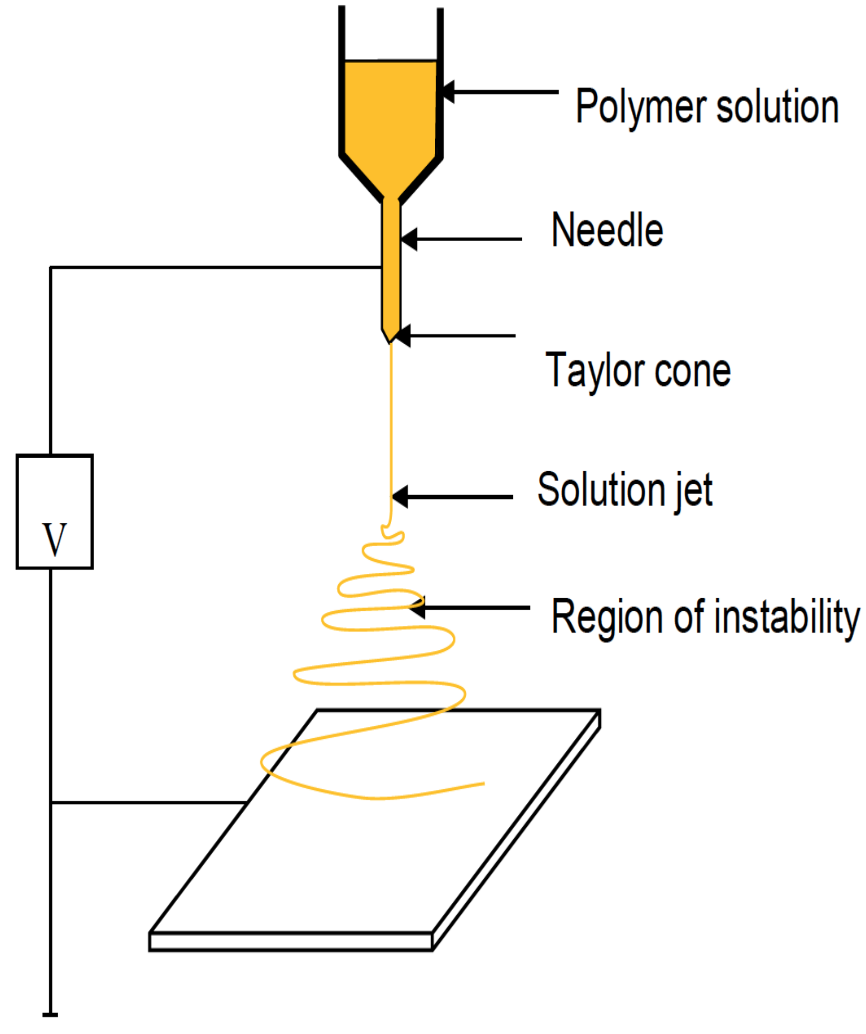
Electrospinning diagram. Image by Joanna Gatford on Wikipedia (CC BY 3.0)

… electrospinning process

Basic principles

Three major components:

1. Power supply
2. Spinneret
3. Collecting plate



… electrospinning process

Solvent used

Two roles:

- Dissolve the polymer molecules
- Carry the dissolved polymer molecules

Polymers	Solvents	Fiber diameter (nm)
Silk fibroin/PEO	Water	590 ± 60
Gelatin	Acetic/Formic acid	109–761
Collagen type I	HFP[a]	100–600
Collagen type II	HFP	496
Gelatin/PVA	Formic acid	133–447
Chitosan	Acetic acid	130
PVA	Water	250–300
Chitosan/PVA	Formic acid , TFA[b], HCl	330
Cellulose acetate	Acetone, DMF[c], Trifluoroethylene (3:1:1)	200–1000
HA/Gelatin	DMF/Water	190–500
Fibrinogen	HFP	80 ± 30
Polyamide-6	m-Cresol + Formic acid	98.3 ± 8.2
Polyurathane	Water	100–500
Polycaprolactone	DMF + Methylene chloride	200
Collagen/chitosan	HFP/TFA	300–500
Chitin	HFP	163
PCL/Gelatin	TFE[d]	470 ± 120
Polyaniline/Gelatin	HFP	61 ± 13

