Thesis for the Degree of Master in Science
with a major in Textile Engineering
The Swedish School of Textiles
2021-02-25 Report no. 2020.14.01

A novel and feasible material recycling technique for end-of-life textiles as All-Cellulose Composites (ACCs)

Belinda Johansson

Examiner: Mikael Skrifvars
Supervisor: Behnaz Baghaei

Visiting adress: Skaraborgsvägen 3 • Postal adress: 501 90 Borås • Website: www.hb.se/ths

ABSTRACT

Today's consumption of textiles generates a large volume of textile waste. Therefore, it is needed to find solutions to re-use the textile waste rather recycling fibers into new fibers. Research using pre- and post-consumer textiles in composites is ongoing and an interesting direction. This thesis reports the recycling of discharged cellulose containing textiles by production of all-cellulose composites (ACCs). ACCs are composites consisting entirely of cellulose. ACCs from discarded denim and polycotton hospital sheets (PCO) were successfully produced with partial dissolution and two-step method. Discarded denim fabrics with 100% cotton (CO) and blend material (BCO) was sorted and shredded into fibers, then made into nonwovens by needle punching. The produced nonwoven laminates and intact hospital sheets were used as the reinforcement in the composites while dissolved cellulose in an ionic liquid (IL), 1-butyl-3-methyl imidazolium acetate ([BMIM][Ac]), was used as the matrix phase. The matrix is then regenerated by removal of the [BMIM][Ac] by washing to form the composite. The washed-out [BMIM][Ac] was collected and recycled in order to study the effect of its reusing as recycled cellulose solvent on mechanical properties of ACCs. Combinations of the different methods, materials and solvents were studied. The mechanical properties – tensile, flexural and impact properties and the void content of the produced composites were determined. Microscopic analysis was done to study the cross-section of the composites. It could be concluded that the achieved values for the mechanical properties can be compared with ACCs found in literature. The results from the research implies that it is possible to find a new purpose for recycled textiles in the form of composites. With this approach, it is possible to avoid unnecessary disposal of textiles containing cellulose.

Keywords: all-cellulose composites, end-of-life textiles, denim fabrics, ionic liquid, mechanical properties, sustainability

POPULAR ABSTRACT

Today's consumption of textiles generates a large volume of textile waste. The recycled textiles are commonly made to fibers, but a lower quality is often maintained when recycling the textiles. It is therefore wanted in this research to examine if end-of-life textiles can be recycled into composites with a higher value. All-Cellulose Composite (ACC) is a composite only consisting of one material. In this way it can enhance the recycling possibility of the composite compared to conventional composites containing a mix of materials. ACCs can be produced by different methods using ionic liquid (IL), in this thesis two different methods are tested – partial dissolution and two-step method. The cellulose material is dissolved by the IL. The IL used for ACC production was washed out, collected and recycled to be able to reuse it. Three different materials were tested, 100% cotton from denim (CO), blend material from denim (BCO) and polycotton fabric from hospital sheets (PCO). Combinations of the materials and methods were tested. Characterization was done to study if the composites could be compared with ACCs found in literature. The mechanical properties and void content were investigated. Microscopic analysis was performed to visually inspect the cross-section of the samples. The results from the mechanical testing showed comparable values compared to ACCs found in literature. Variance between the produced samples could also be detected and analyzed. The results from the research indicates that it is possible find a new purpose for recycled textiles in the form of composites. With this method, it is possible to avoid unnecessary disposal of cellulose containing textiles.

ACKNOWLEDGEMENTS

At first, I would like to thank my supervisor Behnaz Baghaei. Your endless support and positive spirit through the thesis work have been priceless and I could not have asked for a better supervisor. Especially during the circumstances during these tough times.

Also, I want to thank my family, friends and my partner for believing in me and supporting me through my thesis work but also through my years of studying at the University. It would have been difficult without you. At last, I want to take the opportunity to say a special thank you to my dear classmates, through ups and downs, tears and laughs, we made it.

Belinda Johansson
Vårgårda, January 2021

LIST OF ABBREVIATIONS

ACC	All-Cellulose Composite
BCO	In this thesis; blend cotton material from discarded denim
$[BF_4]^-$	Boron tetrafluoride
[BMIM][Ac]	1-Butyl-3-methylimidazolium acetate
[BMIM][Cl]	1-butyl-3-methyl-imidazolium-chloride
$[C4mim]^+ Cl^-$	1-butyl-3-methyl imidazolium chloride
CO	In this thesis; cotton material from discarded denim
DMSO	Dimethyl sulfoxide
FTIR	Fourier Transform Infrared Spectroscopy
IL	Ionic Liquid
O-H group	Hydroxy group
PCO	In this thesis; polyester-cotton fabric from hospital sheets. Polyester in this thesis is referred to Poly (ethylene terephthalate)
$[PF_6]^-$	Hexafluorophosphate
RIL	Recycled Ionic Liquid from the ACC production in this thesis
$[SbF_6]^-$	Fluor antimonic acid

TABLE OF CONTENTS

1. INTRODUCTION

Today's consumptions of textiles generate a large volume of discarded textiles. It is therefore necessary to take action and responsibility for the textile waste. (Shirvanimoghaddam, Motamed, Ramakrishna & Naebe 2020) One of UNs sustainable development goals [1] is – "ensure sustainable consumption and production patterns" (UN 2015). The sustainability goal does not cohere with the increasing consumption of textile products and the number of discarded textiles ending up on landfill or incineration (Periyasamy & Militky 2017; Ponnusamy & Yaashikaa 2018). The development of environmentally friendly materials is therefore of importance, where the development is going towards materials from renewable resources[2] and recycled fibers, polymers and textiles (Sandin & Peters 2018; Soykeabkaew, Nishino & Peijs 2009)

One part of the textile industry which has negative impact on the environment, is the life cycle of denim (Schrott & Paul 2015). For instance, a pair of Levi's 501 jeans requires 3781 liters of water during its life cycle (Levi's Strauss & Co 2015). Luiken and Bouwhuis (2015) claimed that the cutting waste from denim production is between 10% – 15%. Furthermore, the researchers assume in theory, that the post-consumer denim waste are 2.16 million tons per year, where the gathered waste textiles only are 35% – 50%. (Luiken & Bouwhuis 2015) By finding a repurpose for discarded textiles it can reduce several production steps and the need for virgin textile fibers, and indirect the impact on the environmental (Radhakrishnan, 2017; Sandin & Peters 2018). The repurpose does not have to be a new textile fiber, instead the discarded textile can be used to produce artefacts[3] (Sandin & Peters 2018). Previous research has been done where discarded denim was used in the development of biocomposites for structural applications (Temmink, Baghaei & Skrifvars 2018). To proceed with the development of composites with discarded textiles one option could be All-Cellulose Composites. All-Cellulose Composites (ACCs) was first reported by Nishino, Matsuda and Hirao in 2004. The technique for ACCs could be used to enable recycling of discarded textiles into e.g., composites for structural applications. It is therefore interesting in this thesis to investigate if discarded textiles from

[1] United Nations has stated 17 goals for sustainable development in Agenda 2030. Sustainable development involves three dimensions - social, ecological and economical sustainability. (UN 2015)

[2] Materials from renewable resources should have similar properties compared with conventional polymers, effective production and have less impact on the environment compared to petroleum-based polymers (Zhu et al., 2016).

[3] Artefact is a fabricated object by human beings (Nationalencyklopedin n.y.a).

cellulosic materials can be used with the manufacturing technique for ACCs to produce composites.

1.2 Problem description

Denim is a part of today's fast fashion and the post-consumer denim waste follow this trend. To prevent the waste ending up on landfill or incineration, it is needed to find solutions where to use the discarded textiles. One opportunity could be to recycle the textiles into composites. As mentioned in the introduction, discarded denim has been used as reinforcement in biocomposites. Nonetheless, little research has been done where post-consumer textiles has been used as both matrix and reinforcement. It is therefore interesting to study if the technique for producing ACCs can be used as a method to recycle low value textiles[4] into new artefacts, with a higher value. If the denim waste contains a mix of materials it cannot be called an ACC, nevertheless the production method is still the same. In the end, it is wanted to find a repurpose for the discarded textiles so the need for virgin material is reduced and to prevent already produced material just burning up.

1.3 Research questions

The aim of this study is to create ACCs produced from discarded textiles, in the form of nonwoven from denim waste and intact hospital sheets together with ionic liquid in order to investigate if the method can be used to recycle textiles into composites. The mechanical properties of produced composites are determined so it can be compared with ACCs from literature. To fulfill the aim the following research questions are asked:

- Is it possible to use the technique of ACCs, to recycle textile waste from denim fabrics and hospital sheets into composites?
- Can end-of-life cotton fabrics (in the form of nonwoven), be used with the technique for ACCs and achieve comparable mechanical properties - tensile, flexural and impact strength, with other ACCs reinforced with cellulosic fibers?
- Is there a difference regarding the mechanical properties and void content between the different combinations of methods and materials?

1.4 Limitations

The work of the thesis was limited to the resources at the University of Borås due to Covid-19. The quantity of discarded textiles was limited to collected volume.

