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Heikkilä, Pirjo; Määttänen, Marjo; Jetsu, Petri; Kamppuri, Taina; Paunonen, Sara

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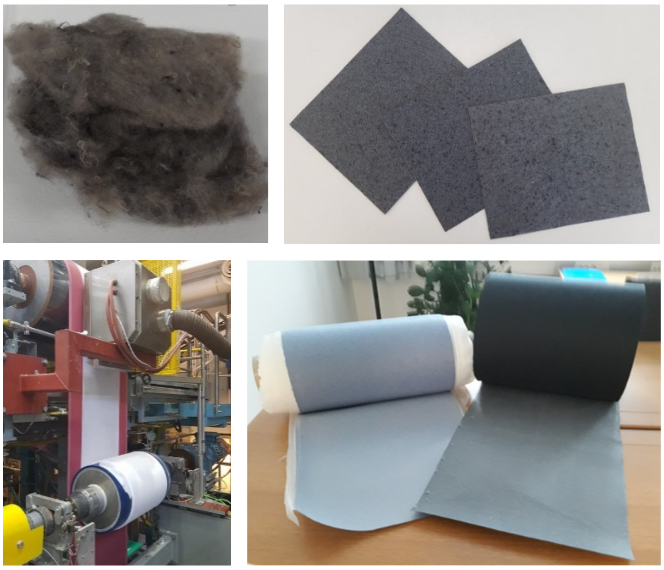

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Authors: Pirjo Heikkilä, Marjo Määttänen, Petri Jetsu, Taina Kamppuri, Sara Paunonen

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Report's title

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Summary

This report summarizes VTT's work carried out in *Circular Nordic Bio Nonwoven in MedTech Applications* (NordicBio) project - a bilateral Swedish Finnish co-operation funded by Vinnova and Business Finland. NordicBio aimed for development and pilot scale demonstration of a cost effective, bio and circular material based materials suitable for medical applications.

VTT work focused on two processes: 1) cleaning of mechanically recycled fibres, and 2) production of nonwoven materials from recycled fibres. Targeted applications were laminated nonwoven products suitable, for example, for bed sheet and absorptive products in hospitals.

Disinfection of textiles can be carried out either in high temperatures with alkaline chemicals and laundry detergents without oxidative chemicals, or in lower temperature together with oxidative chemicals and laundry detergents. It was found that the most effective treatments decreased fibre strength properties, but with certain chemical compositions also removal of some colours was possible. Microbial tests showed that the use of high temperature (80°C) at alkaline conditions is the most effective way to disinfect microbes, even bacterium pores. Lower temperatures (60°C and 40°C) with oxidative detergent reduce the content of other microbes effectively, except bacterium pores.

Nonwovens were prepared from recycled fibres in combination with pulp, regenerated cellulosic fibres and bi-component binder fibres, and using spunlacing, latex treatment and thermally activated bico fibres as binding methods. It was observed that mechanical properties, strength and strain, of nonwovens can be varied widely by fibre content and binding method. We concluded that high recycled cotton content can be achieved with optimized formulation and binding. Slushable materials suitable for recycling were obtained using spunlacing as binding method.

Nonwovens were also prepared in pilot scale for lamination trials. Tensile strength of piloted materials was comparable to commercial references, but their strain was lower and grammages higher than references. Lamination trials were successfully carried out for rolled materials, and lamination improved the mechanical properties.

Confidentiality	Public

Tampere 24.11.2020

Written by	**Reviewed by**	**Accepted by**
Pirjo Heikkilä Senior Scientist	Ali Harlin Professor	Jani Lehto Vice President

VTT's contact address

Pirjo Heikkilä, PL 1300, 33101 Tampere

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VTT, Business Finland, Vinnova, project partners

Preface

This report summarizes VTT's work in *Circular Nordic Bio Nonwoven in MedTech Applications* (NordicBio) project. Project was bilateral Swedish Finnish co-operation funded by Vinnova and Business Finland as part of Joint call Sweden-Finland for *Development of key enabling technologies for biobased products* (call open 20.3.2018-14.6.2018). Main partners were Pure Waste Textile, coordinator of Finnish side of the project, and Wargön Innovation, coordinator of whole project, and Swedish side including also Cellcomb, RISE IVF AB, Fiber-X, Sporda Nonwovens, and Stockholm Läns Landsting (SLL). Duration of the VTT project was 3.11.2018-30.9.2020.

NordicBio aimed for development and pilot scale demonstration of a cost effective, bio and circular material based materials suitable for medical applications. VTT's work focused on cleaning of mechanically recycled fibres and production of nonwoven materials from recycled fibres.

VTT responsible leader was Jani Lehto, project manager and recycling and nonwovens expert was Senior Scientist & Project Manager Pirjo Heikkilä, and project finances was handled by Suvi Järvinen and Seija Laukkanen. VTT's key personnel in project included Senior Scientists Marjo Määttänen, responsible of cleaning process (WP4), and Petri Jetsu, responsible in nonwoven processes (WP6.1) Other VTT experts with expert role in this project were Senior Scientist Taina Kamppuri and Research Professor Ali Harlin.

VTT working group also included large group of laboratory and technical staff carrying out of practical work. Authors would like to acknowledge them: Juha Haakana, Nina Vihersola, Markku Suikka, Petri Mannela, Anne Heikkinen, Marja-Liisa Jalovaara, Saila Orasmaa, Merja Salmijärvi, Satu Salo, Niina Torttila, Janne Airola, Päivi Sarja, Merja Selenius, Sirpa Hokkanen, Heikki Talja, Mervi Raatikainen, Meiju Sinkkonen, Jari-Matti Paatelainen, Roope Siilasto, Daniel Koskelo, Eetta Saarimäki, Ilkka Nurminen, Jari Leino, Jani Lehmonen, Timo Rantanen, Seppo Kovanen, Tiinamari Seppänen, Riitta Pöntynen, Jarmo Kouko, Simo Puikkonen, and Timo Kaljunen.

Authors would also like to thank our project partners Wargön Innovation, RISE and Pure Waste Textiles for providing and processing recycled fibres, and Cellcomb and SLL for challenging us with end-product requirements. We like to thank all partners, including also Fiber-X and Sporda Nonwovens, for fruitful co-operation, and Business Finland and Vinnova for enabling such possibility for bilateral co-operation.

Tampere 24.11.2020

Authors

Contents

Abbreviations and Terminology

Abbreviations

AOB	peracetic acid derived from perborate or percarbonate
BAC	benzalkoniumchloride, antimicrobials active for laundering processes
Bico	Bicomponent binder fibre
BSKP	Bleached softwood kraft pulp
CCA	Cellulose carbamate
CO	Cotton
DDAC	Didecyldimethylammoniumchloride, antimicrobials active for laundering processes
LYO	Lyocell
MedTech	Medical textile applications
MFC	Micro fibrillated cellulose
OE	Open end, spinning method
PE	Polyethylene
PES	Polyester
PET	Polyethylene terephthalate, type of polyester
PF	Peracid Forte, laundry detergent used in cleaning trials
PLA	Polylactid acid
polCO	Polycotton, mixture of cotton and polyester
PP	Polypropylene
PVA	Polyvinyl alcohol
RT	Room temperature
SDS	Sodium dodecyl sulphate, foaming agent in foam laying process
SPB	Select Power Blue, laundry detergent used in cleaning trials
TACN	Triazacyclononane, used in washing detergents
TAED	Tetraacetylethylenediamine, bleach activator

1. Introduction

1.1 MedTech Materials and Nonwovens

The usage of disposable textile and nonwoven items in the health care sector is increasing. These products can be everything from personal care products to sheets clothing and other textiles, operation room materials, sterilization wraps and many others (see Figure 1).

Figure 1 Examples of MedTech products

Increased use disposable instead of durable textiles and using laundry processes is motivated by low cost production and distribution, simplicity and ease of use and level of controlled sterility/cleanness. Currently such disposable items are often made of plastic, and thus fossil based materials, and after use they are wasted and incinerated rather than recycled. Alternatives can be biodegradable, recyclable and/or renewable, all depending on the material specifications.

VTT Competences

VTT has earlier expertise in various areas related to nonwovens in medical purposes and research related to medical environment from nonwoven manufacturing and their surface treatments, clothing and textiles for hospitals, hygiene and other issues related to hospital environment as well as human body modelling (see Figure 2). More details are given in following.

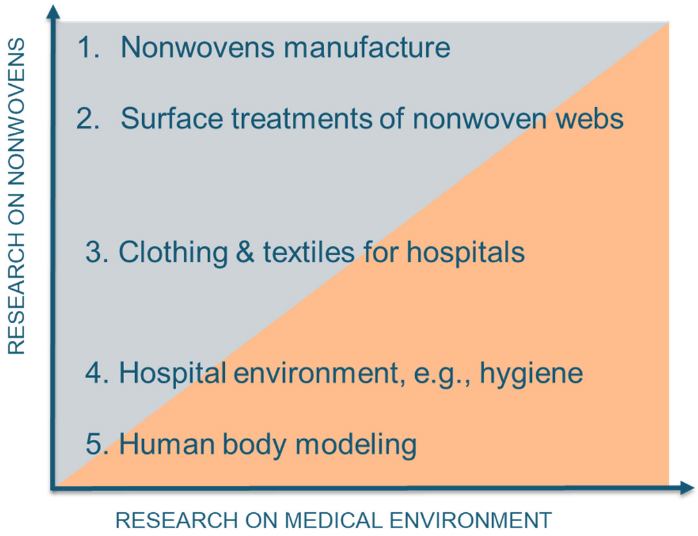


Figure 2 VTT competence areas within nonwovens and medical environment

Nonwovens manufacture. VTT has over 10 years expertise in foam laying process. Principle of foam laying is similar to wet-laying, but transfer media is aqueous foam instead of water. Fibre suspension contains typically 40% - 70% of air. Foam stability can be controlled and typical bubble diameter is ~100 µm. Fibre consistencies can be higher than in wet-laying since small bubbles fill the free space and prevents/reduces fibre flocculation.

The development started over 10 years ago. Technology development has been carried out e.g. in

- KOTVA/ERDF project (2012-2014). Conversion of SUORA
- Foam forming program/ERDF program (2015-2017). Installation of SAMPO
- FFP2020 Future fiber products/ERDF program (2017-2020). New applications

Appearance of foam bubbles and VTT SUORA pilot line is shown in Figure 3, and information of SUORA and SAMPO pilots is collected into Table 1. SAMPO pilot is introduced in Experimental section (Chapter 5).

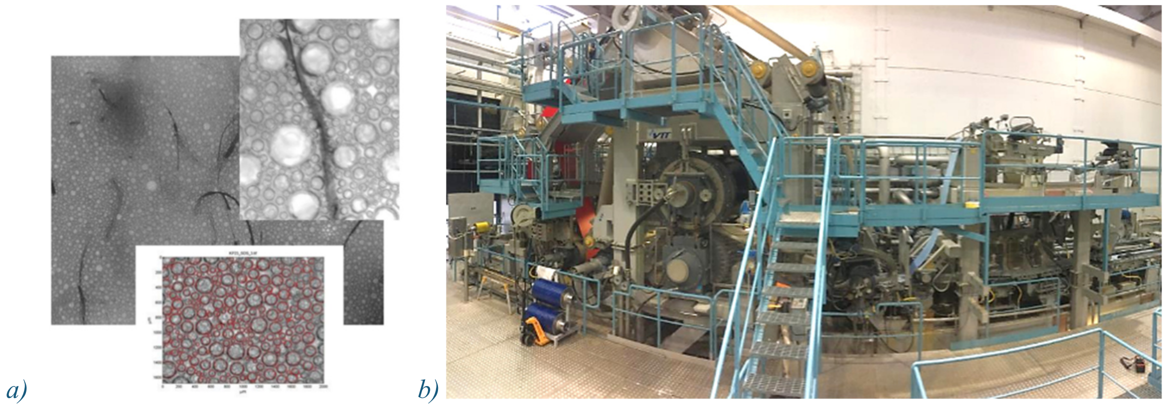


Figure 3 a) Appearance of bubbles and fibres between them, and b) VTT SUORA pilot

Table 1 VTT foam technology pilots

Pilot	SUORA	SAMPO
Description	Pilot-scale research environment for papermaking and board manufacturing processes, and product development. Foam start-up 2013	Pilot-scale research environment for non-pressed materials. Start-up 2017
Technologies	Water-laid & Foam-laid technologies Fourdrinier, hybrid and gap former options Stratified forming option Long nip wet pressing unit Cylinder drying unit (off-line)	Foam Forming Technology Vertical head box Stratified forming option Impingement and through-air dryers
Capacity	Web width 300 mm, max speed 2000 m/min	Web width 600 mm, max speed 200 m/min

Surface treatments of nonwoven webs. VTT has multiple surface treatment technologies that could be suitable for materials used in hospital applications. Examples include

- Foam coating of nanocellulose. Research e.g. in EU-NanoTexSurf-project[1] (2017-2020) included foam properties, anchoring, material properties and performance (see Figure 4a)
- Plasma and corona treatments and activation. Atmospheric plasma treatment (see Figure 4b) can be used for surface activation and plasma coating e.g. hydrophobization with siloxanes (Plastek1 and Plastek2 -projects, 2005-2009). More information e.g. from article written by Nättinen *et al.* (2010)
- Antimicrobial coatings on cubicle (plastic) curtains can be achieved e.g. by corona activation and brush coating with tannins (Sami&Samu-project[2], 2017-2020)

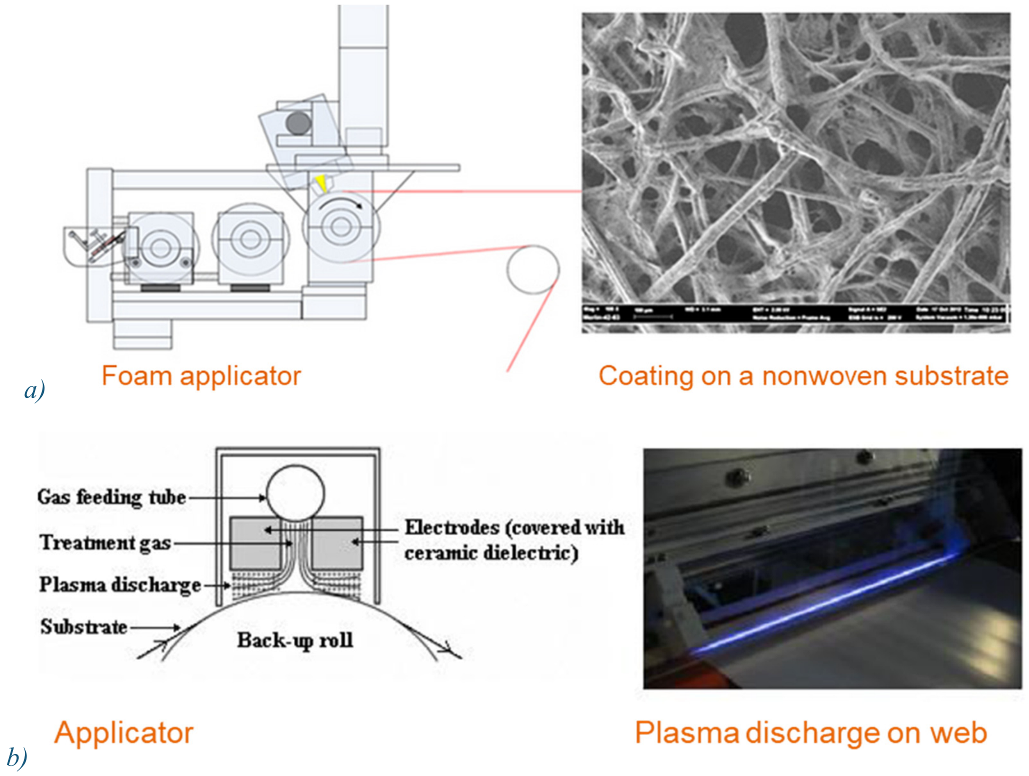


Figure 4 a) Foam coating with nanocellulose, b) atmospheric plasma treatment

1 http://www.nanotextsurf.eu/
2 https://projectsites.vtt.fi/sites/sami-samu/www.vtt.fi/sites/sami-samu/en.html

Clothing & textiles for hospitals. Expertise areas (and project examples) include:

1. Textile products made of new bio-based materials for Hospital Nova e.g., antimicrobial materials (Sami&Samu, 2018-2020)
2. Smart clothing for hospital patients e.g. demand-based heating, sensors integrated to clothes (Smart clothing 2, 2017-2019)
3. Particle emissions from protective clothing, Body-box tests, see Figure 5a (The Finnish high-tech hospital project 2010-2012)
4. Recycling of hospital textiles e.g. a small case study (World of cellulose, 2013)
5. Protective clothing for hospitals & healthcare (Mulfunc, 2004-2007)
6. Clothing for the elderly in hospitals (Easytex, 1997-2000)

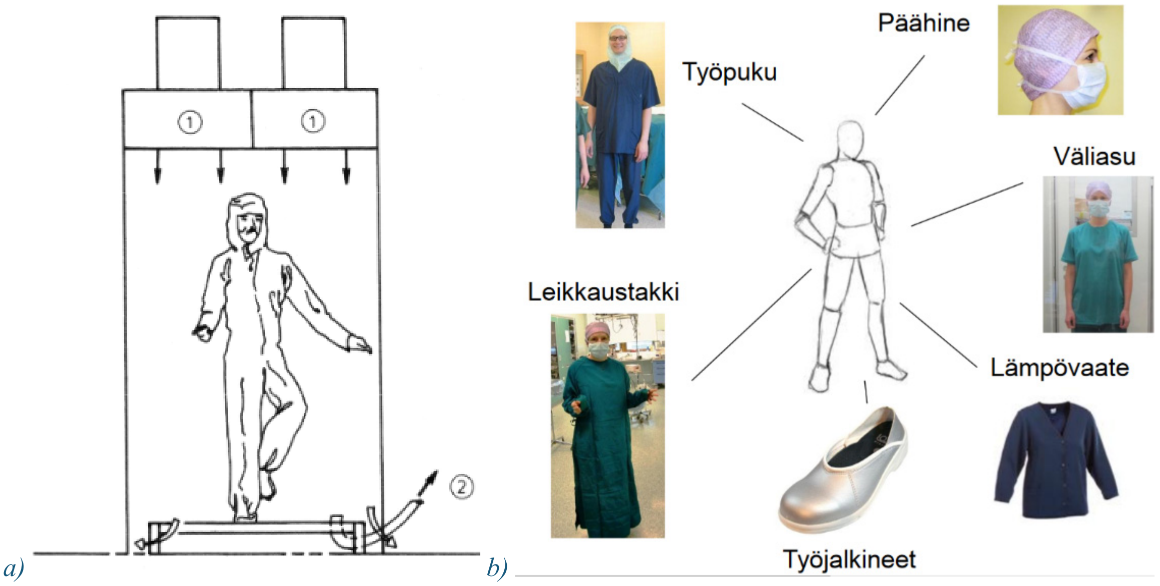


Figure 5 Illustrations related to a) body-box testing, b) surgical ward work clothing concepts

Hospital environment and hygiene. Expertise areas (and project examples) include:

1. Hygiene and cleanliness e.g. The Finnish high-tech hospital forum (HT-hospital forum), 2010-2012
2. Work wear concept created for surgical wards, see Figure 5b (HT-hospital, 2010-2012)
3. Regulations related e.g., to substances and materials used in hospital environment (Sami&Samu, 2017-2020)

Human body modelling. Human body thermal model (see Figure 6) has been studied in various projects for various spaces e.g. for intensive care units (2014-2016), recovery rooms (2016-2018) and waiting rooms.

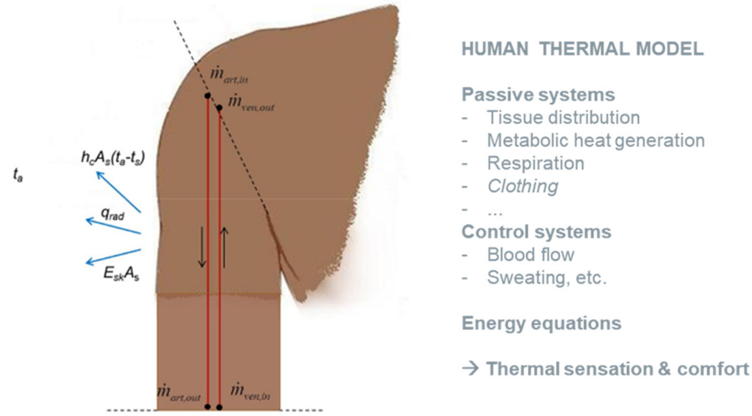


Figure 6 Human body thermal model (Holopainen, 2012)

1.2 Textile and Cotton Recycling

Currently textile collecting and sorting are focusing on re-usable textiles, and there is not yet systematic collection of textile waste for recycling in Nordic counties. That is, however, about the change in 2025, when EU level requirement for separate collecting of waste will come into effect. Since separately collected materials are to be forwarded into recycling, processes needs to be developed and end uses found for recycled textile materials. Replacing synthetic plastics in disposable items seems attractive use for recycled textile fibres.

Separate collection of textile waste to be started in EU member states by 2025 – we can expect to have recycling targets set, similar to those of plastic recycling targets, e.g. for 2030. Textile recycling can be done in fibres level using mechanical processes and by recycling fibre raw materials using chemical and thermal processes, see Figure 7 (Heikkilä *et al.*, 2019a; Kamppuri *et al.*, 2019).

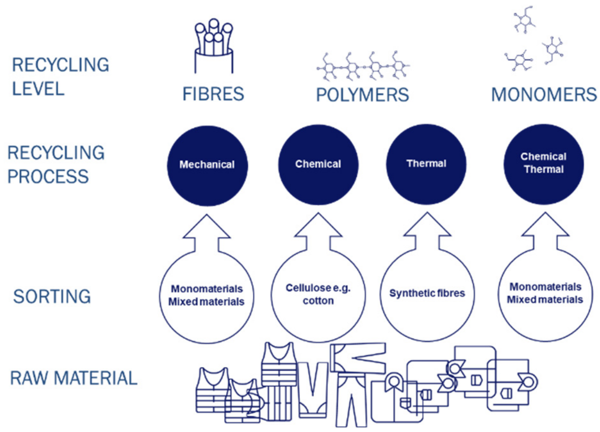


Figure 7 Recycling processes (Heikkilä et al., 2019a; Kamppuri et al., 2019)

Recycling methods

Mechanical processing of textiles materials back into fibre level is in principle a relatively simple process. It includes shredding of materials and opening it into fibres, which affects fibre properties especially by reducing fibre length and strength. In raw materials recycling fibres are disintegrated either into polymer or monomer level and then rebuild back to fibres. These processes may enable restoration of fibre properties into level same or similar to new fibres depending on the process. Fibres obtained from recycling processes can be then used for making textile and nonwoven products. SWOT analysis for mechanical recycling and fibre raw materials recycling processes are included in Figure 8.

Mechanical / fibre recycling	**Strengths** • Low LCA impact compared to other recycling processes • Suitable for monomaterials and blends alike • Only option for natural fibres to preserve their macroscopic/ physical structure	**Weaknesses** • Reduction of fibre length & strength during use and mechanical processing
	Opportunities • Good option especially for pre-consumer and industrial post-consumer materials • Options from paper-nonwoven-yarns depending on fibre quality	**Threats** • Hygiene of post-consumer materials • Consumer acceptance • Finishing agents in fibres - restrictions to use in certain products
Fibre raw materials recycling	**Strengths** • Possibility to restore quality in monomer level recycling equal to virgin materials • Hygiene problems solved	**Weaknesses** • Environmental impact of processes • Polymer level recycling of synthetics difficult to inhomogeneous fractions – lowered quality • Impurities may affect chemical processes
	Opportunities • Some processes suitable for monomaterials and blends alike	**Threats** • Economics of the processes and materials • Amount of recycling cycles not (yet) known

Figure 8 Comparison of fibre recycling and fibre raw materials recycling by SWOT analysis

In mechanical recycling, challenges may arise and quality is influenced by various factors (Heikkilä *et al.*, 2019; Kamppuri *et al.*, 2019). Mechanically recycled material does not undergo any process steps that would affect cleanliness' of the fibres, while chemical and thermal processes can simultaneously be antimicrobial treatments and remove also other impurities. Depending on the use history as well as storing conditions of collected materials they might be contaminated, which may limit their use in sensitive and hygiene applications unless decontaminated somehow. Some textile recyclers may have washing machines available, but in many cases recyclers use materials as they arrive. Currently separate collection of discarded textiles is focused on reusable items, and it can be assumed that consumers returning clothes now to collecting points are likely returning mostly clean items. Cleaning processes might be more important in the future, when textile waste will be collected more systemically and larger crowds, not only the aware consumers, start returning their textile wastes.

Mechanical processing affect properties of fibres materials. Therefore materials should be sorted according to fibre and material type. Durability and, thus, capability of fibres to withstand mechanical processing vary from fibre to fibre. Furthermore, different types of materials require different amount of work to be opened: knitted materials open more easily than woven fabrics and loose weaves easier than tight weaves. Mechanical processing can be optimized for each type of materials, if fibre quality is to be maintained high. This is not that easy take into account in textile sorting in current systems. Therefore opening of heterogeneous materials, such as post-consumer textile waste, typically gives heterogeneous fibre from opening process.

If materials to be recycled include non-textile components, these needs to be removed. This can be done manually with scissors and/or other types of cutters, which is done e.g. by Wolkat[3] (Netherlands) who has sorting and processing plant in Marocco. Automated lines e.g. one in Frankenhuis[4] (Netherlands), are based on cutting textile in small pieces, and those pieces containing non-textile components are separate mainly based on gravity. Doing this manually is labour intensive and, thus, costly phase, while automated lines capable of this require large through-put volumes to be economically viable.

3 https://www.wolkat.com/en
4 https://www.frankenhuisbv.nl/

After removal of non-textile components textiles are ready for mechanical recycling process. It includes cutting, tearing and opening steps. Amount of work determines how well textile structure is disassembled back to individual fibres, but more processing leads to reduction of fibre length. In many cases some kind of compromise between opening quality and fibre length needs to be made especially when aiming for production of yarns. Typically fibre lengths of 25 mm and above are suitable for ring-spinning and 20 mm and above for open-end spinning. Open-end process, which is faster and enable use of shorter fibres, is often used for recycled materials e.g. by Pure Waste Textiles[5].

Nonwoven production methods are not that sensitive to fibre length. Fibres with length of around 10 mm and above are suitable for carding, and carding is well suited for mechanical recycling of textiles and used e.g. by Dafecor[6]. Air- and wet-laying, on the other hand, can utilize also almost dust-like fibre materials. Aero dynamical processes have been developed for very short fibres (dry-paper process) and longer ones alike, and aero dynamical processing lines are available for textile recycling e.g. by Laroche[7]. In wet processing short fibre lengths are actually beneficial for formation, since processing limitations to fibre lengths are related to the maximum length, not the minimum.