Courtesy: A. Hogeman & S. Björquist

… electrospinning process

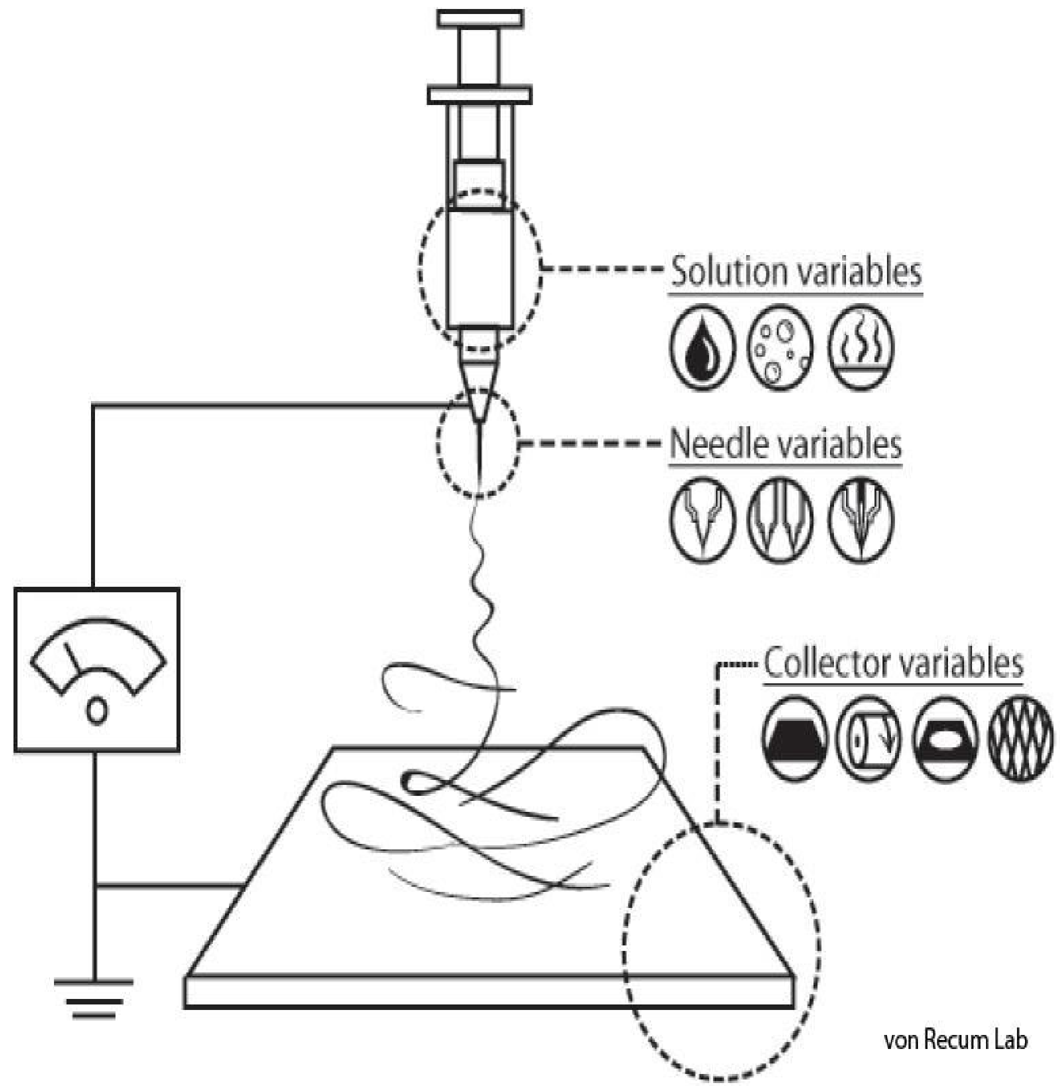
Melt electrospinning

- Advantages:
  - no traces from solvents
  - no required dissolution and recycling of solvents
- Limitations:
  - high viscosity
  - very high processing temperature
  - difficulty in producing fibers in nano range

… electrospinning process

The electrospinning parameters

- Solution parameters
- Process parameters
  - concentration
  - molecular weight
  - viscosity
  - surface tension
  - conductivity
- Ambient parameters



Electrospinning/electrospraying schematic with variations for different processing outcomes.
Image by Delv0n2 on Wikipedia (CC BY-SA 3.0)

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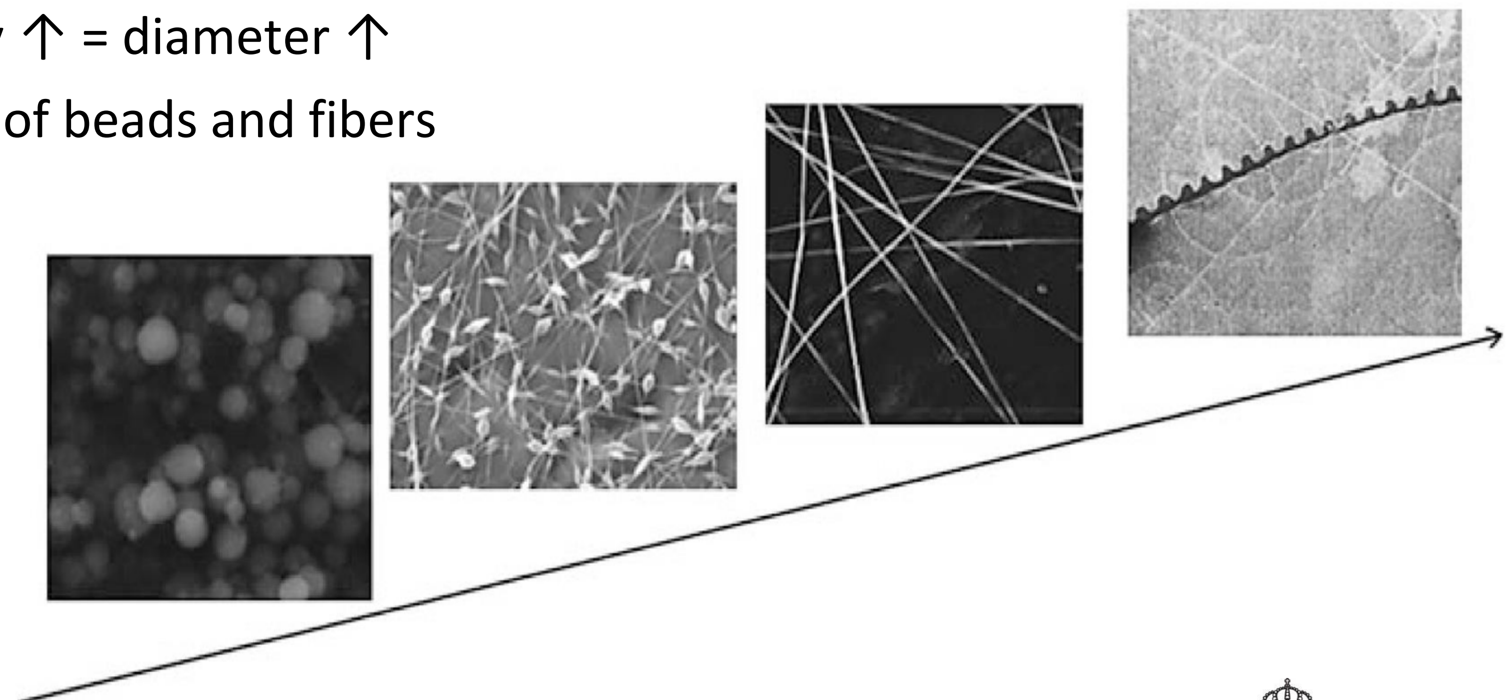
Solution parameter