[4] Low value textiles are most often downcycled, since the quality of the material can get reduced when recycled. Opposite, upcycling is when the recycled material is turned into a new product with a higher value. (Schmidt et al., 2016)

2. LITERATURE REVIEW

2.1 ALL-CELLULOSE COMPOSITES

An ACC is a composite where both the reinforcement and matrix are from cellulose material, this makes the composite a single polymer/monocomponent composite (Nishino, Matsuda & Hirao 2004). Since both parts are of hydrophilic nature it could increase the adhesion at the interface. Which could lead to an increase in performance and mechanical properties of the composite (Nishino, Matsuda & Hirao 2004; Wei et al. 2016). Compared to a composite consisting of different materials with different polarities, e.g., natural fibers with hydrophilic character and hydrophobic polymer matrix. The interface adhesion could therefore be weaker, this due to poor bonding between the two materials. This can decrease among others the mechanical strength of the composite. (Spörl et al. 2018) Huber, Bickerton, Müssig, Pang and Staiger (2013) made ACC laminates to test the flexural strength of it, and then compared it with biocomposites. The researchers could claim that the flexural strength of their produced ACC laminates was better than other biocomposites. Furthermore, no delamination could be detected when inspecting the composite after testing. The result is dependent on the strong adhesion at the interface between the constituents. (Huber et al. 2013) Furthermore, it is beneficial to produce ACCs to simplify the recycling and the possibility to be biodegradable (Nishino, Matsuda & Hirao 2004; Spörl et al. 2018). Spörl, Batti, Vocht, Raab, Müller, Hermanutz and Buchmeiser (2018) could within their research successfully produce an ACC and then recycle it 3 times. The researcher used a "high strength rayon filament yarn" as reinforcement and an ionic liquid *1-ethyl-3-methylimidazolium acetate* [EMIM][OAc] to dissolve the dissolving pulp cellulose for the matrix. Once the composite was made, it was milled to afterward be dissolved and reused. (Spörl et al. 2018)

2.2 MOLECULAR STRUCTURE OF CELLULOSE

The repeating unit in the cellulose polymer is built on two glucose units, which makes the repeating unit, cellobiose, see Figure 1. The repeating units makes a linear cellulose polymer. (McLoughlin, Haye & Paul 2015) The cotton fiber is 35-30% amorphous and 65-70% crystalline. Cotton fibers are hydrophilic but nevertheless not soluble in water due to the hydrogen bonds between the chains (Albertsson, Edlund & Odelius 2012; Hatch 1993).

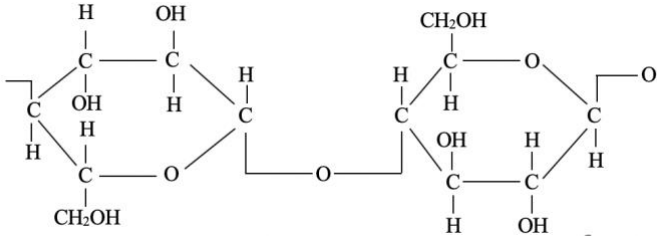

FIGURE 1. STRUCTURE OF CELLULOSE POLYMER INSPIRED FROM HATCH (1993).

The polar hydroxyl groups (OH groups) are the most important chemical group of the cellulose polymer, since it affects the properties of the fiber and can enable the possibility to modify cellulosic textiles. (Caulfield, Feng, Prabawa, Young & Sanadi 1999; Hatch 1993).

2.3 SOLVENTS FOR DISSOLVING CELLULOSE

Multiple solvents can be used to dissolve cellulosic materials when producing ACCs. Some examples are, N,N-dimethylacetamide/lithium chloride (DMAc/LiCl), dimethyl sulfoxide/diethylamide/sulphur-dioxide (DMSO/DEA/SO2) or ionic liquids such as butyl-methylimidazolium chloride ([BMIM][Cl]). (Rebière et al. 2016; McLoughlin et al. 2015) One approach to achieve dissolution of cellulose is to use an aqueous solution containing alkali hydrates and urea hydrates (Cai & Zhang 2005). Another approach to accomplish dissolution is by using ILs. When comparing ILs to conventional chemicals for manufacturing of ACCs, ILs are considered to be more environmentally friendly solvents. (Amde, Liu & Pang 2015) Additionally, ILs are beneficial due to the ability to recycle the solvent (Spörl et al. 2018). ILs are therefore described further in the section below.

To enable the regeneration of cellulose is an anti-solvent also called non-cellulose-solvent used (Rebière et al. 2016). The anti-solvents are described in the section under ILs.

2.3.1 IONIC LIQUIDS

Pringle, Annat, Kar and MacFarlane (2017) defines an IL as "a liquid comprised entirely of ions". Pringle et al. (2017) continue to describe that an IL is of a mixture of salts, that consists of different ions and a typical property of ILs are a melting point below 100°C. There can be a various variant of ILs e.g. 1-butyl-3-methylimidazolium chloride ([BMIM][Cl]) or 1-ethyl-3-methylimidazolium acetate [EMIM][OAc] (Spörl et al. 2018). Most of the ILs are beneficial to work with due to their low flammability and negligible vapor pressure compared to volatile solvents that could be used (Pringle et al. 2017). However, it is important to work with the 'right' ILs. If the IL contains fluorine, have short alkyl side chains or using anions such as, $[BF_4]^-$, $[SbF_6]^-$ and $[PF_6]^-$, it cannot be considered as environmentally friendly. Ionic liquids are therefore dependent on among others their morphology and type, to which extend they are toxic. (Amde, Liu & Pang 2015) Nevertheless, there are some limitations to consider regarding ILs. Three of the limitations are - the high cost compared to common organic solvents, the high viscosity, which could complicate the process in industrial applications and the last is the lack of research regarding the biodegradability. (Meksi & Moussa 2017)

2.3.1.1 Structure of Ionic Liquids

The lignocellulosic complex is disordered by both the anions and cations of the IL, which results in dissolution of the cellulose (Rieland & Love 2020). The high viscosity depends on the strong intermolecular interactions, considering hydrogen bonding, van der Waals forces and Coulombic forces. Where, the high viscosity of ionic liquids can be compared with the consistency of honey. It is possible to reduce the viscosity of the ILs by mixing with molecular solvents. However, adding of solvents will have impact on the flammability and increase in the vapor pressure, compared to ILs without any extra solvent. (Pringle et al. 2017) Dimethyl sulfoxide is one option to use when it is wanted to decrease the viscosity (Lv et al. 2012).

2.3.1.2 Recycling of Ionic Liquids

As mentioned above, the costs of using ionic liquids are high, it is therefore desired to recover and reuse the ILs. Different methods have been used for recovery of ILs, where some of them are; distillation, extraction and membrane-based methods. Distillation and extraction are the methods most commonly used for recovering of ILs. (Mai, Ahn & Koo 2014)

2.3.3 Anti-solvents

Anti-solvents/non-cellulose-solvents are used for regeneration of the cellulose. Regeneration of cellulose means that the cellulose is separated from its solvent. Anti-solvent is needed to control the properties and structure of it, e.g. the crystallinity of the regenerated cellulose (Tan, Chen, Li & Xie 2019). Water, ethanol and acetonitrile are commonly used anti-solvents (Tan, Chen, Li & Xie 2019; Huber, 2012a). Since the solvents consists of at least one hydrogen atom, it enables the hydrogen bonding in the cellulose (Scheiner 1997). When using water as the anti-solvent a more crystalline-structured and organized cellulose will be created. This reaction is due to polarity of water and hydrogen bonding between water and cellulose molecules. Where ethanol has less polarity compared to water. (Tan et al. 2019) A study has showed that the diffusion coefficient has impact on the crystallinity of the regenerated cellulose. Where anti-solvents with a lower diffusion coefficient resulted in a more crystalline cellulose. (Fan et al. 2017) However, the result from Tan et al. (2019) showed that water gives a higher crystallinity contradictory to Fan et al. (2017) result which showed that water with a high diffusion coefficient results in a less crystalline cellulose. The difference in result could depend on the IL and cellulose used in the experiments, since the researchers used different materials. Hence, the choice of ILs and anti-solvents can have impact on the crystallinity (Fan et al. 2017).

2.4 Steps for dissolving and regeneration

In general, five steps are used to dissolve and regenerate the cellulose sample with ILs. The first step is to pre-dry the sample to decrease the content of

moisture in the sample. This is done to prevent any hinder of the dissolution. The second step is to dissolve the cellulose. This is done under specific conditions depending on material of cellulose sample. The third step is regeneration of the dissolved cellulose, where a *non-cellulose-solvent* is added. The fourth step is to remove the solvent residuals by filtering and washing the regenerated sample. The fifth and final step is to dry the sample and ensure no residuals are left in the sample. (Rebière et al. 2016)

2.5 Fabrication methods for all-cellulose composites

Two methods are commonly used for productions of ACCs; first method is dissolution of cellulosic material in a solvent which then are added on a cellulosic reinforcement (two-step method) and the second method is partial dissolution (one-step method) (Nishino, Matsuda & Hirao 2004). Figure 2 illustrates the two different methods.

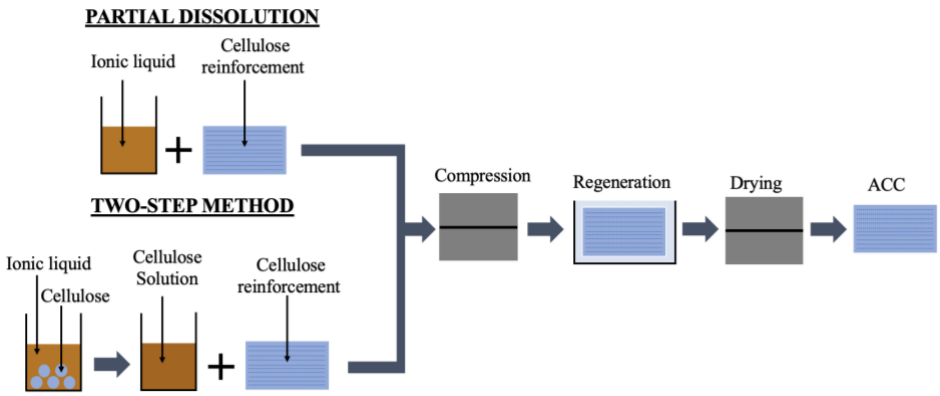

Figure 2. Illustration of the methods partial dissolution and two-step method (figure inspired from Baghaei and Skrifvars 2020).