VTT experience in textile recycling and circular economy topics have been gathered e.g. following projects:

- TEKI/Tekes project, also known as The Relooping Fashion Initiative (2015-2017) Modelling of circular ecosystem for textiles, development of chemical (cellulose carbamate - CCA) recycling process for cotton (https://cris.vtt.fi/en/publications/the-relooping-fashion-initiative).
- Repotex/Tekes project (2016-2017) Commercialization of CCA process
- Trash-2-Cash: Designed high-value products from zero-value waste textiles and fibres via design driven technologies (H2020-NMP-2014-646226, 2015-2018) (www.trash2cashproject.eu) Chemical recycling of cotton and mixed cotton-polyester textiles
- Telaketju Tekes/Tekes project (2017-2019) Building circular textile ecosystem to Finland (https://cris.vtt.fi/en/publications/telaketju-towards-circularity-of-textiles)
- Telaketju 2 BF/Business Finland project (2019-2021) Business from circularity of textiles (https://telaketju.turkuamk.fi/en/front-page/)

Cotton and its recycling

Properties of cotton fibres, such as moisture absorption and pleasurable touch, makes them preferable raw material in various applications, especially clothing. In some of these applications cotton can be replaced with regenerated cellulosic fibres without losing performance, but replacing cotton with synthetics cannot provide similar properties and function.

Cotton from textile materials can be recycled mechanically or chemically. In the mechanical recycling, textile material (or product) is torn down into fibres. After opening, these fibres can then be processed into nonwovens and yarns. The origin of textile waste determines the possible uses of the recycled fraction.

Pre-consumer cotton waste, such as cutting waste from textile industry, has almost similar strength compared to original virgin fibres, and only slightly reduced length. Therefore, such cotton fibre fractions are well suited for the production of yarns as such (Esteve-Turillas & de la Guardia, 2017) and when mixed with virgin cotton fibres (Wanassi *et al.*, 2016). Composition of pre-consumer waste is typically known, so sorting according to fibre types is typically not a necessity, and if materials of different colours are kept separated, the original colour remains in a new product.

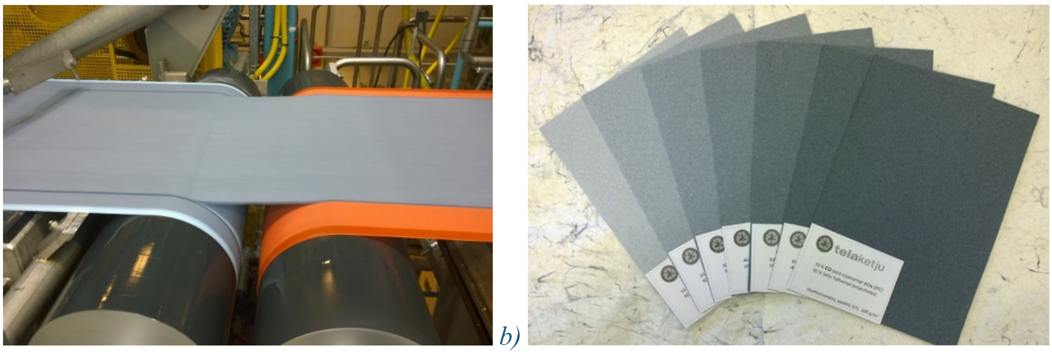
[5] www.purewaste.org
[6] https://dafecor.fi/
[7] http://www.laroche.fr/en

Post-consumer cotton waste, however, has worn during use and laundry, and therefore quality is reduced and there is variation in fibre properties (Palme, 2014). It requires sorting according to fibre type, since consumer waste contains mixed textile materials. Furthermore, coloration of pre-consumer materials are mixed, which affects appearance of materials unless sorted by colour as well. Furthermore, cleanliness and hygiene issues of post-consumer waste also needs to be taken into account in processing and especially in end-products.

Cotton materials can also be chemically recycled by dissolving and the obtained cellulose solution is used for the production of regenerated cellulosic fibres (Heikkilä *et al.*, 2018a; Heikkilä *et al.*, 2018b). Typical raw material for regenerated cellulosic fibres is dissolving grade pulp. Since degree of polymerization of cellulose in cotton is higher than that of dissolving pulp, worn cotton is well suited for the dissolution processes. One significant benefit of chemical recycling also is that fibre length and strength can be restored, thus regenerated cellulosic fibres from recycled cotton are long and strong enough to be suited for the production of yarns. Lenzing has launched Refibra fibres containing certain portion of recycled cotton as its raw materials. R&D activities are going on in making dissolving pulp from post-consumer cotton, as well as to adjust various regenerated cellulose processes for using recycled cotton as raw material, these include e.g. lyocell (Haule *et al.*, 2016) and cellulose carbamate processes (Heikkilä *et al.*, 2018b). Further benefit of the chemical recycling processing is that hygiene problems are solved simultaneously since microbes, mould etc. do not survive in the chemical processes involved.

End-products in mechanical recycling is typically down-cycling into low value nonwoven products using carding or air-laying processes. Making nonwoven products from mechanically recycled cotton by carding require certain fibre length. While air laying is capable of handle also shorter fibres, it has limited production speed and it do not enable production of thin materials. Typical fibre lengths in carding is 15-50 mm, and in air laying 5-30 mm. With wet laying process even shorter fibres are required.

The foam laying process is developed from the wet-laying/paper making process. In foam process water is replaced by aqueous foam which enables better formation and reduces water use compared to wet-laying, but the production process remains high-speed compared to other nonwoven processes. Foam laying process is very versatile. It is capable of utilizing residues that are waste to most mechanical recycling processes, i.e. very short fibre fractions, mixtures of fibre materials and even residual dust from a shredding process. Properties of the foam laid materials vary from paper-like thin structures to nonwoven-like structures and thicker webs. We have used recycled cotton in foam laying process earlier in Telaketju project (Figure 9). Product appearance and feel changed from a paper-like material to a softer, felt-like material with increasing the cotton content. Furthermore, with increasing amount of cotton, the strength and stiffness of the structure decreased (Figure 10).



a) b)

Figure 9 Web formation in foam laying in process and b) appearance of foam laid nonwovens from mixtures of recycled cotton and pulp (Heikkilä et al., 2019a & 2019b)

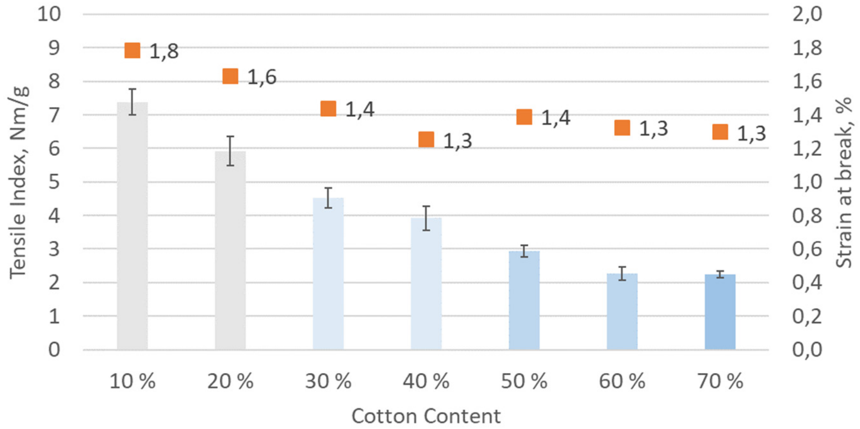


Figure 10 Tensile properties - tensile index and strain at break - as a function of amount of recycled cotton in CO/pulp mixture (Heikkilä et al., 2019a & 2019b)

The mechanically opened cotton fibres can also be used for yarn spinning. Depending on the remaining fibre length and its distribution mechanical recycled fibres can be used as mixture with virgin fibres or fibres recycled by other methods. Furthermore, open-end (OE) spinning is more suitable for shorter fibres and ring spinning. Pure Waste have tested the mechanically recycled post-consumer cotton fibres OE-spinning in industrial scale. The OE-yarn made from recycled fibres with the production scale machinery consisted of post-consumer textile waste (20 %), pre-consumer textile waste (40 %) and recycled polyester, r-PET (40 %). Yarn and fabric quality did not deviated from typical commercial OE yarns, and such grade has been commercialized (Figure 11).

a)


b)


Figure 11 a) T-shirt made of recycled fibres - material composing of 20 % of post-consumer cotton, 40 % of pre-consumer cotton and 40 % of polyester from PET bottles. (Heikkilä et al., 2019b), and b) corresponding commercial products by Pure Waste[8]

1.3 NordicBio Project

This report is summarizing work carried out by VTT within *Circular Nordic Bio Nonwoven in MedTech Applications* (NordicBio) project. NordicBio project aimed for development and pilot scale demonstration of a cost effective, bio and circular material based materials suitable for medical applications. Work was carried out in Swedish-Finnish co-operation project coordinated by Wargön Innovation. Finnish side of the consortium including VTT technical Research Centre of Finland Ltd. (VTT) and Pure Waste Textiles (Pure Waste) was funded by Business Finland and Swedish side including Wargön Innovation (Wargön), Cellcomb, RISE IVF AB (RISE), Fiber-X, Sporda Nonwovens (Sporda), and Stockholm Läns Landsting (SLL) was funded by Vinnova. Project composed of 12 work packages of which are listed in Table 2 as well as participants.

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8 https://www.purewaste.org/post-waste-era-by-pure-waste/post-waste-shirt.html

Table 2 Work packages (WPs) and participants in NordicBio project

No	Main themes of work packages	Leader and other partners
1	Market requirements & potential in MedTech application	Cellcomb, Wargön
2	Textile waste material collection & sorting	Wargön
3	Shredding efficiency	Pure Waste, RISE
4	Cleaning technologies	VTT
5	Production technologies, yarn	Pure Waste, VTT, RISE
6.1	Production technologies of nonwoven (foam & air)	VTT
6.2	Production technologies of nonwoven (carding & wet lay)	RISE, Fiber-X, Sporda
7	New MedTech product creation and testing	Cellcomb
8	Sales and usage analysis in MedTech application	Cellcomb, Wargön, SLL
9	Market and usage analysis related to yarns and fabrics	Pure Waste, SLL, Wargön
10	Project management	Wargön, Pure Waste
11	Communication	Wargön, Pure Waste

VTT's work focused on cleaning technologies and nonwoven production with foam and air laying processes. The other stages of the mechanical processing described above were not studied. This report summarizes work carried out in VTT's sub-project within Finland-Sweden bilateral NordicBio project. Description of work closely related to VTT sub-project, but carried out by other partners, is included for clarification. Goal and scope is described in Chapter 2, description of work and approaches are given in Chapter 3. Results are divided by the topic - cleaning process development as Chapter 4, nonwoven development as Chapter 5 and nonwoven piloting as Chapter 6. Summary and conclusions are made in Chapter 7.

2. Goal and Scope

Objectives of NordicBio project in general were:

- Develop circular and bio-based textile alternatives to fossil based materials in medical disposable items
- Development and scaling up of required recycling technologies
- Ensure low cost, ease-of-use, and required cleanness

Material flows and processes of the project are shown in Figure 12. Scope of the project includes both recycled fibres from post-consumer textile waste as well as natural plant fibres. Recycled materials require cleaning process prior using them in production of nonwovens or yarns for medical textile products. In the case of nonwovens recycled textile fibres was planned to be used as such or as blend with other fibres including regenerated cellulose fibres and cellulose pulp. Even though it would be ideal to be able to recycle disposable hospital items as a raw materials, such closed loop recycling was shortly studied, but not a part of the piloting work carried out within the project.

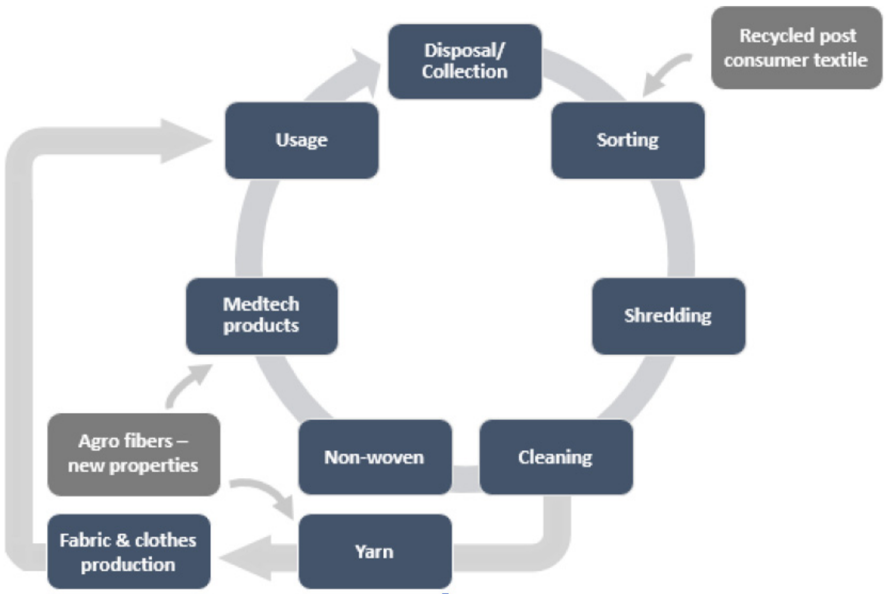


Figure 12 Material flows and processes in NordicBio project

Expected project outcome is novel circular and/or biomaterials meeting the requirements of hospital environments, but which would be equally applicable for related industry sectors like food, cosmetics and pharmaceutical. Furthermore, developed cleaning processes would enable wider use of mechanically recycled fibres in general.

VTT's project focuses on the development and piloting of cleaning process for mechanically recycled fibres and nonwoven manufacturing processes. VTT's project targets were:

- Up-scaled cleaning method for mechanically recycled fibres meeting medical textile criteria
- Pilot scale production of nonwoven material for medical application
- Carry out this work given budget in a way that also converting and testing of these materials can be carried out by given project duration

VTT work had direct linkages to work carried out by other partners as described in Chapter 3.

3. Overview of VTT Work and Linkages to Other Partners

VTT role focused on the development and piloting of cleaning process and testing of foam and air laying technologies for production of nonwovens from recycled fibres, and production of nonwoven materials in pilot scale for making medical products. Summary of VTT work including linkages with other work packages and partners is shown in Figure 13.

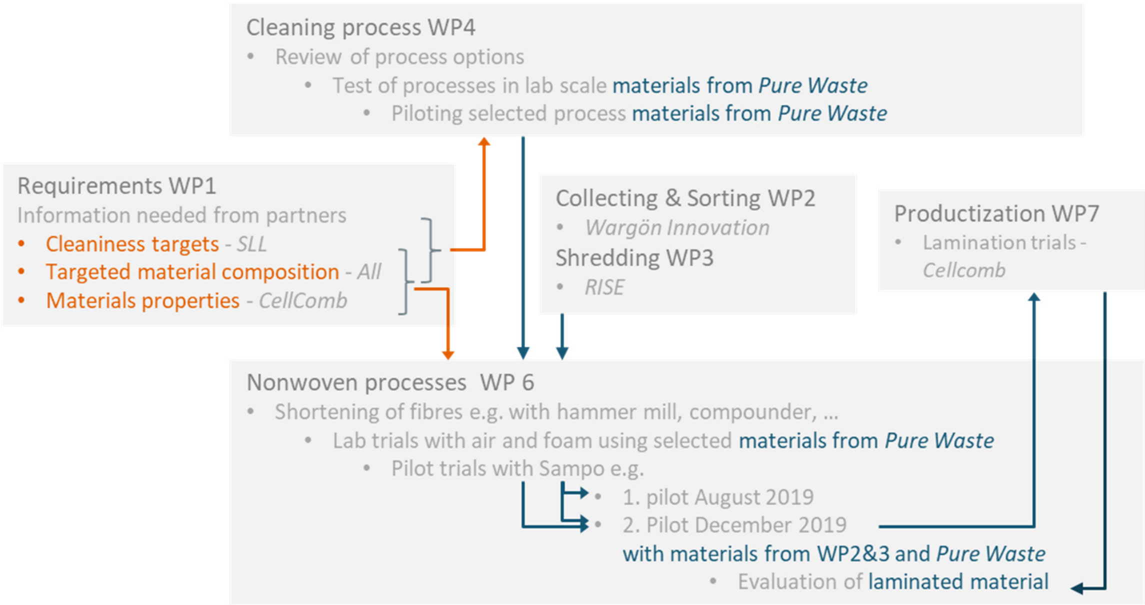


Figure 13 Summary of VTT work and its information (orange) and material linkages (blue) to other WorkPackages and partners

Material and process requirements for VTT part determined in WP1 are described in Chapter 3.1 and recycled raw materials obtained from project partners is summarized in Charter 3.2. VTT work and results, including evaluation of materials laminated by Cellcomb, are described within Chapters 4-6.

3.1 Requirements for Medical Nonwovens

Nonwoven materials can be used in wide range of applications within hospitals ranging from disposable garments, bed sheet to wound care and absorptive products. Medical textiles have specific requirements concerning their composition and properties. The most important standards to fulfil are ISO 13485:2016, the standard for medical devices and SS-EN 13795:2011+A1:2013. There is also a standard for textiles, SS 8760023, but it is not applicable for disposables. Quality control and technical specifications of disposable products were available from website of Swedish national agency for public procurement (Upphandling myndigheten, 2019).

Targets for the materials developed in NordicBio projects were agreed in kick-off meeting with Cellcomb, who is a producer of disposable medical and hospital textiles. Their main interest for this project was to find alternatives for laminated textile products suitable for bed sheet and absorptive products. Main property sought in this project was softness and hand feel. Bed sheets should have sufficient strength in use, but numerical value was not determined. Materials should not lint. Eco friendliness and antimicrobial activity were also included as beneficial properties. Furthermore, these materials should be clean enough, but sterilization or clean room type products are not needed. Preferred colour was white(ish), which could be obtained by sorting of textiles. However, colour removing treatments were considered also interesting, and it was, thus, included into lab scale testing plans.

Hydroentanglement (i.e. spunlacing) would be the best binding method for hospital textiles, since no additional chemicals are needed. That method is available in lab scale at VTT, and in large scale at

Sporda. Traditional latex bonding was used in experiments for practical reasons, but it is not preferred in hospital applications. However, latexes could be replaced in future by similar organic bonding agents. Thermal bonding using bicomponent (bico) fibres is also acceptable, but not a preferred binding option. Target minimum length for produced nonwoven rolls was 200 meters that laminating tests can be executed in Cellcomb.

3.2 Recycled Raw Materials

Recycled textile fibres used in NordicBio trials at VTT included pre-consumer i.e. post-industrial material obtained from Pure Waste and post-consumer materials obtained from Swedish partners. VTT obtained 5 batches of fibres:

Pure Waste material used in the project was mechanically opened black cotton from cutting wastes of t-shirt jersey.

- Cotton batch (COpi1) - obtained 2018 - black, used for fibre shortening trials, and lab scale air-laying and foam-laying trials
- Cotton batch 2 (COpi2) - 50 kg, obtained 06/2019 (opened at RISE) - white, used for fibre cutting tests and first nonwoven pilot
- Cotton batch 3 (COpi3) - 165 kg, obtained 08/2019 - black, used for cleaning pilot, second nonwoven pilot, and in laboratory scale bacteria and discoloration tests including material discoloured to Pure Waste

Post-consumer textile materials were collected and sorted by Wargön, where sorting was carried out manually. Materials were shredded and mechanically opened by RISE. Opened fibre batches included:

- Cotton-polyester batch 1 (polCOpc1) - 42 kg, obtained 06/2019, estimated fibre ratio approx. 70/30, white, used for first nonwoven pilot
- Cotton-polyester batch 2 (polCOpc2) - 250 kg, obtained 12/2019, mixture of two grades with polyester/cotton ratios of 65/35 (working pants) and 50/50 (hospital clothes), used for second nonwoven pilot

Fibre coding: CO = cotton, polCO = polycotton = mixture of cotton and polyester, pi = post-industrial = pre-consumer, pc = post-consumer

4. Cleaning Process

This chapter describes literature review (4.1), lab scale process development and trials (4.2), and piloting of cleaning process (4.3).

4.1 Literature Review

The main objectives of literature review was evaluate how different kinds of contaminants such as dirt, stain and particularly micro-organism can be removed from used waste textiles without sacrificing mechanical fibre properties. To some extent colour/dye removal of textiles was also reviewed. The strategies to remove different kinds of contaminants and additives from textiles are summarized at the end of this chapter in Table 3.

Removal of contaminants

Industrial laundering employs standardized processes for effective dirt and stain removal and ensure a sufficient hygienic reconditioning of textiles with minor harmful effects on textile properties. The most effective way to remove dirt, stain, soil and most of the micro-organism is to use high over 60°C washing temperatures. If high temperatures are not allowed, lower temperature, 40°C, combined with longer washing time, use of surfactants or oxidative (bleaching) detergents can be applied depending on the type of contamination. For example, in the case of soil an extended washing at 40°C has reported to give the same cleaning performance than standard washing at 60°C (Janczak *et al.*, 2010). Also, surfactants are effective detergents for basic cleaning and the removal of hydrophobic soil, but not for microbial load (Brands *et al.*, 2016).

In Finland and Sweden, so called thermal disinfection at laundries is typically carried out at 70°C or more for at least 10 minutes (Forss, 2017; Rahunen, 2019; Tano & Melhus, 2014). High washing temperatures for long periods of time result in a significant environmental impact through large energy expenditure and can shorten lifetime of the fabrics. A common alternative is to use chemical disinfection (or chemothermal disinfection) i.e. wash at lower temperatures and combine this with a biocide types of chemicals such as oxidative chemicals typically peracid containing detergents at slightly alkaline conditions (Forss, 2017; Rahunen, 2019; Tano & Melhus 2014). The amount of oxidative chemicals required for disinfection increases with decreasing temperature (Rahunen, 2019). According to Bockmühl (2017) laundering below 60°C without AOB (peracetic acid derived from perborate or percarbonate and bleach activator tetraacetylethylenediamine TAED) or chlorine does not guarantee efficacy against all kinds of micro-organisms, especially in critical cases such as dermatomycoses or gastrointestinal infections. Peracetic acid possesses broad-spectrum biocidal activity at low temperatures and in the presence of soil. When TAED is used as activator, peracetic acid can be generated *in situ*, thus eliminating the direct handling of peracetic acid. Peracetic acid solutions are unstable and volatile, evolving pungent fumes at low concentrations. They are potentially explosive and extremely corrosive (Jones, 2007). In USA, some laundries use ozone as oxidative chemical for disinfection. Ozone gas is dissolved in cold (ambient) washing waters and washing is carried out at lower pH under 9 (Rice *at al.*, 2009).

The prolongation of the wash cycle at low temperatures (under 50°C) does not complete the elimination of micro-organism, so increased time can only partly compensate for decreasing temperatures (Honisch *et al.*, 2014). Mechanical action during washing by removing microbial cells on the textile surface during laundering both at low and elevated temperatures is beneficial even with the use of oxidative chemicals (Brands *et al*., 2016). In addition, tumble drying after washing textiles at 60°C or 70°C has reported to reduce the number of bacteria as much as washing (Tano & Melhus, 2014).

Benefit of higher temperature (≥ 60°C) is that it ensures complete inactivation of non-enveloped virus such as poliovirus and norovirus, and with bleaching such temperature also inactivate fungal infections. Due to this the European standard for chemical-thermal textile disinfection (DIN EN 16616) applicable in nursing homes, hospitals, hotels, and food processing premises, requires more testing of materials washed temperatures below 60°C (bacteria including *P. aeruginosa*, *Esherichia coli*, *S. aureus*, *E. hirae* and yeast *C. albicans*, and if possible also: mould *Aspergillus brasiliensis* and mycobacteria) than those washed in temperatures of 60°C and above (only bacteria *Enterococcus faecium*). However, bacteria *Clostridium difficile* (causing hospital infections) can be found even after laundry at 71°C. (Lakdawala *et al.*, 2011)

Removal of dyes and cross-linked textile finishing chemicals

Cellulose based textile can be dyed with different kinds of dyes. Dye types attach differently to cellulose: reactive dyes form covalent bonds between the dye molecule and the hydroxyl groups of cellulose (Lewis, 2007) and direct dyes attach through van der Waals forces and hydrogen bonding (Lim & Hudson, 2004). Vat dyes are trapped mechanically inside the cotton structure during the dyeing process, in which the water-soluble leuco form of the vat dye penetrates the fibre structure and becomes trapped inside when it is oxidized back to an insoluble form (Aspland, 1992). According to Aspland (1997), complete chemical stripping, i.e. removing dyes from coloured fabrics, may be carried out either with reduction or oxidation or with combination of these methods depending on the dyes. In the past, direct dyes were removed by boiling the fabric in alkaline sodium hydrosulphite or bleaching with sodium hypochlorite. Vat dyes were treated in high temperature reduction bath containing caustic soda and sodium dithionite or sodium hydrosulphite and quaternary ammonium salt as stripping assistant or a substance that was responsible to combine with the dye molecule, so it does not re-attach with textile material once it has been stripped out (Chavan, 1969; Sapers, 1971).

Nowadays, oxidative chemicals such as ozone, hydrogen peroxide and peracids have replaced chlorine and permanganate-based treatments (Winkler *et al.*, 1997; Walger *et al*., 2015; Eren *et al.*, 2016). With bi-hetero type reactive dyes 89-94 % stripping efficiency were achieved when reductive treatment with the combination of sodium hydroxide and sodium hydrosulphite was used at elevated temperatures 80°C and 100°C (Uddin *et al.*, 2015). In addition to effective dye removal, a sequential alkali-acid or acid – alkali stripping of reactive dyes from cotton fabrics has been reported to remove cross-linked textile finish (Haule *et al.*, 2014; Wedin *et al.*, 2018). If hydrogen peroxide treatment was added into the acid-alkali sequence, over 90 lightness values was got with Tencel fabric dyed with azo-based reactive colours, Black 5 and Red 228, but anthraquinone-based reactive dye was highly resistant for the removal (Bigambo *et al.*, 2018). Määttänen *et al.* (2019) have studied the combination of different chemical treatments (alkaline, acid, hydrogen peroxide, ozone and dithionite) on the removal dyes from direct, reactive and vat dyed cotton textiles and found that to some extent colour can be removed all of them, most easily and with highest brightness from direct dyed cotton.

Influence of disinfection and bleaching on fibre properties

High concentrations of hydrogen peroxide (> 6.5 ml 30% hydrogen peroxide agent/kg clothes) at high temperature (≥ 70°C) with low bath ratio (1:3) has reported to cause mechanical and chemical damages for cotton textiles in laundering after 50 washing cycles (Fijan *et al.*, 2007). Also bleaching with TAED at 40°C after 50 washing cycles caused tensile strength loss and damages especially if Mn-TACN or TACN salt were used as activator (Reinhardt, 2006). With increase of concentration of reductive stripping chemicals (up to 10 g NaOH and hydrosulphate /l) and temperature (80°C => 100°C) damages to the cotton fabric strength has reported to increase. The maximum strength loss was 10% (Uddin *et al.*, 2015).

Table 3 Strategies to remove different kinds of contaminants and additives from textiles

Contaminant / chemical residue to be removed	Methods and additional notes
Dirty/stain/soil	Laundry type process • 60…90°C washing • 40°C washing + longer time + surfactants + bleach/oxidative detergent (per acid / peroxide/ oxygen /ozone-based bleaching)
Micro-organism	Main methods • High temperature ≥ 60°C • Lower temperature ≤ 60°C and oxidative bleaching Additional effect with • Cationic antimicrobial activators such as quaternary ammonium compounds (BAC, DDAC) are added after washing in rinsing. They form interaction between textile surface reducing the microbial burden from washing machine biofilm • Mechanical action in washing machine for removal of microbial cells • Tumble drying Mechanical and chemical damage with higher concentrations of oxidative chemical after several washing cycles
Dyes	• Bleaching: reductive, alkaline, oxidative treatment and combination of them depending on dye type • Removal might increase absorption properties, when covalent bonds between dye and cotton fibre are removed • With higher temperatures and chemical concentrations detrimental effect on fibre mechanical properties increases
Cross-linkers from finishing treatments	• Alkaline + acid / acid- alkaline treatment

Conclusions: Disinfection of textiles from different king of micro-organism should be done either with high temperature, ≥ 60°C, or with lower temperature together with oxidative chemical.