Concentration

- Concentration ↑ = viscosity ↑ = diameter ↑
- Concentration ↓ = mixture of beads and fibers



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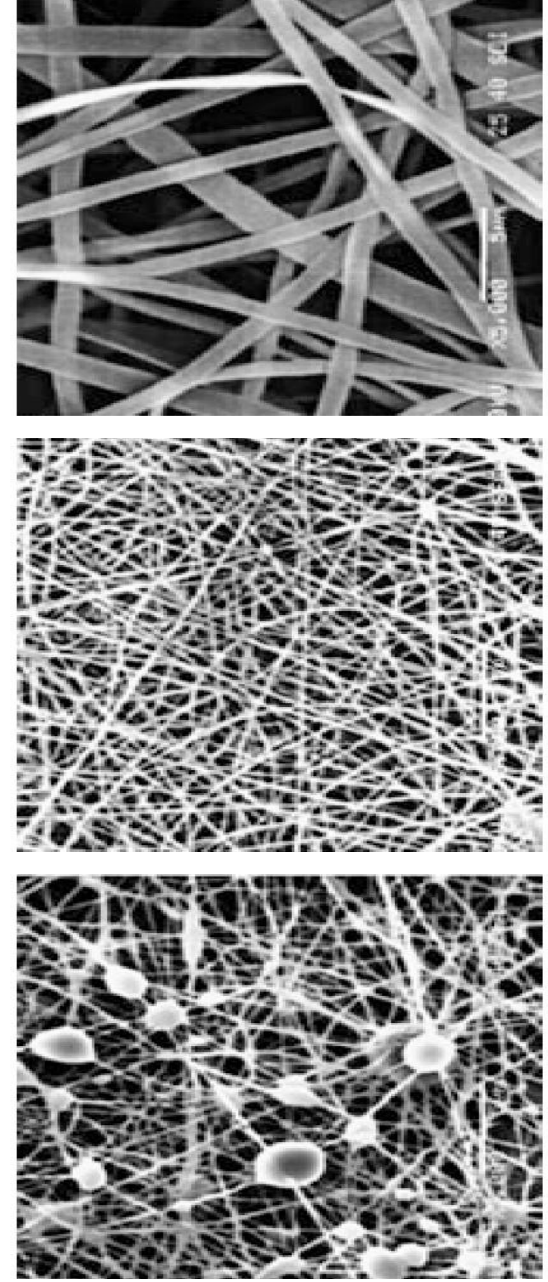
Solution parameter

Molecular weight

- Molecular weight ↑ = desired viscosity, no beads, large average diameter
- Molecular weight ↓ = beads forming rather than fibres
- Increasing molecular weight



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… electrospinning process

Solution parameter

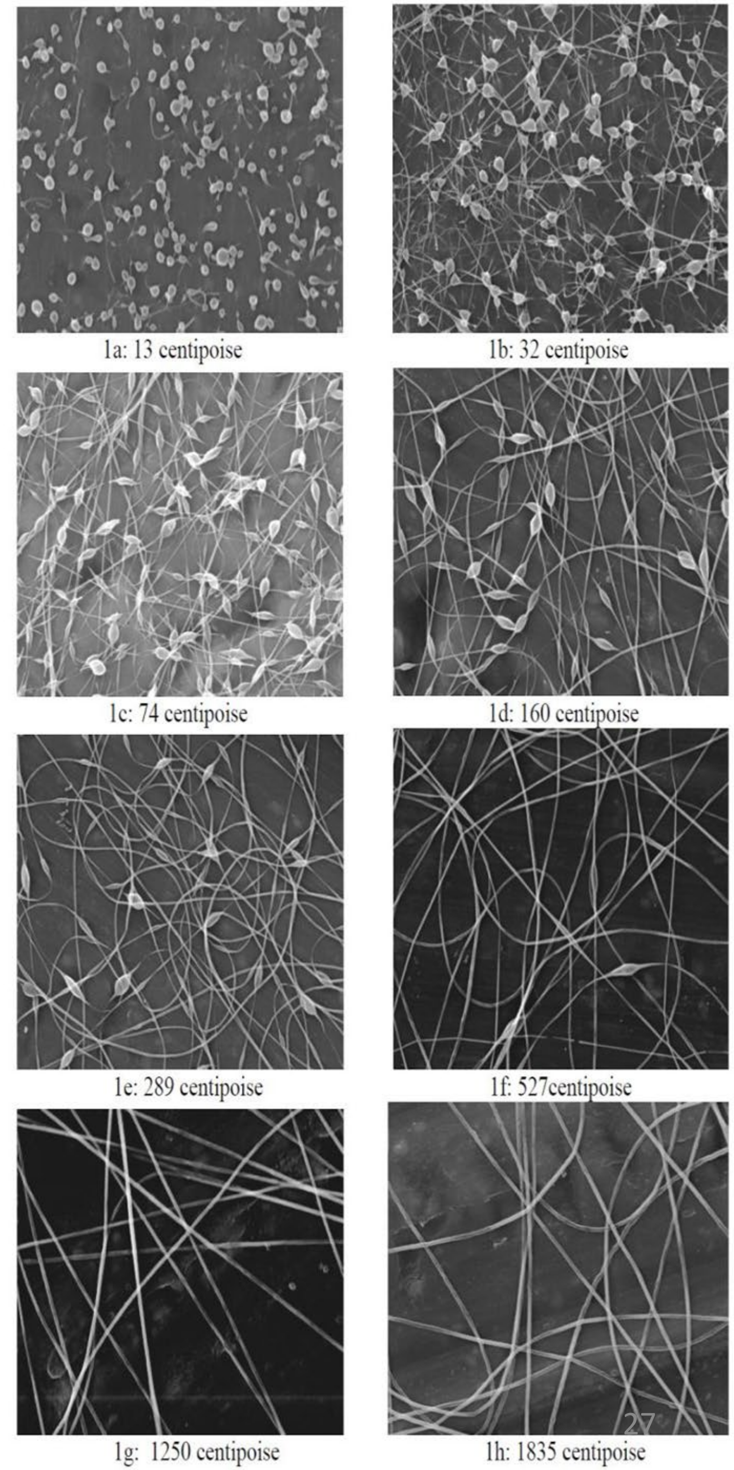
<u>*Viscosity, η*</u>

- very ↑ η = no fiber formation
- very ↓ η = no ejection of jet

Increase in solution viscosity

-> uniform fiber diameter, few beads

Viscosity is polymer specific!



