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REPORT

D4.3 Report describing processing windows for production processes of fabrics made of sustainable fibers (e.g. CelluNova fibers).

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Introduction

The aim of this report was to evaluate possible sustainable alternatives to cotton. A screening on the market was done for biobased fibers and this report describes existing and future potentially more sustainable fibers.

Washing tests have been performed at Swerea IVF on some of the materials described in this report. The results from those tests are presented in the report *D4.4 Report on washability of the developed products*.

Materials and Methods

The table below gives information on the materials that have been used in this report.

Table 1. Materials and background information

Materials	Background information
Cotton	Conventional material in need of biobased complementing alternatives due to the fact that "Peak Cotton" is probably already reached.
Viscose	Regenerated wood cellulose fiber.
Lyocell	Regenerated wood cellulose fiber, closed process. Stronger and less polluting process than fibers from the Viscose process.
Bamboo Viscose	Viscose fiber made from bamboo cellulose pulp.
CelluNova	Prototype fiber from the CelluNova/ForTex projects (2011-2014). Not yet optimized. Regenerated wood based cellulose fiber from using the CelluNova process, which could outperform Viscose and Lyocell from environmental and economical perspective according to ForTex calulations.
Milk protein fiber	Casein based fiber from milk. Qmilk produce from waste milk only. It has possibility to be a sustainable fiber. (Yet to be proven.)
Soybean	Protein fiber with yellow appearance. Difficult to dye.
Seaweed fibers	This regenerated fiber product was made by adding small amounts of seaweed powder to wood cellulose pulp in the viscose process.

CelluNova fibers

CelluNova fibers, which were used in samples described in this report, were produced at IBWCH in Lodz, Poland August 2011.
The owner of these fibers is the For Tex project. But Mistra Future Fashion has got permission to use some of the batches to manufacture fibers into textile samples and demonstrators and to disseminate the results. Unfortunately the amount of fiber that was available was limited. That is why only small scale trials could be performed at this stage.

Nonwoven samples

Staple fibers were manually cut to 38 mm. A defined weight of fiber was placed at the carding machine at Swerea IVF. The fiber web was carded two times, second time with reverse feeding direction. After carding two times the web was needled one time from each side. Fiber used for this was CelluNova prototype fibers. CelluNova was also blended with polyester, modacrylic and lyocell to see how the material behaves together with other fibers. Later on the nonwoven test was repeated with bamboo viscose and bamboo modal, this time without blending.

CelluNova works well to blend together with the other materials, and has a nice feeling. These CelluNova fibers are thin, ~2dtex, that contribute to a smooth feeling in the nonwoven product. The lyocell fiber that was available for this trial had a higher titer, ~7dtex, than CelluNova and therefore not completely comparable to the CelluNova fiber. The thicknesses of the fiber have more influence on the feeling than the material performance in this case.

Yarn twisting techniques

At Swerea IVF, there is a yarn twisting machine, Agteks DirectTwist® 2D6 (see Figure 1). This equipment can twist yarn or multifilament in different ways. It can also twist several yarns together or let one material be the core and let another thread cover it, yarn covering, see Figure 1. It could be a way to improve the strength of a yarn if blending it together with a stronger yarn, for example cotton or polyester. Trials show that the strength increases if blending CelluNova with a stronger material. But for fashion industry most yarns are not twisted any further than from the yarn spinning step. And if a blend is desired that will be done already before the yarn spinning step, after the bale opening step.

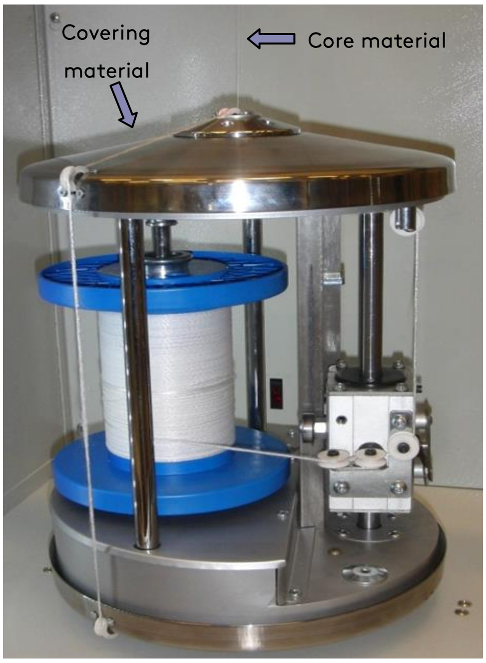

Figure 1: Yarn in twisting process.
One material in the core and another material covering it.

Flat knitting samples

A flat knitting machine with gauge 6 is available at Swerea IVF. In this trial the threads are too thin for optimal result in gauge 6. Multifilament from CelluNova was twisted according to table 2. No testing were done on these samples that were produced only for demonstration.

