

# MAN-MADE TEXTILE FIBRE OF NON-WOOD SIDESTREAM BIOMASS

## - GRØNNE HOSER INBIOM PROJECT -

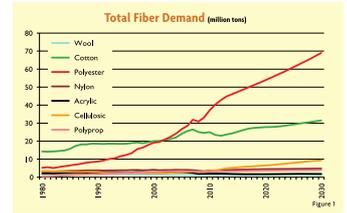
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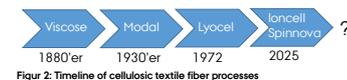
## Introduction

- Global production of textile materials, one of the most polluting industries with an annual production of 100mill tons, is predicted to increase steadily and substantially in the future (fig1)
- Production of cotton, known as "the thirsty fiber" is stagnating (fig1).
- Man-made cellulosic textile fibre, at present 7% of total global fibre volume, is a promising candidate, to meet the increasing global textile fiber demand; as cotton substitute with similar technical properties, but for better environmental score, and as bio-based supplement to polyester (at present 50% of global textile volume)
- Man-made cellulosic fibre are at present mainly made of wood-cellulose, and to a minor degree from bamboo and textile waste. Using wood poses an increased burden to forest eco-system, in particular endangered forest, but also to ecosystem and -diversity by increasing the area of industrial mono-culture forest. Using biomass cellulosic sidestream fractions offers a promising and eco-friendly cellulose source.
- Man-made cellulosic fibre can be made by a number of different methods; the earliest and most widespread one being the viscose-process (fig2). Where the later modal (or HWM) process is a modification of the viscose process, then the Lyocell® process is fundamentally different; direct dissolution of cellulose by green solvent (NMMO), in stead of indirect dissolution in viscose/HWM-process. At present, development of new and improved processes are undertaken in different initiatives, and of different states of commercial maturity (fig2).
- Pulping (refining) non-wood biomass to "marked pulp" (pure cellulose), requires less "harsh" chemical and lower processing temperatures than wood pulping.
- There is an increased demand for green protein for food, feed and non-food-applications, as alternative to meet and soy-protein. Green Protein from grass and other protein crops are promising candidates.
- To have environmentally and economically sound green protein production, and in the future bio-economy in general, valorising the biomass sidestreams is crucial.
- Using the cellulosic sidestream from protein- and plantwax production, for manmade textile fibres offers a promising, sustainable solution to valorise biomass sidestreams at the same time as offering a solution to an increasing demand for a functional, bio-based sustainable solution in the textile industry.
- This project explores the technical and environmental potential of using biomass sidestream from production of plantwax (wheatstraw) and plant-protein (grass, lucerne, and others), by characterising and refining the biomass and using it for lab-scale textile fiber manufacturing. Thorough characterisation of the textile fibres demonstrate technical properties in line with commercially available counterparts.

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**Figure 1: Total fiber demand.** Expected demand of textile fibre, until 2030. Production of cotton is expected to stagnate. Increased demand of textile is expected to be met by substantial increase in production of polyester.  
From <https://www.thefiberyear.com/fileadmin/pdf/TFY2018TOC.pdf>



**Figure 2: Timeline of cellulosic textile fiber processes**

## Methods

**Sidestream biomass: Wheatstraw cellulose (WSC)** obtained by plantwax- and bio-compound extraction and bleaching (outlined to the left), from Jena Trading. Cellulose-content ~95%.

**EmimOAc** (N-ethyl-N-methyl-imidazolium acetate) from Sigma (fig4A).

**Viscosity determination** (ISO 5351:2010; *Pulps -- Determination of limiting viscosity number in cupri-ethylenediamine (CED) solution*) in brief: Viscosity of 0,5% WSC, dissolved in 0,5M cupriethylenediamine (CED), determined by capillary viscometer method.

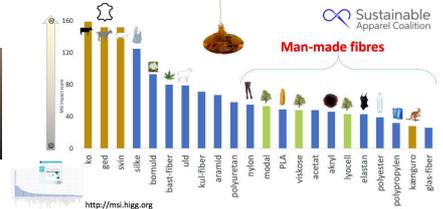
**"Spinnability"**: To determine suited cellulose-concentration and temperature of spinning solution: 5, 10, 15 and 20% WSC dissolved in EmimOAc (fig4B). Elastic and viscous moduli (Pa) determined with Kinexus Rheometer, plotted (fig4B) against angular frequency.

**Textilefiber manufacture:** Air-gap dry-jet wet-spinning with WSC (12%) in EmimOAc, on bench-scale spinning instrument (fig4E), at Swerea IVF. Instruments consists of spin pump, a spin bath and take-up rolls (Fig4E). Extrusion performed through multi-filament spinneret (fig4D) (33 holes, 120 µm capillary diameter) over 10 mm air-gap into spin bath of water or isopropanol at 15°C, and at 3 different draw ratios ( $V_{tu}/V_e$ , fig4E). Fibres were then washed in water for 24 then dried at 80°C for 45 min.

**Tensile testing of textile fibre.** Fiber tenacity (cN/tex), force (cN) and titer (dtex) measured on Vibroskop/Vibrodyn (Lenzing Instruments, Austria) on temperature and humidity conditioned fibres with extension rate of 20 mm min<sup>-1</sup> and gauge length of 20 mm.

**Scanning electron microscopy.** Executed at Navitas, by project group of mechanical Engineer students.

Diameter of textilfibre determined from these microscopy pictures (fig5), of fibre length and cross-section, respectively.