Dyes removal effect depends on the used dyeing method and combination of chemicals used for removal. With sequential treatment higher removal can be achieved.

Both disinfection and bleaching treatments can be harmful for the mechanical properties of textile material. In laundry type of testing typically 50 washing cycles are run before damage analysis therefore influence of single treatment is not known.

4.2 Lab Scale Development and Trials

4.2.1 Effect of cleaning process on fibre strength properties

Objective:

Evaluation the influence of cleaning (disinfection) conditions (see Table 4) on cotton and polyester fibre strength properties. Based on the results (strength properties) experiment points with dyed PureWaste cotton **COpi3** for sheet property tests were selected (see 4.2.2).

Materials and Methods:

Materials used in these trials were virgin staple cotton (vCOref) and new polyester filaments (vPESref). (see Figure 14) in order to provide sufficient initial length for fibre strength testing. Polyester filaments were cut manually into 40-50 mm length. The mechanical properties of the fibres were determined according to the ISO 1973 and ISO 5079 standards using a Textechno Favegraph testing machine for individual fibres. Cotton fibres were measured at Tampere University of Technology and polyester fibres at VTT.

Figure 14 Virgin cotton staple fibres (vCOref) and polyester filament fibres (vPESref) used for studying effect of cleaning process conditions

The used detergents and chemicals are listed below and cleaning conditions described in Table 4. All treatments were done in buckets at water bath with manual mixing. Cleaning process was carried out for 60 min at 10% fibre consistency.

- Peracid Forte (PF), commercial laundry bleaching and disinfection detergent by Christeyns
  - Mixture of peracetic acid (10-20%), hydrogen peroxide (15-30%) and acetic acid (15-30%)
  - Preliminary recipes for disinfection treatments at different temperatures, based on bacteria fabric tests carried out in MetropoliLab, were got from Christeyns
- Select Power Blue (SPB), alkaline washing detergent without oxidative agent by Christeyns
  - NaOH content 30-50%
  - Preliminary recipe for disinfection treatment at 70°C, based on bacteria fabric tests carried out in MetropoliLab, was got from Christeyns
- Pure NaOH, H_2O_2 treatments were tested as alternative disinfection and bleaching chemicals for commercial laundry detergents

Table 4 Conditions of cleaning treatments of vCOref and vPESref. Dosages are given either as % on fibres or as concentration g/l

Treatment	Temperature, °C	Dosage, % or g/l	Comment
Laundry detergent without oxidative bleach activator (Select Power Blue, SPB)			
SPB-1.3/60 SPB-1.3/70	60 70	1.3%	60°C limit temperature for micro-organism (*Enterococcusfaecium)*testing acc. DIN EN 16616 70°C used in Finland and Sweden for disinfection without oxidative agent
Laundry detergent with oxidative bleach activator (Peracid Forte, PF) (pH adjusted 9-10 with SPB)			
PF-1.2/40 PF-6/40 PF-1.2/60 PF-6/60 PF-1.2/70 PF-6/70	40 60 70	1.2% and 6%	40-60°C temperatures for chemical disinfection 70°C can give more effective discoloration
NaOH			
NaOH-40/60 NaOH-40/70	60 and 70	40 g/l	Discoloration effect 6 was got with this concentration in Trash-2-Cash project
H_2O_2 (pH adjusted >10 with NaOH)			
P-0.5/40 P-1.2/40 P-0.5/60 P-1.2/60 P-0.5/70 P-1.2/70	40 60 70	0.5% and1.2%	Chemical disinfection and discoloration

Results:

The fibre strength properties of polyester fibres after oxidative and alkaline cleaning in different temperatures are presented in Figure 15. Clearly increase of temperature decreased the fibre strength properties with all chemicals (oxidative and alkaline). At the same temperature cleaning without oxidative chemical resulted lower strength properties. The biggest reduction in strength properties, 20%, was got with alkaline chemicals (SPB and NaOH) at 70°C. The effects of Peracid Forte and hydrogen peroxide on fibre properties were very similar.

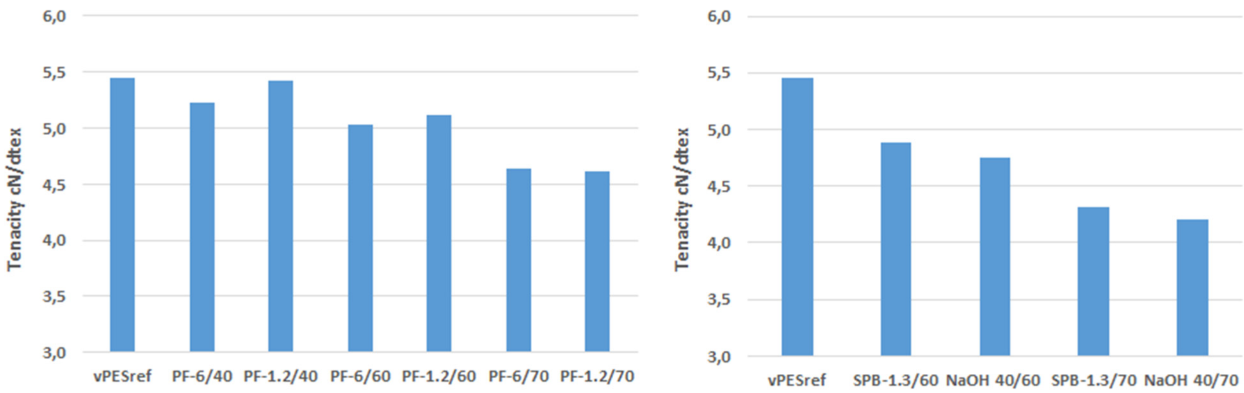


Figure 15 Effect of cleaning conditions on the polyester fibre strength properties. Raw material vPESref

In the case of cotton fibres there were lot of variations in the fibre tenacity values already in the original starting material. The most stable and highest fibre strength properties were achieved with Peracid Forte treatments Figure 16. It seems that higher temperature is not as detrimental for cotton strength properties as it is for polyester's. Cleaning with high alkali concentration resulted the lowest fibre strength properties Figure 17.

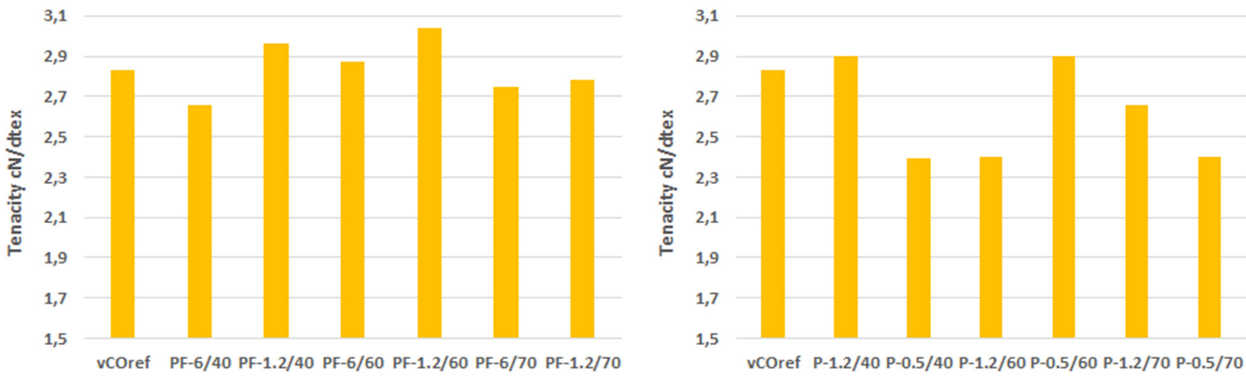


Figure 16 Effect of oxidative cleaning treatments (Peracid Forte (PF) and hydrogen peroxide (P)) on the cotton fibre strength properties. Raw material vCOref

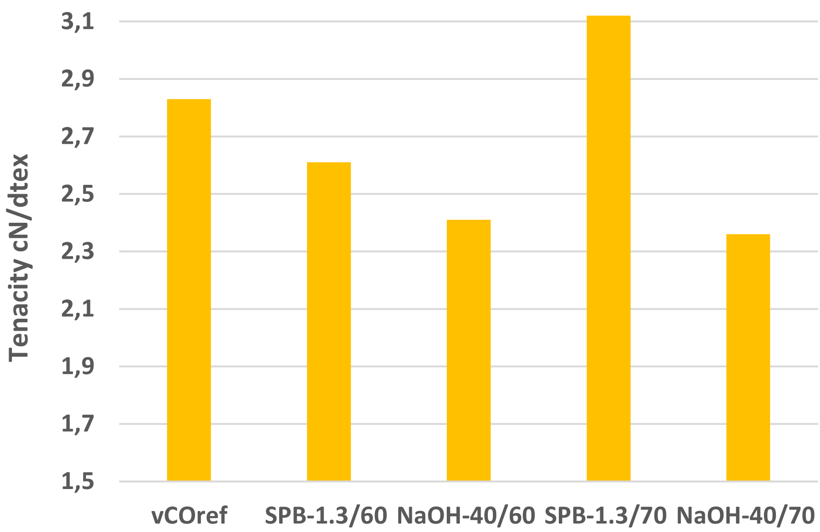


Figure 17 Effect of thermal cleaning with different alkaline chemicals (SPB and NaOH) on the cotton fibre strength properties. Raw material vCOref

Based on the results experiments with the dyed PureWaste cotton COpi3 for sheet property tests were decided to do mainly at temperature 60°C without hydrogen peroxide treatments.

Conclusions: High temperature and high alkali concentration decreased fibre strength properties => 60°C was selected for the main temperature in the following cleaning trials.

The influence of Peracid Forte and hydrogen peroxide treatments on fibre strength properties were rather similar. With cotton material Peracid Forte resulted more stable performance, therefore Peracid Forte was selected as oxidative treatments in the following cleaning trials.

4.2.2 Effect of cleaning process on sheet properties

Objective:

Evaluation the influence of cleaning (disinfection) conditions on foam formed hand-sheet appearance made from cleaned cotton fibres. Additional target was to check how the selected cleaning conditions removed colour from the reactive dyed cotton fibres.

Materials and Methods:

Materials used in these trials was cotton batch 3 (COpi3) from PureWaste. The first set of cleaning test was done from the material as such, but after hand sheet preparation at Jyväskylä (manufacture of foam formed hand sheets is described in Chapter 5.3), it was noticed that fibre length was too long for preparing good quality sheets for evaluation. Therefore, cotton was shortened by granulator and part of test points were repeated. The conditions and chemicals of cleaning test are presented in Table 5. Experiments were carried out in mixing reactor with 300 g (oven dry/100%) cotton / batch. Yield, brightness (ISO 2470) and viscosity (ISO 5351) were determined from the cleaned fibres. The tenacity of the fibres was also planned to measure, but the dark colour of fibres made it impossible to set fibres to the clamp of the measure instrument.

*Table 5 The conditions and chemicals of cleaning experiments for hand sheet tests. Textile raw material was COpi3. Experimental point marked with * were repeated with shortened fibres.*

Treatment	Temperature, °C	Dosage, % or g/l	Comment
Laundry detergent without oxidative bleach activator (Select Power Blue, SPB)			
SPB-1.3/60* SPB-6/60	60	1.3%	60°C limit temperature for micro-organism (only *Enterococcusfaecium)*testing acc. DIN EN 16616, no big strength lost seen in 4.2.1 trials Higher dosage tested for colour removal effect
Laundry detergent with oxidative bleach activator (Peracid Forte) (pH adjusted 9-10 with SPB)			
PF-1.3/60* PF-6/60	60	1.3% and 6%	At 60°C no big strength lost seen in 4.2.1 trials Higher dosage tested for colour removal effect
NaOH			
NaOH-20/60 NaOH-40/60 NaOH-40/80*	60 and 80	20 and 40 g/l	Effect of alkali concentration and temperature tested for colour removal
O_3 (pH adjusted <9 with SPB, consistency 12% and reaction time 7.5 - 15 min depending on O_3-dosage			
O_3-0.5/RT O_3-1/RT*	RT	0.5% and 1%	Chemical disinfection and discoloration

Results:

The handling long fibres in cleaning trials were rather non-homogenous. Only O_3 and alkali treatments removed very slightly the colour of the fibres. The highest increase in brightness, 8 ISO-%, was got with NaOH-40/80. Typically, viscosity values of cotton materials were between 1400-1500 ml/g. Only higher PF dosage (PF-6/60) and O_3 treatments decreased cotton's viscosity around 1000 ml/g indicating chemical damages in fibre structure (Fijan *et al.*, 2007), which can also influence on the mechanical strength properties of fibres. As mentioned before the quality of the foam formed hand sheets (Figure 18) made from the long fibres was too poor for evaluation.

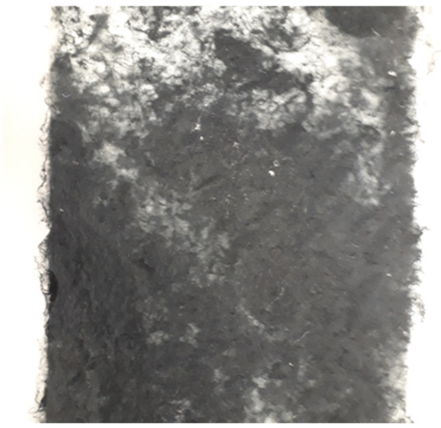


Figure 18 The original fibre length of COpi3 was too long for the preparation of good quality hand sheets for evaluation.

Shortening of fibres (more information in Chapter 5.1) improved handling of material in cleaning treatments, which improved the homogeneity of the treatments. The results of fibre properties after treatments are presented in Table 6. The highest yield loss and highest brightness were resulted from the high concentration NaOH treatment at 80°C (NaOH-40/80). The viscosity of the cotton fibres were decreased in O_3 and NaOH treatments.

Table 6 COpi3 (shortened) fibre properties after cleaning trials

Test point	Yield, %	Brightness, %	Viscosity, ml/g
SPB-1.3/60	100.3	1.9	1470
PF-1.3/60	99.8	2.5	1430
O_3-1/RT	98.8	3.4	1110
NaOH-40/80	95.9	15.8	1180

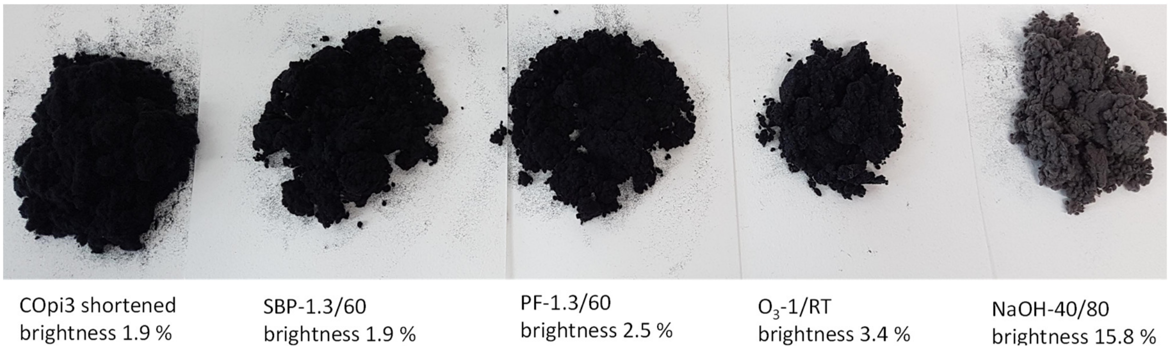


Figure 19 COpi3 fibres before and after cleaning treatments

The appearance of foam formed sheets based on shortened and cleaned cotton fibres is shown in Figure 20. NaOH treatment at 80°C led to clearly lightest colour of sheet. All cleaned fibre grades worked well in foam forming process and the formation of sheets was otherwise rather good, but clear cotton fibre clumps existed in sheets (more info related to fibre clumps is presented in Chapter 6.2). The strength of sheets was very low, so any mechanical tests could not carry-out. Low strength level also led to break-up of some sheets in sheet handling, which can be seen as missing parts. Based on these experiences it was decided to refine part of cleaned cotton fibres for better bonding in pilot trials.

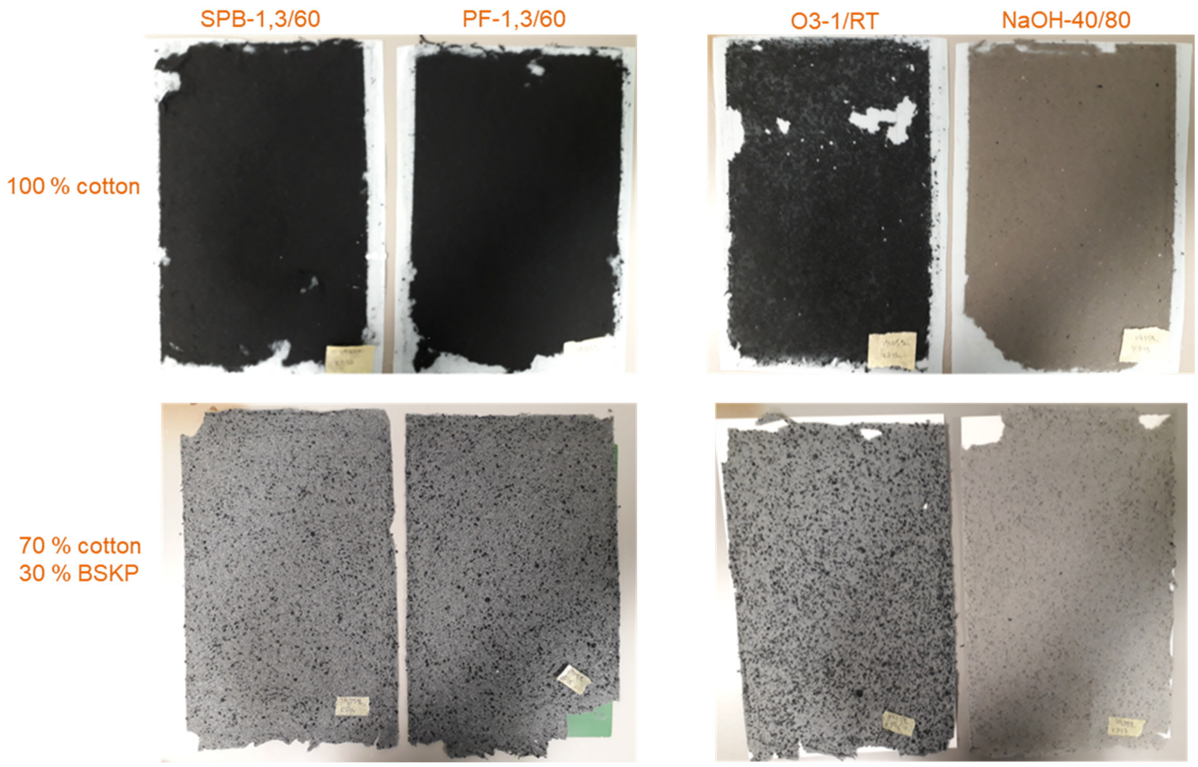


Figure 20 Appearance of sheets based on shortened and cleaned cotton fibres

Based on the results Peracid Forte treatment (PF-1.3/60) was selected for the Pilot Cleaning runs. It contained double disinfection effect thermal and oxidative. And fibres' viscosity value indicated that cleaning was not harmful for fibre strength properties.

Conclusions: Shortening of cotton fibres improved handling of material in cleaning trials and improved formation of hand sheets. The strength of sheets was very low, so any mechanical tests couldn't carry-out, therefore it was decided to refine part of cleaned cotton fibres for better bonding in pilot trials.

The highest discoloration effect (brightness 15.8 ISO-%) was got with NaOH-40/80. Unfortunately it also resulted the lowest cotton viscosity and the lowest treatment yield.

Peracid Forte treatment (PF-1.3/60) was selected for the Pilot Cleaning runs due to its double disinfection effect: thermal and oxidative.

4.2.3 Discolouration of recycled cotton

Objective:

Evaluation the colour removal of reactive dyed cotton using reductive bleaching and combination of reductive and oxidative bleaching.

Materials and Methods:

Materials used in these trials was cotton batch 3 (COpi3) from PureWaste. Discoloration experiments were carried out in buckets at water bath with manual mixing. Reductive bleaching was done using dithionite ($Na_2S_2O_4$, marked Y in exp. points) and either NaOH or SPB as alkali source. Oxidative bleaching with hydrogen peroxide (H_2O_2, marked P in exp. points) was done for selected samples after dithionite stage. The used bleaching conditions are presented in Table 7. Treatment consistency in dithionite stage was only 4% because of the avoiding mix air into pulp suspension, which would decompose dithionite and decrease its bleaching efficiency. In industrial processes dithionite treatment is carried out under vacuum and in higher consistency. The higher consistency enables to decrease chemical dosages with same or even better discoloration response, which would make the bleaching more economic. Analyses carried out were yield, brightness from carded sample and fibre strength.

Table 7 Conditions of reductive and oxidative bleaching stages

Treatment	Dosage, % on fibres	Alkali dosage, % on fibres	Consistency, %	Temp., °C	Time, min	Comment
Reductive bleaching with dithionite (Y)	2, 3.5, 5, 7.5, 10, 20	pH 6.5 with H_2SO_4 or SPB: 1.3 and 3.3 or NaOH: 1, 2.5, 5, 10, 20, 48	4	60, 80	60 120	Only some exp. at 60°C with 60 and 120 min Most exp. at 80°C with 60 min
Oxidative bleaching with H_2O_2 (P)	1.5	NaOH:1.1% and Epsom 0.5%	8, 10	70	180	Most exp. at 8% consistency due to better mixing

Results:

Both alkalinity and dithionite dosage had influence on discoloration of reactive dyed cotton in the reductive bleaching stage (Figure 21). Alkalinity influenced on the tone of the colour. The higher alkalinity more bluish was the tone after discoloration. Cotton yield after dithionite treatments were typically between 95.5-98.8%. Lower with higher NaOH concentration.

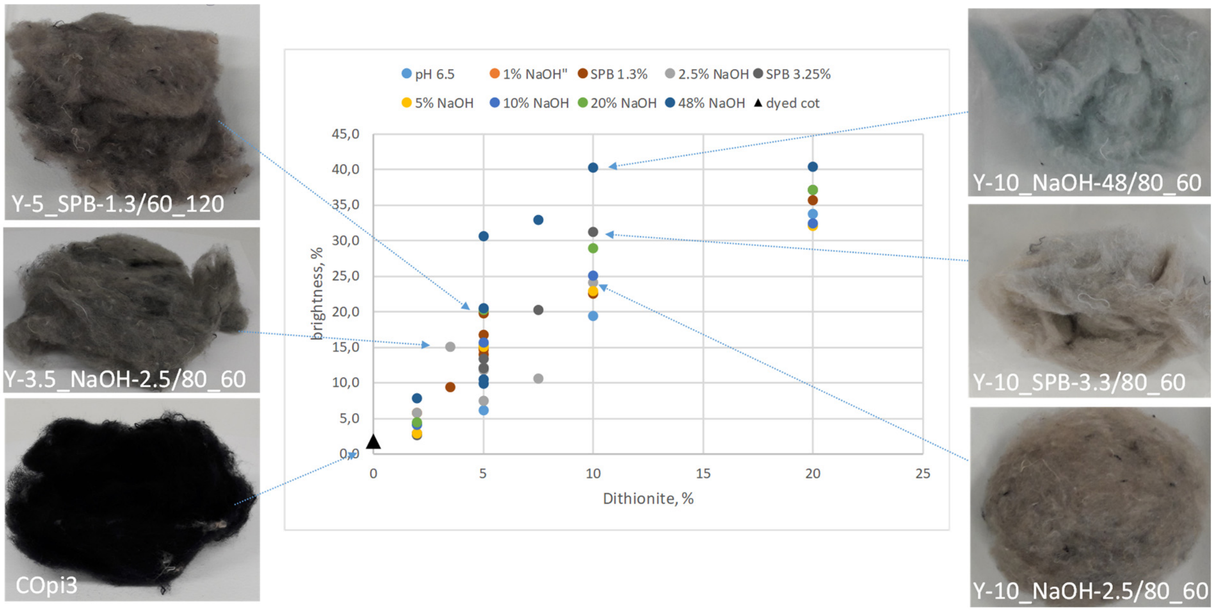


Figure 21 Influence of dithionite and alkali dosage on cotton brightness. In the experiment point labels is first dithionite dosage, then alkali source and its dosage followed by temperature and treatment time

Oxidative hydrogen peroxide treatment gave only few units increase in the brightness of reactive dyed cotton (Figure 22). The average cotton yield after peroxide treatments was 99%. Hydrogen peroxide treatment turned the tone of the colour reddish. Fibre strength properties (tenacity) measured from selected samples did not show clear effect of different discoloration conditions on fibre properties.

Based on the results reductive bleaching Y-10_SPB-3.3/80_60 was selected as discoloration treatment of larger amount (3 kg) of COpi3 material for yarn spinning trials to PureWaste. Its tone of the colour was best for re-dying after yarn making.

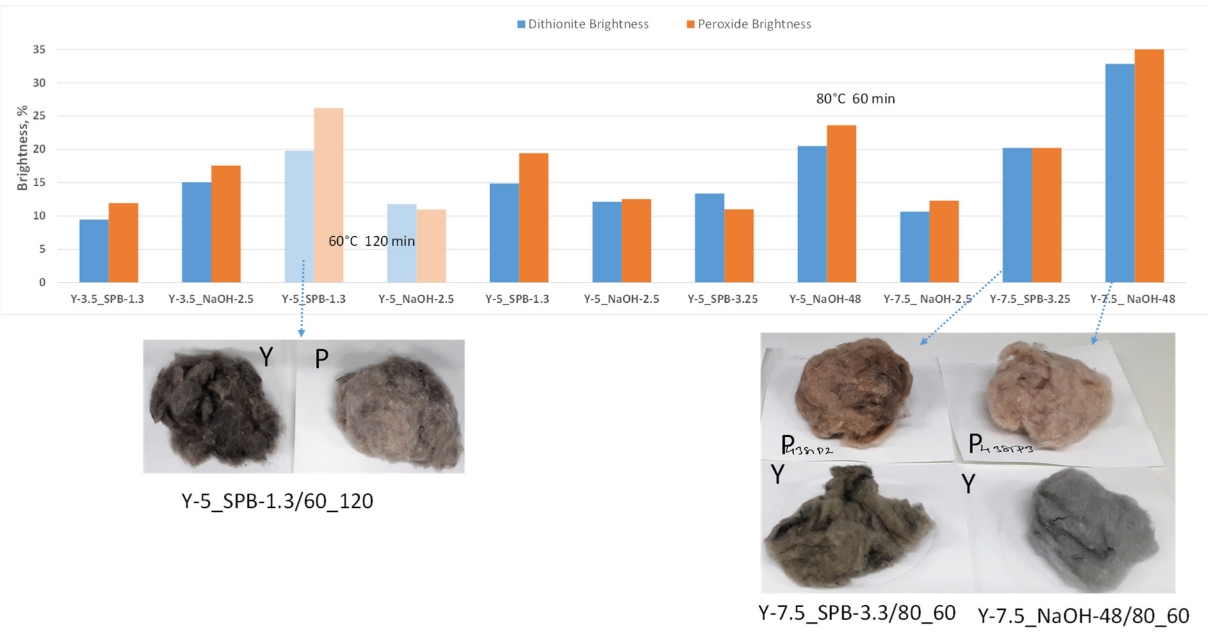


Figure 22 Influence of sequential reductive and oxidative bleaching treatments on cotton discoloration. In the experiment point labels is first dithionite dosage, then alkali source and its dosage followed by temperature and treatment time. Processing conditions were constant in peroxide stage, therefore they are not marked

Conclusions: Reductive bleaching with dithionite discoloured reactive dyed cotton. Higher dithionite and alkali dosage resulted higher brightness. The higher alkalinity the more bluish was the tone of the colour after discoloration.