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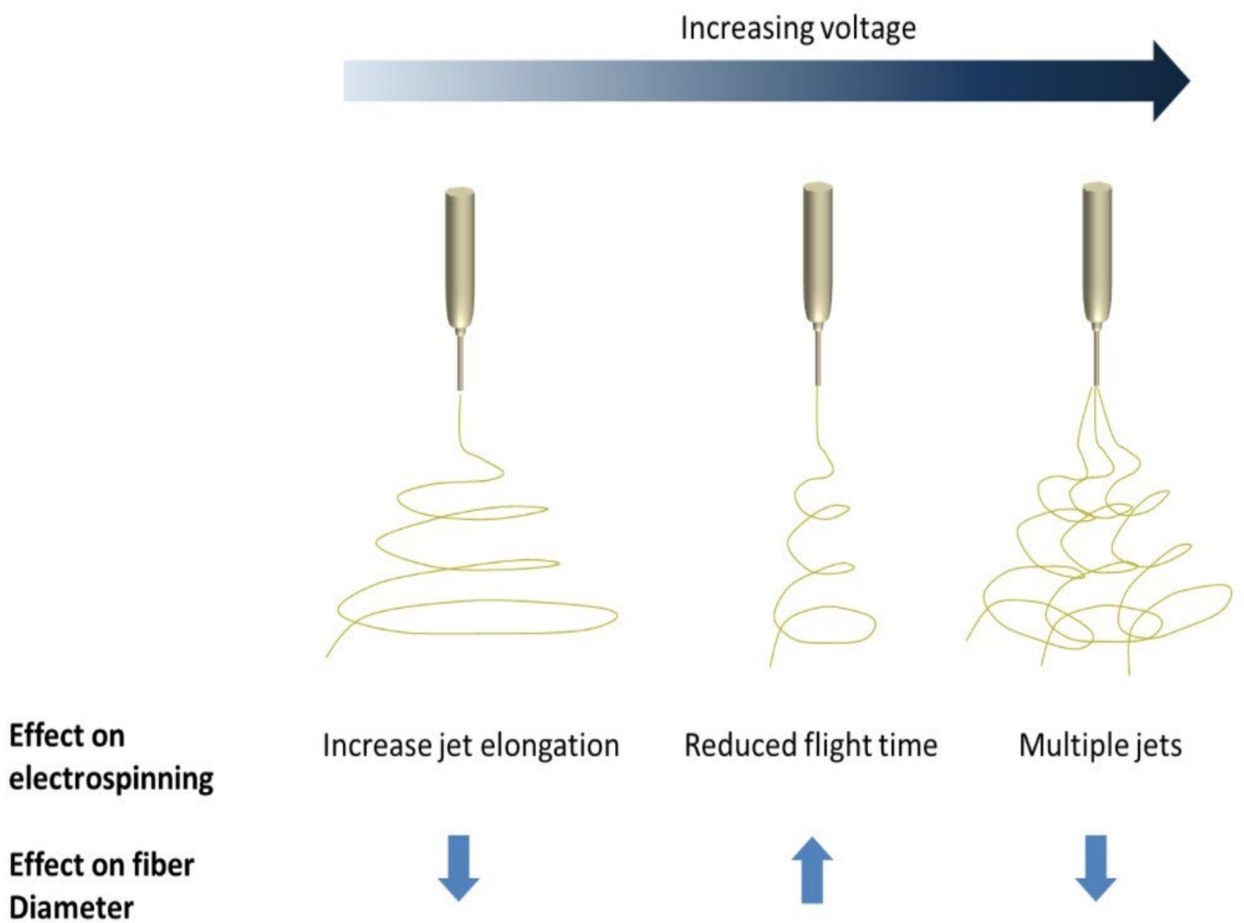
… electrospinning process

<u>The electrospinning parameters</u>

- ❖ Solution parameters
- ❖ Process parameters
  - ➢ applied voltage
  - ➢ feed rate/flow rate
  - ➢ types of collectors
  - ➢ tip to collector distance
- ❖ Ambient parameters