**Figure 3: Environmental assessment of textile materials.** Data for environmental impact by production of different textile materials, from MSI. Material Sustainability Index, MSI-data are based on technical, published environmental assessments, that have been evaluated by the global organisation "Sustainable Apparel Coalition", and is freely available. The higher the MSI score, the higher the negative environmental impact. Green columns: regenerated cellulosic fibre. Yellow columns animal leather. Inset, in left corner is data on all materials in MSI, as well as screenshot of the frontpage of the database.

## Results/discussion

**Viscosity of WSC:** 157 ml/g, which is lower than typical value for spinning pulp (typically 400 – 500ml/g).

**Spinnability assessment** indicate that cellulose concentration around 20% falls in the interval of preferred values (blue shading in fig4B).

**Textile fiber manufacture** can be seen in fig4D; example of manufactured WSC-fiber in overview outline above.

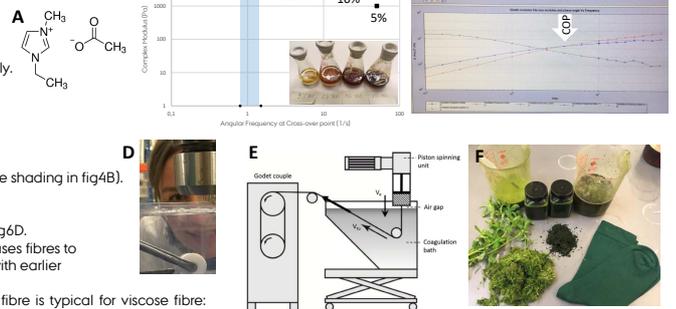
**Tensile testing** of textile fibre results is shown in fig6A-C.

**SEM microscopy** pictures are shown in fig5. Diameters of textile fibres, determined from SEM pictures, are shown in fig6D.

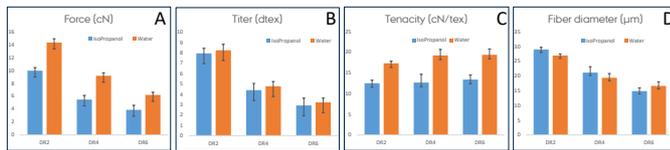
Generally, both strength and titer (linear density) is reduced, with increasing draw ratio. Using water as spin bath causes fibres to ave higher strength and a more regular round shape, than when isopropanol is used as spin bath. This is consistent with earlier experience (pers. comm. with Jenny Bengtson, RISE).

SEM investigation was undertaken on commercially available viscose filament from ENKA. Morphology of these fibre is typical for viscose fibre: irregular cross section and striatal lines lengthwise (data not shown). According to information from provider, the tenacity of the ENKA filaments are 19cN/tex. The strength of the manufactured in this study is in same range (fig6C): strength of IPA-solidified fibre is generally a bit lower, whereas strength of water-solidified fibre generally are similar (fig6C), regardless of draw ratio.

It is interesting that the WSC-fibre made in this study performs so well, with respect to strength, when taking into account that the cellulose is not absolutely pure, the viscosity lower than what is reported to be more ideal and the concentration of WSC gives COP-values outside of the "spinnability" interval (blue areas in fig4B). With improvements on these aspects, for future fiber spinning work, there is good reason to believe that higher-strength fibre can be obtained; fibre with properties well suited for many applications typically meet by conventional viscose fibre.



**Figure 4: spinning of cellulose fibre**  
A) Molecular structure of EmimOAc; a green innovativ strong cellulose solvent. B) Cross-over points of dynamic viscosity measurements at different concentrations of cellulose in EmimOAc. In inset is shown dissolved cellulose i 5, 10, 15 and 20% in EmimOAc. C) Screenshot for measurements of dynamic viscosities. Arrow indicate Cross Over Point (COP) where G' and G'' have identical modulus (Pa). D) Close-up of fiber-spinning in lab-scale, from spinneret, via the air-gap and into the antisolvent solution. E) Schematic illustration of lab-scale air-gap wet-spinning set-up. F) Illustration of different stages in the process from green biomass to final textile product. Fresh lucerne, presscake and protein and protein-juice after screw-pressing of fresh biomass, green socks.



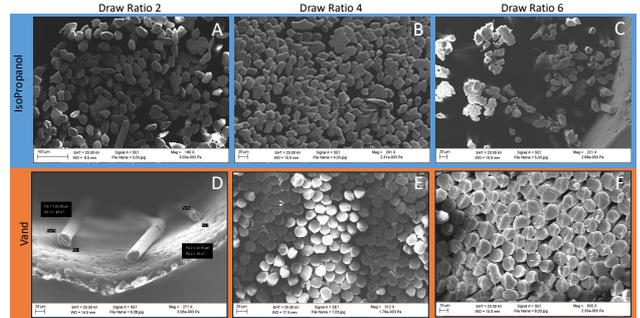
**Figure 6: Technical testing of manufactured cellulosic fibre**

## Conclusion

Textile fiber manufacture of cellulosic fibre, from non-wood sidestream biomass, here wheat straw cellulose, by direct dissolution with innovative green chemistry solvent, EmimOAc, has been demonstrated. Fiber characterisation demonstrates strength, size and morphology to be in the range of cellulosic fiber-alternatives, like ENKA filament, on the market. With rather simple and known improvements, it is most likely that the fiber strength will be even higher, and hence usefull for a broader range of commercial applications.

## Actknowlegdements

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**Figure 5: Scanning electron Microscopy of manufacture cellulosic fibre.**