Oxidative hydrogen peroxide treatment after dithionite stage gave only few units increase in the brightness. Hydrogen peroxide treatment turned the tone of the colour reddish.

Reductive bleaching with 10% dithionite dosage and 3.3% SBP dosage (as alkali) at 80°C for 60 min (Y-10_SBP-3.3/80_60) was selected as discoloration treatment of larger amount (3 kg) of COpi3 material for yarn spinning trials to PureWaste.

4.2.4 Efficiency of the removal of micro-organism

Objective:

Evaluate the efficiency to remove micro-organisms with selected cleaning/disinfection treatments.

Materials and Methods:

Screening the efficacy of different disinfection / decontamination / cleaning methods was performed using worst case scenario (=high concentration of various microbe types was added to cotton before post treatments). Textile material used in these trials was cotton batch 3 (COpi3) from PureWaste. Cotton samples in 15 g portions were contaminated with six different microbes by mixing microbes into cotton one hour before disinfection treatment. After disinfection the amount of survived microbes were analysed from the samples. Reference material was treated with micro-organism cocktail without disinfection treatment, and also effect of foaming agent SDS was studied. Microbes, their culture collection number and concentration used are presented in Table 8. The used disinfection treatments are presented in the Table 9.

Table 8 Information of microbes used in trials

Microbe	VTT's culture collection number	Concentration, cfu/ml	Additional information
Mould, *Aspergillus niger*	VTT D-81078	$3.0x10^6$	black mould, common in indoor air.
Bacterium spore, *Bacillus atrophaeus* spores	VTT E-052737	$2.8x10^7$	very resistant form of microbes, surrogate for pathogenic *Bacillus anthracis*
yeast, *Saccharomyces cerevisiae*	VTT C-96203	$1.3x10^8$	yeast for making bred
bacterium gram negative, *Pseudomonas fragi*	VTT E-98200T	$4.1x10^3$	slime forming bacteria
bacterium gram positive, *Micrococcus luteus*	VTT E-91474	$2.4x10^7$	common on human skin
virus, MS2 bacteriophage	DSM 13767	1.5 ml/sample, 10^8 cfu/ml	surrogate for harmful viruses, needs *E.coli* for host

Table 9 Treatment conditions of micro-organism removal tests

Treatment	Codes	Dosage, % on fibres	Alkali dosage, % on fibres	Consis-tency, %	Temp, °C	Time, min	Comment
Reference	COpi3						No disinfection treatment
SDS	SDS-0.6/RT	0.6 g/l	-	1.5	RT	2	Simulation of SDS addition in foam forming
Peracid Forte	PF-1.6/40 PF-1.6/60	1.6	SBP 1.3	8	40 60	60	PF-1.6/60 was used at pilot trails
Reductive dithionite (Y) bleaching	Y-10/80	10	SPB 3.3	4	80	60	3 kg sample to Pure waste was done with this
Sequential Y-P bleaching	Y-10/80+ P 1.3/70	1.3	NaOH 1% Epsom 0.5%	8	70	60	Conditions in Y-stage same as above
Vaporised H_2O_2 at RT	VHP-low/RT				RT		VTT's reference treatment

Results:

The amounts of survived microbes found from the samples after disinfection/ cleaning treatments are presented in Table 10. The growing of *Pseudomonas fragi* (bacterium gram negative) did not succeed well in microbe mix, therefore the number of units is lower than with other microbes. The detection limit for virus was 2700 PFU/sample and for the other microbes 340 CFU/sample. The most effective disinfection treatments for all microbes were the combination reductive and oxidative discoloration treatments at high temperatures (70°C and 80°C). The second effective was reductive discoloration at 80°C. These two treatments killed effectively the resistant bacterium spore due to the use of high temperature. In addition, oxidative treatment using laundry detergents at lower temperatures (40°C and 60°C) were effective for other microbes except for bacterium spore.

Table 10 Amount of survived microbes detected from samples after disinfection treatment

Code	*A. Niger* (mould)	*B. atrophaeus* (bacterium spore)	*S. Cerevisiae* (yeast)	P. fragi (bacterium gram -)	M. Luteus (bacterium gram -)	MS2 phage (virus)
COpi3	780 000	19 000 000	36 000 000	20 000	20 000 000	500 000 000
SDS-0.6/RT	130 000	510 000	340 000	6 800	1 500 000	17 000 000
PF-1.6/60	< 340	130 000	< 340	< 340	< 340	< 2 300
PF-1.6/40	2 700	410 000	< 340	< 340	< 340	< 2 300
Y-10/80+P-1.3/70	< 340	< 340	< 340	< 340	< 340	< 2 300
Y-10/80	< 340	680	< 340	< 340	< 340	< 2 300
VHP-low/RT	24 000	510 000	510 000	< 340	300 000	25 000

The logarithmic reduction of microbes after treatments are presented in Table 11. In general, reductions smaller than one log unit are not significant. Reduction bigger than three log units are considered effective in surface disinfection. Effective disinfection in liquid phase can be achieved if reduction is bigger than 3-5 log units depending on microbe. In liquid microbes can escape in all directions therefore, reduction efficiency should be higher. As can be noticed the discoloration treatment at 80°C are suitable for liquid disinfection with all the tested microbes.

Table 11 Logarithmic reduction of microbes after cleaning/ disinfection treatments

Code	*A. Niger* (mould)	*B. atrophaeus* (bacterium spore)	*S. Cerevisiae* (yeast)	P. fragi (bacterium gram -)	M. Luteus (bacterium gram -)	MS2 phage (virus)
SDS-0.6/RT	0.8	1.6	2.0	0.5	1.1	1.5
PF-1.6/60	3.4	2.2	5.0	1.8	1.1	5.3
PF-1.6/40	2.5	1.7	5.0	1.8	4.8	5.3
Y-10/80+P-1.3/70	3.4	4.7	5.0	1.8	4.8	5.3
Y-10/80	3.4	4.4	5.0	1.8	4.8	5.3
VHP-low/RT	1.5	1.6	1.8	1.8	1.8	4.3

Conclusions: Microbial tests showed that the use of high temperature (80°C) at alkaline conditions is the most effective way to disinfect microbes, even bacterium pores.

Lower temperatures (60°C and 40°C) with oxidative detergent reduce the content of other microbes effectively, except bacterium pores.

4.3 Piloting

Objective:

Objective was to clean recycled textile waste in pilot scale and prepare cleaned raw material for the second nonwoven pilot.

Pilot procedure:

Textile material used in cleaning pilot was cotton batch 3 (COpi3) from PureWaste. Cotton fibres were shortened with granulator before cleaning. The cleaning pilot was done at KCL in Espoo. Piloting steps are shown in Figure 23.

In pilot 134 kg of COpi3 was fed in pulper and diluted with hot tap water to 8.2% consistency with mixing. The feeding of cotton caused dust. Next 2.8 kg of alkaline Select Power Blue was diluted in 1:10 and added with mixing in the pulper. After that the same amount of 1:10 diluted Peracid Forte was added. After chemicals dosing the target consistency, 8%, was achieved. Due to lower cleaning consistency at pilot compared to laboratory scale the dosages of chemicals were slightly higher. Temperature and pH were checked after each chemical dosing (Table 12). pH target was 9-10 and temperature target 60°C. Due to slightly too high temperature, 65°C, heating of the pulper was omitted. After 30 minutes cleaning time the temperature was checked once more and noticed that it was 67.7°C, probably because of mixing. Cold tap water added in order to cool the temperature. Cleaning was continued for 30 minutes and after that cotton was diluted with hot tap water and pumped into another vat, where it was diluted to 1.2% consistency. Diluted cotton was fed to Eimcobelt drum filter for dewatering. Dry matter content after Eimcobelt was about 17%. The dilution-dewatering washing was repeated with cold tap water targeting to pH ~8. After cleaning pilot about 34 kg of the cleaned cotton was sent to refining in order to improve fibres bonding ability in nonwoven pilot. The rest of the cleaned cotton fibres (100 kg if yield loss is estimated to be 0%) were sent straight to nonwoven pilot trials.

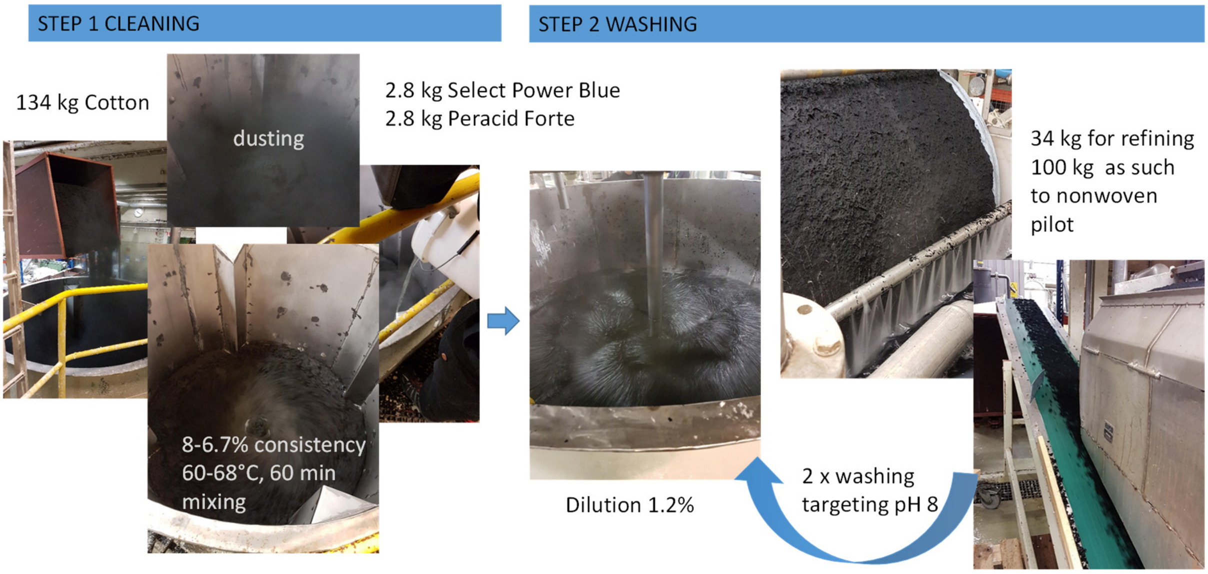


Figure 23 Cleaning pilot process at KCL

Table 12 Process condition in the cleaning pilot

	Amount, kg	Consistency, %	Temperature, °C	pH	Time, min
Step 1 Cleaning					
COpi3	134	8.2	60		
Select Power Blue (SDS)	2.8 (28*)	8.1	60	11.4	
Peracid Forte (PF)	2.8 (28*)	8.0	65	10.3	0
Checking pH and temperature			67.7	9.7	30
Cold tap water	310	6.7	65		30
End of cleaning		6.7	67	9.9	60
Step 2 Washing					
Hot tap water		1.2		9.2	
Cold tap water		1.2		8.2	

*diluted amount

Conclusions: Piloting went mainly as planned. Only temperature increase because of mixing was an unexpected incident, which was not noticed in laboratory trials.

5. Nonwoven Development

This chapter summarizes work related to nonwoven process development. Chapter 5.1 focuses on technologies that can be used to reduce fibre length into suitable range for air-laid and foam-laid products. Next chapter focuses on work carried out in lab scale to test material processability and recipes for nonwoven materials in air laying (Chapter 5.2) and foam laying (Chapter 5.3). The piloting of nonwoven process are described in Chapter 6.

5.1 Shortening of Fibres

Objective:

To study different up-scalable methods for shortening recycled fibres in order to homogenize raw material and improve formation in nonwoven processes sensitive to fibre length.

Materials and Methods:

Cotton fibres used in the shortening tests were black pre-consumer cotton from Pure Waste (COpi1).

Three grinders suitable for reducing fibre length and which are available at VTT were tested. Tested machines were Atrex CD650 G55 (grinder), Pallman PHM 4-7.5 (hammer mill) and Rapid GK640-U (granulator).

Approximately one kilo of cotton material was treated with all three machines. Cut cotton fibres were tested in lab scale foam laying device. The utilized fibre mixture was 70% cotton and 30% BSKP. Target grammage of sheets was 100 g/m^2.

Results:

Appearance of foam laid sheets made from mixture of three types of shortened cotton fibres and BSKP are shown in Figure 24. Clearly best formation was achieved with cotton fibres processed by Rapid granulator and therefore it was selected as cutting device for the further studies.

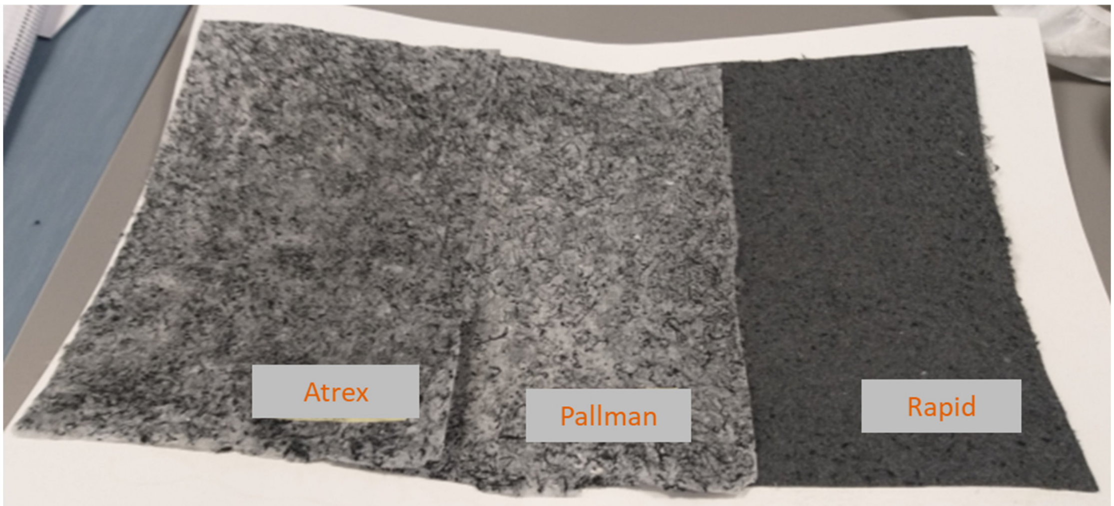


Figure 24 Appearance of foam laid sheets made with fibres shortened by Atrex, Pallman and Rapid machines

Conclusions: Comparison of different cutting methods showed that the best machine for shortening recycled cotton fibres was Rapid granulator.

5.2 Nonwoven Production by Airlaying

Objective:

Aim was to demonstrate the use of recycled cotton in nonwovens made by using air laying technology.

Materials and Methods:

Materials in air laying tests were:

- CO: Recycled cotton - dust from mechanically recycled cotton jeans, longer fibres as such and shortened fibres
- Bico: Bi-component fibre PE/PP (12 mm) from FiberVision
- Lyocell (LYO): Tencel fibre (10 mm) from Lenzing
- Fluff pulp

The desired amount of fibres was inserted inside the air lying drum. The drum was let to roll for 5 minutes during which the fibres were collected on stationary wire under the drum. The air laying set up is shown in Figure 25.

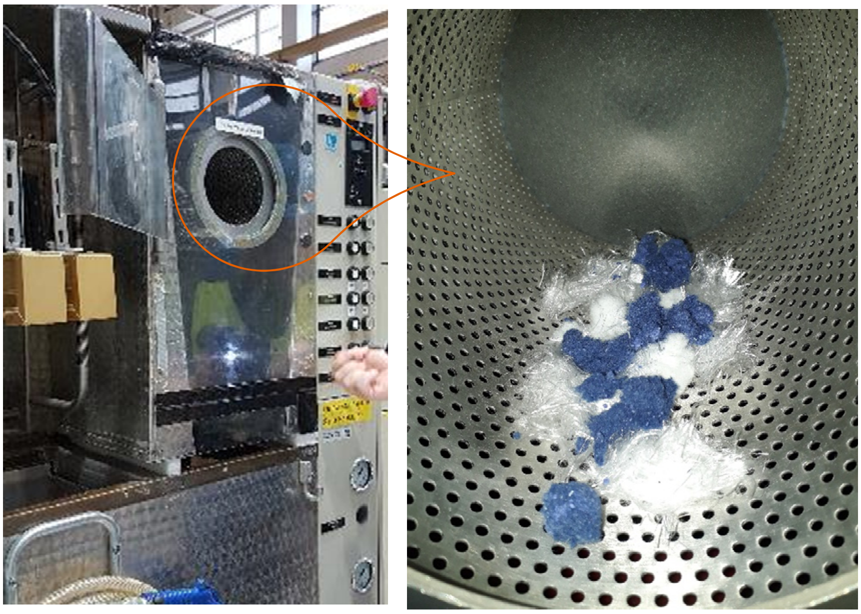


Figure 25 Lab scale air-laying nonwoven setup used in NordicBio project (left) and inside view from the drum with the fibres inside (right)

Hydroentanglement was carried out using lab scale device (see Figure 30a). First pre-treatment with 10 bars on one side was executed and then 20 bars for both sides. Thermal bonding (partial melting of bico fibres) was carried out in oven at 136°C for 2 min.

Results:

Airlaying setup used in these trials was newly obtained to VTT from Tampere University of Technology, and it was used commenced in the beginning of this project. The first samples were made during the implementation from materials from VTT's reserve (dust like fibres, not collected within the project) and the appearance of the sheets is shown in Figure 26.

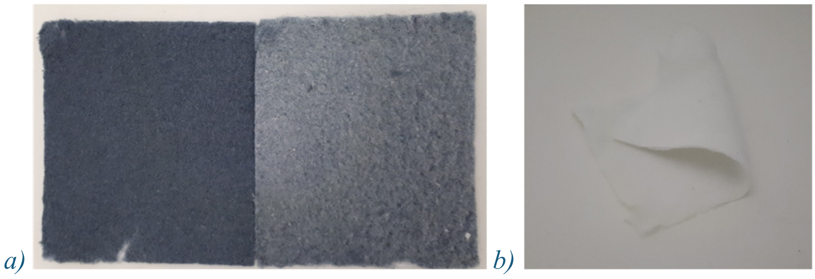


Figure 26 The first sheets were made of mechanically recycled cotton jeans (blue), hammer milled pulp fibres and bico with targeted grammage of 100 g/m^2. a) CO-pulp-bico 2:2:1 - both sides of the sample, and b) pulp-bico 4:1. Bico fibres were thermally activated at 165°C

In the air laying demos, sheets with 40-70% of longer recycled cotton fibres combined with bico, lyocell fibres and fluff pulp were produced with the targeted grammage 80 g/m^2. Trial points are shown in Table 13. The airlaid sheets with bico fibres were thermally bonded and the sheets with lyocell fibres were hydroentangled.

Table 13 Trial and material combinations used in air laying demos

Trial	Cotton, %	Bico, %	Lyocell, %	Fluff pulp, %
1	50	30		20
2	70	30		
3	40		40	20

The shortening of the recycled cotton was necessary, but caused fibre entanglements that is seen as knots /dots in nonwoven sheets. This caused also clear sidedness to the sheets – knots were heavier and clearly come out of the drum first and formed bottom side of the sheet. In this case the recycled material was dark and the other materials white which highlighted the knots and sidedness, Figure 27. The bonding method applied, had a clear effect on the structure: thermally bonded sheets were fluffy and thick and hydroentangled sheets were very thin, Figure 28.

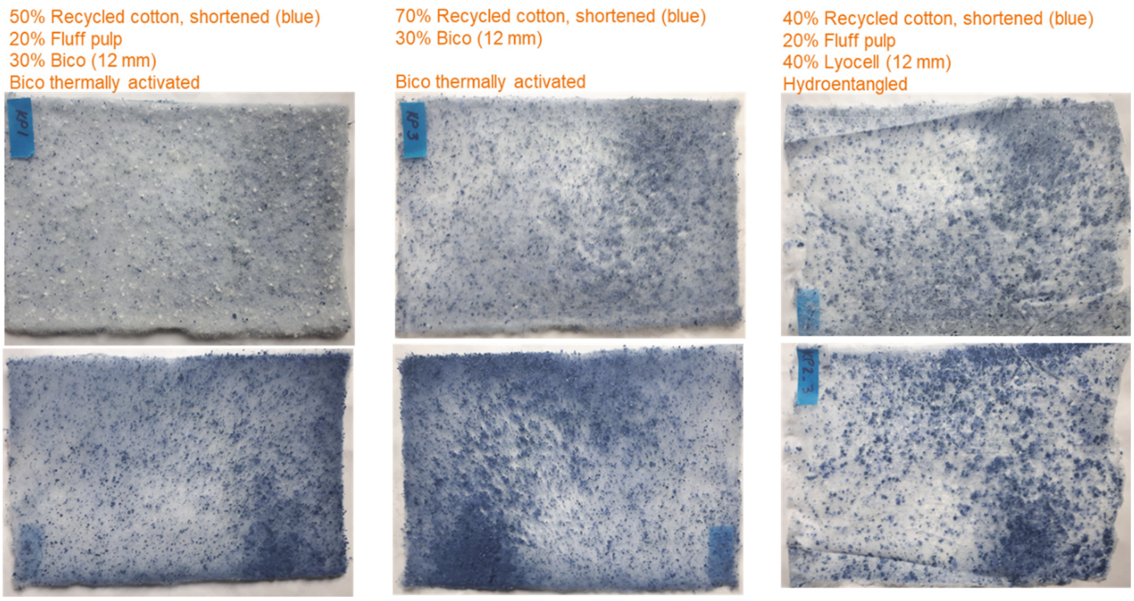


Figure 27 Images of air laid sheets from both sides

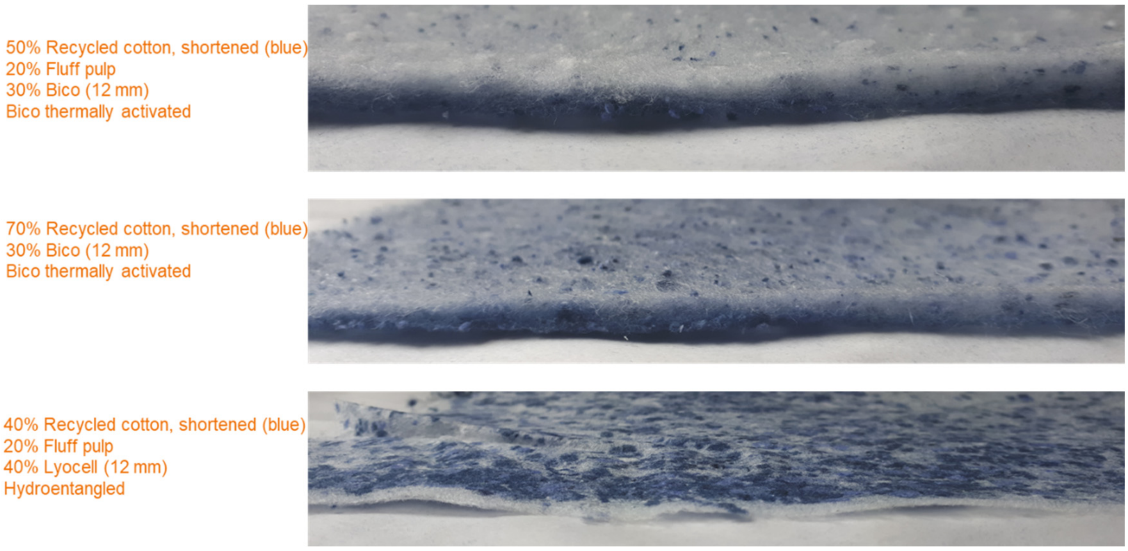


Figure 28 Side profiles of the airlaid sheets. Two upper sheets thermally activated and bottom sheet hydroentangled

Conclusions: Air laying was suitable method to form nonwovens from recycled cotton. Further optimisation of the pre-treatment of fibres is needed to overcome the knots in nonwoven, caused by the fibre entanglements.

5.3 Foam Laying in Lab Scale

Objective:

Test different material combinations and bonding methods for preparation of nonwovens from recycled and bio-based materials.

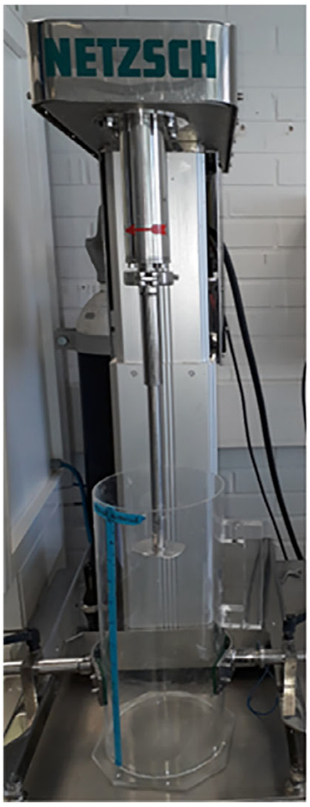
Materials and Methods:

Materials used in foam laying lab tests were:

- BSKP: Bleached softwood kraft pulp
- CO: Rapid granulator processed recycled cotton from Pure Waste (black, pre-consumer) - COpi1
- Bico: Bi-component fibre PE/PP (12 mm) from FiberVision
- LYO: Tencel fibre (6 mm) from Lenzing
- SDS: Sodium dodecyl sulphate, surfactant from (Sigma-Aldrich)
- PVA: Polyvinyl alcohol (POVAL™ 6-88) from Kuraray, surfactant + tested as bonding agent + antidusting agent
- MFC: microfibrillated cellulose produced by VTT, tested as bonding agent + antidusting agent
- Cationic starch (Raisamyl 50021) from Chemigate, tested as bonding agent + antidusting agent

The fibre foam was prepared by axially agitated mixing of water, surfactant and fibres. The mixing was carried out in a cylindrical tank (Figure 29, left). The dosage of SDS was 0.3 g/l and PVA 2 g/l. Cotton fibres were dosed to foam at the end of foaming phase. The starting volume of the pulp suspension was a constant 1 l. The mixing was continued until the air content of the foam was approximately 65%. The fibre foam was poured into a deep mould with a wire bottom (Figure 29, right) and drained with vacuum.

The wet sheet was removed from the mould on the wire and dried with a laboratory drier (KRK rotary drier).



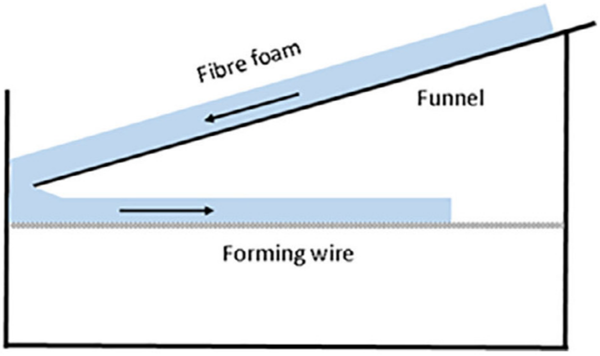



Figure 29 On the left, the mixer and mixing vessel for generating fibre foam. On the right top, the sheet mould and on the right bottom, the schematic picture of the pouring phase of fibre foam

In addition to chemical bonding (PVA, MFC and starch), binding was carried out by spunlacing and bico fibres (thermal bonding).