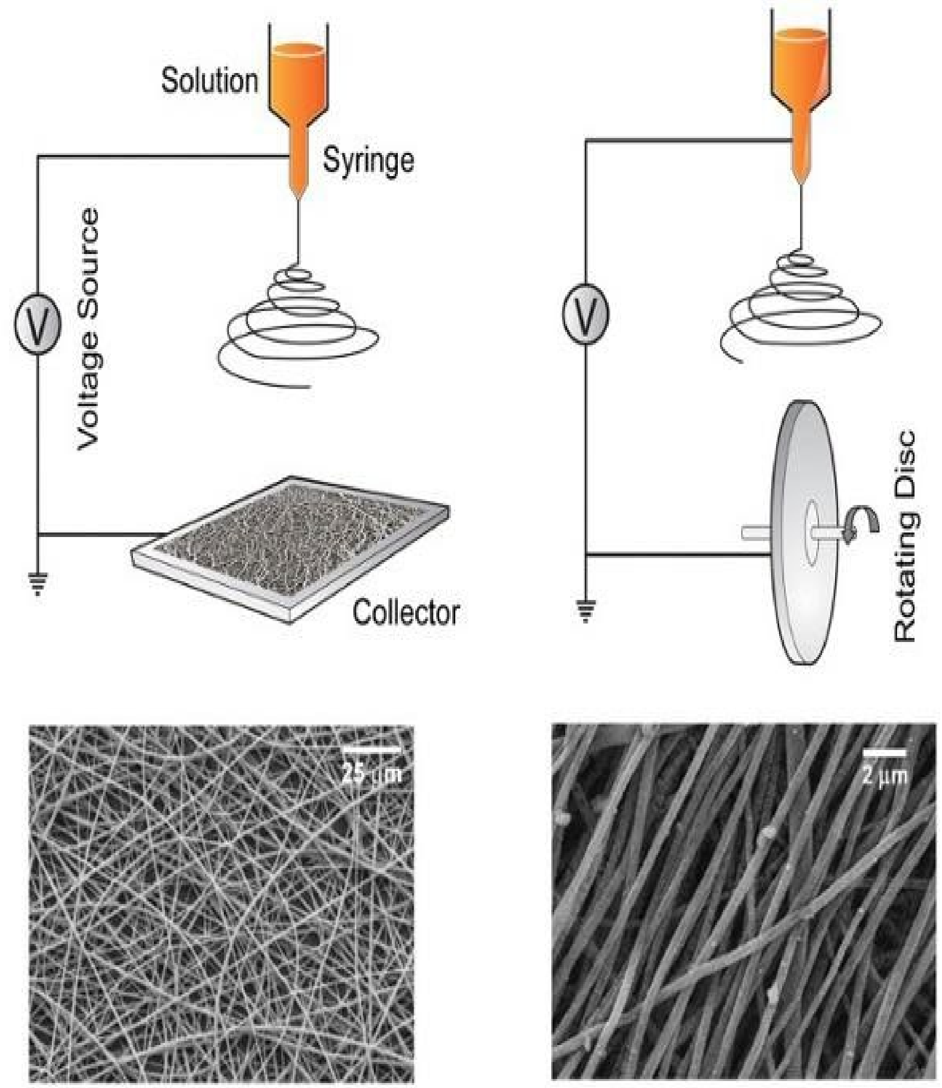
… electrospinning process

<u>The electrospinning parameters</u>

Types of collectors

Arrangement and final structure

↓ conductive area = beaded fibres



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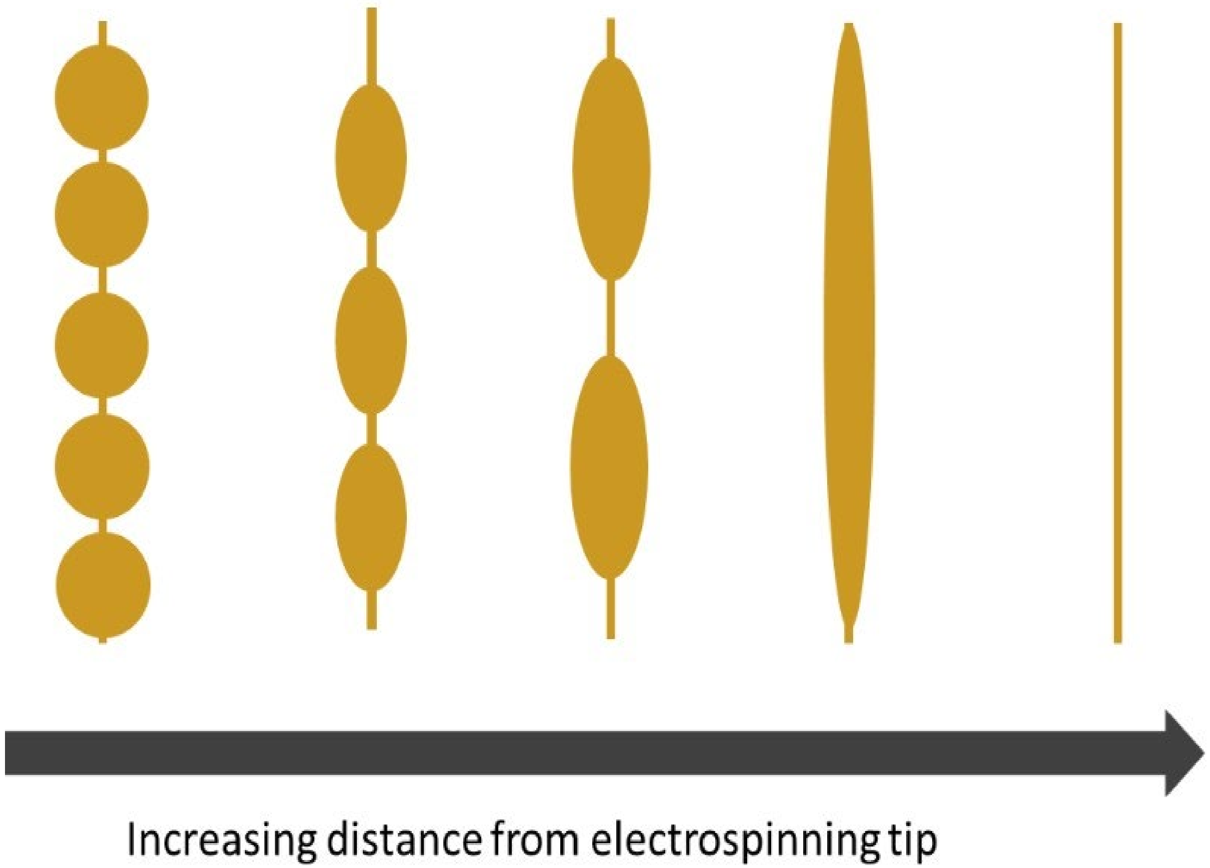
… electrospinning process

<u>The electrospinning parameters</u>

Tip to collector distance

- Solvent evaporation
- Beads forming



Regenerated Fibres

Introduction

- RF is a class of man-made fibres which are produced from naturally occurring polymers.
- These polymers don’t occur naturally as fibres and several physical and chemical processing steps are needed to convert them into fibre form.
- The starting materials for regenerated fibres are cellulose and protein.