Table 2. Materials in flat knitting samples

Cotton	CelluNova	CelluNova+Lyocell	CelluNova + Polyester
4 threads 100 tpm	1 thread 200 tpm	1+1 thread 250 tpm	1+1 thread 250 tpm
79 tex	154 tex	192 tex	194 tex

Ring spinning staple yarn, knitting and dyeing of CelluNova fiber

The first trial with making yarn of CelluNova fiber was done within the ForTex project at the School of Textiles in Borås. The fibers were cut manually into staple fibers and put through the ring spinning process. The multifilament was not crimped before this process. It was therefore necessary to card the fiber web several times to achieve an even web. As a consequence of this, the fiber length was reduced and the spinning process was negatively affected. The fabric became uneven and it was difficult to find any parts good enough for testing. Therefore no tests have been performed on this material.

The first ring spinning trial with CelluNova was not successful. An uneven yarn was created that caused several failures in the knitting process. If the CelluNova multifilament were crimped before cutting into staple fibers it could generate a better result if repeating the test. Therefore a new trial was made. CelluNova multifilament from, made at IBWCH in Lodz, Poland August 2011, was used for this trial. The multifilament was given a crimp at Swerea IVF in SL, Extrusion, Systems, Ltd equipment. This was done with no additional heat. See Figure 2. After the multifilament was crimped, a cutting process was done manually. The fibers were cut into 38 mm staple fibers. This was done by winding the multifilament with a winch and then cut it in the middle. 38 mm fibers with a crease were the result of this cutting process. This crease was easily stretched out during next step. To separate the fibers from each other as much as possible they were placed in a plastic bag and air was added under pressure to blow them apart.

Figure 2: CelluNova multifilament; crimped filaments, at the top, compared to non treated filaments, at the bottom.

A new ring spinning trial was done at the School of Textiles in Borås. 15 grams of staple fibers were fed into the carding machine. A thin fiber web was collected at the big drum, this web was fed again into the carding machine crosswise to the fiber direction. Two times was enough to create an even fiber web.

The fiber web was folded on the middle and rolled together to a thick sliver. The sliver was fed into the stretching unit and stretched to 350%. After folding, another 350% stretching was applied. A sliver was created to go into the ring spinning step. New fibers were fed into the carding machine and after same process as above, 10 slivers were created.

The machine was adjusted to create a Ne 30 yarn, which is a common and suitable dimension for single jersey knitting. Four slivers were placed in the ring spinning equipment at the same time. After some trials and fine adjustments of the machine settings, the thread was measured to a linear density of 20 tex, which corresponds to Ne 30. Ten brown small bobbins with approximately 7-9 gram each were produced.

Circular knitting machine Camber Velnit with gauge 18, at the Swedish School of Textiles in Borås was chosen for knitting trials. Nine of the bobbins, with the ring spun CelluNova yarns, were placed in the circular knitting creel. Due to a slight variation in linear density between the bobbins a, striped effect can be seen in the fabric. Approximately 350 mm of CelluNova fabric were knitted without any holes, see Figure 3. Before the knitted CelluNova material, a Viscose thread, Ne 30, were knitted and after the CelluNova a Cotton thread, Ne 30, were knitted to have as reference material.

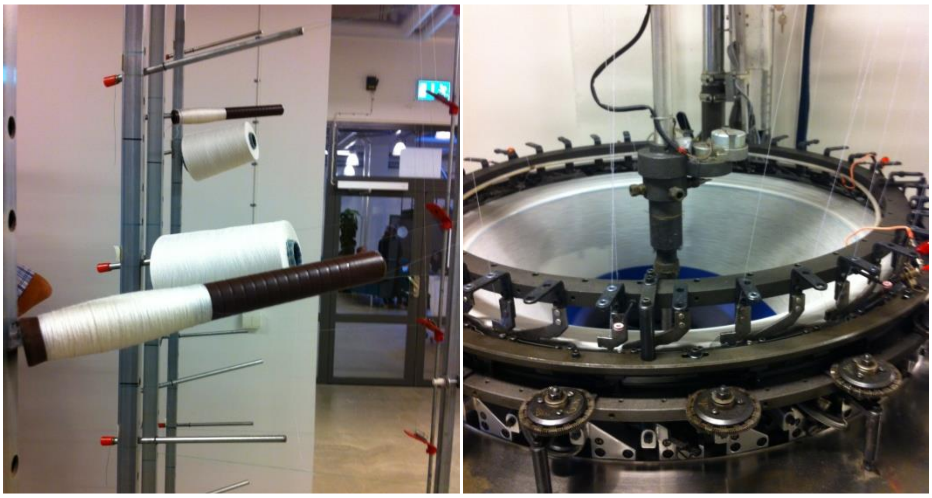

Figure 3: Left: one bobbin in the creel. Right: fabric produced in Camber Velnit circular knitting machine.

After knitting a tube with Viscose, CelluNova and Cotton fabric was produced. This tube was washed before further testing. Standard detergent and 40°C program was chosen.