The purpose of partial dissolution/one-step method is to dissolve the surface of the fibers and then regenerate the cellulose to form the matrix and bond to the undissolved fiber. In the two-step method, cellulose is dissolved in a solution and then added on the undissolved cellulose reinforcement. (Huber et al. 2012b) The concentration of cellulose in the solvent is of importance, since a cellulose concentration above 4% can give a poor impregnation due to high viscosity of the solution (Qin, Soykeabkaew, Xiuyuan & Peijs 2008). Nevertheless, the following production steps are not depending on previous methods mentioned. The dissolution of cellulose is done by using heat and pressure. The pressed composite/laminates are then placed in an anti-solvent to enable regeneration. The last step is to dry the laminates with heat and pressure. Furthermore, it is possible to collect the used IL from the regeneration solution and reuse it for a new fabrication of ACCs. (Huber, Bickerton, Müssig, Pang & Staiger 2012a) The time and temperature during dissolution and the applied compressing pressure with high temperature, are important process factors which has impact on the properties on the created ACC laminae (Mat Salleh, Magniez, Pang, Dormanns & Staiger 2017). Huber

et al. (2012a) reported that the highest tensile strength was achieved by using a pressure on 0.20 MPa, 60 min and hot press at 95°C. Similar result was achieved by Mat Salleh et al. (Mat Salleh et al. 2017) where the highest value of Youngs modulus was reached at 60 min, 105°C and a pressure of 0.25 MPa. When the ACC laminae has a more uniform and homogeneous structure and less voids it will result in higher mechanical properties (Mat Salleh et al. 2017). Hence, the researcher's findings indicate, that the applied pressure will have impact on the mechanical properties of the ACC laminae.

2.6 SUSTAINABILITY

The definition *sustainability* implies that both society and organizations use and relates to earth's resources, without affecting the environment and humans negative (Sweet 2017). Sustainable development contains three dimensions – ecological, economic and social sustainability. One of UNs sustainable development goals is – "ensure sustainable consumption and production patterns". Where one subgoal is – "by 2030, sustainably reduce the waste generation through prevention, reduction, recycling and reuse". (UN 2015) The ecological dimension refers to biodiversity and the resources of the nature, the social dimension concerns the social expectations, culture and politics, and the economic dimension is about economic (Gröndahl & Svanström 2011). Furthermore, to work towards sustainable development, the expression *cradle to cradle* can be applied, also called circular economy. The aim of circular economy is to imitate the cycle of the nature, where disposals are considered to be a new resource instead of waste. By doing a clever design from the beginning, where choices are made that can simplify the recycling and reuse of the materials, it can lead to a decrease in e.g., costs. (Lerpold & Nordbrand 2017 p.191) It is therefore necessary to recycle textiles and find sustainable solutions in all dimensions to ensure that the goals will be realized.

2.7 RECYCLING OF TEXTILES

There are different methods for how to recycle textile material, either chemical or mechanical recycling. The collected discarded textiles can be made to fibers again by the mechanical process, shredding. (Östlund et al. 2015) The quality of the shredded fibers, such as fiber length and strength depend on which type of textile waste was used. Fibers with low quality are most often used in nonwoven applications, reinforcement, automotive sound insolation etc. (Leonas 2017) The denim waste from production often has high quality since the materials in the fabric is known, compared to the post-consumer waste where the compositions could be unknown and therefore a lower quality could be the case. (Luiken & Bouwhuis 2015)

2.7.1 DENIM

The fabric *denim* is twill weave made out of cotton, usually colored blue. Denim was first used for work wear and then developed into a fashion garment (Nationalencyklopedin n.y.b). Since denim is one of the most commonly used material worldwide, it has a significant impact on the environment. Considering the life cycle of a pair of jeans, from fiber production to post-consumer phase. (Radhakrishnan 2017) Now days, denim can consist of a combination of fibers and not only cotton.

2.8 RECYCLED TEXTILES IN COMPOSITES

Discarded textiles can be recycled and find a repurpose by using the material in composites. This enables the possibility to create new products from waste material. Previous research about composites with recycled denim has been reported. Temmink, Baghaei and Skrifvars (2018) developed biocomposites from denim waste and thermoset bioresins, such as bio-epoxy and acrylated epoxidized soybean oil resin. The biocomposites were created by doing compression moulding, vacuum infusion, resin transfer moulding and hand lay-up. In the research the researchers used denim consisting of 100% cotton. The denim was kept intact and cut in appropriate size. (Temmink, Baghaei & Skrifvars 2018) Baghaei, Compiet and Skrifvars (2020) developed ACCs from discarded cotton textiles. The reinforcement was from post-consumer cotton denim, where the fabric was kept intact and cut into squares, and the matrix phase was from two different cellulose resources - recovered and virgin cotton fibers, dissolved in IL. (Baghaei, Compiet & Skrifvars 2020) Meng et al. (2019) used denim waste and epoxy resin to produce a composite. The researchers used a needle-punching technology to create a nonwoven fabric from the denim waste. The denim waste was converted to fibrous form by a “cutting-opening-carding-needling process”. The nonwoven is created by needles penetrating the fiber web and are therefore securing the fibers. (Meng et al. 2019)

2.9 APPLICATIONS OF ACCS

The applications available for ACCs and composites with natural fibers are in general numerous. Both aircraft and automotive industry are working and developing natural fiber composites. It is beneficial due to the lower price, possibilities for anisotropic properties and the specific properties that can be obtained with natural fibers. (Ho et al. 2012) For example, composites consisting of wood and/or cotton fibers are used in interior, exterior and trunk floor parts in cars. The mechanical properties required for structural applications depend on the particular application. (Thomas et al., 2013) Tervahartiala et al. (2018) succeeded to produce ACCs by using different kinds of pulps. Their results indicate that the produced ACCs have potential to be used for corrugated board applications. Furthermore, the composites can be used for e.g., sound absorption (Ho et al. 2012).

2.10 MECHANICAL PROPERTIES OF ALL-CELLULOSE COMPOSITES

The mechanical properties of ACCs depend on different factors such as, the material used as reinforcement and conditions during processing. Since the production methods, parameters and type of cellulose varies between produces ACCs, it can be difficult to compare the mechanical properties between the composites. (Singh et al. 2015)

A summary of methods used for production of ACCs and the measured mechanical properties can be seen in Table 1.

TABLE 1. LITERATURE REVIEW ON ACCS PRODUCTION METHODS AND THEIR MECHANICAL PROPERTIES.

Composition	**Solvent**	**Anti-solvent**	**Method**	**Mechanical properties**			**Ref**
Reinforcement: post-consumer cotton denim Matrix: recovered and virgin cotton	[BMIM][Ac]	Water	2-step	Young's modulus [GPa] 2.5 to 4.3 Flexural modulus [GPa] 1.6 to 2.1	Flexural strength [MPa] 25 to 29 Charpy energy [kJ/m^2] 31 to 37		5
Rayon Twill weave	[BMIM][Ac]	Distilled water	Partial dissolution	Flexural modulus [GPa] 3.72+/-0.34	Flexural strength [MPa] 135.24+/-11.16	Charpy energy [kJ/m^2] 41.54+/-4.44	6
Flax Nonwoven	[BMIM][Cl]	Water	Partial dissolution	Young's modulus [GPa] 4.6+/-0.5	Tensile strength [MPa] 34+/-2		7
Lyocell Nonwoven	[BMIM][Cl]	Water	Partial dissolution	Young's modulus [GPa] 7.2+/-1	Tensile strength [MPa] 78+/-4		7

5: Baghaei, B., Compiet, S. & Skrifvars, M. (2020). Mechanical properties of all-cellulose composites from end-of-life textiles. *Journal of polymer research,* 27(9). doi:10.1007/s10965-020-02214-1

6: Huber et al., (2013). Flexural and impact properties of all-cellulose composite laminates. *Composites Science and Technology,* 88, pp. 92-98. doi:10.1016/j.compscitech.2013.08.040

7: Gindl-Altmutter et al., (2012). All-cellulose composites prepared from flax and lyocell fibres compared to epoxy–matrix composites. *Composites Science and Technology*, 72(11), pp. 1304-1309. doi:10.1016/j.compscitech.2012.05.011

Rayon Twill weave	[BMIM] [Ac]	Distilled water	Partial dissolution	Young's modulus [GPa] 4.1	Tensile strength [MPa] 91.12	8
Rayon Twill weave	[BMIM] [Ac]	Distilled water	Partial dissolution	Young's modulus [GPa] 1.08	Tensile strength [MPa] 64.99	8
Rayon Twill weave	[BMIM] [Ac]	Distilled water	Partial dissolution	Young's modulus [GPa] 2.71	Tensile strength [MPa] 71.61	8
Microcrystalline cellulose	[BMIM] [Cl]	Water	Partial dissolution	Young's modulus [GPa] 3.86 to 7.16	Tensile strength [MPa] 33.9 to 76.8	9
Reinforcement: ramie Matrix: craft pulp from coniferous trees	LiCl/DMAc	Methanol	2-step	Young's modulus [GPa] 45	Tensile strength [MPa] 480	10
Lyocell Twill weave	$[C4mim]^+ Cl^-$	Distilled water	Partial dissolution	Young's modulus [GPa] 1.7	Tensile strength [MPa] 45	11
Reinforcement: ramie Matrix: ramie	LiCl/DMAc	Methanol	2-step	Young's modulus [GPa] 25	Tensile strength [MPa] 540	12
Bocell	LiCl/DMAc	Methanol	Partial dissolution	Young's modulus [GPa]	Tensile strength [MPa]	

8: Huber et al., (2012). Solvent infusion processing of all-cellulose composite materials. *Carbohydrate Polymers*, 90(1), pp. 730-733. doi:https://doi.org/10.1016/j.carbpol.2012.05.047

9: Zhou, L. (2010). All Cellulose Composite prepared in an inoic liquid. Diss. University of Minnesota.