Spunlacing i.e. hydroentanglement was carried out using lab scale device (see Figure 30a). First pre-treatment with 10 bars on one side was executed and then three different treatment levels for both sides of sample was done:

- 20 bars on 2*both sides (code HE20 in tables)
- HE20 + 40 bars on 2*both sides (HE40)
- HE40 + 60 bars on 2*both sides (HE60)

Bico fibres were included into web and they were activated in oven (Figure 30b) 5 min at 135°C temperature.

a)  *b)* 

Figure 30 a) Lab scale devices for hydro entanglement and b) oven used in thermal bonding i.e. activation of bico fibres

5.3.1 Preliminary Trials with Recycled Cotton Fibres

In the first stage, lab test trials focused on the testing different fibre compositions. Surfactant was SDS with fixed 0.3 g/l dosage and targeted grammage 100 g/m^2. Trial points are shown in Table 14. Samples were produced for visual/hand feel inspection and testing of basic properties including grammage, thickness, density, and tensile strength.

Table 14 Trial points (TP) for foam laying lab scale (L) testing of different fibre compositions. Surfactant SDS 0.3 g/l target grammage 100 g/m^2

TP	BSKP, %	Cotton, %	Bico, %	Lyocell, %	Bonding
L4	50	50			None, HE20, HE40, HE60
L5	40	60			None, HE20, HE40, HE60
L6	30	70			None, HE20, HE40, HE60
L7	20	80			None, HE20, HE40, HE60
L8	10	90			None, HE20, HE40, HE60
L9		70	30		Thermal 135°C, 5 min
L10		80	20		Thermal 135°C, 5 min
L11		90	10		Thermal 135°C, 5 min
L12		50		50	HE20, HE40
L13		60		40	HE20, HE40
L14		70		30	HE20, HE40, HE60
L15	10	60		30	HE20, HE40, HE60
L16	10	70		20	HE20, HE40, HE60

Dusting of bico bonded samples seemed to be less than those containing pulp and, as expected, they had more textile like feel. Sample made of cotton and bico (L11) produced very textile like material, but it was observed that the sample release a lot of dust. Also hand&feel of sample made of mixture of cotton and lyocell fibres (L12) was more textile like. In order to produce more textile like samples with good mechanical properties and reduced dusting additional set of samples were made according to Table 15.

Table 15 Trial points (TP) for selected samples with good hand feel and strength, and reduced dusting

TP	BSKP %	Cotton %	Bico %	Lyocell %	Surfactant & dosage	g/m^2	Bonding
L17		45	10	45	SDS 0.3 g/l	70	Thermal 135°C, 5 min
A							Thermal 135°C, 5 min + HE20
B							HE20 + thermal 135°C, 5 min
L18	30	70			SDS 0.3 g/l	100	Starch 2% surface treatment (1% for both surfaces)
L20		90	10		SDS 0.3 g/l	70	Thermal 135°C, 5 min
L21	30	70			PVA 2 g/l	100	Polyvinyl alcohol
L22	30	70			SDS 0.3 g/l	100	MFC 2% pulp dosage
L23		90	10		SDS 0.3 g/l	50	Thermal 135°C, 5 min

Results - cotton and pulp containing sheets:

Hydroentanglement reduced the grammage of samples (see Figure 31). Grammage reduction was 12-28 % between untreated and heavily treated samples. Tensile strength decreased with decreasing pulp content, and increased with hydroentanglement (Figure 32). Tensile strength of heavily spunlaced samples was 39-63 % higher compared to untreated ones. Some samples with low pulp content and mild spunlacing (L7, L8, L8-HE20) were too were too weak for tensile testing. Strain increased clearly with increasing spunlace intensity, but pulp:cotton ratio did not have clear effect on strain (Figure 33).

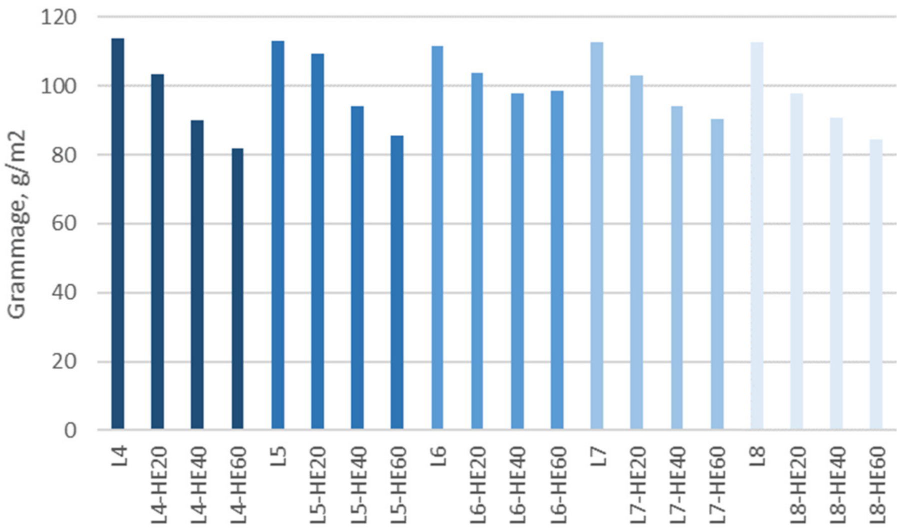


Figure 31 Effect of hydroentanglement on grammage with samples containing pulp and cotton in ratios of 5:5 (L4), 4:6 (L5), 3:7 (L6), 2:8 (L7) and 1:9 (L8) with no bonding and with HE20, HE40 and HE60 hydroentanglement treatments

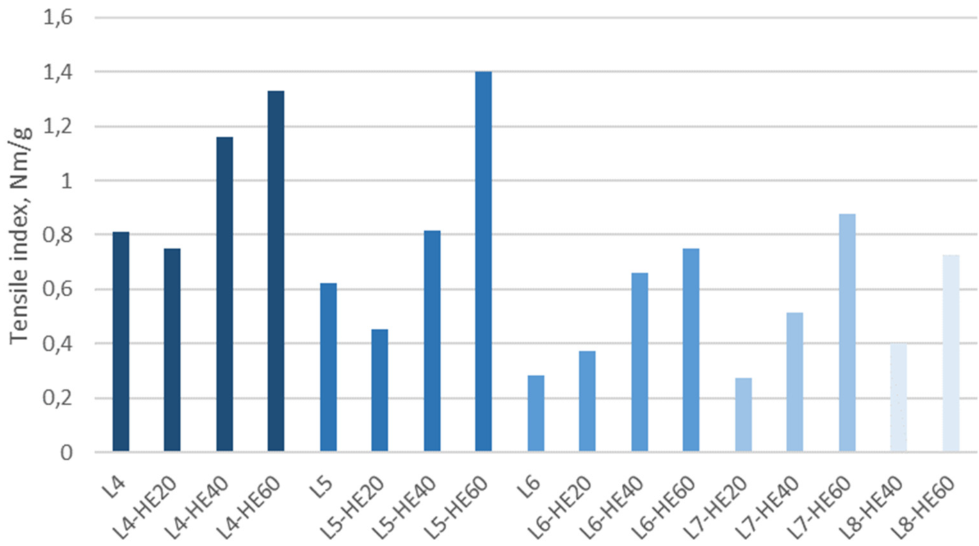


Figure 32 Effect of pulp and cotton in ratios of 5:5 (L4), 4:6 (L5), 3:7 (L6), 2:8 (L7) and 1:9 (L8) and hydroentanglement (none, HE20, HE40 and HE60 treatments) on strength (tensile index / Nm/g)

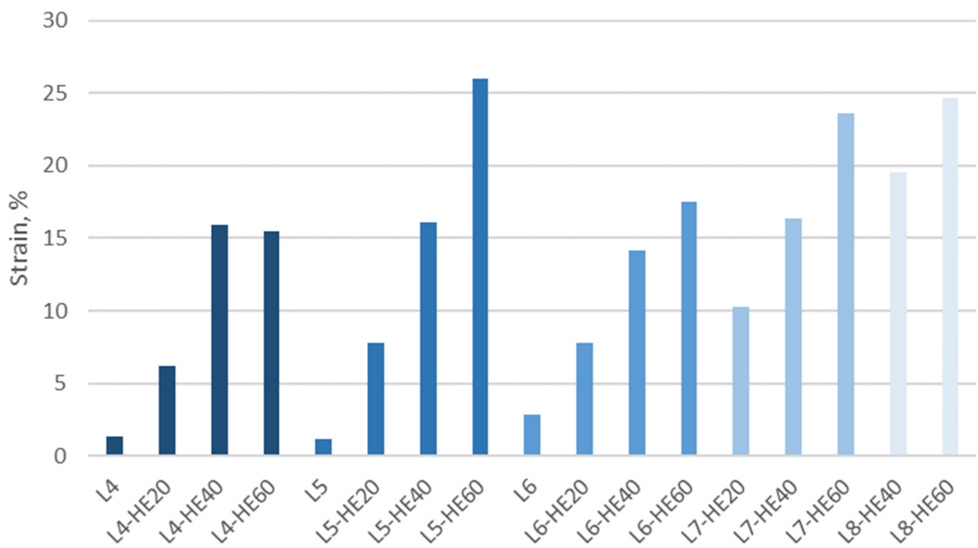


Figure 33 Effect of pulp and cotton in ratios of 5:5 (L4), 4:6 (L5), 3:7 (L6), 2:8 (L7) and 1:9 (L8) and hydroentanglement (none, HE20, HE40 and HE60 treatments) on strain

Results - cotton and bico fibre containing sheets

Bico fibre content was a clear effect on strength i.e. higher content led to higher strength, but no effect on strain, see Figure 24.

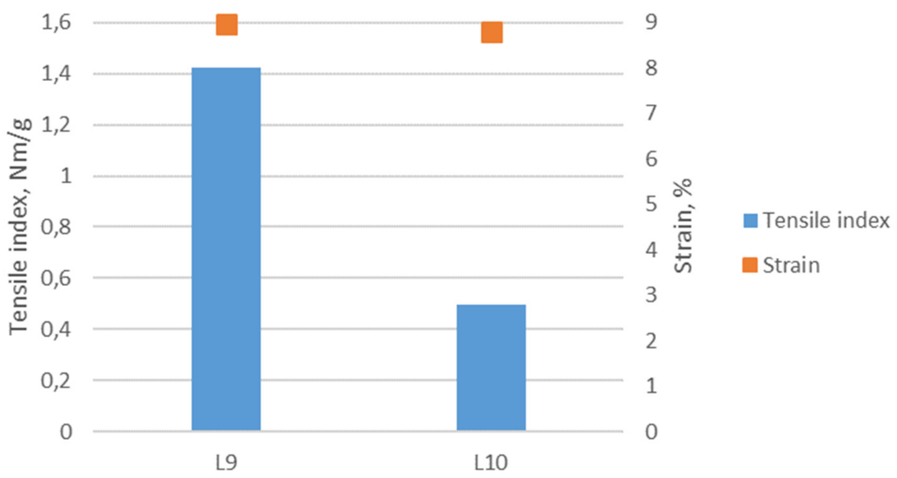


Figure 34 Effect of bico fibres on tensile strength and strain. Content of bico fibres 30 % (L9) and 20 % (L10)

Results - lyocell containing sheets:

Lyocell fibres were added to recipes in order to increase long fibre fraction giving textile like feel. These samples also contains recycled cotton and some of them also pulp or bico fibres in order to bind sheets. Effect of fibre composition and hydroentanglement process on tensile index and strain of samples are shown in Figure 35 and Figure 36, respectively. Cotton/lyocell ratio had no clear effect on strength, but small amount of BSKP improved strength slightly. Hydroentanglement improved the strength and the highest strength was achieved with combining hydroentanglement treatment and bico fibres. Some produced samples were too weak for tensile testing (L12-13-14-15 with HE20, L15-HE40).

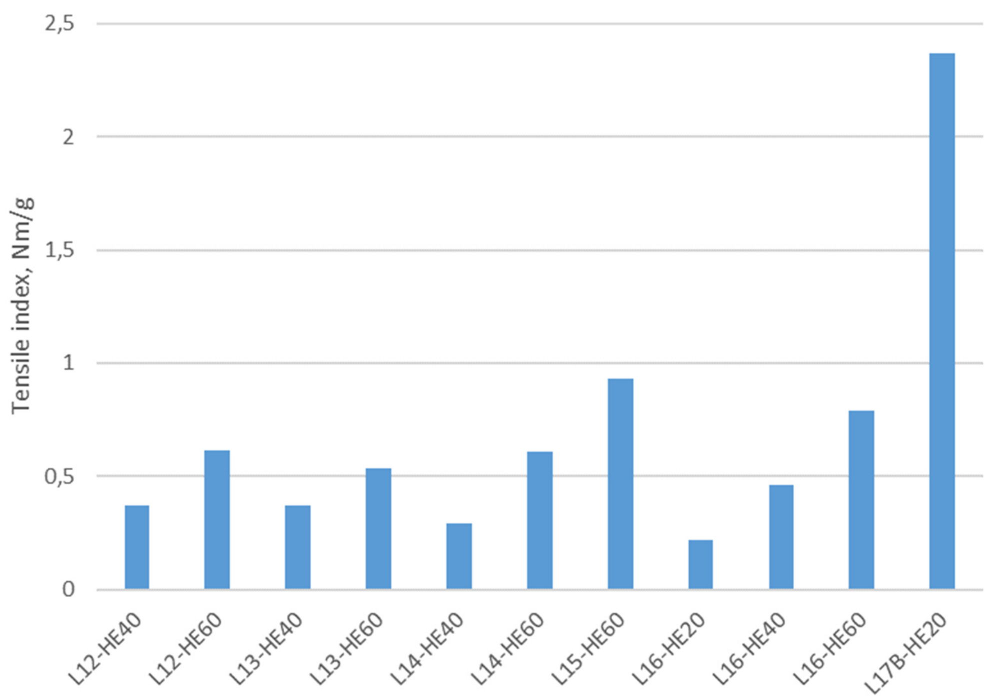


Figure 35 Effect of fibre composition and hydroentanglement of Lyocell containing samples on tensile strength (tensile index / Nm/g). Samples with CO-LYO ratios 5:5 (L12), 6:4 (L13) and 7:3 (L14); with pulp-CO-LYO ratios of 1:6:3 (L15) and 1:7:2 (L16); and CO-bico-LYO ratio of 45:10:45 (L17). Hydroentanglement treatments HE20, HE40 and HE60

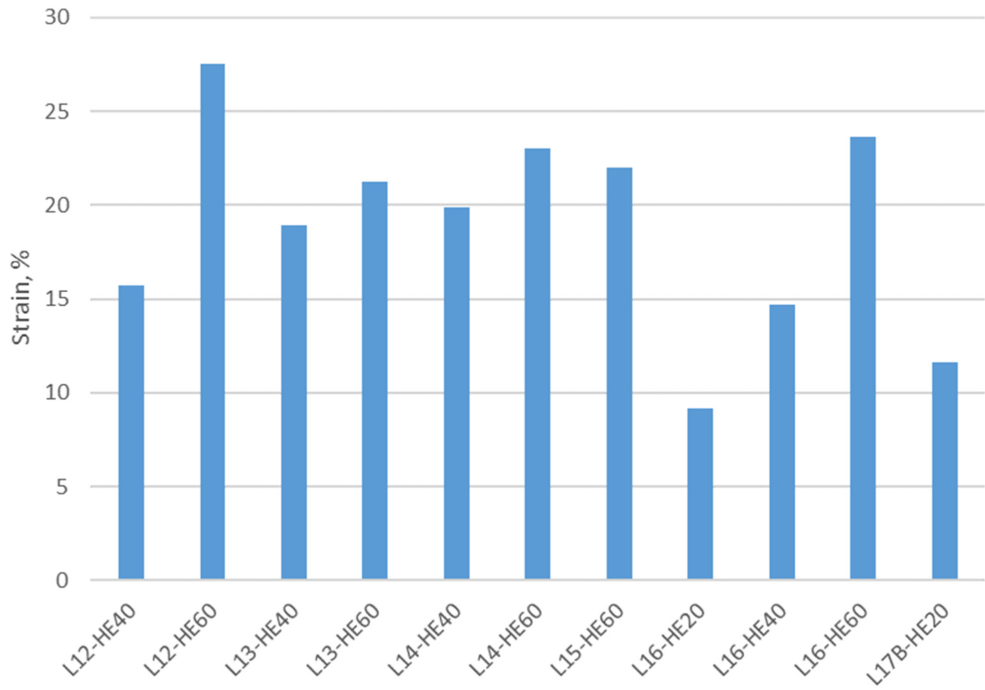


Figure 36 Effect of fibre composition and hydroentanglement of lyocell containing samples on strain. Samples with CO-LYO ratios 5:5 (L12), 6:4 (L13) and 7:3 (L14); with pulp-CO-LYO ratios of 1:6:3 (L15) & 1:7:2 (L16); and CO-bico-LYO ratio of 45:10:45 (L17). Hydroentanglement treatments HE20, HE40 and HE60

Results - additional tests for better strength and reduced dusting:

Chemical bonding with starch, PVA and MFC was tested against the mechanical bonding (hydroentanglement) for samples which pulp/cotton ratio was 30:70. Starch (2 %) was impregnated on the surfaces of sample (1% for both surfaces), MFC (2 %) was dosed to pulp as well as PVA (PVA act as a foaming agent as well). Clearly highest strength was achieved with surface sizing with starch (Figure 37), whereas hydroentanglement level was dominating parameter effected on strain (Figure 38).

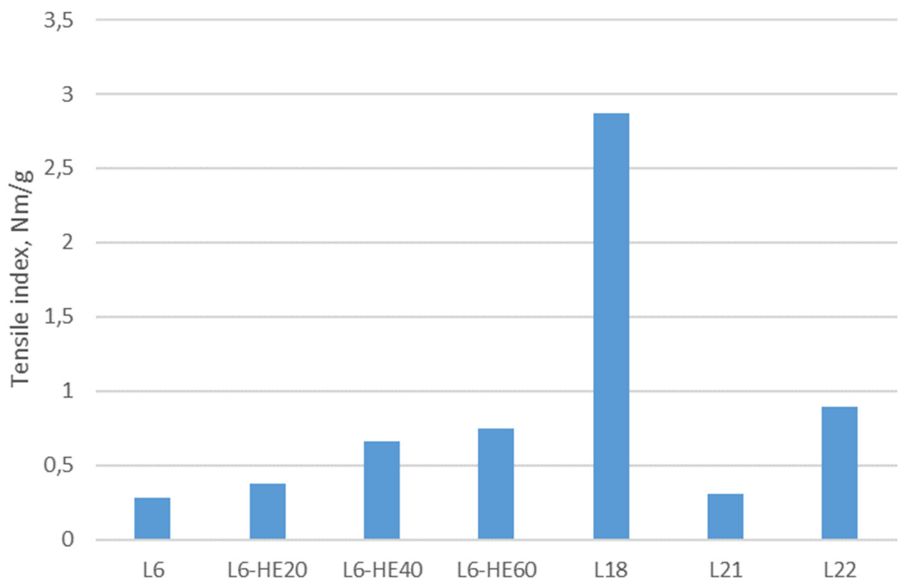


Figure 37 The effect of mechanical and chemical bonding on tensile strength. Hydroentanglement treatment levels for sample L6 none, HE20, HE40 and HE60. Chemical bonding 2% starch (L18), PVA (L21) and 2% MFC (L22). Pulp-CO ratio 3:7 in all samples

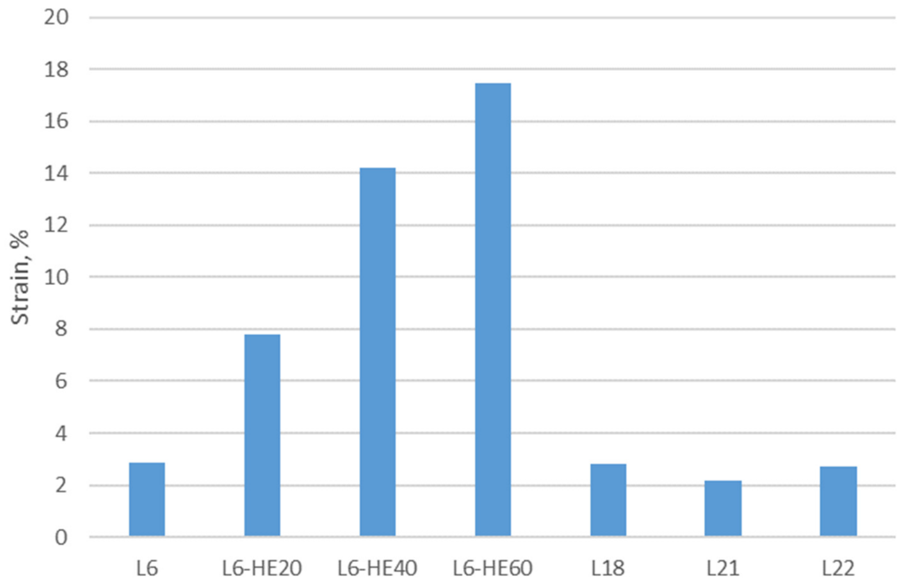


Figure 38 The effect of mechanical and chemical bonding on strain. Hydroentanglement treatment levels for sample L6 none, HE20, HE40 and HE60. Chemical bonding 2% starch (L18), PVA (L21) and 2% MFC (L22). Pulp-cotton ratio 3:7 in all samples

Three trial points were selected for making feel inspection samples for partners. Recycled cotton fibres were mixed with other fibres, namely cellulose pulp, bico fibres and lyocell fibres, in order to obtain materials with varying strength and touch properties. Produced materials were

- Low strength / low strain material: BSKP 30 %, Cotton 70 % (PVA bonded)
- Low strength / medium strain material: Cotton 90 %, bico 10 % (thermally bonded)
- High strength / medium strain material: Cotton 45 %, bico 10 %, lyocell 45 % (mechanically (HE20) and thermally bonded)

Appearance of three samples produced with different kinds of recipes in shown in Figure 39. Detailed compositions and tensile properties of these samples are shown in Figure 40.

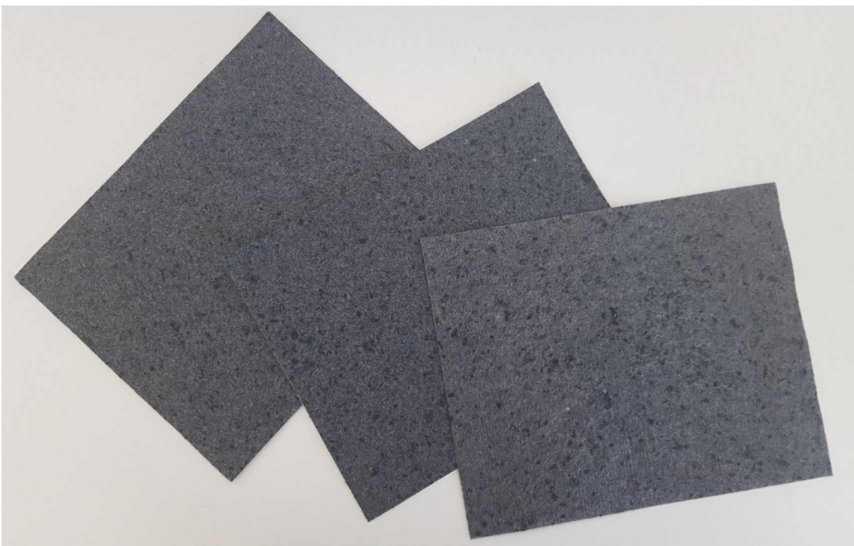


Figure 39 Appearance of selected partner samples (Heikkilä et al., 2019b)

Sample	Fibres/bonding	Grammage (g/m²)
L21	COpi1 70 % BSKP 30 % PVA bonded	100
L20	COpi1 90 % bico 10 % Thermally bonded	70
L17B	COpi1 % lyocell 45 % bico 10 % Mechanically (HE20) and thermally bonded	70

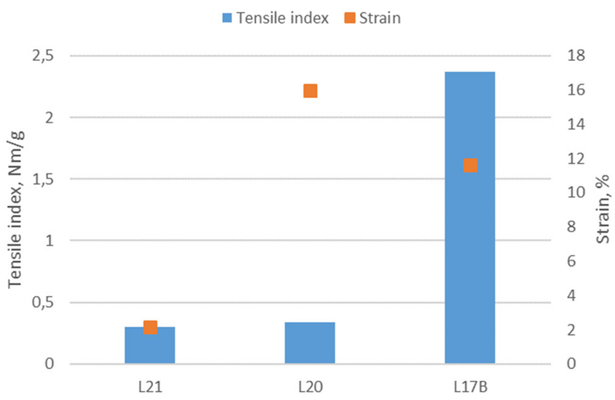


Figure 40 Composition and tensile properties of selected partner samples (Heikkilä et al., 2019b)

Conclusions: Tensile strength increased with increasing pulp content. The highest tensile strengths were achieved with combining hydroentanglement treatment and bico fibres or with surface sizing with starch.

Hydroentanglement treatment caused a clear reduction to grammage, increased tensile strength a little and dominating the development of strain.

High recycled cotton content can be achieved with optimized formulation and binding.

5.3.2 Foam Laying of PLA, Bico and Flax Fibres

Materials and methods:

PLA bico fibres (Ingeo fibre, 4.4 Dtex x 51 mm, supplier Max Model S.A.S.) ($bico_{PLA}$) were used to demonstrate totally biodegradable nonwoven solution with sufficient strength level. The length of PLA bico fibres (51 mm, Figure 41a) was too long to be used in foam laying process as such and therefore fibres were cut approximately to 10 mm fibre length (Figure 41b) before the foam laying phase. PLA bico fibres were not optimized to water based process (as foam laying) and therefore some challenges existed in mixing phase. Partial disintegration of bico fibre bundles in mixing phase led to poor formation of bico fibres (Figure 41c).

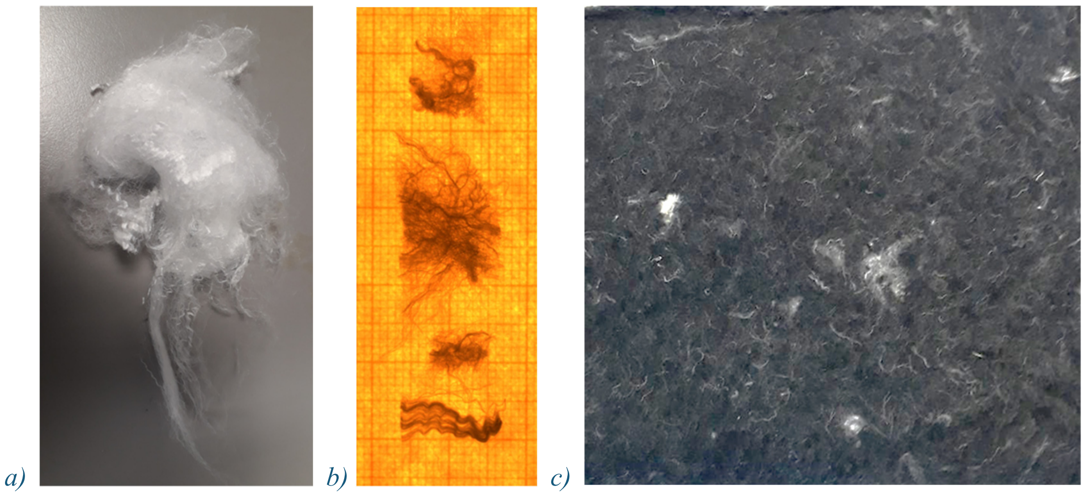


Figure 41 a) PLA bico fibres, b) cut PLA bico fibres on the millimetre paper, and c) appearance of PLA bico fibre containing sheet

Flax fibres were tested as one potential bio-based fibre alternative. The length on staple flax fibres was approximately 12-13 mm (Figure 42).