One part of the tube was cut off and dyed with Cibacron F color according to dyeing process for cellulosic fibers. Color pigment Ciba-Geigy CIBACRON ROT F-B from Ciba Specialty Chemicals was used. Bath ratio was chosen to 1:20, the fabric weight was 66 g so the total dyeing bath was calculated to 1200 ml. 72 g NaCl was added (60g/l). 12 g Na_2Co_3 was added after half dyeing time, 10g/l. The dyeing was performed at Swerea IVF in a big pan at 60°C for a total time of 90 min. After dyeing and washing , the fabric was iron dried.

A red color was the result of this dyeing trial. The dyed CelluNova material is shown in Figure 4.

Figure 4: Circular knitted CelluNova fabric left: dyedand right: undyed.

Further trials CelluNova fibers

One further trial with producing yarn, from CelluNova fibers, and make a knitted sample was done in October 2013. Two other batches of CelluNova prototype fibers produced at IBWCH in Lodz, Poland August 2011, were used.

Same parameters and approach as previous described, but instead of 15 gram fiber 25 gram fiber was used in each carding set, and in the stretching step, each fiber web was stretched three times instead of two times. The sliver became thinner and it facilitated the ring spinning step so the thread became more even. Still the yarn count varies on the continuing yarn and causes stripes in the knitted fabric, but less than the previous trial. A single jersey, gauge 18, was produced at the School of Textiles in Borås. The yarn amount was enough to produce approximately 600 mm circular knitted CelluNova fabric.

To see if CelluNova takes up more color than other cellulose based materials, a dyeing test was done. For comparison cotton, viscose, bamboo viscose and lyocell were chosen as references. A dyeing bath was prepared and split in 4 different pots (bamboo viscose and standard viscose were placed together) according to table 3 below.

Table 3. Materials and dyeing parameters.

Material	Weight fabric [g]	Dyeing bath [ml]	Na2CO3 [g]
CelluNova	124	2480	25
Lyocell	115	2300	23
Cotton	109	2180	22
Bamboo viscose + Standard viscose	132	2640	27

Bath ratio was chosen to 1:20, which generated 9600 ml in total. So 10 liters dyeing bath was prepared and 500 g NaCl was added (50g/l). Na_2Co_3 was added after half dyeing time, 10g/l. Reactive dyes are suitable for cellulose fibers. The color pigment was chosen to be a turquoise blending between these colors:
30 g Drimaren K-2B, Clariant
5,5 g Cibacron Brilliant Green, Ciba Specialty Chemicals
2,0 g Cibacron C-5G, Ciba Specialty Chemicals

The dyeing baths, including fabric, were heated to 60°C. The dyeing time was in total 90 minutes. After dyeing the fabrics were washed, first in warm water (60°C) then in cold water (20)°C and finally with standard detergent from Testgewebe GmbH.

Resulting color was measured in Konica Minolta Spectrophotometer CM-3600d at Swerea IVF. See Figure 5. In table 4 the color measurement results of the five different materials are shown.

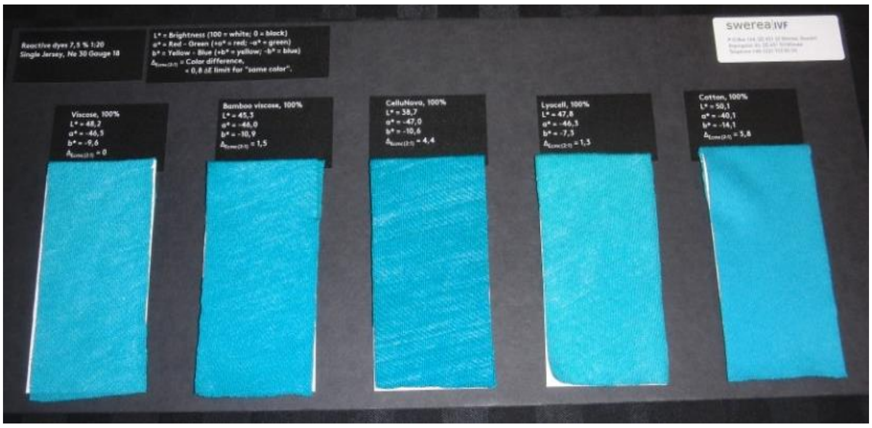

Figure 5: Five different materials dyed during the same conditions, from left to right.

Table 4. Color measurements on the five dyed materials.

Lab measurements	Standard viscose	Bamboo viscose	CelluNova	Lyocell	Cotton
L*	48,2	45,3	38,7	47,8	50,1
a*	-46,5	-46,0	-47,0	-46,3	-40,1
b*	-9,6	-10,9	-10,6	-7,3	-14,1
ΔEcmc (2:1)	0	1,5	4,4	1,3	3,8

L* = Brightness (100 = white; 0 = black)
a* = Red - Green (+a* = red; -a* = green)
b* = Yellow - Blue (+b* = yellow; -b* = blue)
$\Delta_{Ecmc\ (2:1)}$ = Color difference, < 0, 8 ΔE limit for "same color"

CelluNova fabric gets darkest (lowest L*) and it has the highest value in ΔEcmc which is a color difference index, this time compared to Viscose. It is also visible with the eye that the CelluNova fabric is more intense in color. The cotton fabric goes more blue (-b*) than the other materials and it is least intense in color.