10: Nishino, T., Matsuda, I. & Hirao, K. (2004). All-Cellulose Composite. *Macromolecules*, 37(20), pp. 7683-7687. doi:10.1021/ma049300h

11: Dak, B. & Mukhopadhyay, S. (2018). All-cellulose composite laminates with low moisture and water sensitivity. *Polymer*, 141, pp. 79-85. doi:https://doi.org/10.1016/j.polymer.2018.02.065

12: Qin, C., Soykeabkaew, N., Xiuyuan, N. & Peijs, T. (2008). The effect of fibre volume fraction and mercerization on the properties of all-cellulose composites. *Carbohydrate Polymers*, 71(3), pp. 458-467

3. MATERIALS AND METHODS

3.1 MATERIALS

In this study, three different materials were used - 100% cotton from discarded denim (CO), blend material with a main content from cotton from discarded denim (BCO) and polycotton fabric from hospital sheets (PCO). The post-consumer denim fabrics were provided by Texaid (Switzerland). The hospital sheets were a plain weave fabric, maintained from Textilia AB, Sweden. The materials, fabric types and areal weight of used materials can be seen in Table 2. An ionic liquid, 1-Butyl-3-methylimidazolium acetate (Sigma-Aldrich) was used to enable the dissolution of the cellulose. Dimethyl sulfoxide (Sigma-Aldrich) was added to decrease the viscosity of the solution of ionic liquid and cellulose. Distilled water was used for regeneration of the samples. The [BMIM][Ac] used for production of ACCs was collected in flasks and recycled.

TABLE 2. MATERIAL, ABBREVIATION, FABRIC TYPE AND AREAL WEIGHT OF THE MATERIALS USED FOR PRODUCTION OF ACCS SAMPLES.

Material	Abbreviation	Fabric type	Areal weight (g/m^2)
100% cotton from denim	CO	Nonwoven	130-160
Blend material denim	BCO	Nonwoven	Approx. 180
Polycotton	PCO	Plain weave	157.5

3.1.1 1-BUTYL-3-METHYLIMIDAZOLIUM ACETATE

The [BMIM][Ac] used in this research has a relative density of 1.055,00 g/cm^3 at 25°C and the melting point is < - 20°C (Sigma Aldrich, 2018). The [BMIM][Ac] was used for dissolution of the cellulosic material in this research.

3.1.2 DIMETHYL SULFOXIDE

The DMSO used in this research has a relative density 1.1 g/cm^3 and a melting point at 18.4°C (Sigma Aldrich, 2019). DMSO is chosen as co-solvent since it decreases the viscosity of ILs (Lv et al. 2012). However, when adding a co-solvent, it can demolish the possibility to recover the [BMIM][Ac].

13: Soykeabkaew, N., Nishino, T. & Peijs, T. (2009). All-cellulose composites of regenerated cellulose fibres by surface selective dissolution. *Composites Part A: Applied Science and Manufacturing*, 40(4), pp. 321-328. doi:https://doi.org/10.1016/j.compositesa.2008.10.021

3.2 METHODS

3.2.1 SAMPLE PREPARATION

Shredding was chosen as method to recycle the discarded denim into fibers again, since shredding enables the possibility to recycle different types of fabrics e.g., damaged jeans. The collected denim was sorted after fiber content, denim with 100% cotton and denim with blend materials. Zippers, buttons, seams and hard parts etc. were removed from the jeans. The denim was cut into smaller pieces, since the cutter had a limit of processing fabric samples with sizes less than 1 m^2. A shredding machine from New Shun Xing Environmental Technology with four drums was used for the shredding of the cut samples. The first drum was an NSX-FS1040 with 8 mm long saw teeth, the following three drums were NSX-QT310 with 4 mm saw teeth. The fiber length measurement was done by picture analysis from optical microscope. The length of the shredded fibers before carding was 7.02 mm and after carding 11.15 mm, where the short fibers got stuck on the fiber drum during carding (Figure 3). The hospital fabric was kept intact and cut into squares of 0.19 x 0.19 m.

3.2.2 CARDING

Carding was done to disentangle the fibers, position the fibers parallel to each other and to produce fiber webs. 60 g of fibers was collected to create each fiber web. A *Mesdan Lab Carding Machine* (Brescia, Italy) was used for the production of fiber webs. CO fibers were first carded, to avoid unwanted fibers in the CO web. BCO fibers were carded afterwards. The fibers were carded once due to difficulties during carding, as mentioned in section 3.2.1. The machine was cleaned between each carding run, to prevent cloth formation on the drum, as seen in Figure 3. The environment during carding was: 53% humidity and room temperature 22°C.

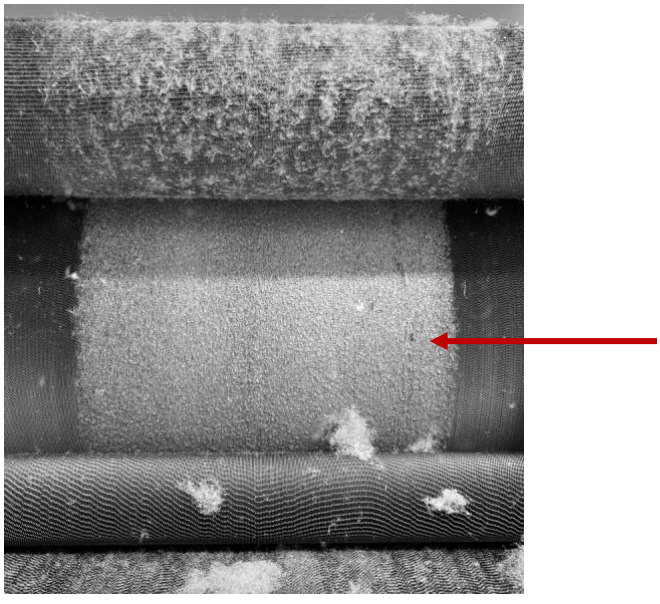

FIGURE 3. RECYCLED DENIM FIBERS STUCK BETWEEN THE NEEDLES ON THE DRUM IN THE CARDING MACHINE.

3.2.3 NEEDLE PUNCHING

Needle punching is a widely used production method of nonwovens. The nonwoven is mechanically created by needles working up and down through the web, which generates entanglements of the fibers and gives a cohesive nonwoven. To produce the nonwoven a needle punching machine was used. The fiber webs were placed in the same fiber direction as from the carding procedure. Each fiber web was needle punched once. The environment during production of the nonwoven was: 53% humidity and room temperature 22°C. The produced nonwoven was cut into squares with the dimension 0.19 x 0.19 m.

3.2.4 MANUFACTURING

Two methods are used for manufacturing of the ACCs, partial dissolution and dissolution of cellulose in a solvent added on a cellulosic reinforcement. In this thesis both methods are tested to compare if there is a difference between the methods. Table 3 shows which samples is produced with which method and solvent and the fiber content of each composite. The fiber content was calculated by Equation 1.

$$V_F = \frac{v_f}{v_c} * 100 \qquad (1)$$

V_f: ratio of fiber wt%
v_f: volume of fibers
v_c: volume of composite

The ACCs produced with partial dissolution had a higher weight as composite compared to dry nonwoven laminates, which indicates that there is still some [BMIM][Ac] or water content in the composite. Therefore, the fiber wt% cannot be calculated.

TABLE 3. SAMPLE, METHOD AND SOLVENT USED FOR PRODUCTION OF THE ACCS. THE FIBER CONTENT OF EACH COMPOSITE IS PRESENTED.

Sample	Method	Solvent/Solution	Fiber content (wt%)
CO1	Partial dissolution	[BMIM][Ac]	-
CO2	Two-step	[BMIM][Ac]+ CO	94
CO3	Two-step	[BMIM][Ac]+ CO + DMSO	87
BCO1	Partial dissolution	[BMIM][Ac]	-
BCO2	Two-step	[BMIM][Ac]+ CO	90
BCO3	Two-step	[BMIM][Ac]+ CO + DMSO	87
PCO	Partial dissolution	[BMIM][Ac]	-
RILCO	Partial dissolution	Recycled [BMIM][Ac]	-

The direction of the fibers in the nonwoven sheets were kept in the same direction as the carding and needle punching, which gives unidirectional composites. The total number of layers was dependent on the total weight of 50 g material. The prepared samples were dried in an oven (Termaks, Bergen, Norway) at 80°C for 24 h before use, to remove moisture content. *A 20 tonne manual bench press* (Rondol Technology Ltd, United Kingdom) was used for pressing of the composites. The nonwoven sheets were placed between two *Kevlar* sheets and then placed between two metal plates, with a metal mold in between with a cut out with the measure 0.19 x 0.19 m.