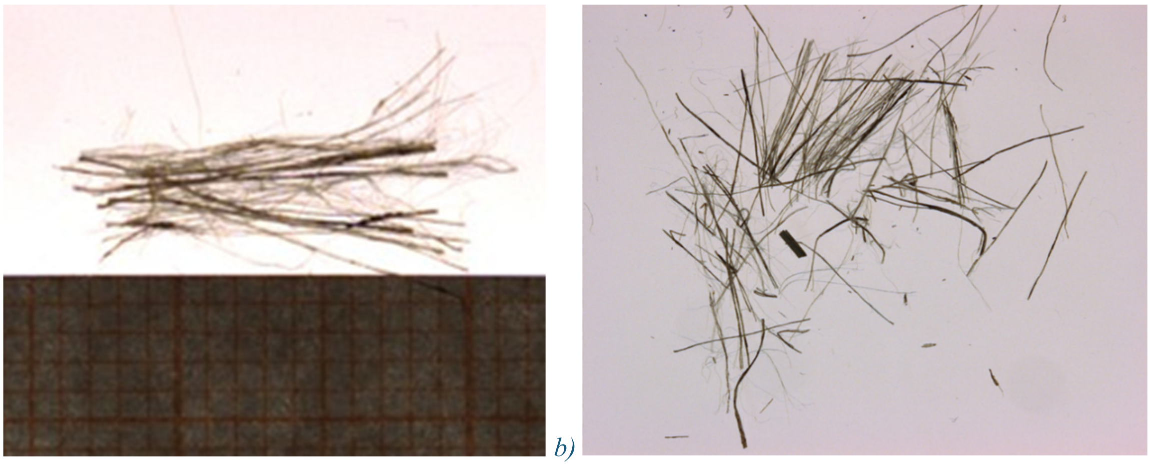


Figure 42 a) Staple flax fibres next to millimetre paper, and b) general appearance of staple flax fibres

In these tests surfactant was SDS with fixed 0.3 g/l dosage and targeted grammage 100 g/m^2. PE/PP type bico fibres (bico$_{PE/PP}$), used in other trials, were also used in this series for comparison. Trial points are shown in Table 16.

Table 16 Trial points (TP) for foam laying lab scale (L) testing of different fibre compositions. Surfactant SDS 0.3 g/l target grammage 100 g/m^2

TP	BSKP %	Cotton %	Flax %	Bico$_{PLA}$ %	Bico$_{PE/PP}$ %	Bonding
L24		70		30		Thermal 145°C, 5 min
L25		80		20		Thermal 145°C, 5 min
L26		90		10		Thermal 145°C, 5 min
L27	30		70			None, HE60
L28			90		10	Thermal 135°C, 5 min
L29			90	10		Thermal 145°C, 5 min
L30		45	45		10	Thermal 135°C, 5 min
B						HE20 + Thermal 135°C, 5 min

Results:

The tensile strength of sheets increased with the higher PLA bico fibre content (Figure 43). However, the use of PLA bico fibres led to lower strength levels than with PE/PP bico fibres. This could be due to differences in bico fibres mechanical properties or bonding capability or inhomogeneous fibre distribution in PLA bico fibre case. The strain of PLA bico fibre containing sheets was higher or at least at the same level than sheets containing PE/PP bico fibres.

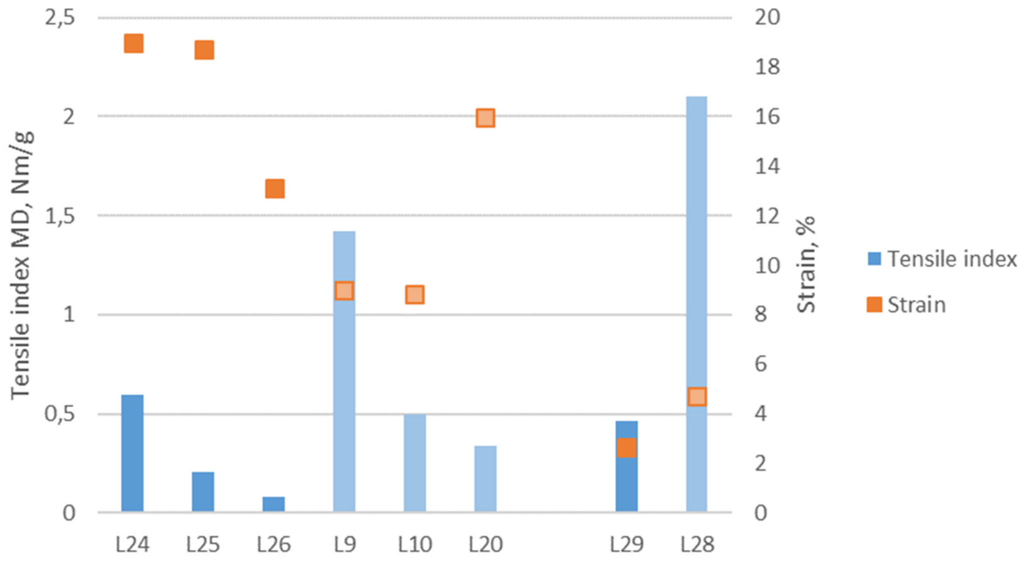


Figure 43 The effect of different bico fibres on tensile properties: PLA based bico shown as dark colour and PE/PP bico as lighter colour. Samples L24-L20 composed of CO and bico - content of bico$_{PLA}$ fibres 30% (L24), 20% (L25) and 10% (L26), and content of bico$_{PE/PP}$ fibres 30% (L9), 20% (L10) and 10% (L20). Last samples contain 90 % flax and 10 % bico fibres: bico$_{PE/PP}$ in L28 and and bico$_{PLA}$ in L29

The higher tensile strength levels were achieved with flax fibres than recycled cotton or lyocell fibres (Figure 44). However the strain of sheets containing flax fibres was at the same level than in cotton or lyocell fibre containing sheets.

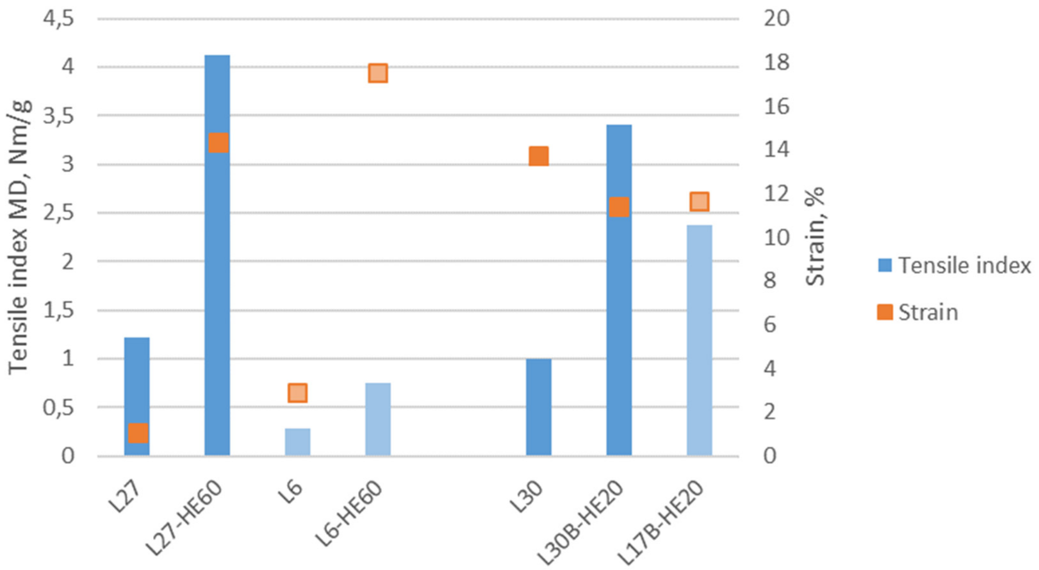


Figure 44 The effect of flax fibres on tensile properties compared to cotton. Dark colour represents results of flax fibres and light colour cotton (L6, L6-HE60) or lyocell (L17B-HE20) fibres. Hydroentanglement treatment levels for samples L27, L6 and L30 none and HE60 and for sample L17 HE20. Fibre mixtures were BSKP-flax 3:7 (L27), BSKP-CO 3:7 (L6), CO-flax-bico$_{PE/PP}$ 45:45:10 (L30), and CO-LYO-bico$_{PE/PP}$ 45:45:10 (L17)

Conclusions: Tensile strength increased with the higher PLA bico fibre content.
Lower tensile strength level was achieved with PLA bico fibres than with PE/PP bico fibres.
Higher tensile strength level was achieved with flax fibres than with recycled cotton or lyocell fibres.

5.3.3 Recycling Trials of Foam Laid Materials

Materials and methods:

In this part the recyclability of nonwovens based on recycled cotton fibres was tested. The tested subjects were

- Recyclability of sheets based on cotton fibres (no extra binding): 1 to 4 slushing cycles
- Recyclability of spunlaced web
- Recyclability of latex bonded web

The utilized raw materials in the samples were

- Cotton fibres: unrefined post-consumer cotton and refined post-consumer cotton (50/50 % mixture) from batch COpi3
- Surfactant: sodium dodecyl sulphate (SDS)
- Latex: Primal Eco 358 ER (20 %)

Results:

The test procedure related to recyclability of cotton based sheets was sheet making 1 -> slushing 1 -> sheet making 2 -> slushing 2 -> etc. Every slushing consists of 30 min pre-soaking phase and slushing phase with 30 000 revolutions (L&W disintegrator). Produced sheets were

- Reference (60 g/m^2, not slushed)
- Once slushed
- Twice slushed
- Three times slushed
- Four times slushed

The appearance of those sheets is presented in Figure 45 and formation results in Figure 46. It seems that one slushing cycle even can improve the formation and decrease the flock size, but after that with the additional slushing cycles, the formation as well as the flock size will stay at constant level.

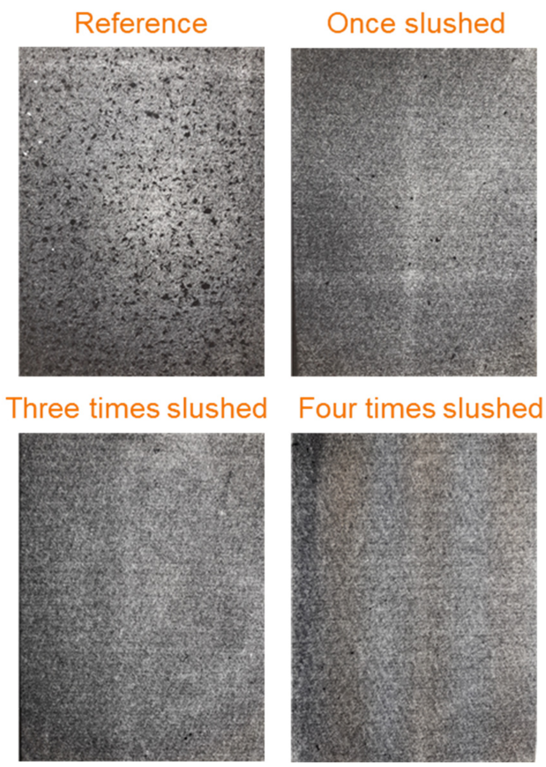


Figure 45 Appearance of slushed sheets

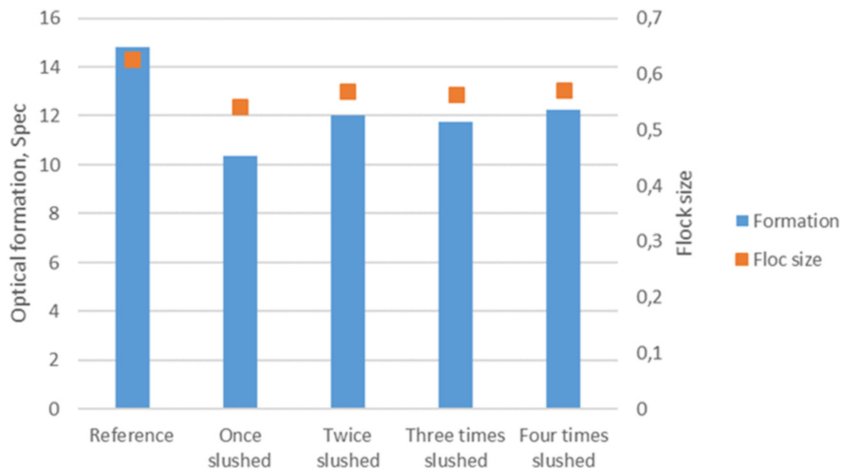


Figure 46 Optical formation and flock size as a function of slushing cycles

One slushing cycle improved the tensile strength because of improved formation (Figure 47). However, the tensile strength decreased with additional slushing cycles. The main reason for this could be the loss of fines, which can be seen in Figure 48. It is known that the amount of fines effects greatly on the mechanical properties of sheet.

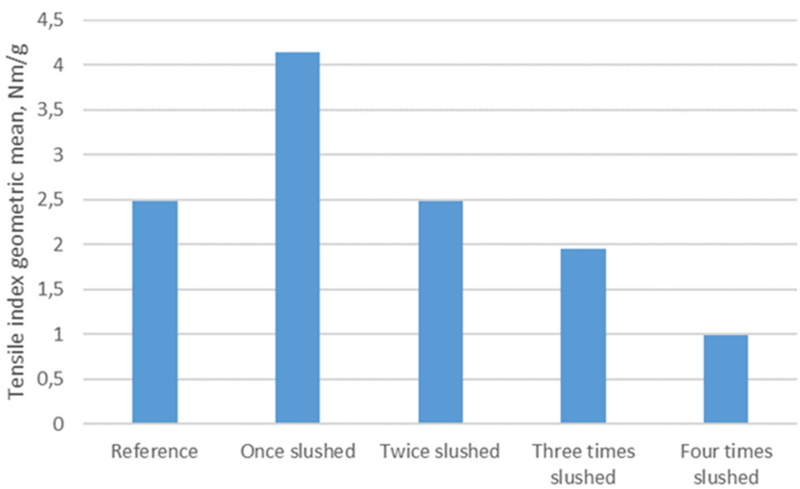


Figure 47 Tensile strength as a function of slushing cycles

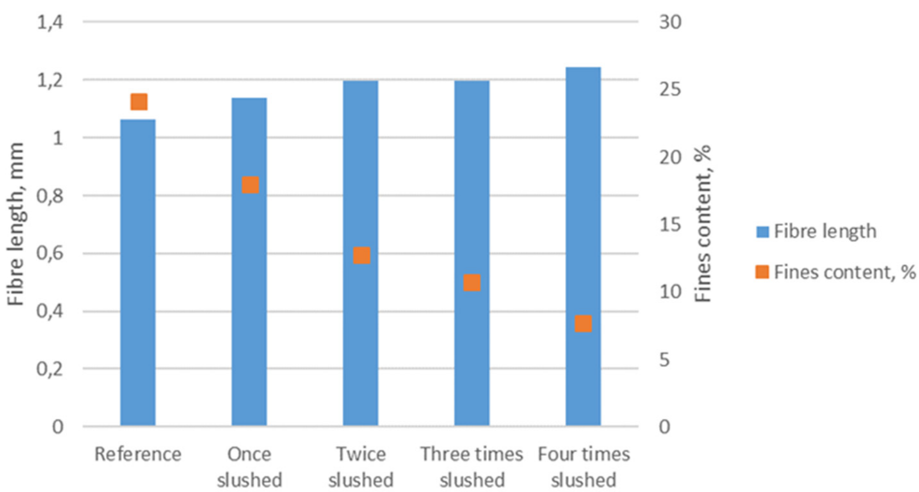


Figure 48 Fibre length and fines content as a function of slushing cycles

Spunlace treatment did not effect on the recyclable of sheet. The appearance of sheet based on slushed spunlaced web is more or less the same than once slushed sheet without spunlacing (Figure 49). Spunlaced parameters were 1 x 10 bar + 2 x 20 bar and both surfaces were treated at the same way.

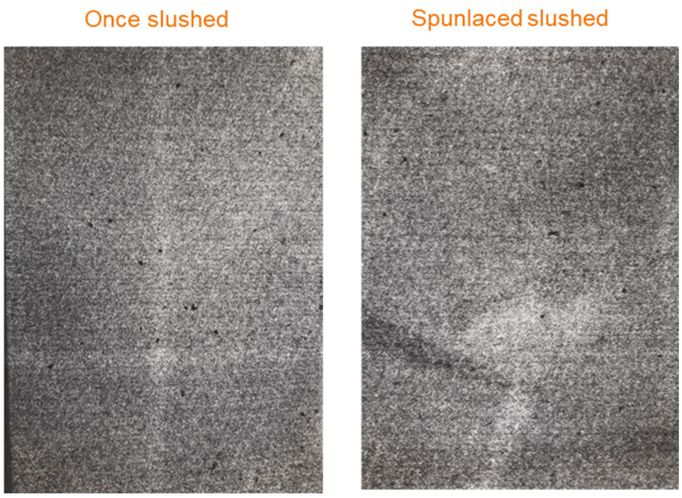


Figure 49 Appearance of once slushed (without spunlacing) and spunlaced and then slushed sheets. Spunlaced parameters were 1 x 10 bar + 2 x 20 bar and both surfaces were treated at the same way

Latex bonding deteriorated slushability clearly. Clear web pieces were seen after water slushing (Figure 50) and even after NaOH (10 % concentration) assisted slushing (Figure 51). NaOH concentrations 2%, 4%, 6%, 8% and 10% and mixing times 10 min and 30 min were tested.

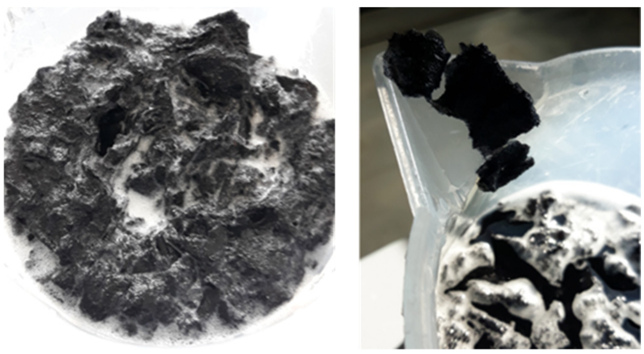


Figure 50 Sheet appearance after water slushing

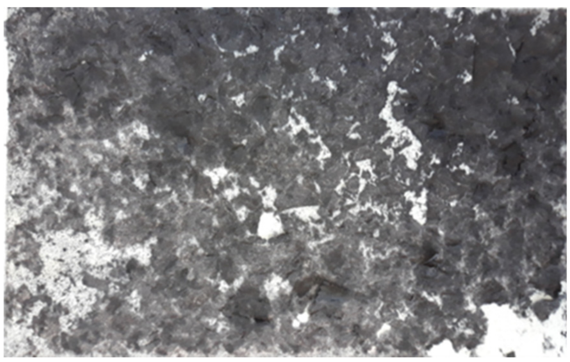


Figure 51 Sheet appearance after NaOH assisted slushing. 10 % NaOH concentration

Conclusions: One slushing cycle improved the formation and after that formation was at constant level.

One slushing cycle improved tensile strength because of improved formation. However, tensile strength decreased with additional slushing cycles, because of the loss of fines.

Spunlace treatment did not effect on slushability, while latex bonding deteriorated slushability clearly.

6. Nonwoven Piloting

Piloting was carried out in SAMPO pilot line (Figure) located at VTT. The main specifications of SAMPO pilot line are

- Design speed 1000 m/min
- Sampling speed < 200 m/min
- Sample width 600 mm (rolls or sheets)
- Vertical headbox
- Two impingement/through air dryer units

Figure 52 SAMPO pilot

6.1 First Pilot - Testing Recipes

Objective:

The first pilot run focused on testing different formulations.

Materials and trial plan:

The target of the first trial was test different recipes based on two recycled cotton fibres: polycotton (polCOpc1) and cotton (COpi2) in combinations with BSKP pulp, bico (PE/PP 12 mm) and lyocell (6 mm).

Both fibre grades were opened at RISE and cut with Rapid granulator at VTT. Foam laying was first tested in lab scale to ensure that processed fibres will work properly in foam laying process. Appearance of lab sheets based on two fibre grades is shown in Figure 53.

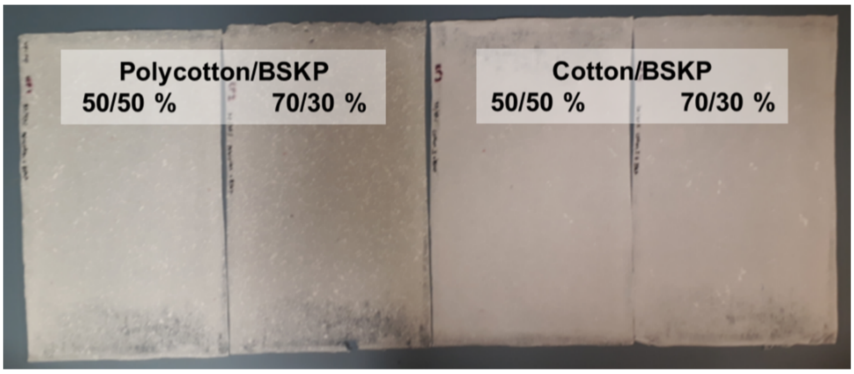


Figure 53 Appearance of lab sheets obtained from materials selected to be used in the first pilot

Chemical bonding with latex (Primal Eco 358 ER), thermal bonding with bico fibres and mechanical bonding with spunlace process were used to improve mechanical properties of produced materials.

Phases of spunlacing were

- Pre-treatment with 10 bars on one side
- 20 bars on 2*both sides (HE20)
- HE20 + 40 bars on 2*both sides (HE40)
- HE40 + 60 bars on 2*both sides (HE60)

Bico fibres were dosed into web in web formation phase and they were activated in oven 2 min at 136°C. Latex was impregnated into web afterwards and activated in oven 2 min at 180°C.

Table 17 Trial points (TP) of the first pilot (1P) trial were

TP	polCO %	CO %	BSKP %	Bico %	Lyocell %	Bonding
1P1	50		50			None, HE20*; latex 20 %
1P2	70		30			None, HE20*; latex 20 %
1P3	40		40	20		Thermal 136°C, 2 min
1P4		50	50			None, HE20*; latex 20 %
1P5		70	30			None, HE20, HE40*; latex 20 %
1P7		40	20		40	None, HE20, HE40*; latex 20 %
1P8		70		30		Thermal 136°C, 2 min
1P9		40	40	20		Thermal 136°C, 2 min
1P11		33	33		33	None, HE20, HE40, HE60; latex 20 %

*Higher spunlace pressures were not possible

Trial points were run to longer rolls which basis weight decreased towards the end of roll i.e. from each trial points at least 30 g/m^2 and 40 g/m^2 grammage samples were collected. From some trial points also 15 g/m^2 grammage samples were collected.

Results:

General outcome was that both recycled cotton based fibre grades worked well in foam forming process, but all produced samples contain high amount of fibre clumps. We could not affect on the amount or shape of fibre clumps by process parameters during the pilot trial. We noticed that some fibre clumps were already seen in cut fibre fractions after fibre cutting phase. We did not know the composition of fibre clumps i.e. were they only entangled cotton or polyester fibres or was there also some amounts of melted polyester. To clarify the composition of fibre clumps SEM images from three disintegrated fibre clump samples from polycotton grade and one sample from cotton grade were taken (Figure 54). The main observations were

- Clumps did not contain melted polyester, only fibres
- There were mechanical reasons for the formation of fibre clumps like fibre entanglements, fibre loops, damaged fibre surfaces and damaged fibre heads etc.
- There were also clear depressions in fibres, which probably originate from textile processing

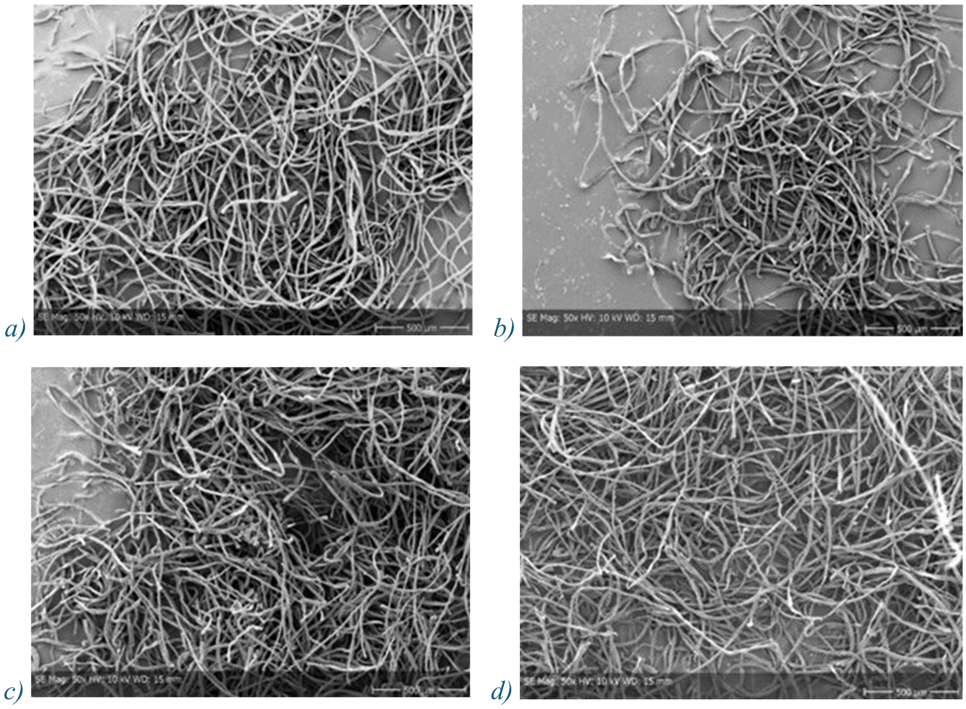


Figure 54 SEM images from disintegrated fibre clumps: a-c) polycotton and d) cotton

Mechanical properties of non-bonded samples are presented in Figure 55. Overall the strength level was very low and many samples were too weak for measurement. It seems that the grammage had a quite small effect on the mechanical properties of non-bonded sheets.