Fiber to fabric

To illustrate all the steps that are needed to produce a ring spun yarn and knit a fabric a demonstrator with these eight steps was developed. The eight steps are the following and can be seen in Figure 6, from left to right:

1. Multifilament from wet spinning process
2. Crimped multifilament
3. Cut into staple fibers
4. Carded into a fiber web
5. Drawing process
6. Ring spinning to yarn
7. Knitting
8. Dyeing

Figure 6: Eight steps from fiber to knitted fabric.

First CelluNova garment

A piece of the turquoise CelluNova fabric was manufactured to a doll size dress (See Figure 7). The small size for the first prototype garment in CelluNova was chosen because of the limited amount of CelluNova fiber available. However, the fabric feels soft and drapes well. The stripy effect due to uneven threads influences less when the fabric is assembled to a garment. When CelluNova threads are produced in industrial scale the thread will have the same count continuously and the stripy effect will not be seen.

Figure 7: CelluNova dress on a doll.

Milk protein fiber yarn

Milk based fiber was obtained as a possible sustainable textile fiber alternative. According to the German company Qmilk, milk protein fiber can be made of waste milk, which is not allowed for consumption. Qmilk describes how to produce milk fiber on their homepage:(http://en.qmilk.eu/). The milk is allowed to go sour and is then dried to a powder. This powder together with water and unknown "natural ingredients" makes up the spinning solution. Through a melt spinning procedure at 80°C, milk protein fibers is produced. It is claimed that it takes only 3 liters of water to produce 1 kg fiber. According to info letter from Qmilk, they are building up their production plant and were planning to start fiber production in April 2014. Unfortunately no material was made available for this research project so far.

When looking further for milk protein fibers, a yarn agent called Swicofil in Switzerland was identified. They could offer milk fiber yarn produced in China. 20 kg Ne 30 Milk protein yarn were ordered. Unfortunately, very little information was provided together with the material regarding properties and production parameters. The material was undyed, off-white.

A thicker yarn, Viking Pure Milk, for hand knitting was ordered from Viking Garn in Norway but also this yarn was produced in China. This yarn was available in many colors but as the color of P4 is light purple this color was chosen.

Cardigan, milk protein fiber

As a prototype from the milk protein yarn a children's cardigan was produced at Swerea IVF on a flat knitting machine with gauge 6. This was done with the purple hand knitting yarn.

Figure 8: A cardigan is under construction in milk protein yarn.

Milk protein cuff for milk protein fiber allergy test

Does milk protein fiber irritate the skin on a milk protein allergic person? To do a simple test on that a pair of milk protein cuffs were knitted, see Figure 9. These cuffs were placed on a highly milk protein allergic person. After some hours with the cuffs no irritation on the skin was obtained. So the indication from this test with only one person, is that the milk protein does not cause harm to a milk-allergic person.

Figure 9: Knitted hand cuff in 100% Milk protein fiber yarn.

Knitted milk protein fiber fabric

Knitting trials, with the off-white Ne 30 milk protein fiber yarn, was done at the School of Textiles in Borås in Camber Velnit knitting machine. A single jersey fabric with gauge 18 was produced.

Some reference materials were also knitted and afterwards all fabrics were washed at 40°C with standard detergent. One reflection after washing was that the milk protein material dried faster than the other materials. No further study of moisture absorption/evaporation has been performed.

Jacquard knitted milk fiber fabric

A cow illustration from artist Emma Cowlam was used as pattern in a three color jacquard knitting machine at the School of Textiles in Borås See Figure 10. Milk protein fiber, in off-white, knits at the front side. The cow pattern line, in black thread, is in viscose. White viscose is knitting on the back side to fill up the third color. This generates a fabric containing approximately 40% milk protein fiber and 60% viscose.

Different fabrics with cows were produced, some with small cows on different places and one with a big cow.