3.2.4.1 Partial dissolution/one-step method

Table 4 shows the temperature, pressure and time applied for each sample produced by partial dissolution.

TABLE 4. TEMPERATURE, PRESSURE AND TIME FOR EACH SAMPLE WITH PARTIAL DISSOLUTION.

Sample	Temperature [°C]	Pressure [Pa]	Time [min]
CO1	110	0.25, 0.50	60
BCO1	110	0.25	60
PCO	110	0.50, 0.75	30, 30
RILCO	110	0.25	60

The weight of the [BMIM][Ac] was 2.5 times the weight of nonwoven sheets (g). The impregnation was performed by pouring the pure [BMIM][Ac] on each nonwoven sheet, until all nonwoven sheets were impregnated (Figure 4). The same procedure was done with the recycled [BMIM][Ac]. Except the method for recovering of the used [BMIM][Ac], which can be found under section 3.2.3.

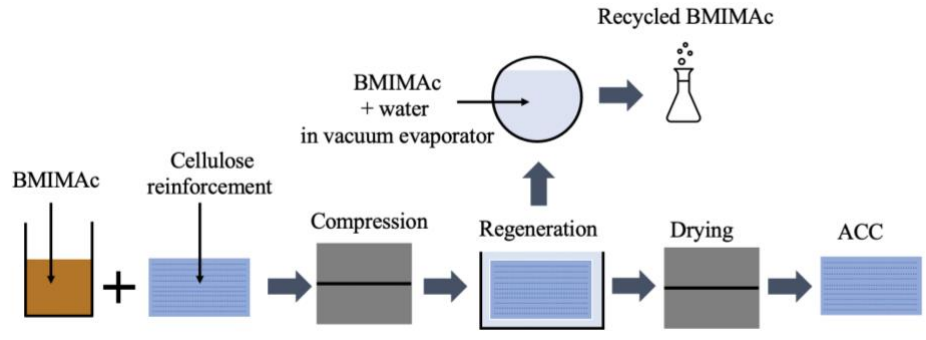

FIGURE 4. ILLUSTRATION OF THE METHOD FOR PARTIAL DISSOLUTION.

3.2.4.2 Two-step method

The material and chemicals used with the two-step method can be seen in Table 3, under section 3.2.4. The weight of the [BMIM][Ac] was 6 times higher than the weight of the laminates. Weight of cellulose for dissolution was 1.7 wt.% of the [BMIM][Ac]. The cellulose was dissolved in the

[BMIM][Ac] during stirring at a temperature of 100°C, for 60 - 90 min or until complete dissolution of the cellulose. The solution was poured and spread over the laminates with the same procedure as for the one-step method (Figure 5). The temperature in the press was 110°C, the pressure was between 0.05 - 0.125 MPa and the time in the press was between 30 - 50 min.

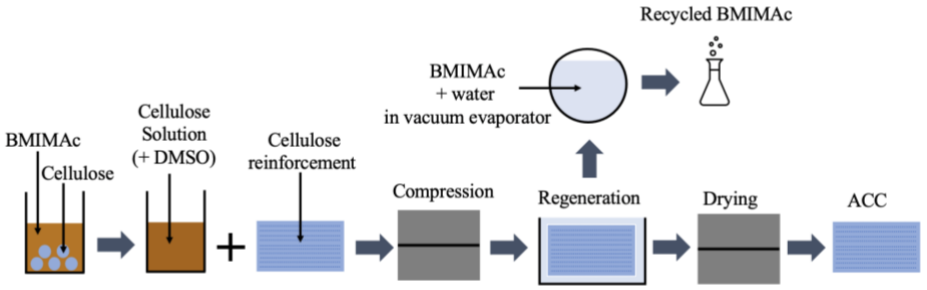

FIGURE 5. ILLUSTRATION OF THE TWO-STEP METHOD.

The materials used for the two-step method with pure [BMIM][Ac] and DMSO was CO and BCO. The total weight of solution was six times the weight of the sheets. The weight of the DMSO was 20% of the total solution. The weight of [BMIM][Ac] was weight of total solution minus weight of DMSO. Weight of cellulose for dissolution was 1.7 wt.% of the [BMIM][Ac]. The mixture of [BMIM][Ac] and DMSO were stirred for 5 min before adding the cellulose. The dissolution of the cellulose was performed at a temperature of 100°C, 60-90 min or until complete dissolution of the cellulose during stirring. The same procedure from pressing from two-step with pure [BMIM][Ac] was used (Figure 5). The temperature, pressure and time applied for each sample is presented in Table 5.

TABLE 5. TEMPERATURE, PRESSURE AND TIME FOR EACH SAMPLE WITH TWO-STEP METHOD.

Sample	Temperature [°C]	Pressure [MPa]	Time [min]
CO2	110	0.125 - 0.375	60
CO3	110	0.050 - 0.125	30
BCO2	110	0.050 - 0.125	30, 50
BCO3	110	0.050 - 0.125	30

3.2.4.3 Equivalent for all samples

All samples were handled equivalent after pressing. The sample was placed in a box with deionized water for regeneration. A weight was placed on each sample. The water was changed frequently each day, until the [BMIM][Ac] content was removed from the composites. The water with [BMIM][Ac] was collected to enable recycling of the [BMIM][Ac]. The samples were dried in the bench press at 110°C, with a pressure of 2.5 MPa (100kN) for 5-6 hours. A weight was then placed on the metal plates to reduce the risk for an

'uneven' composite and to avoid shrinkage, and to enable the cool down of produced composite.

3.2.3 Recycling of [BMIM][Ac]

To enable the recycling of the [BMIM][Ac] a *Heidolph rotary evaporator* (Heidolph Instruments GmbH & CO, Schwabach, Germany) was used. The temperature was 80°C, the globe had a constant rotation of 15 rpm and a pressure of 1 bar. The mixture of [BMIM][Ac] and water was continuous refiled. *A Rotavapor R-114* and a *Waterbath B-480* from *Büchi* (Flawil, Switzerland) and a *Diaphragm vacuum pump MZ 2C* from Vacuubrand were used to remove the last content of water. The temperature range was between 45-80 °C, pressure of approx. 0.20 bar and the RPM was at a constant speed.

3.3 Characterization

The test samples were cut with a Laser cutter *Spirit-402-01* (GCC Innovation, Taiwan) by SVEMA Industriteknik AB in Borås. Fourier transform infrared spectroscopy (FTIR) was done to analyze the hydrogen content in the used [BMIM][Ac]. Tensile, flexural and impact testing were done on the composites to characterize the mechanical properties. Furthermore, pictures from microscopy were taken to analyze at the cross-section of the composites and void content of the composites was measured.

3.3.1 Fourier Transform Infrared Spectroscopy

FTIR can characterize the wanted solution by absorbing or transmitting the infrared light. Depending on in which regions the light is absorbed, different functional groups can be detected. A *Nicolet iS10* from *Thermo Scientific* with a SMART iTX diamond was used to perform FTIR on the recycled [BMIM][Ac]. To collect the data, the software *Omnic* (Thermo Scientific) was used. 64 scans were used in each run and the resolution was 4. The recycled [BMIM][Ac] was compared with pure [BMIM][Ac] by analyzing the O-H groups in the solvent.

3.3.2 Tensile testing

The composite Young's modulus (GPa) and tensile strength (MPa) can be determined by performing tensile test. The tensile tests were performed on a *Tinius Olsen H10KT* (Salfords, UK) machine. A 5 kN cell/force transducer was used. A clamp *Epsilon Company SV E99524, 3542-050M-010*-57 was placed on specimen before each testing. The specimens were extended at a rate of 10 mm per minutes. The dimensions of the dog bone shaped specimens were - width 10 mm, length 150 mm and a thickness of 1.5 – 3 mm depending on the composite. Each sample had six test specimens tested.

3.3.3 Flexural testing

The flexural testing was done by a 3-point bending test on the same machine as the tensile testing. Elongation and corresponding strain and force of the

samples can be detected by flexural testing. The flexural strength (MPa) and flexural modulus (GPa) can then be calculated from the results. A bent rate of 10 mm per minute was applied until the sample broke or till it reached its lowest point. The dimensions on the flat specimens were - length 80 mm, width between 14.4 – 14.7 mm and thickness between 1.5 – 3 mm depending on the composite. Each sample had six test specimens tested.

3.3.4 IMPACT TESTING

Impact test is performed to detect the impact energy required to fracture a composite. The impact testing was completed on a *Cometech testing machine* (Taichung city, Taiwan). The impact pendulum had a capacity of 5 J. The composites were tested edgewise. The results were calculated in the program *Meteorite*. The test specimens had a flat shape with the dimensions - width 10 mm, length 80 mm and thickness 1.5 – 3 mm, depending on the composite type.

3.3.5 MICROSCOPY

Microscopy is used worldwide for different applications where it is wanted to observe a structure or surfaces. It can for example be used to study the cross-section of a composite and furthermore detect voids. A *Nikon SMZ800 stereomicroscope* (Tokyo, Japan) was used with a zoom of 4 times, together with the software *NIS Element* (Nikon, Tokyo, Japan).