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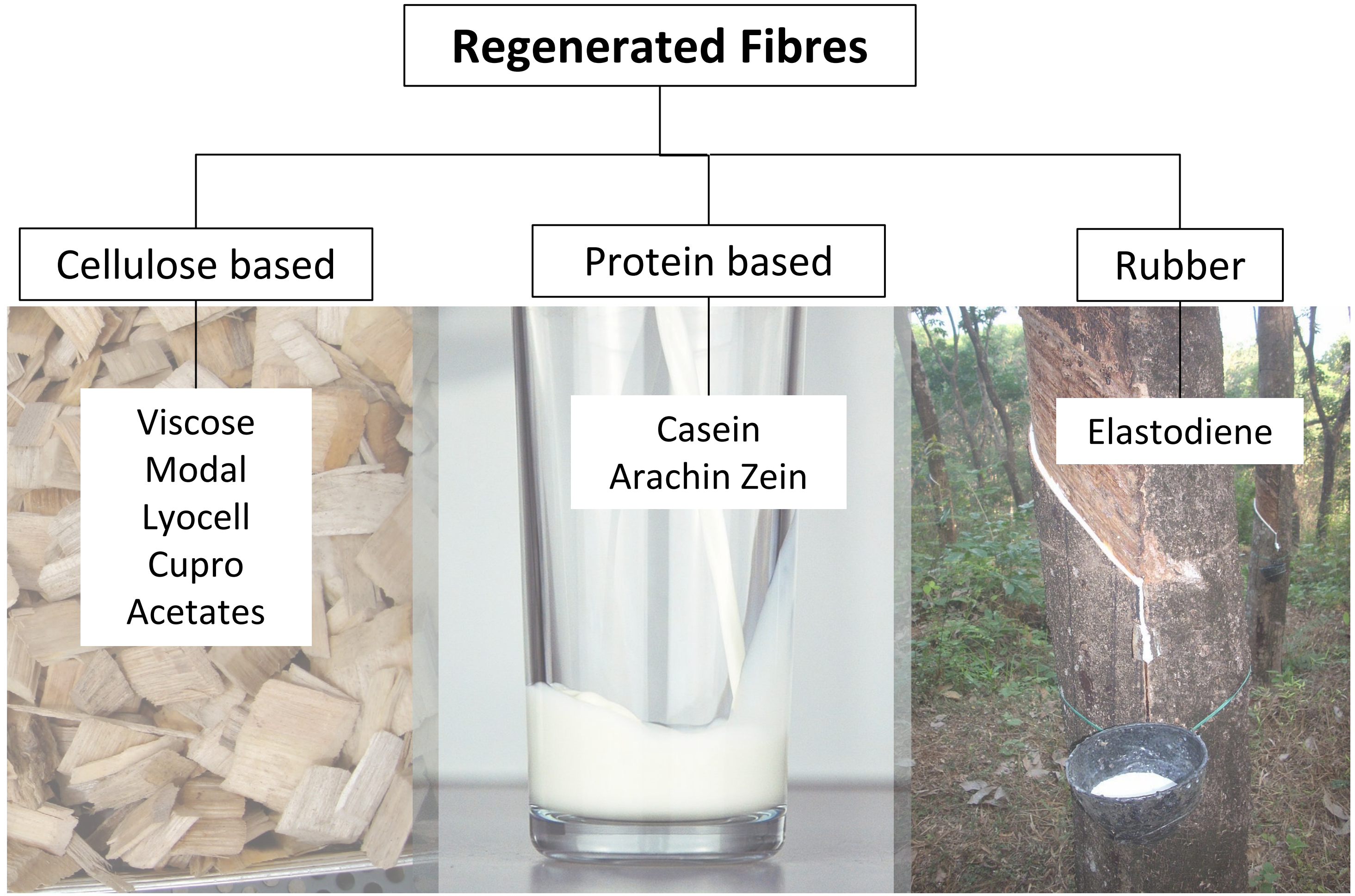
Types of regenerated fibres

The most important of them are ***cellulose*** based regenerated fibres, being widely used for apparel and industrial applications



Photos : Wood chips by SusTexEdu. Milk glass by Stefan Kühn on Wikipedia (GFDL) and Rubber tree by Manojk on Wikipedia (CC BY-SA 3.0)

Regenerated cellulose fibres

- Regenerated fibres of cellulose origin, are made of cellulose originated from mainly wood, cotton and bamboo.
- These are the first man-made fibres and also known as ***artificial silk*** during the early years of production.
- RCF have ***advantages*** over natural cellulosic fibres and can be produced with ***different properties***.
- However, the ***environmental*** and ***sustainability*** issues are major concerns.

Photo by SusTexEdu

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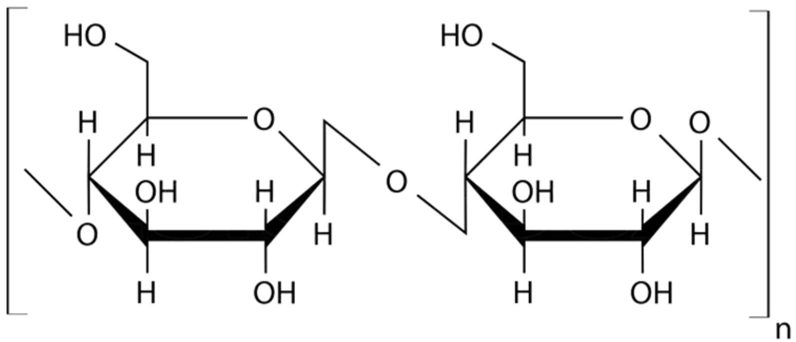
Viscose Rayon Fibres

- **Viscose rayon** fibres were first developed during the 1850s and production process was patented in 1892.
- The generic name, **Rayon,** was adopted by U.S. Department of Commerce in 1942. "**Ray-**" (ray of light) indicates fibre brightness and "**-on**" represented the cotton like structure.