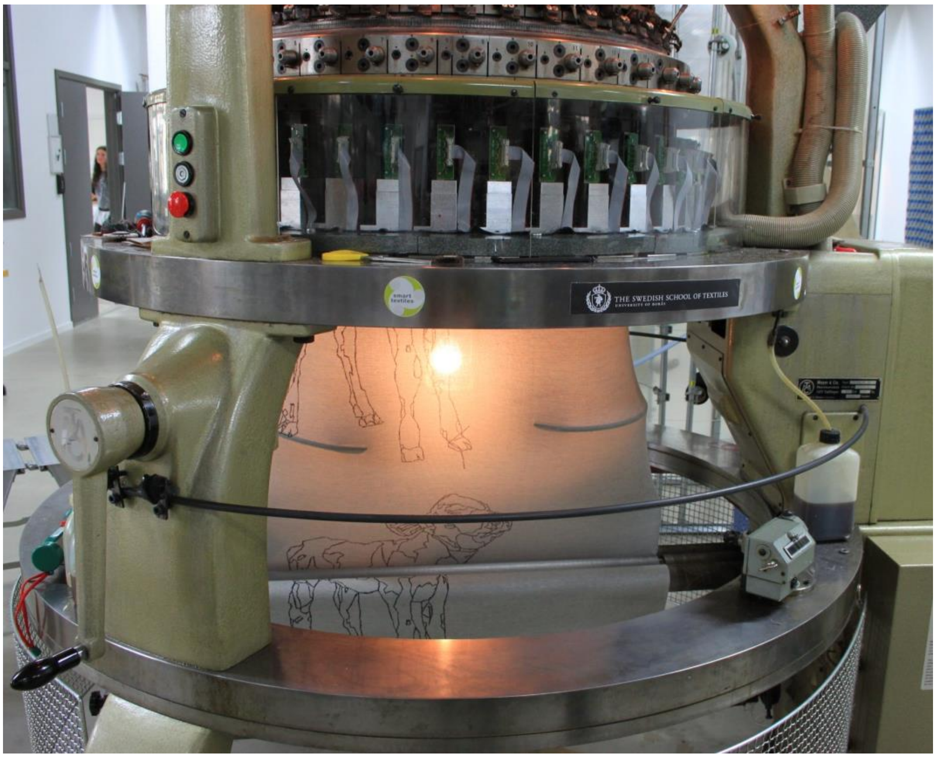

Figure 10: The big cow comes knitted out of the machine.

Children’s hoodie

With the fabric described above a children’s hoodie was manufactured. The pattern parts were chosen to match the cow pattern in the best looking way, see Figure 11. Black cuff fabric in cotton was chosen for the cuffs, Figure 12 shows the finished hoddie.

Figure 11: Fabric with small cows, pattern parts to the hoddie was carefully placed on the fabric.

Figure 12: Children’s hoodie in milk protein and viscose, cuffs in cotton.

Woven milk protein fiber fabric

A trial was done with weaving a milk fabric. Two milk threads were twisted into one yarn, whichwas used in the insert. A weaving machine with a black cotton warp was chosen for the trial. Weft dominated twill gives a lighter upside due to the off-white insert and a black reverse because of black warp.

This combination of material increases the strength compared to pure milk protein fabric. In an abrasion test this material manages 30 000 cycles in Martindale equipment which is a good result.

Weaving single yarn insert in cotton warp

Single milk protein yarn was used as insert in a white cotton warp. However, the thread was too weak as a single thread, and just a small amount of fabric went through the machine before failure. The material produced before failure is included in the washing test that is described in report *D4.4 Report on wash ability of the developed products.*

A 100% soybean thread was also used as insert in the same cotton warp.
This material is more yellow than the milk protein fiber fabric. The soybean fiber is difficult to dye and most dye houses is not accepting this fiber. In this part of the project, a decision was taken that the soybean plant is most suitable for food and not for textile purpose. Therefore no further tests have been made on this material.

Dyeing of protein based materials

A trial with dyeing fabric with acid colors was done at the Swedish School of Textiles in Borås in Pyrotec MB2 equipment. An orange color, 2% Brilliant orange GR from Zenit AB was chosen and the dyed materials were;

100% milk protein, knitted single jersey
40% milk protein, 60% cotton, woven fabric
100% silk, woven fabric
100% Wool, woven fabric
100% CelluNova

The CelluNova material is not protein based and therefore it did not take up much color in this dyeing test. CelluNova and cotton is cellulose based and dyes best with reactive colors as shown in test above. The cotton warp in the woven fabric was therefore lighter than the insert milk yarn. The milk fabric worked well to dye; it took up color as predicted. Silk and wool reflects light different from cellulose based fabric. This is most due to the cross-section of the fibers. Therefore no color measurement comparisons were done on these materials.

Fibers from seaweed

Fibers from seaweed ("fibers from the sea") has come up as a possible sustainable textile fiber for the fashion market. It turns out to be difficult to find any suppliers of this. Smart Fibers in collaboration with Lenzing has has developed a fiber product they call SeaCell, the following information text can be found on their homepage:http://www.smartfiber.info/seacell 2014-06-02.

"The health-promoting properties of the maritime natural substance are being used by producing a unique fiber that combines cellulose with seaweed. The exceptional about SeaCell™ is how the seaweed has been permanently incorporated into the fiber, locking the effects of the marine substances into the fiber for good." It says also that the fiber does well for the skin and the health.

What can be further understood from the brief information given is that the fiber is produced in the Lyocell process in which powder from seaweed has been added. The seaweed content is low, and the main part of the fiber is a lyocell fiber (i.e. main raw material is dissolving pulp from wood cellulose). This material is not included in this study due to difficulties to get hold of the fibers.