3.3.6 DENSITY AND VOID CONTENT

The density of the composite samples was measured by first weighing the sample in air and then weigh it in a 95% ethanol solution (VWR Chemicals) according to Equation 2 below.

$$\rho_t = \frac{W_a}{(W_a - W_e)} * \rho_e \qquad (2)$$

ρ_t: density of composite (g/cm^3)
W_a: weight in air (g)
W_e: weight in ethanol (g)
ρ_e: density of ethanol (0.79 g/cm^3)

The measured density is used to be able to calculate the void content of the composite samples. Equation 3 was used for calculating the void content.

$$V = 1 - \frac{\rho_t}{\rho_c} * 100 \qquad (3)$$

V: void content (%)
ρ_t: density of composite (g/cm^3)
ρ_c: density cotton (rule of mixture) (1.54 g/cm^3)

3.4 STATISTIC ANALYSIS OF THE MECHANICAL PROPERTIES

It is tested if each specific combination of material and method have a statically significant influence on the mechanical properties - tensile, flexural and impact properties. One-way ANOVAs (analysis of variance) with a 95% confidence interval. Table 6 shows the different groups for statistical analyzing. The software *IBM SPSS statistics 26* from IBM was used when conducting the statistical analysis.

TABLE 6. GROUPS FOR STATISTICAL ANALYSIS.

Groups for comparison	Sample
Influence of ionic liquid	CO1 & RILCO
Influence of method	CO1 & CO2
Influence of material	CO1 & BCO1
Influence of material and method	CO2, CO3, BCO2 & BCO3
Influence of DMSO	CO2 - CO3 & BCO2 - BCO3
Influence of cotton and polycotton	CO1 & PCO

To proceed with the statistical method, it is needed to state a null hypothesis (H_0) and an alternate hypothesis (H_a).

H_0: There is no influence of the chosen combination of material and method on the mechanical properties of the ACCs.
H_a: There is an influence of the chosen combination of material and method on the mechanical properties of the ACCs.

To decide whether the null hypothesis can be rejected or not, the significance is needed. Where a significance below 0.05 gives evidence for the alternate hypothesis and the hypothesis can be rejected**.** Then the opposite, a significance level above 0.05 indicates that the hypothesis cannot be rejected.

4. RESULTS AND DISCUSSION

The influence of material type, different solvents and type of method were investigated by doing tensile, flexural and impact testing, void content and observation on the cross-section with microscope was accomplished. Furthermore, the OH-content in the recycled [BMIM][Ac] was detected by FTIR.

4.1 FOURIER TRANSFORM INFRARED SPECTROSCOPY

The result from FTIR was observed in the band position 3200-3650 cm^{-1}, since the O-H groups can be detected here. Figure 6 shows the result from the FTIR on the recycled [BMIM][Ac]. The red line represents pure [BMIM][Ac] (PIL). The purple line (OH_0) in the bottom was taken from the first step of

distillation. This indicates a large content of O-H groups compared to PIL. The green line shows a decrease in OH content after 3 h (OH_3) and the blue line shows a decrease in O-H groups after 5 h (OH_5) in the evaporator. Nevertheless, the solution was still contaminated. The pink line (RIL) shows that the O-H bindings has decreased even more and are closest to PIL. However, the difference between the solvents indicates that small amounts of water was still remained in the RIL sample. Yet, it might be possible to remove even more OH content by extending the time in the evaporator.

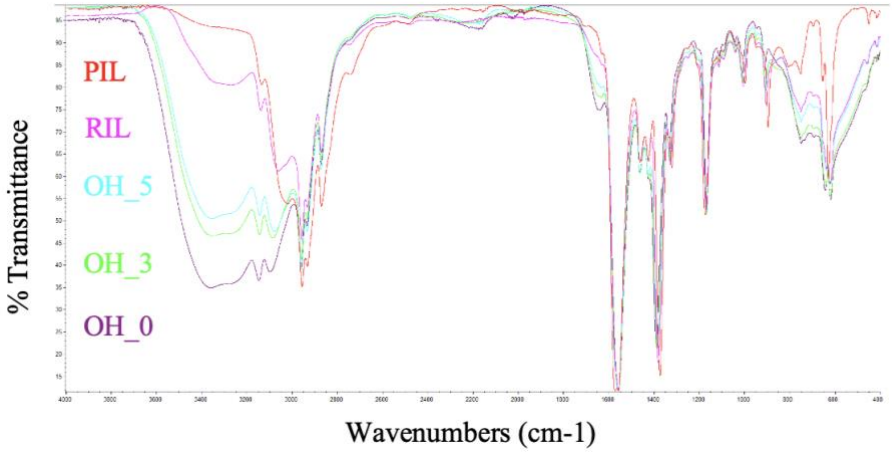

FIGURE 6. VISUAL DECREASE OF OH-CONTENT IN THE [BMIM][AC]-WATER SOLUTION.

4.2 MECHANICAL CHARACTERIZATION

This section presents the results from the mechanical characterization – tensile, flexural and impact properties.

4.2.1 TENSILE PROPERTIES

The results from the tensile testing are presented in this section. Figure 7 shows an overview of the result from measuring the E-modulus. It can be seen that the lowest E-modulus was measured by CO2 (1.27±1.03 GPa) while BCO2 measured the highest (5.75±0.25 GPa).

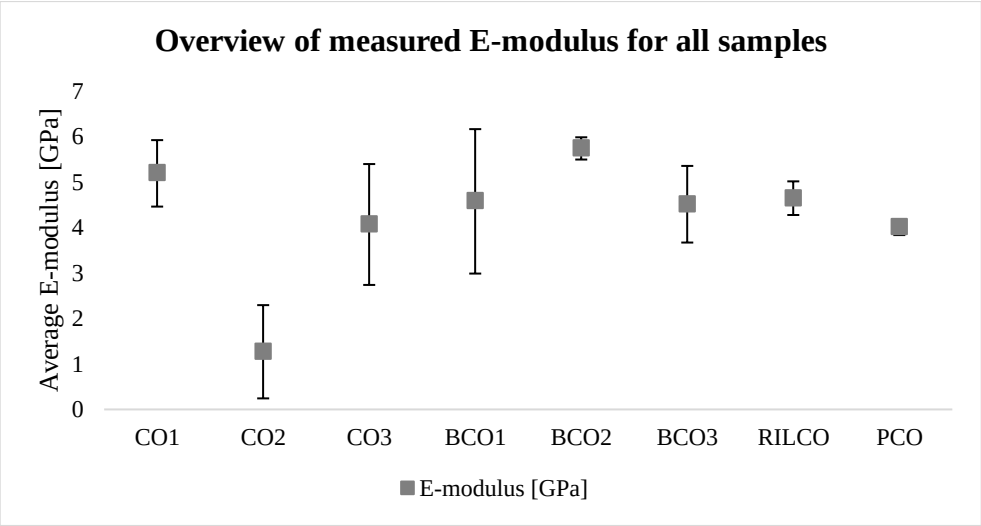

FIGURE 7. OVERVIEW OF MEASURED E-MODULUS FOR ALL SAMPLES.

Figure 8 shows the measured tensile strength, where the highest tensile strength was obtained by PCO (73.3±4.6 MPa) while the lowest was obtained by RILCO (20.1±6.8 MPa).

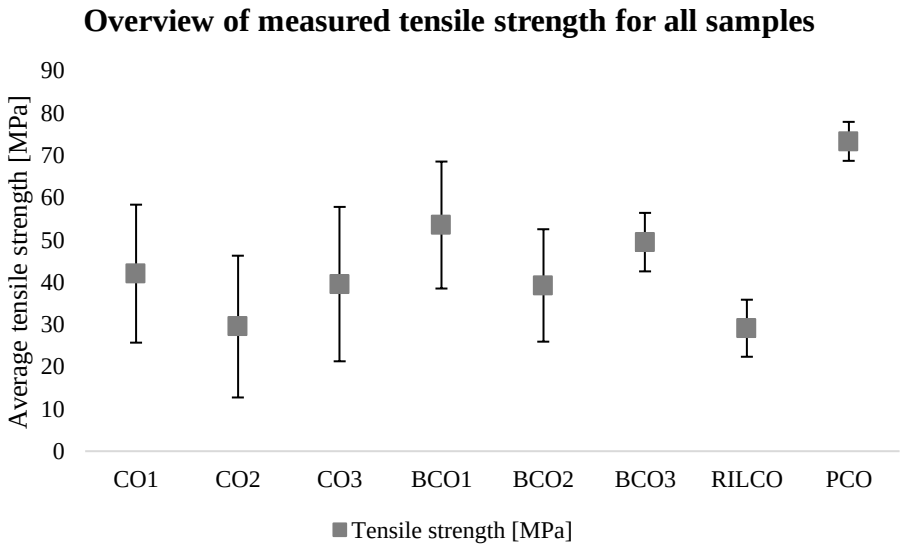

FIGURE 8. THE OBTAINED TENSILE STRENGTH FOR ALL SAMPLES.

4.2.1.1 Influence of ionic liquids on tensile properties

When comparing between CO1 and RILCO, it can be seen that both highest E-modulus (5.95±0.73 GPa vs 4.65±0.37 GPa) and tensile strength (42.0±16.3 MPa vs 20.1±6.8 MPa) was achieved by CO1. When analyzing statistically, the significance (p-value > 0.05) indicates that the choice of pure [BMIM][Ac] or recycled [BMIM][Ac] does not have a significant influence on the tensile strength and the E-modulus of these samples. Since the denim fibers in the nonwoven is the reinforcement, it can influence the tensile properties of the composite (Baghaei et al., 2020). The reinforcement used in CO1 and RILCO has the same structure which can result in the non-influence of [BMIM][Ac] in this case.