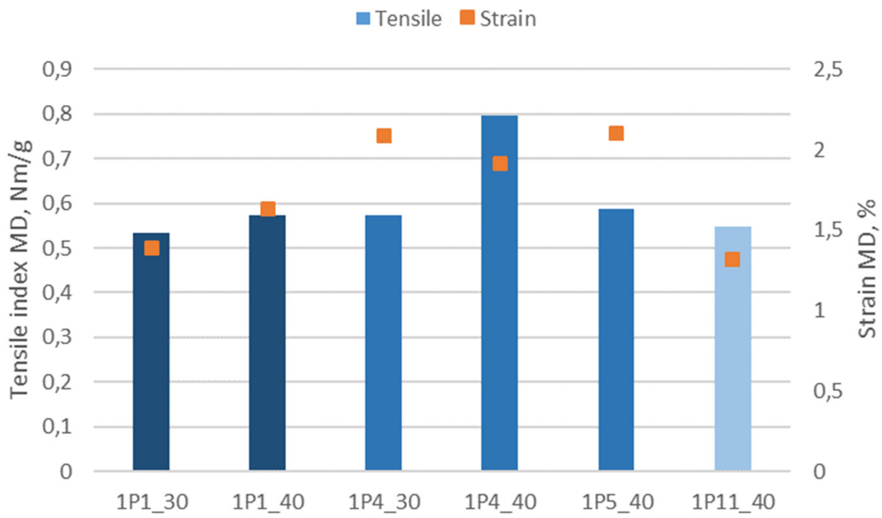


Figure 55 Mechanical properties of non-bonded samples. Fibre mixtures were polCO-BSKP 1:1(1P1), CO-BSKP 1:1 (1P4), CO-BSKP 7:3 (1P5), CO-BSKP-LYO 1:1:1 (1P11). Number after trial point mark (30 or 40) represent the grammage of sheets

However the effect of grammage on strength properties is much clearer when thermally bonded sheets are compared i.e. higher grammage led to higher strength (Figure 56).

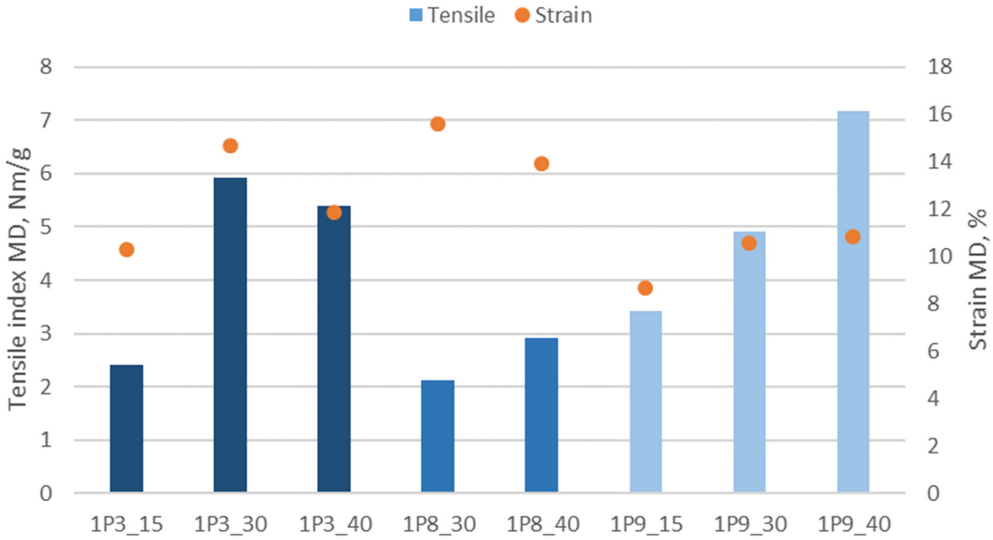


Figure 56 Mechanical properties of thermally bonded samples. Fibre mixtures: polCO-BSKP-bico 2:2:1 (1P3), CO-bico 7:3 (1P8), CO-BSKP 7:3 (1P5), CO-BSKP-bico 2:2:1 (1P9). Number after trial point mark (30 or 40) represent the grammage of sheets

When comparing different bonding methods we can notice that the strongest nonwovens were achieved by latex impregnation (marked with L). The second best option was to utilize bico fibres. Spunlace treatment increased strain, but not so much strength (Figure 57, Figure 58 and Figure 59).

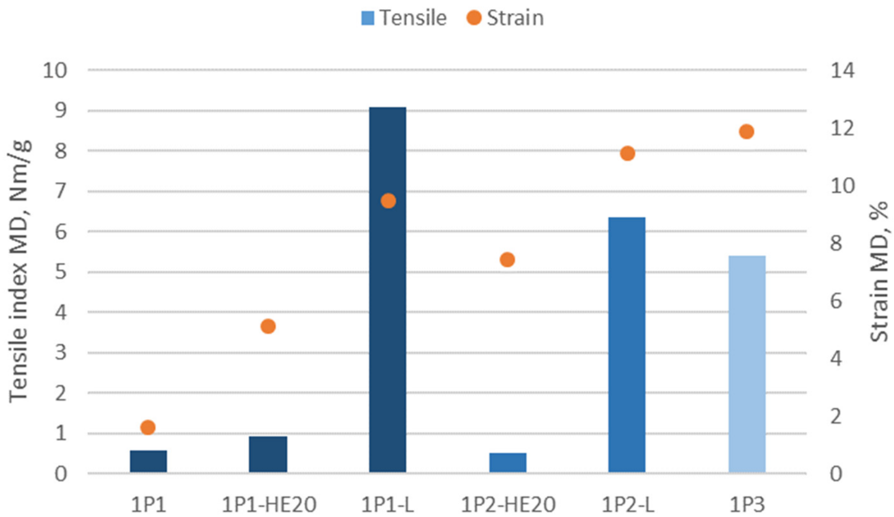


Figure 57 The effect of bonding method on mechanical properties of samples based on polycotton. Fibre mixtures were polCO-BSKP 1:1 (1P1), polCO-BSKP 7:3 (1P2), polCO-BSKP-bico 2:2:1 (1P3). Hydroentanglement treatment level HE20. L after trial point mark represent latex bonded sheets

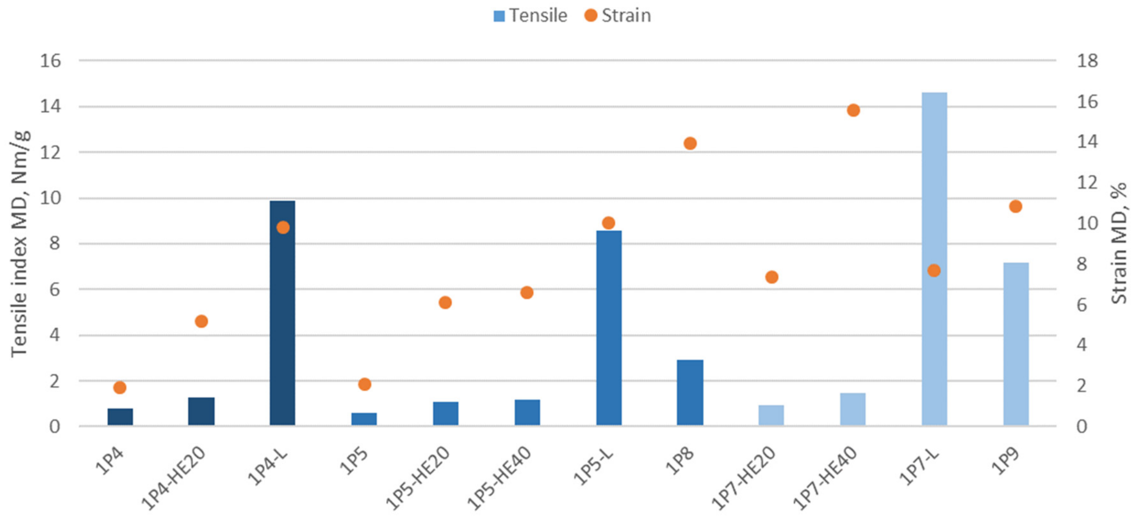


Figure 58 The effect of bonding method on mechanical properties of samples based on cotton. Fibre mixtures were CO-BSKP 1:1 (1P4), CO-BSKP 7:3 (1P5), CO-bico 7:3 (1P8), CO-BSKP-LYO 2:1:2 (1P7), CO-BSKP-bico 2:2:1 (1P9). Hydroentanglement treatment levels HE20 and HE40. L after trial point mark represent latex bonded sheets

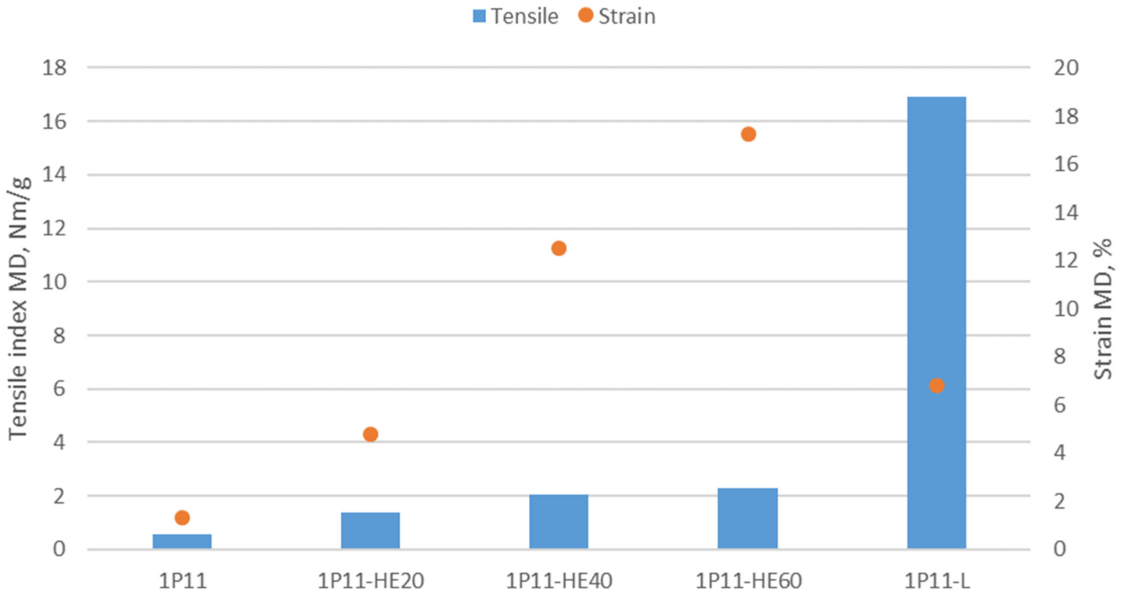


Figure 59 The effect of bonding method on mechanical properties of samples based on cotton, BSKP and lyocell in ratio of 1:1:1 (1P11). Hydroentanglement treatment levels HE20, HE40 and HE60. L after trial point mark represent latex bonded sheet

Hydroentanglement reduced the grammage of samples and especially samples containing high amount of cotton fibres (Figure 60).

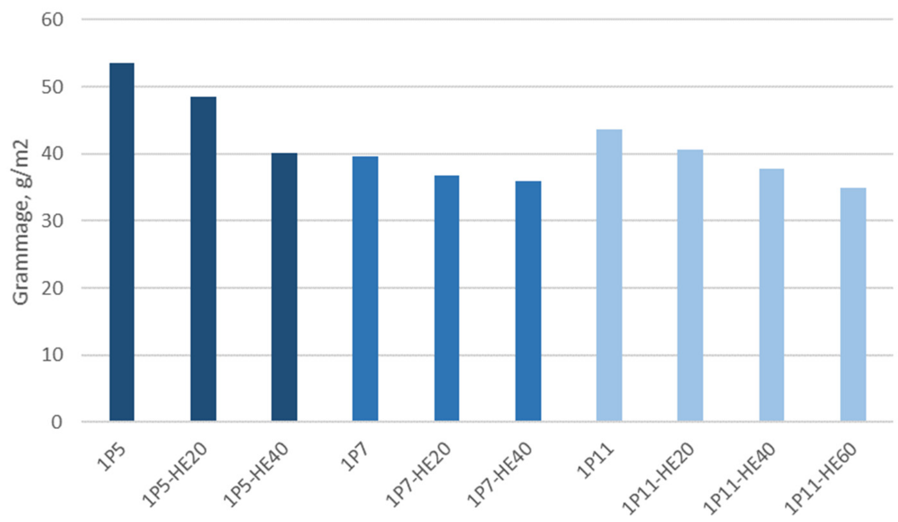


Figure 60 Effect of hydroentanglement on grammage with samples containing pulp and cotton in ratio of 3:7 (1P5) and containing pulp, cotton and lyocell in ratios of 1:2:2 (1P7) and 1:1:1 (1P11) with no bonding and with HE20, HE40 and HE60 hydroentanglement treatments.

Conclusions: Recycled polycotton and cotton fibre fractions worked well in foam forming process, but a lot of fibre clumps exist in nonwoven webs.

Spunlace treatment decreased the grammage.

The strongest nonwovens were achieved by latex impregnation and the second best option was to utilize bico fibres. Spunlace treatment increased strain, but not so much strength.

6.2 Process Improvements

Objective:

Improving the quality of nonwovens webs namely decreasing the amount of fibre clumps in cut fibre fraction as well as in final sheet.

6.2.1 Fibre cutting tests

Materials and methods:

Tests were carried out in Rapid granulator, laboratory L&W disintegrator, laboratory foam generator and laboratory sheet mould. In the first test, screen hole diameters of granulator were 6 mm and 10 mm, and rotor speeds 30 Hz and 50 Hz. Utilized raw materials were BSKP and cotton (COpi2). Different pre-treatments (slushing and soaking) before foaming and sheet making were also tested. Tested pre-treatments were

- Untreated: no slushing or soaking before foaming and sheet making
- Slushed: slushed with 10 000 revolutions (L&W disintegrator) before foaming and sheet making
- Soaked + slushed: soaked overnight and slushed with 10 000 revolutions (L&W disintegrator) before foaming and sheet making
- Longer slushing: Slushed at low consistency (half consistency compared to earlier points) with longer time (30 000 revolutions, L&W disintegrator) before foaming and sheet making

Results:

Appearance of some sheets obtained from cut fibres are shown in Figure 61. It was observed that screen hole diameter had a clear effect on the uniformity of sheets, smaller being the better, but rotor speed had no effect. The best result was achieved with 6 mm screen hole diameter and fast slushing without extra soaking.

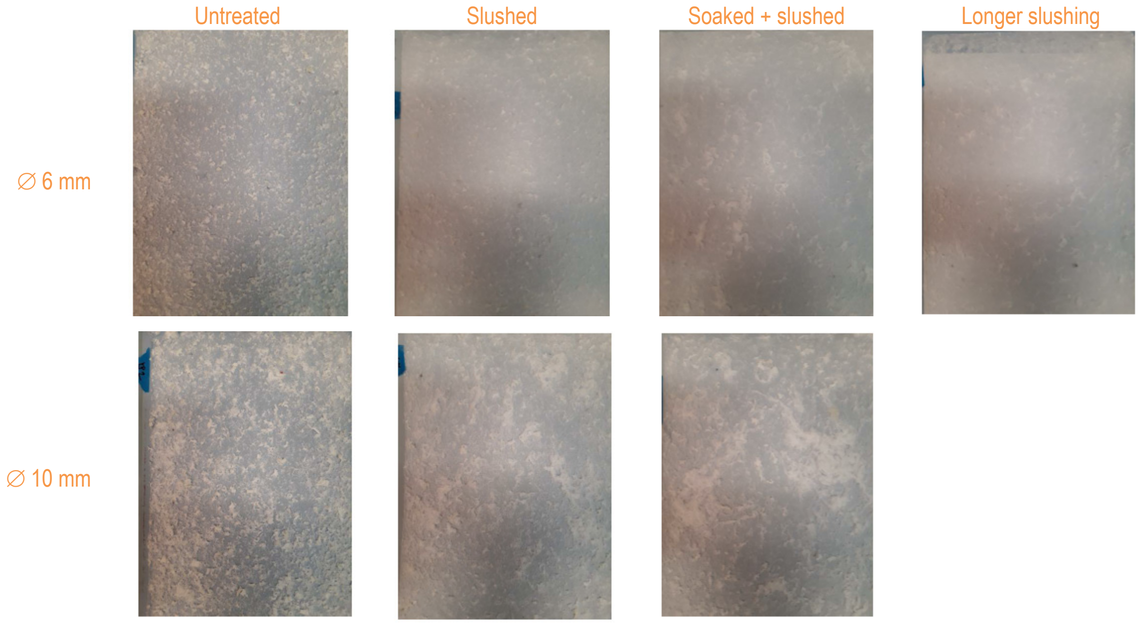


Figure 61 Appearance of sheets obtained in the first cutting optimization test - variables: screen hole diameter (6 mm and 10 mm) and pre-treatments (untreated, slushing and soaking) before foaming and sheet making

In the second test, screen hole diameters of granulator were 3 mm and 6 mm, rotor speed 50 Hz and raw material cotton (COpi2). Fibres were cut without and with vacuum assisted fibre fraction exhausting.

Appearance of sheets obtained from cut fibres are shown in Figure 62. It was observed again that hole diameter had a clear effect on the uniformity of sheets, smaller being the better, and that vacuum in exhausting system was not beneficial for the cutting process. The best result was achieved without vacuum in exhausting system and with 3 mm screen hole diameter and fast slushing.

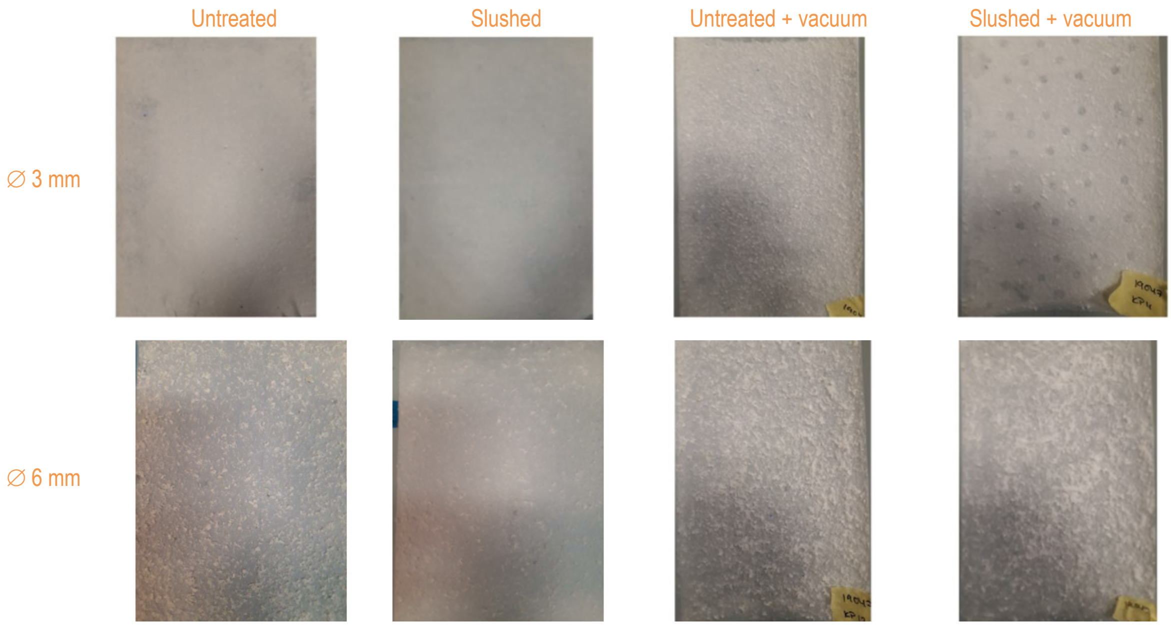


Figure 62 Appearance of sheets obtained in the second cutting optimization test - variables: screen hole diameter (3 mm and 6 mm), vacuum level in exhausting system (no vacuum and vacuum) and pre-treatments (untreated and slushed) before foaming and sheet making

6.2.2 Wet refining

Materials and methods:

Another approach to remove clumps was wet-refining. Three refining levels low, medium and high were tested. The estimated refining energy for those three levels are approximately 0.5 SEC MWh/t, 1.5 SEC MWh/t and 2 SEC MWh/t.

Results:

Appearance of sheets obtained from wet-refined fibres are shown in Figure 63. Wet refining disintegrated hard fibre clumps effectively. However some fibre flocks were observed in sheets produced with our normal foaming vessel volume (1 litre) and mixer speed (3800 rpm) used in laboratory.

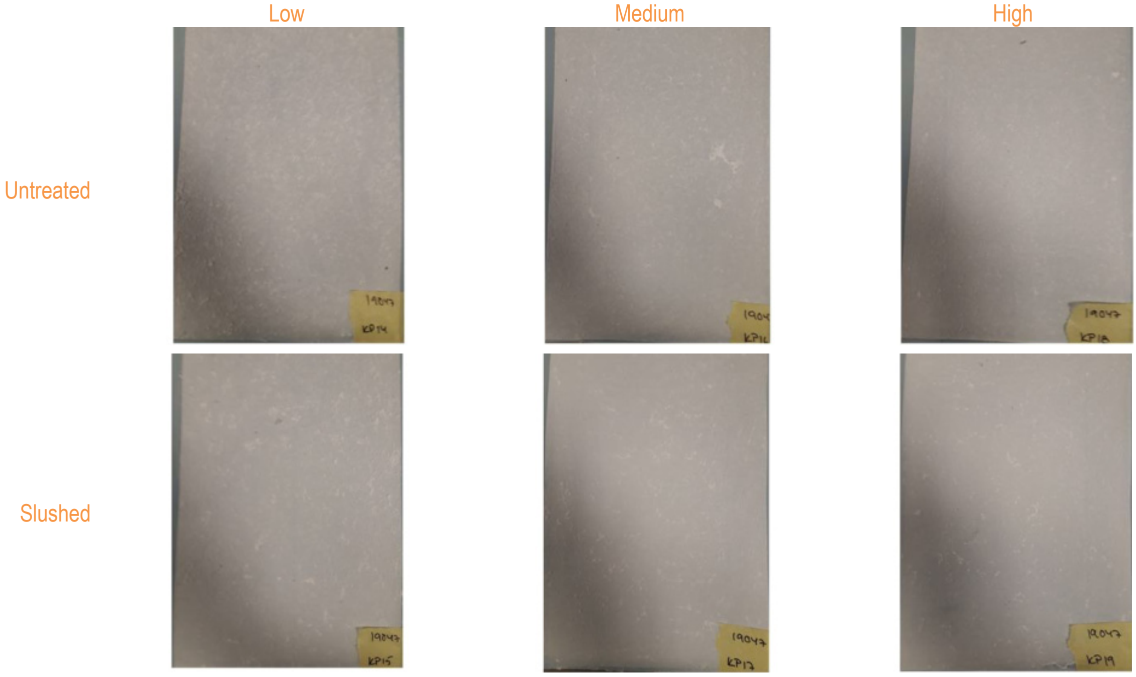


Figure 63 Appearance of sheets obtained in wet-refining test - variables refining level (low, medium, high) and pre-treatments (untreated, slushed) before foaming and sheet making

We noticed that wet refining increased the strength of sheets based on recycled cotton significantly (Figure 64). We can assume that even sheets based on 100 % of post-consumer cotton can be produced. However, at the same time softness disappeared and material had a paper-like touch.

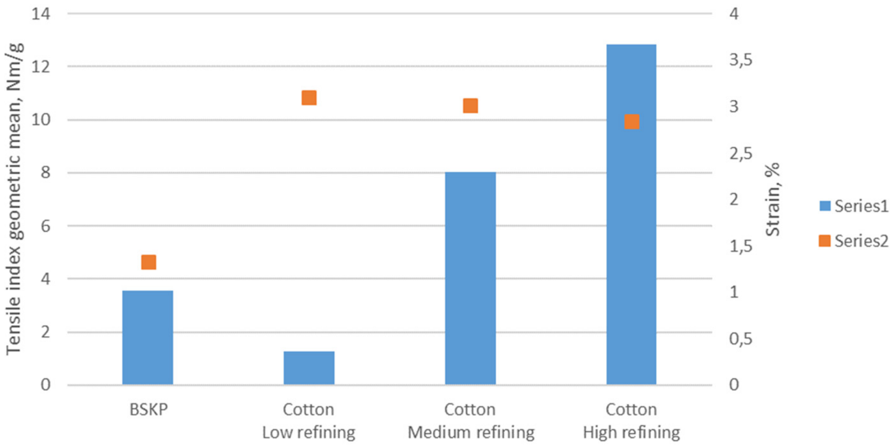


Figure 64 The effect of refining on tensile properties of sheets based on recycled cotton, BSKP as reference. Refining energies: low 0.5 SEC MWh/t, medium 1.5 SEC MWh/t and high 2 SEC MWh/t.

6.2.3 Foam volume testing

Materials and methods:

In order to prevent fibre flocculation and thus improve formation we tested with unrefined cotton fibres larger vessel volume (3 litres) and higher mixer speed (4600 rpm) in foaming phase. The effect of SDS dosage was also tested. Low foam air content was achieved with 0.3 g/l SDS dosage and high air content with 0.6 g/l dosage. With these changes the amount of fibre flocks were reduced, probably because of reduced fibre contacts with the rod of mixer (reduced roping effect), higher intensity of mixing (increased disintegration of fibre flocks) and higher air content (reduced flocculation).

Results:

Utilization of 3 mm screen hole diameter in cutting phase and slushing before foaming phase together with large foaming volume, high air content and high mixing intensity led to best result, where no hard fibre clumps and no fibre flocks were not observed in sheet (Figure 65).

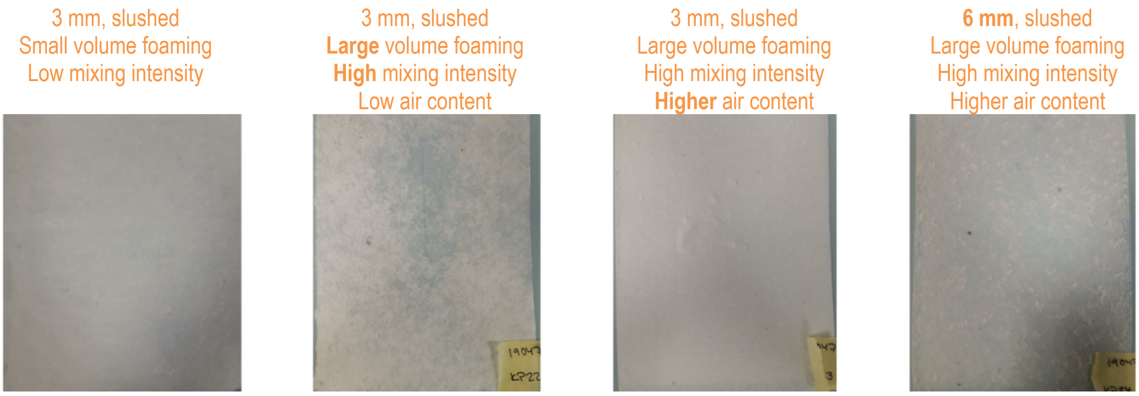


Figure 65 Appearance of sheet obtained in foaming parameter test - variables: screen hole size (3 mm and 6 mm), foaming volume (small/large), mixing intensity (low/high) and air content (low/high)

Conclusions. The amount of fibre clumps in sheets based on cut recycled cotton can be reduced by using granulator screen with small hole diameter in cutting phase, by wet refining of cut fractions and by slushing of cut fractions before sheet making phase.

Fibre flocculation can be reduced by optimizing the foaming phase i.e. with high intensity mixing and optimized tank and mixer dimensions.

Wet refining increase the strength of web so much that sheet based on 100% recycled cotton can be produced. However at the same time softness disappeared (paper like feeling).

6.3 Second Pilot - Making Rolled Materials

Objective:

The target of the second pilot trial was to produce nonwoven rolls based on two recycled cotton fibre grades, for lamination tests executed in Cellcomb.