Continued searching for seaweed fibers results in a fiber called 100% Seaweed. One sample bobbin was ordered in Ne 26 via Swicofil, yarn agent in Switzerland. This material is produced in China and according to the agent "not produced in a very environmental way". When the fiber arrived, information from the agent said that it only contains 15% of the seaweed substance and that the rest is ordinary viscose. Probably, the seaweed powder is added to the dissolving pulp at an early stage of the viscose process.

An NMR test on the fiber was performed at Chalmers University of Tehcnology, Göteborg, Sweden; this test should characterize the cellulose structure in the material. The result of this test was that it contains regenerated cellulose e.g. viscose. No trace of other cellulose structures than regenerated cellulose was obtained. However, the method was most looking for evaluating cellulose content and seaweed can include different types of cellulose as well as protein, carbonates and mineral substance.

Fiber Testing

Mechanical test of the fiber shows that this seaweed fiber produced in China is very similar to ordinary viscose in both tensile strength and elongation. Five threads of each material were measured according to standard SS EN-ISO 2062:1995.

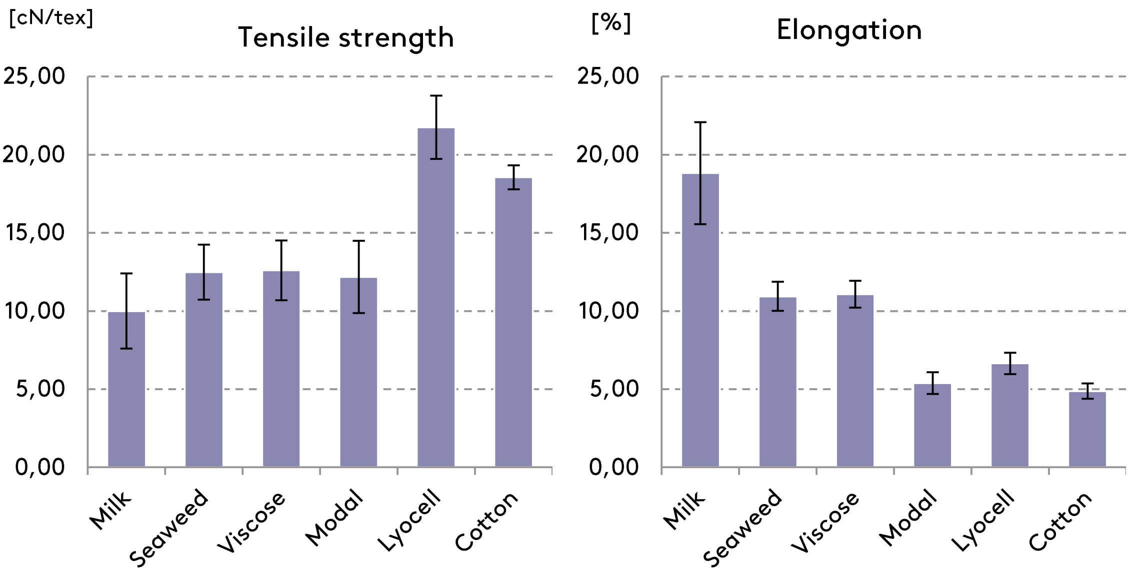

Figure 12: Tensile strength and elongation for Milk protein, Seaweed and some other cellulose based threads.

Textile testing

To investigate and compare these textile materials following tests have been performed.

Pilling
Test according to standard SS-EN ISO 12945-2:2000
Pilling is rated at different number of cycles. All materials start at rate 5. CelluNova and Milk protein managed best after 5000 cycles. Cotton was rated worst when the test was finished. See diagram in Figure 13.