4.2.1.2 Influence of method on tensile properties

By comparing ACCs manufactured by partial dissolution (CO1) and two step method (CO2), the statistical analysis indicates no significant difference in tensile strength between manufacturing methods (p-value > 0.05). The E-modulus, however had a significant difference between CO1 and CO2 (p-value < 0.05), where CO1 achieved a higher E-modulus. This could be due to the fact that the fiber skin of the reinforcement is transformed into matrix phase. Which could improve the transverse tensile properties, due to stronger interfacial interaction when using partial dissolution.

4.2.1.3 Influence of material on tensile properties

When comparing CO1 and BCO1, the result was quite even for the E-modulus. The tensile strength was higher for BCO1 (53.5±15.0 MPa vs 42.0±16.3 MPa). The highest E-modulus was obtained by CO1 (5.95±0.73 GPa vs 4.58±1.59 GPa). Nonetheless, the significance (p-value > 0.05) indicates that there is no influence of the material with partial dissolution on the tensile strength nor Young's modulus. Equivalent to the result from comparing [BMIM][Ac], the reinforcement used in CO1 and BCO1 has the same structure which can result in that the choice of material does not influence the tensile properties in this case.

4.2.1.4 Influence of method and material on tensile properties

ACCs manufacture via two-step method were compared with the ones via two-step method with DMSO and also with the different materials – CO2, CO3, BCO2 and BCO3. BCO2 measured the highest E-modulus while CO2 measured the lowest (Figure 9). BCO3 measured the highest tensile strength while CO2 continued to obtain the lowest values (Figure 10). The significance (p-value > 0.05) indicates that the tensile strength was not influenced by the combination of method and material. Opposite to the statistical analysis for the E-modulus, where the significance level (p-value < 0.05) indicates that the combination of material and method could have an influence on the E-modulus. The undissolved blend content in BCO2 and BCO3 could influence the E-modulus, since the blend content contains among others polyester fibers, which can act as reinforcement.

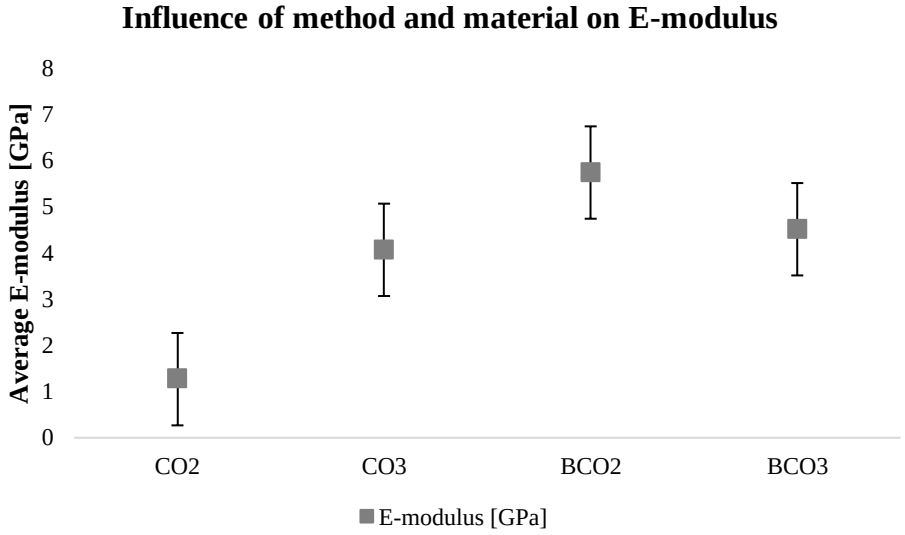

FIGURE 9. MEASURED E-MODULUS FOR COTTON (CO2) AND BLEND COTTON (BCO2) PRODUCED WITH TWO-STEP METHOD AND COTTON (CO3) AND BLEND COTTON (BCO3) PRODUCED WITH TWO-STEP METHOD WITH DMSO.

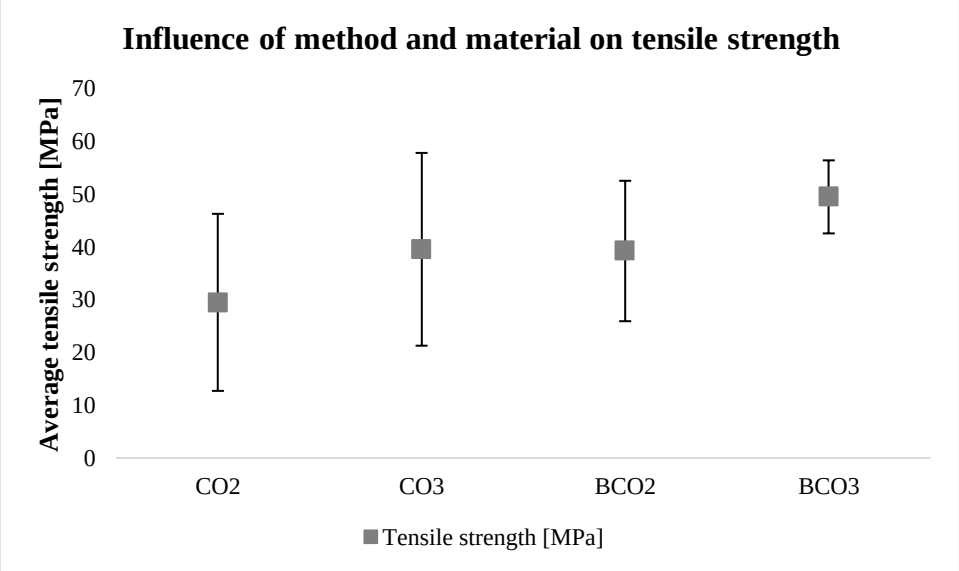

FIGURE 10. MEASURED TENSILE STRENGTH FOR COTTON (CO2) AND BLEND COTTON (BCO2) PRODUCED WITH TWO-STEP METHOD AND COTTON (CO3) AND BLEND COTTON (BCO3) PRODUCED WITH TWO-STEP METHOD WITH DMSO.

4.2.1.5 Influence of DMSO on tensile properties

When comparing if there was an influence of addition of DMSO with two-step method, it can by statistical analysis be validated that the Young's modulus was influenced by DMSO (p-value > 0.05) between both CO2 - CO3 and BCO2 - BCO3. DMSO is added to reduce the monomer friction in cellulose which decreases the viscosity (Lv et al., 2012) in the [BMIM][Ac] + CO solution. The reduction of viscosity appears to have an influence on the Young's modulus on produced ACCs. It could be due to improved bonding at the interface (Huber et al., 2013). The tensile strength was however not influenced by the addition of DMSO (p-value > 0.05) neither for CO2 - CO3,

and BCO2 - BCO3. Furthermore, the statistical analysis indicates that there was no influence of the choice of material with DMSO - CO3 and BCO3, for both E-modulus and tensile strength (p-value > 0.05). Hence, it could be the role of the reinforcement that contributes to the tensile properties.

4.2.1.6 Influence of cotton and polyestercotton on tensile properties

PCO and CO1 were compared to each other. The results show that a higher tensile strength was achieved by PCO and opposite, a higher E-modulus was obtained by CO1. The significance (p-value < 0.05) indicates that the choice of material - cotton or polyester cotton, had an influence on both the Young's modulus and the tensile strength. The PCO sample contains undissolved polyester and continuous filaments in the woven structure, which act as reinforcement and could contribute to the strength and modulus of the PCO sample. Polyester has a higher strength and elasticity compared to cotton (Hatch, 1993).

4.2.2 Flexural properties

Figure 11 shows the measured flexural modulus and Figure 12 shows the maintained flexural strength. The CO2 sample could not be tested for its flexural properties and therefore the sample was removed from this section. The RILCO sample measured the highest flexural modulus, while CO3 measured the lowest (Figure 11). Highest flexural strength was obtained by BCO1 and the lowest by RILCO (Figure 12). Strong interfacial adhesion in ACCs can contribute to superior flexural properties (Baghaei & Skrifvars 2020).

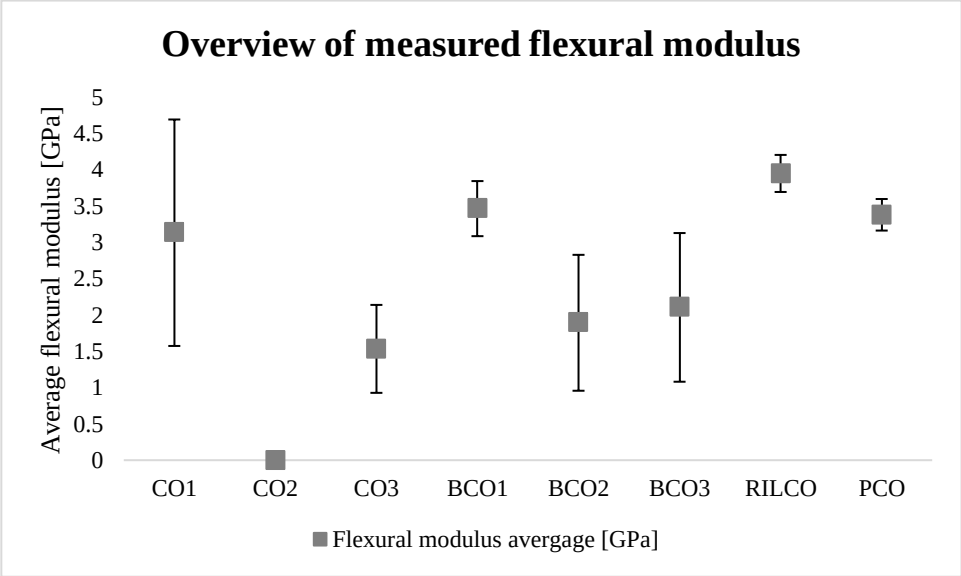

Figure 11. Overview of maintained flexural modulus for all samples.