Materials and trial plan:

The utilized cotton based fibre grades were

- Polycotton (polCOpc2)
- Cotton (COpi3)

Polycotton was opened at RISE and both fibre grades were cut with Rapid granulator at VTT. After cutting phase cotton grade was cleaned at KCL and further 35 kg cleaned fibre batch was wet refined at VTT. Addition to these fibre grades bico fibres were used to increase the strength of nonwovens.

Trials points of second pilot in shown in Table 18. At the first trial day four fibre mixtures and two grammage levels were tested to clarify the most promising fibre mixture for roll making. At the second day three rolls were produced. Produced rolls were first dried in cylinder drying unit and then two of them were thermally bonded (bico activation) in surface treatment line. Both mentioned actions were executed at VTT. Finally two activated rolls were sent to Cellcomb for lamination tests.

Table 18 Trial points (TP) of the second pilot (2P) trial

TP	Polycotton %	Unrefined CO %	Refined CO %	BSKP %	Bico 12 mm %	Comments
2P1	50			40	10	Fibre mixture test
2P2	50			30	20	Fibre mixture test
2P3	50			20	30	Fibre mixture test
2P4	65			15	20	Fibre mixture test
2P5	50			20	30	Roll
2P6	50			20	30	Roll for lamination test
2P7		25	45		30	Roll for lamination test

Results:

Fibre mixture tests showed that the highest Bico content led to highest strength and strain, so the fibre mixture polycotton 50%, BSKP 20% and Bico 30% (2P3) was selected to roll making (Figure 66 and Figure 67).

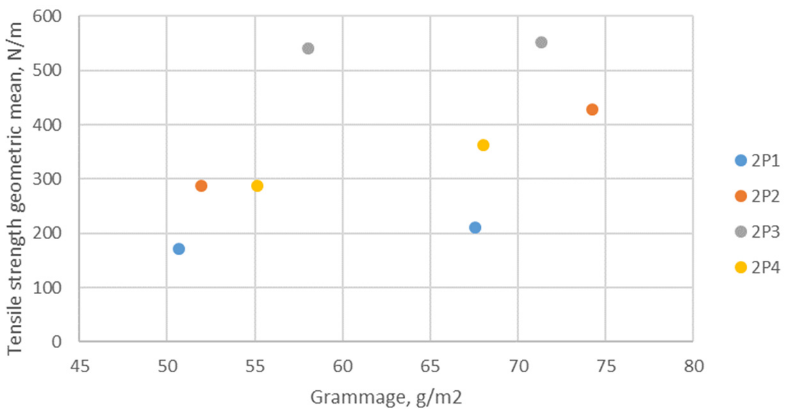


Figure 66 Tensile strength of different fibre mixtures as a function of grammage. Polycotton:BSKP:bico ratios were 5:4:1 (2P1), 5:3:2 (2P2), 5:2:3 (2P3), 65:15:20 (2P4)

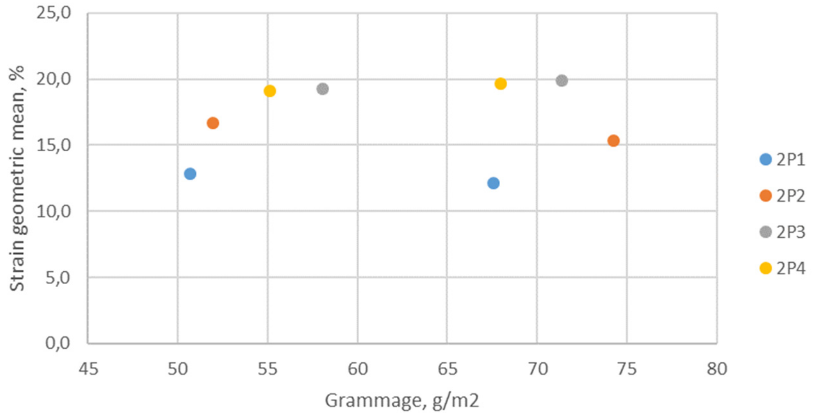


Figure 67 Strain of different fibre mixtures as a function of grammage. PolCO-BSKP-bico ratios were 5:4:1 (2P1), 5:3:2 (2P2), 5:2:3 (2P3), 65:15:20 (2P4)

For benchmarking tensile properties of five commercially used nonwovens (delivered by Cellcomb) were analysed and compared against the strength of produced NordicBio rolls on second day. The grammage of commercial nonwovens varies from 30 g/m^2 to 50 g/m^2 and the grammage of NordicBio rolls were 68 g/m^2 (2P5), 91 g/m^2 (2P6) and 60 g/m^2 (2P7). We can notice from Figure 68 that tensile strength of NordicBio materials was comparable to commercial references, but strain was much lower.

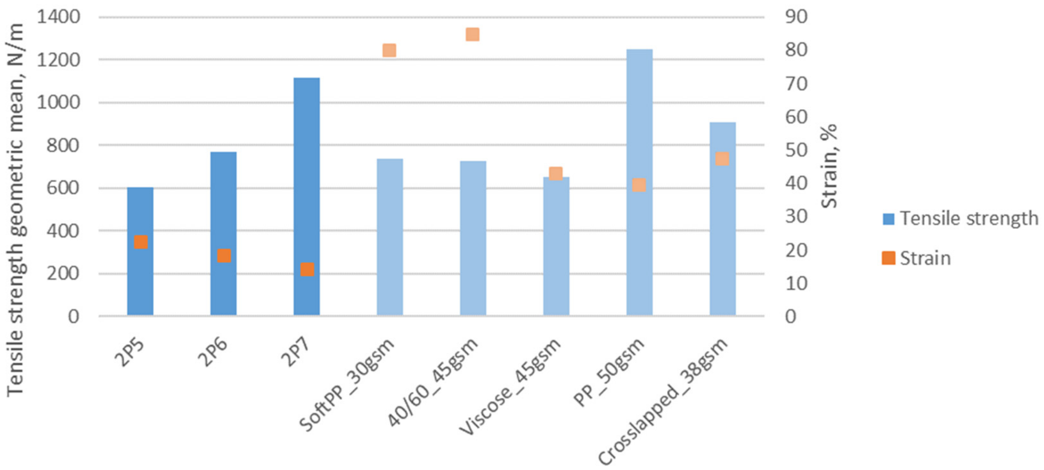


Figure 68 Tensile strength and strain of NordicBio materials compared to commercial references

Conclusions. High bico content led to high strength and strain. Tensile strength of NordicBio materials was comparable to commercial references, but strain was much lower.

6.4 Lamination Trial

The purpose of the trial was a processability test to see if recycled textiles, formed on rolls, will be able to be laminated with a barrier film in the standard lamination process.

Materials and methods:

Lamination test was executed in production scale lamination line with 50 m/min speed at Cellcomb (Figure 69). Starch based film (width 900 mm, grammage 12 g/m^2 and thickness 10 µm) was laminated by hot melt glue (synthetic polymer, 2 g/m^2) on the cotton based nonwovens at Cellcomb laminating line. Two rolls of 400 mm width produced in pilot trial 2 were processed at Cellcomb (Table 19).

Figure 69 Lamination of rolls at Cellcomb

Results:

Two laminated rolls were produced in trial (Figure 70). Grammages of rolls 1 and 2 were 104 g/m^2 and 84 g/m^2, and lengths 20 m and 100 m, respectively (Table 19). The tensile strength of laminated webs is approximately 20 % higher than in non-laminated webs (Figure 71).

Table 19 Rolls sent to Cellcomb and properties before and after lamination

Roll		**Roll 1 (2P6)**	**Roll 2 (2P7)**
Fibre mixture		polCO 50 %, BSKP 20 %, bico 30 %	unrefined CO 25 %, refined CO 45 %, bico 30 %
Before lamination	Grammage	90 g/m^2	60 g/m^2
	Length	~300 m	~600 m
After lamination	Grammage	104 g/m^2	84 g/m^2
	Length	20 m	100 m

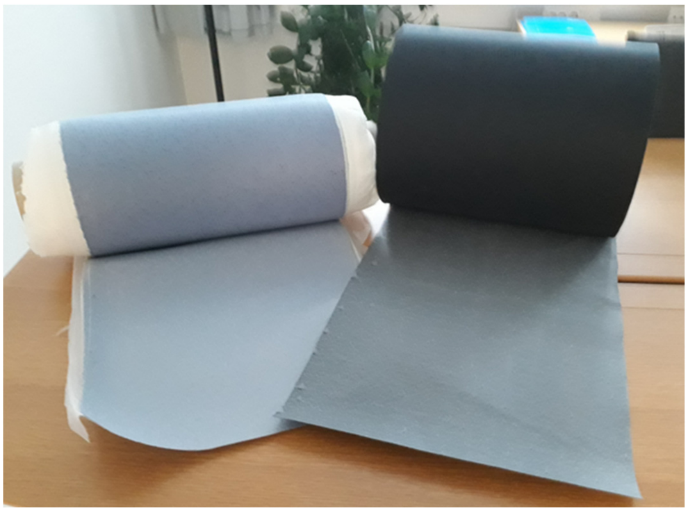


Figure 70 Laminated rolls

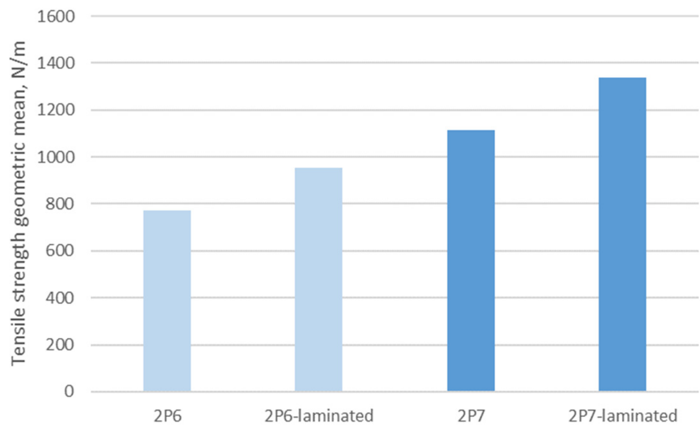


Figure 71 Tensile strength of non-laminated and laminated webs of roll 1 (2P6) and roll 2 (2P7)

Conclusions: The processability of rolls in laminating line and the adhesion of film were good.

The strength of NordicBio webs was at good level and met the expected target values. However the grammage of webs was higher than commercial nonwovens meaning that in the long run the strength/grammage ratio has to be improved.

7. Summary and Conclusions

This report summarizes VTT's work carried out in *Circular Nordic Bio Nonwoven in MedTech Applications* (NordicBio) project - a bilateral Swedish Finnish co-operation funded by Vinnova and Business Finland. NordicBio aimed for development and pilot scale demonstration of a cost effective, bio and circular material based materials suitable for medical applications.

VTT work focused on two processes: 1) cleaning of mechanically recycled fibres and 2) production of nonwoven materials from recycled fibres. Targeted applications were laminated nonwoven products suitable for bed sheet and absorptive products in hospitals. The main tasks of VTT research work related to cleaning consisted on literature study, laboratory testing of several possible cleaning processes and piloting of selected process. The main tasks related to nonwoven development consisted studies of fibre cutting options and of air-laying process, laboratory testing of various material formulations and binding methods for adjusting the nonwoven properties, and piloting of selected recipes.

Cleaning

The main objectives of literature review was evaluate how different kinds of contaminants such as dirt, stain and particularly micro-organism can be removed from used waste textiles without sacrificing mechanical fibre properties. To some extent colour/dye removal of textiles was also reviewed. Disinfection of textiles from different king of micro-organism should be done either with high temperature, ≥ 60°C, or with lower temperature together with oxidative chemical. Dyes removal effect depends on the used dyeing method and combination of chemicals used for removal. With sequential treatment higher removal can be achieved. Both disinfection and bleaching treatments can be harmful for the mechanical properties of textile material. In laundry type of testing typically 50 washing cycles are run before damage analysis therefore influence of single treatment is not known.

Evaluation the influence of cleaning (disinfection) conditions on cotton and polyester fibre strength properties was studied in laboratory trials. Testing included two commercial laundry detergents, Peracid Forte (PF) and Select Power Blue (SPB), and pure NaOH, H_2O_2 treatments were tested as alternative disinfection and bleaching chemicals. High temperature and high alkali concentration decreased fibre strength properties => 60°C was selected for the main temperature in the following cleaning trials. The influence of PF and hydrogen peroxide treatments on fibre strength properties were rather similar. With cotton material PF resulted more stable performance. Shortening of cotton fibres improved handling of material in cleaning trials and improved formation of hand sheets.

Colour removal was of reactive dyed cotton using reductive bleaching and combination of reductive and oxidative bleaching. Reductive bleaching with dithionite discoloured reactive dyed cotton. Higher dithionite and alkali dosage resulted higher brightness. The higher alkalinity the more bluish was the tone of the colour after discoloration. Oxidative hydrogen peroxide treatment after dithionite stage gave only few units increase in the brightness. Hydrogen peroxide treatment turned the tone of the colour reddish.

Test series was also carried out in order to evaluate the efficiency to remove micro-organisms with selected cleaning/disinfection treatments. Cotton samples were contaminated with six different microbes: mould, bacterium spore, yeast, bacterium gram negative, bacterium gram positive and virus. Microbial tests showed that the use of high temperature (80°C) at alkaline conditions is the most effective way to disinfect microbes, even bacterium pores. Lower temperatures (60°C and 40°C) with oxidative detergent reduce the content of other microbes effectively, except bacterium pores.

Peracid Forte treatment (2.0% and 60°C) was selected for the Pilot Cleaning runs due to its double disinfection effect: thermal and oxidative, and SPB for adjustment of pH. The cleaning pilot was done at KCL in Espoo for 134 kilos of recycled cotton fibres, which were shortened with granulator before cleaning. Process included 1) cleaning phase carried with SPB & PF, in consistency of 8%, at 60°C for 60 minutes, and 2) washing phase composing of diluting and de-watering steps until pH reduced to pH ~8. Piloting went mainly as planned, slight increase of temperature was observed in mixing, which was corrected by diluting with cold water. Most of the fibres from cleaning pilot was taken to nonwoven pilots as such and about 34 kg was refined in order to improve fibre bonding ability in nonwoven pilot.

Nonwovens

We studied different up-scalable methods for shortening recycled fibres in order to homogenize raw material and improve formation in nonwoven processes sensitive to fibre length. Comparison of different cutting methods showed that the best machine for shortening recycled cotton fibres was Rapid granulator. We also tested lab scale airlaying setup for preparation of nonwovens. Preliminary trials showed that our setup was not suited for making nonwovens from mixtures of recycled fibres and pulp with existing drums. Therefore studies were continued with foam laying process.

Nonwovens were prepared from recycled fibres in combination with pulp, regenerated cellulosic fibres and bi-component binder fibres, and using spunlacing, latex treatment and thermally activated bi-co fibres as binding methods. It was observed that mechanical properties, strength and strain, of nonwovens can be varied widely by fibre content and binding method. Tensile strength increased with increasing pulp content, while the highest tensile strengths were achieved with combining hydroentanglement treatment and bico fibres or with surface sizing with starch. Hydroentanglement treatment caused a clear reduction to grammage, increased tensile strength a little and dominating the development of strain. It was clear that high recycled cotton content can be achieved with optimized formulation and binding.

We also tested recyclability of foam laid nonwovens by slushing trials. Spunlace treatment did not effect on slushability, while latex bonding deteriorated slushability clearly. One slushing cycle improved the formation and after that formation was at constant level. One slushing cycle also improved tensile strength because of improved formation. However, tensile strength decreased with additional slushing cycles, because of the loss of fines.

Piloting was divided into two separate piloting occasions several months apart. The first pilot run focused on testing different formulations. Recycled fibres used included polycotton and cotton. Those were mixed pulp, bico and/or lyocell fibres with bonded afterwards with latex, spunlacing and/or heat, similarly to lab scale trials earlier. Recycled polycotton and cotton fibre fractions worked well in foam forming process, but a lot of fibre clumps exist in nonwoven webs. Spunlace treatment decreased the grammage. The strongest nonwovens were achieved by latex impregnation and the second best option was to utilize bico fibres. Spunlace treatment increased strain, but not so much strength.

Between pilots we focused on improving the quality of nonwovens webs namely decreasing the amount of fibre clumps in cut fibre fraction as well as in final sheet. It was observed that the amount of fibre clumps in sheets based on cut recycled cotton can be reduced by using granulator screen with small hole diameter in cutting phase, by wet refining cut fractions and by slushing cut fractions before sheet making phase. Fibre flocculation can be reduced by optimizing the foaming phase i.e. with high intensity mixing and optimized tank and mixer dimensions. It was also observed that the wet refining increased the strength of web so much that sheet based on 100% recycled cotton could be produced. However at the same time softness disappeared and sheet had more paper like feel.

The target of the second pilot trial was to produce nonwoven rolls based on two recycled fibre grades to produce high quality materials for lamination tests executed in Cellcomb. Recycled fibres included post-consumer polycotton and cleaned pre-consumer cotton. Polycotton was mixed with pulp and bico, and roll was obtained with 50/20/30 % composition. Another roll was prepared from recycled cotton composing of unrefined and refined cotton blended with bico with ratio of 25 % / 45 % / 30 %. High bico content of 30 % led to high strength and strain. Tensile strength of piloted materials was comparable to commercial references, but their strain was lower and grammages higher than references.

Rolls were sent to Cellcomb for lamination trials. It was observed that the processability of rolls in lamination line and the adhesion of film were good. The strength of laminated NordicBio webs were at good level and met the expected target values. However, the grammage of webs was higher than commercial nonwovens meaning that in the long run the strength/gsm ratio has to be improved.

Outcome and future prospects

NordicBio project addressed to growing amount waste coming from nonwoven medical disposables which currently are mostly based on fossil resources, and are incinerated after use. Post-consumer textile waste poses a huge opportunity to replace fossil raw materials and thus reduce the environmental effect of such disposables. VTT's target in this project was development and piloting of

1) purification/bleaching process mechanically recycled fibres as well as 2) development of medical grade nonwovens from natural fibre, recycled fibre and/or pulp mixtures.

Within this work we have tackled one of the challenges of recycled textiles, possible contamination of materials which can be solved via cleaning process optimized for textile wastes. We have also shown that tensile properties similar to commercial products can be achieved with foam laying process, and nonwovens were suitable for productisation via lamination. Further product and process development would be needed for improvements that could enable commercialization of this concept. Main development needs are related to fibre processing e.g. cutting and refining, and web properties, e.g. reduction of basis weight and increase of strain. In addition, more efficient removal of colour with the same process is use to disinfection of material would be beneficial for MedTech application.

References

Aspland J.R. (1992) Vat dyes and their application, *Textile Chemist & Colorist* **24**(1)22-24

Aspland J.R. (1997) Reactive dyes: Their Application and chemistry, Chapter 9 in *Textile Dyeing and Coloration*, Research Triangle Park, NC, American Association of textile Chemists and Colorists, p. 130

Bigambo P., Liu H., Broadbent P.J., Carr C.M., Rigout M. (2018) Surface chemical and calorimetric analysis of reactively dyed cellulosic fabrics. The effect of ISO 105 C09 laundering and the implications for waste cellulosic recycling, *Dyes and Pigments* **148** 91-98

Brands B., Brinkmann A., Bloomfield S., Bockmühl, D.P. (2016) Microbicidal action of heat, detergents and active oxygen bleach as components of laundry hygiene. *Tenside Surfact Det* **53** 1-8

Bockmühl D.P. (2017) Laundry hygiene - how to get more than clean, *Journal of Applied Microbiology* **122** 1124-1133

Chavan, R.B. (1969) Stripping of Dyestuffs from Textiles, *Rayon Industries of India*, February 1969, 167-170

Eren S., Gümüs B., Eren H. (2016) Color stripping of reactive-dyed cotton by ozone treatment, *Color. Technol.* **132** 466-471

Esteve-Turrillas F.A., de la Guardia M. (2017), Environmental impact of Recover cotton in textile industry, *Resources, Conservation and Recycling* **116** 107-115

Fiberyear, The Fiber Year 2017, available https://www.thefiberyear.com/

Fijan S., Turk S.S., Neral B., Pušić T. (2007) The influence of industrial Laundering of hospital textiles on the properties of cotton fabrics. *Textile Research Journal* **77**(4)247-255

Forss S. (2017) email discussion 28.9.2017, Lindström group

Haule L.V., Carr C.M., Rigout, M. (2014) Investigation into the removal of an easy-care crosslinking agent from cotton and the subsequent regeneration of lyocell-type fibers, *Cellulose* **21** 2147-2156, DOI 10.1007/s10570-014-0225-3

Haule L.V., Carr C.M., Rigout M. (2016) Preparation and physical properties of regenerated cellulose fibres from cotton waste garments, *Journal of Cleaner Production* **112** 4445-4451

Heikkilä P., Fontell P., Määttänen M., Harlin A. (2018a) Review of textile Recycling Ecosystem and a Case of Cotton. In K. Niinimäki (Ed.) Sustainable Fashion in a Circular Economy (pp. 192-219). Aalto University

Heikkilä P. (ed.), Fontell P., Kamppuri T., Mensonen A., Määttänen M., Pitkänen M., Raudaskoski A., Vehmas K., Vehviläinen M., Harlin A. (2018b) The Relooping Fashion Initiative, VTT Research Report No VTT-R-01703-18, available https://cris.vtt.fi/en/publications/the-relooping-fashion-initiative

Heikkilä P., Cura K., Heikkilä J., Hinkka V. Ikonen T., Kamppuri T., Knuutila H., Kokko M., Lankiniemi S., Lehtinen L., Mäkiö I., Pitkänen M., Saarimäki E., Virta M., Zitting J., Harlin A. (2019a) Telaketju towards circularity of textiles, VTT Research Report VTT-R-00062-19 https://cris.vtt.fi/ws/portalfiles/portal/24459480/Telaketju_Towards_Circularity_of_Textiles_PublicReport.pdf

Heikkilä P., Kamppuri T., Saarimäki E., Pesola J., Alhainen N., Jetsu P. (2019b) Recycled Cotton Fibres in Technical and Clothing Applications, 4th International Conference On Natural Fibers Porto/Portugal, 1-3 July 2019

Holopainen R. (2012) A human thermal model for improved thermal comfort: PhD Dissertation, VTT Science 23, https://www.vttresearch.com/sites/default/files/pdf/science/2012/S23.pdf

Honisch M., Stamminger R., Bockmühl D.P. (2014) Impact of wash cycle time, temperature and detergent formulation on the hygiene effectiveness of domestic laundering. *J Appl Microbiol* **117** 1787-1797

Honisch M., Weide M.R., Speckmann D., Bockmühl D.P. (2016) Antimicrobial efficacy of laundry detergents with regard to time and temperature in domestic washing machines. *Tenside Surfact Det* **53** 547-552

Izadifar Z., Chen X., Kulyk W. (2012) Strategic Design and Fabrication of Engineered Scaffolds for Articular Cartilage Repair, *Journal of Functional Biomaterials* **3** 799-838, DOI 10.3390/jfb3040799

Janczak F., Stamminger R., Nickel D., Speckmann D. (2010) Energy savings by low temperature washing. *SOFW J* **136** 75-80

Jones T. (2007) Peracetic acid: a biocide for the future. *INFORM Technology Collection* **18**(1)63-65

Kamppuri T., Pitkänen M., Heikkilä P., Saarimäki E., Cura K., Zitting J., Knuutila H., Mäkiö I. (2019) Tekstiilimateriaalien soveltuvuus kierrätykseen, VTT Tutkimusraportti VTT-R-0091-19, https://cris.vtt.fi/ws/portalfiles/portal/24225719/VTT_R_00091_19.pdf

Lakdawala N., Pham J., Shah M., Holton J. (2011) Effectiveness of low-temperature domestic laundry on the decontamination of healthcare workers' uniforms. *Infect Control Hosp Epidemiol* **32** 1103-1108

Lewis D.M. (2007) Dyeing cotton with reactive dyes under neutral conditions, *Coloration Technology*. **123** 306-311. DOI: doi.org/10.1111/j.1478-4408.2007.00099.x

Lim S.H., Hudson S.M. (2004) Application of a fibre-reactive chitosan derivative to cotton fabric as a zero-salt dyeing auxiliary, *Coloration Technology*. **120** 108-113. DOI:doi.org/10.1111/j.1478-4408.2004.tb00215.x

Määttänen M., Asikainen S., Kamppuri T., *et al.* (2019), Colour management in circular economy: decolourization of cotton waste, *Research Journal of Textile and Apparel* **23** 134-152, https://doi.org/10.1108/RJTA-10-2018-0058

Nättinen K., Nikkola J., Minkkinen H., Heikkilä P., Lavonen J., Tuominen M. (2011) Reel-to-reel inline atmospheric plasma deposition of hydrophobic coatings, *Journal of Coatings Technology and Research* **8** 237-245, DOI 10.1007/s11998-010-9292-z

Palme A., Idström A., Nordstierna L., Berlid H. (2014) Chemical and ultrastructural changes in cotton cellulose induced by laundering and textile use, *Cellulose* **21** 4681-5691

Rahunen M. (2019) email discussion 28.3.2019, Oy Christeyns Nordic Ab

Reinhardt G. (2006) Fingerprint of bleach system. *Journal of molecular catalysis A: Chemical* **251** 177-184

Rice R.G., DeBrum M., Cardis D., Tapp C. (2009) The ozone Laundry Handbook: a Comprehensive Guide for the proper application of ozone in the commercial Laundry Industry. Ozone: Science & Engineering, **31**(5)339-347, DOI: 10.1080/01919510903091318

Sapers I. (1971) Products and process for removing organic pigments and dyes from dyed and printed natural and synthetic textile materials, U.S. Pat. No. 3,591,325

Tano E., Melhus Å. (2014), Level of decontamination after washing textiles at 60°C or 70°C followed by tumble drying, *Infection ecology & Epidemiology* **4**(1)24313, DOI:10.3402/iee.v4.24314

Uddin M.G., Islam M.M., Islam M.R. (2015) Effects of reductive stripping of reactive dyes on the quality of cotton fabric, *Fashion and Textiles* **2**(8)12, DOI 10.1186/s40691-015-0032-y

Upphandlings myndigheten (2019) Engångstextilier för sjukvården. Available: https://www.upphandlingsmyndigheten.se/hallbarhet/stall-hallbarhetskrav/sjukvard-och-omsorg/engangstextilier-for-sjukvarden/engangstextilier-for-sjukvarden/ (visited 4th March 2019)

Walger E., Rivollier C., Marlin N., Mortha G. (2015) Activated hydrogen peroxide decolorization of a model azo dye-colored pulp, *Holzforschung* **69**(6)677-683

Wanassi B., Azzouz B., Hasse M.B. (2016) Value-added waste cotton yarn: Optimization of recycling process and spinning of reclaimed fibers, *Industrial Crops and Products* **87** 27-32

Wedin H., Niit E., Mansoor Z.A., Kristinsdottir A.R., de la Motte H., Jönsson C., Östlund Å., Lindgren C. (2018) Preparation of viscose fibers stripped of reactive dyes and wrinkle-free crosslinked cotton textile finish, *Journal of Polymers and the Environment* **26** 3603-3612

Winkler J., Smith E.R., Compton R.G. (1997) A study of the Mechanism of Bleaching Cotton Using Peracids and Hydrogen Peroxide as Model Systems. *Journal of Colloid and Interface Science* **195** 229-240