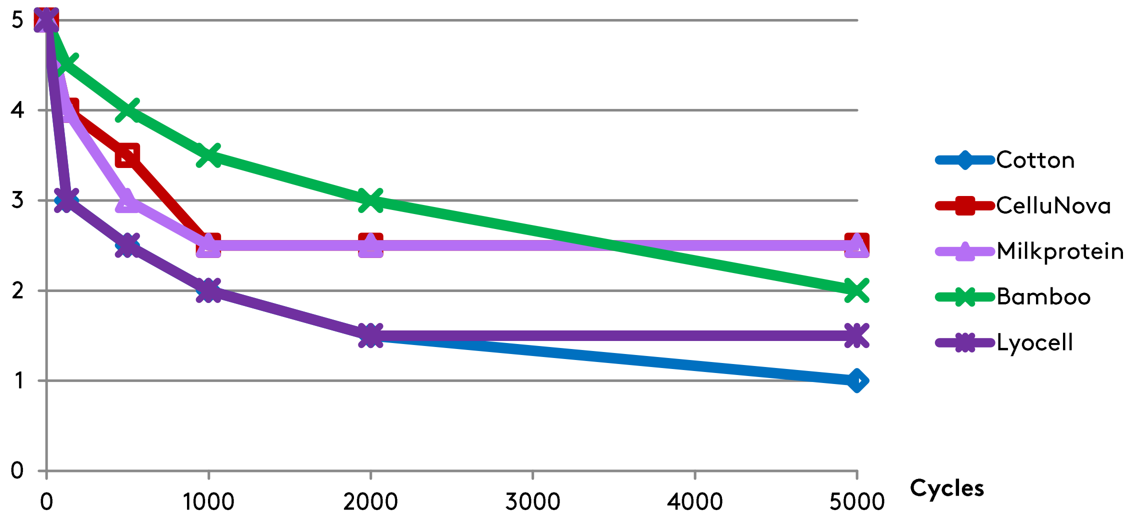

Figure 13: Pilling rate at various cycles, 5 is no change and 1 is severe pilling.

Figure 14: CelluNova sample after 5000 cycles in Pilling test.

Abrasion

Test against abrasion was done according to standard SS-EN ISO 12947-2:1999. Samples were assembled in Martindale equipment and were tested until material failure occured. The samples were checked with defined intervals according to the standards. Table 5 below shows number of cycles before thread breaks or holes appear in the samples.

Table 5. Number of cycles before material failure.

Fabric	Cotton woven	Standard viscose, woven	Bamboo viscose, woven	CelluNova knitted	Milk protein, single jersey (knitted)	Milk protein /viscose, knitted	Milk protein woven
No of cycles	30 000	22 000	20 000	4 000	3 000	8 000	30 000

The cotton fabric managed most cycles before failure (30 000 cycles). The woven milk fabric actually reached as high abrasion resistance as the cotton fabric but it should be noted that the Milk protein fabric contains cotton in the warp. Neither of the knitted fabrics (from CelluNova or Milk protein fibers) performed well in abrasion test. However, the woven structure is stronger and has a more even surface which increases the abrasion resistance compared to a knitted structure which means that these tests does not tell whether a woven CelluNova fabric would have poorer abrasion resistance than e.g. a woven viscose fabric.

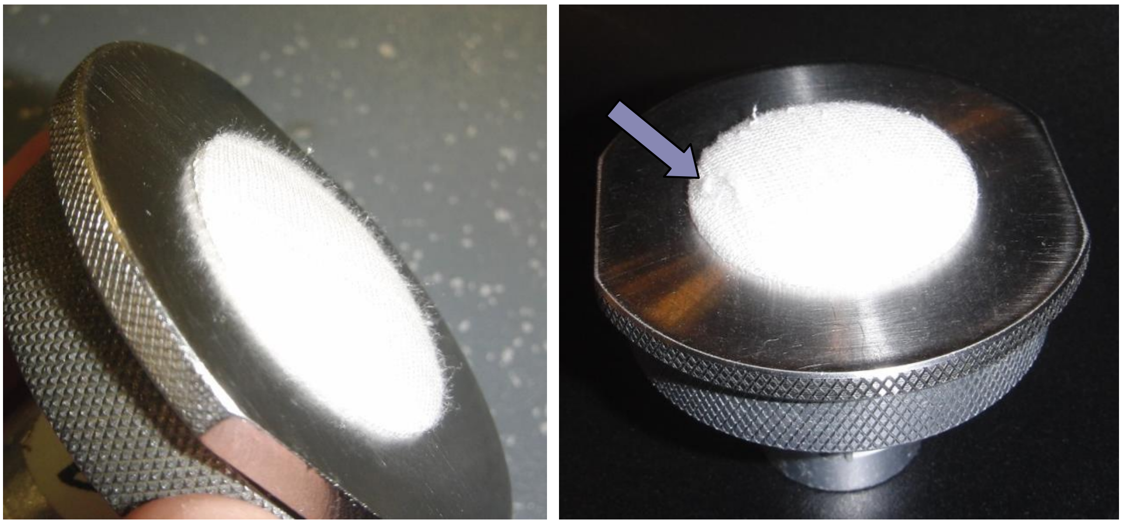

Figure 15: CelluNova after material failure in Martindale equipment for abrasion test.

Color fastness to rubbing

Dyed CelluNova fabric was compared to dyed viscose and cotton fabrics.
Color fastness to rubbing was determined according to the standard SS EN ISO 105-X12.

Table 6. Rating 5 is excellent, 1 is bad performance.

Fabric	Rubbing dry	Rubbing wet
CelluNova	5	2,5
Viscose	4,5	3
Cotton	4,5	3,5

Color fastness to laundering

According to standard SS EN ISO 105-C06.
CelluNova fabric was compared to viscose and cotton.
Milk protein fabric was compared to other natural protein fiber fabrics (silk and wool).

Table 7. Rating 5 is excellent and 1 is bad performance.

Material	Staining	Color change
CelluNova	4,9	5
Viscose	4,9	4,5
Cotton	4,7	4
Milk single	4,8	4,5
Milk woven	4,7	4,5
Silk	4,9	4
Wool	4,7	4,5

Figure 16: CelluNova staining sample.