Cellulose

- It has faced a strong challenge from synthetic fibres like nylon, polyester and acrylics but retained its place as major textile fibres.

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VIDEOS to be found with keywords e.g.: *The Viscose Fibre Line* and *Viscose Rayon, manufacturing process, properties and uses*

… viscose rayon fibres

- Rayon fibres have *bright, smooth* and *lustrous* appearance much like silk, *hygroscopicity* and easy *dyeability.*
- They can be produced with wide range of properties, particularly mechanical properties, so far unmatched by any other fiber, natural or manufactured.
- The common sources for rayon production are *wood pulp* and *cotton lint.*
- Highly purified wood pulp consists of *90-95%* cellulose called *chemical cellulose* and dissolving pulp.
- *Cuprammonium hydroxide* was first used as solvent for cellulose dissolving which was replaced with viscose process due to high cost, lack of stability and solvent handling.
- Viscose process is still the predominant method for rayon production.

… viscose rayon fibres

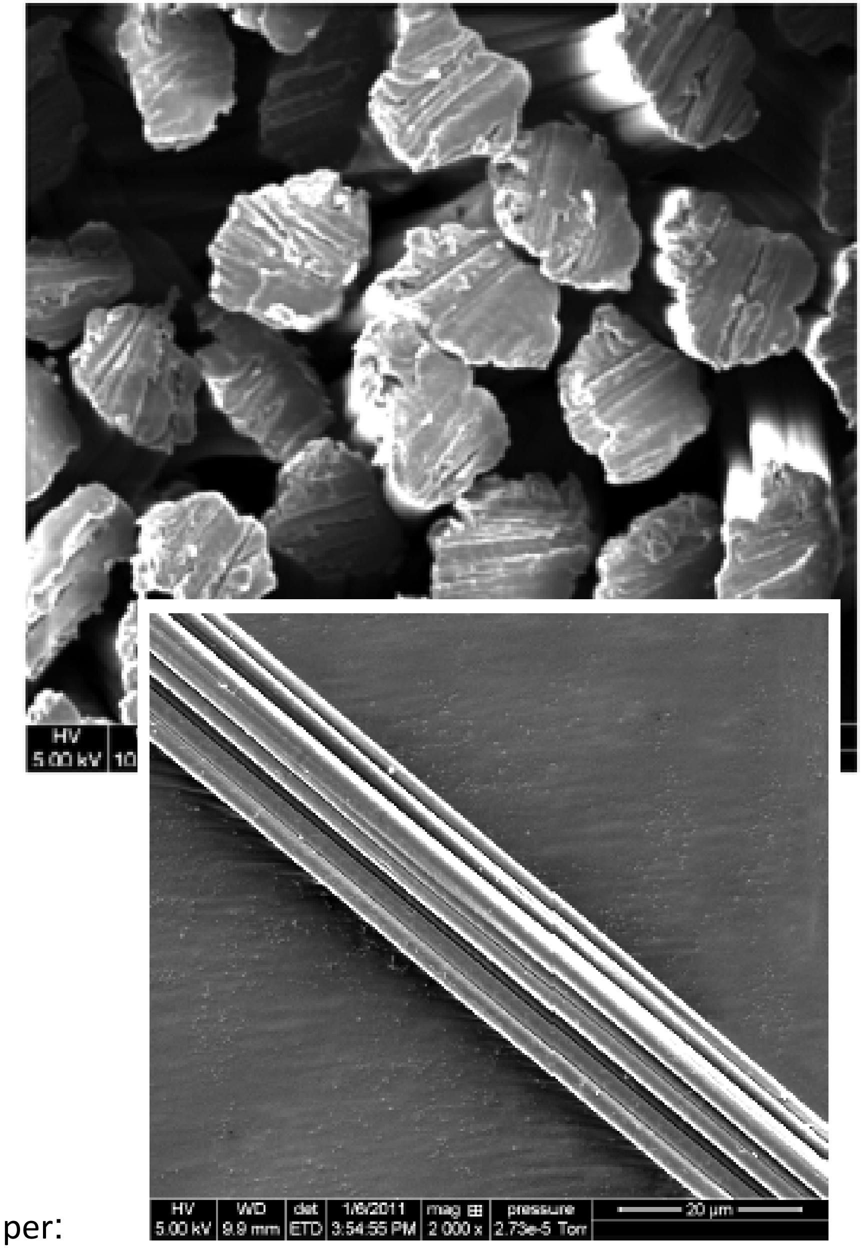
Environmental and Sustainability Concerns

- Rayon is produced from naturally polymers obtained from *softwood trees* (fast growing) and mature trees from forest.
- *Clear cutting* of trees has diversified effects on environment.
- Wood pulp processing uses large quantity of acid and other chemical that may pollute air and water.
- Air emission of *sulfur, nitrous oxides, carbon disulfide* and *hydrogen sulfide* produce high pollution.
- *Chemical removal* and reuse them makes overall process *costly.*
- With advancement, *99% waste liquor* can be *recovered.*
- Viscose is *biodegradable* and also recyclable.
- Disposal of viscose products used as disposable diapers or sanitary products is problem.

Properties of viscose rayon fibres

Physical structure of Rayon

- Regular viscose is characterized by lengthwise lines called *striations.*
- Cross-section resembles a *distorted circle* with a serrated contour.
- Filament rayon yarns range from 80 to 980 filaments per yarn and vary in size from 40 to 5000 denier.
- Commercial used fineness of staple fibres range from 1.5 to 15 denier.



Images from full paper:
High Temperature Composites From Renewable Resources: A Perspective on Current Technological Challenges for the Manufacturing of Non-Oil Based High Char Yield Matrices and Carbon Fibers

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... properties of viscose rayon fibres

Mechanical properties

- Tenacity ranges between 2.6 to 3 g/den when dry and 1.2 to 1.8 g/den when wet.
- Has lower tensile strength under wet conditions than dry conditions but have high elongation up to 30% under wet conditions, which make fabrics stretchable.

Table 4.1 Viscose rayon fiber tensile strength and elongation

Fiber	Tenacity (g/denier)		Elongation (%)	
	Dry	Wet	Dry	Wet
Viscose	2.6–3.1	1.2–1.8	20–25	25–30
HWM	4.1–4.3	2.3–2.5	13–15	13–15
Tencel	4.8–5.0	4.2–4.6	14–16	16–18
Cotton	2.4–2.9	3.1–3.6	7–9	12–14
Polyester	4.8–6.0	4.8–6.0	44–45	44–45

Courtaulds Fibers Inc., 1999.

Lyocell fibres

- Lyocell fibre (U.S. brand name *Tencel*) another type of regenerated cellulose fibres made from wood pulp.
- The production method is totally different than viscose process.
- Due to environmental concerns of viscose process and an environmental friendly solvent ***N-methylmorpholine-N-oxide (NMMO)*** is used for cellulose dissolution.
- Its new generation of regenerated fibres with environmentally friendly processing and improved properties.
- Only **5%** of the rayon fibre market is lyocell.

Lyocell fabric. Photo by SusTexEdu

VIDEOS to be found with keywords e.g.: *TENCEL, Lyocell, production*

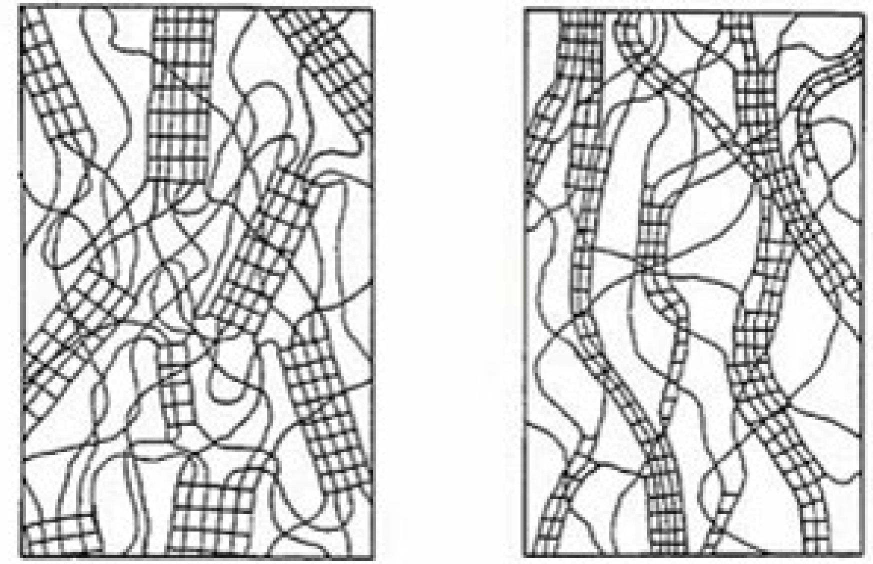
… lyocell fibres

- The dissolution of cellulose in NMMO was started during *1960s and in 1992*, in Mobile Alabama in USA, the maximum production of lyocell was started with trade name *"**TENCEL**".*
- ***Lenzing AG*** in Austria is another major *European* company engaged in the production of lyocell fibre.
- As compared to viscose process, it is a ***green technology*** that eliminates toxic chemical used for chemical reactions and reduce air and water emissions.
- **Lyocell fibre appearance**
  - lyocell fibre has a close to *circular cross-section.*
  - longitudinal surface is very smooth and cylindrical *without any striation.*
  - due to different fibre appearance than viscose, lyocell rayon fabrics exhibit better fabric feel and drape.

Properties of lyocell fibres

Mechanical Properties

- Lyocell fibres with *higher dry* and *wet tensile* strength can be produced with lyocell spinning technology.
- *Dry density* of lyocell is *larger* than that of *viscose* and *HWM rayon* fibres and equivalent to *polyester* fibres.
- It has ability to keep the *85% of dry tenacity* in wet conditions.
- Has *lower elongation* value as compared to viscose rayon fibres.
- Due to high crystallinity, lyocell fibre features a fibrillar structure.



(a) Fringed micelle (b) Fringed fibril.

Fibrillation of lyocell fibre. Image by W. Zhang on Wikipedia (CC BY 3.0)

Sustainability of lyocell fibres

Environmental and sustainability concerns

- *Closed loop* manufacturing process that recovers and reprocesses *99.5%* of the amine oxide solvent.
- *Non-toxic* and *non-corrosive* chemicals are used.
- Fast growing *eucalyptus* trees are used for lyocell production.
- *Biodegradable* and recycled.

Lyocell®

Uses of lyocell fibres

Applications of lyocell

- Lyocell is *more expensive per pound* as compared to viscose rayon.
- Use in professional business wear, hosiery, casual wear, and window treatment fabrics.
- *Blends* with wool, cotton and other manufactured fibers is made to get required properties.
- In manufacturing of glass and other items, lyocell is used in conveyer belts because of its *strength and softness.*

Photos from Wikipedia:
Label of a coat, by Nevermind2 (Public domain)
Lyocell fibres in the mattress cover, by Dobrozhinetsky (CC BY-SA 3.0)

Learning material

1. *"Pearson New International Edition: Textiles"* by Sara J. Kadolph. (ebook)
2. *"Textiles and Fashion, Materials, Design and Technology"* by R. Sinclair, ISBN: 978-1-84569-931-4.
3. *"The Chemistry of Textile Fibers"*, by Robert R. Mather, 2nd Edition

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Visit the project website to see all the intellectual outputs of the project.


Image by M. Amgwerd 2023

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