All of the fabrics performed well in staining, color change and rubbing tests. CelluNova and Milk protein both seem competitive to conventional materials in these aspects.

Further tests

CelluNova was compared to viscose and cotton in Kawabata equipment.
Kawabata measurements for surface properties/friction were determined with Kawabata KES-FB-4. Measurement for bending rigidity was determined with Kawabata KES-FB-2. In these tests no significant deviations were found.

Test with Skin model SS-EN ISO 31092:1993. Thermal Resistance & Water Vapor Resistance were also performed on these three materials. Neither here were any significant changes between the materials obtained.

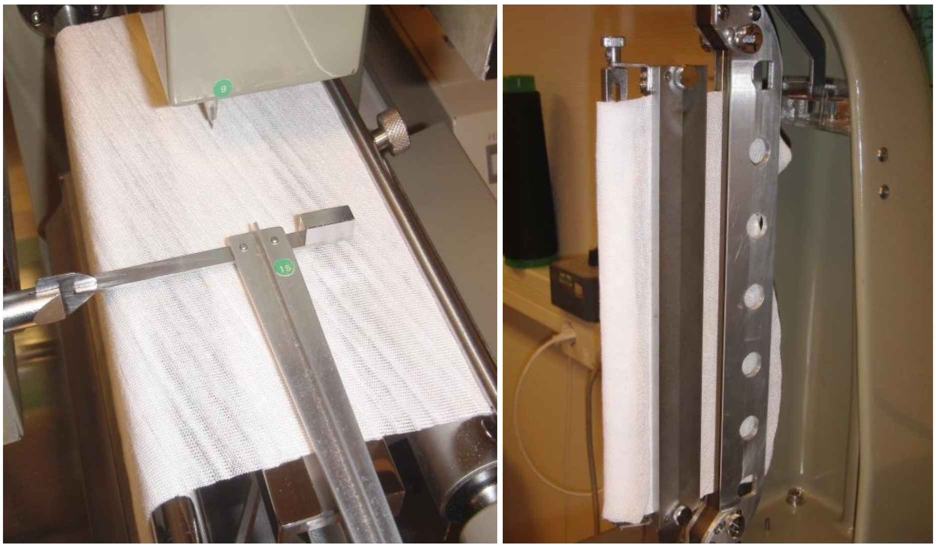

Figure 17: Kawabata measurements on CelluNova fabric.

Coming activities / Future work

This report describes the development work within Mistra Future Fashion June 2011 until June 2014. The project will continue one more year (ends May 2015) and the following activities can be of interest to look deeper into.

- Perform further test in larger scale on the CelluNova material if and when more fibers are available from the last optimized CelluNova process.
- Create woven fabrics of the milk protein fiber yarn with the cow pattern and test the properties of these fabrics, mainly tensile testing and other mechanical tests.
- Perform testing of fibers from Qmilk. If Qmilk is interested, an LCA could be performed.
- Milk protein fibers are known as a compostable material. It would be interesting to study the material during these conditions both as single material but also in blends.
- Keep searching for other possible sustainable materials. Source other suppliers for algae or seaweed fibers.

All CelluNova fibers described in this report were produced at IBWCH in Poland under contract within the CelluNova project 2011. Further CelluNova fibers available for Mistra Future Fashion will be produced within the ForTex project and with some different parameters compared to the IBWCH fiber (generally better performace). If and when fibers are available, more tests will be performed.

Conclusion

As a conclusion from the tests described in this report, CelluNova fiber is competitive as an alternative to cotton and viscose fiber. However, the CelluNova fiber spinning process used for the fibers described in this report was not at all optimized and the process is still to date not fully optimized.
In the dyeing test, CelluNova gets most intense in color compared to the material in the study. This could result in lower consumption of dyeing chemicals in order to obtain the same appearance as for example cotton or conventional viscose fabrics.

Regarding milk protein fiber, it would be interesting to evaluate fibers from Qmilk produced in the new factory in Germany. The tests done so far were with fibers produced in China on which was given very little information. It is unclear how this material has been produced and therefore it is hard to say how sustainable the material is. Even Qmilk has potential to be a sustainable alternative that fulfils the quality demands, for a new fashion fiber. Milk protein fiber is made of protein, Casein, and would be benchmarked against both wool and silk regarding color uptake and appearance. But the material can also be an alternative to replace cotton in fashion clothes.

About Mistra Future Fashion

The purpose of the Mistra Future Fashion Program is to deliver knowledge and solutions that the Swedish fashion industry and its stakeholders can use to improve the fashion sector's environmental performance and strengthen its global competitiveness. The program is structured so that it leverages the expertise and networks of leading Swedish and international research institutes and universities. Stakeholders engaged in the program include governmental agencies, voluntary organisations, and companies within the entire textile value chain: forestry, pulping, textile manufacturing and recycling. To find out more please visit www.mistrafuturefashion.com.