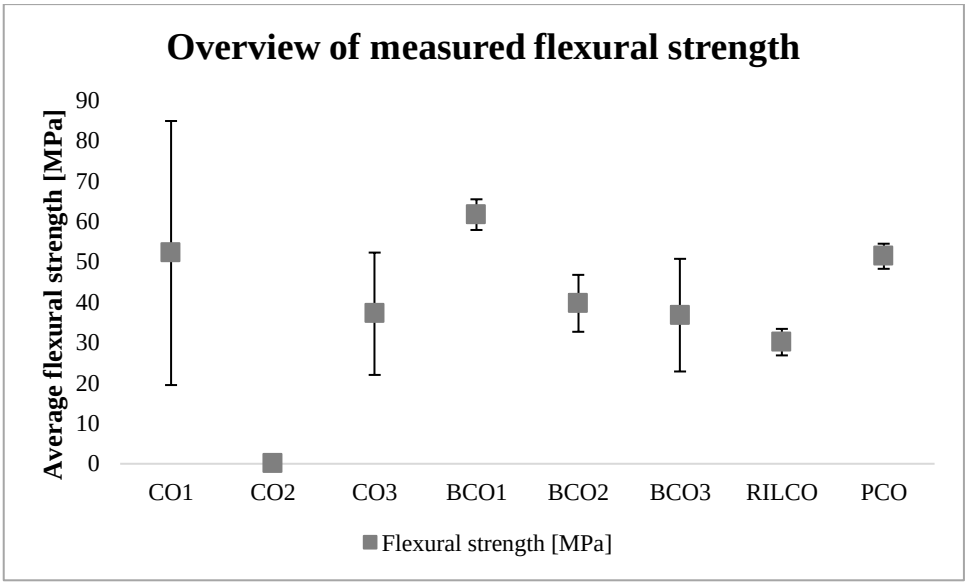

FIGURE 12. OVERVIEW OF MEASURED FLEXURAL STRENGTH FOR ALL SAMPLES.

4.2.2.1 Influence of ionic liquids on flexural properties

When studying if the choice of ionic liquid had influence on the flexural modulus and strength, the statistical analysis confirms that there was no significant difference when using pure [BMIM][Ac] or recycled [BMIM][Ac] (p-value > 0.05). Consequently, the construction of the nonwoven was the same in both samples and it could be that the reinforcement influences the flexural properties.

4.2.2.2 Influence of method on flexural properties

By statistical analysis it could be concluded that the choice of partial dissolution and two-step method with blend cotton had an influence (p-value < 0.05) on both the flexural modulus and flexural strength. It appears like the dissolution with two-step method (BCO2) weakens the reinforcement which results in a decrease in the flexural modulus and strength. Comparing to partial dissolution (BCO1), where the dissolved surface of the fibers contributes to a higher strength and modulus. This since BCO1 achieved higher flexural properties compared to BCO2.

4.2.2.3 Influence of material on flexural properties

Partial dissolution with different materials – CO1 and BCO1, were compared to each other. CO1 measured a lower flexural modulus and strength compared to BCO1. The significance (p-value > 0.05) however indicates that the choice of material in this case did neither influence the flexural modulus nor the strength. The result can depend on that the same construction of nonwoven was used.

4.2.2.4 Influence of method and material on flexural properties

The highest flexural modulus was measured by BCO3 while CO3 had the lowest. Whereas BCO3 measured the lowest flexural strength and BCO2

measured the highest flexural strength. By comparing ACCs manufactured by two-step method with and without DMSO and the two different materials, CO and BCO, the statistical analysis indicates that there was no significance difference (p-value > 0.05) in neither flexural modulus nor strength.

4.2.2.5 Influence of DMSO on flexural properties

The influence of DMSO on the flexural properties for the blend cotton samples was studied. The statistical analysis confirms that there was no influence of DMSO on the flexural properties when comparing BCO2 and BCO3 to each other (p-value > 0.05). Meaning that the addition of DMSO does not influence the flexural properties.

4.2.2.6 Influence of cotton and polyestercotton on flexural properties

The flexural modulus and flexural strength for CO1 and PCO achieved similar results. Where the PCO sample measured a slightly higher flexural modulus and the CO1 sample measured a higher flexural strength. Nevertheless, the significance (p-value > 0.05) for both properties indicates that the material does not influence the flexural properties in this case. The material composition and type of reinforcement are different between these samples, and yet there was no significance difference in flexural properties between the samples. The result could depend on the strong interfacial adhesion formed in the ACCs.

4.2.3 IMPACT PROPERTIES

An overview of the results from the impact testing can be seen in Figure 13. The highest energy absorption was obtained by BCO1 while CO3 obtained the lowest. Further statistical analysis between the samples were conducted, as previous sections. The continuous matrix of cellulose can contribute to good impact properties (Baghaei & Skrifvars 2020).

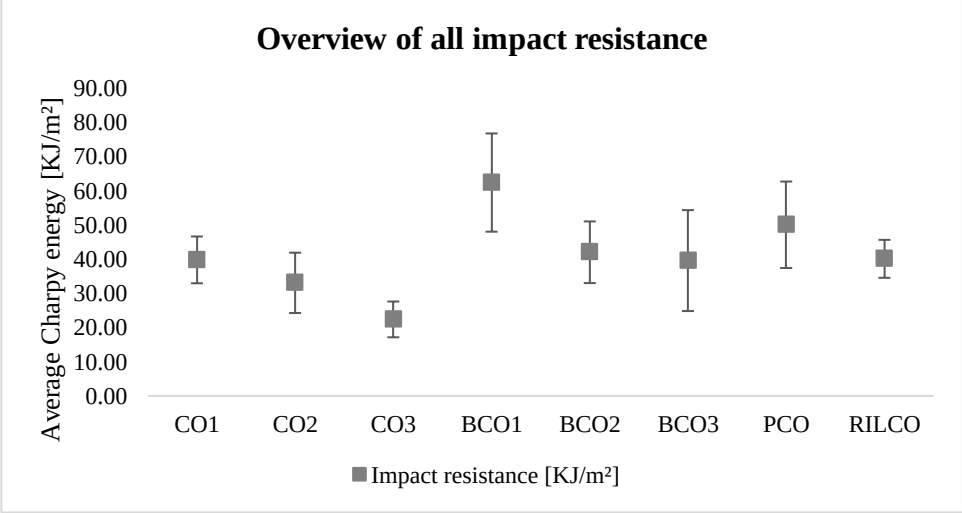

FIGURE 13. THE MEASURED IMPACT RESISTANCE FOR THE DIFFERENT COMPOSITES.

4.2.3.1 Influence of ionic liquids on impact resistance

When studying the influence of ionic liquid between cotton with pure [BMIM][Ac] and cotton with recycled [BMIM][Ac] it was found that both composites had similar energy absorption. Where CO1 had an energy absorption of 39.79±6.85 kJ/m^2 and RILCO has 40.09±5.55 kJ/m^2. Statistical analysis shows no significant difference between the composites (p-value > 0.05). This means that the choice of ionic liquid - pure [BMIM][Ac] or recycled [BMIM][Ac], does not influence the impact resistance.

4.2.3.2 Influence of method on impact resistance

Cotton with partial dissolution achieved a higher energy absorption (39.79±6.85 kJ/m^2) compared to measured energy absorption for cotton by two-step method (33.07±8.82 kJ/m^2). Statistical analysis indicates no significant difference in impact resistance between manufacturing methods (p-value > 0.05), which indicates that there was no influence of the chosen methods on the impact resistance.

4.2.3.3 Influence of material on impact resistance

The influence of material on impact resistance was studied, where partial dissolution with pure [BMIM][Ac] was used with cotton and blend cotton. BCO1 achieved highest energy absorption (62.41±14.37 kJ/m^2) compared to CO1 (39.79±6.85 kJ/m^2). Statistical analysis confirms a significant difference in impact resistance between CO1 and BCO1 (p-value < 0.05) which means that the choice of cotton and blend cotton have influence on the impact resistance.

4.2.3.4 Influence of method and material on impact resistance

The highest energy absorption was maintained by BCO2 (42.02±9.02 kJ/m^2) while CO3 manintained the lowest (22.37±5.24 kJ/m^2). Statistical analysis verifies that there was no significance different between material and method (p-value > 0.05). The result indicates that the material and method in this case does not influence the impact resistance.

4.2.3.5 Influence of DMSO on impact resistance

The influence of DMSO on the impact resistance was studied for both CO and BCO. The statistical analysis was performed between CO2 - CO3, BCO2 - BCO3 and CO3 - BCO3. The result from the analysis showed no influence of the DMSO on the impact resistance for neither one of the comparisons (p-value > 0.05).

4.2.3.5 Influence of cotton and polyestercotton on impact resistance

The impact resistance for CO1 was 39.79±6.85 kJ/m^2 and 50.07±12.65 kJ/m^2 for PCO, where PCO achieved the highest impact resistance. However, the

significance (p-value > 0.05) indicates that the different materials, cotton and polyestercotton, does not influence the impact resistance.

4.2.4 Summary and discussion of mechanical properties

The research by Baghaei et al. (2020) using sheets of denim waste to produce ACCs resulted in a given Young's modulus of 2.5 to 4.3 GPa, flexural modulus 1.6 to 2.1 GPa, flexural strength 25 to 20 MPa and Charpy energy 31 to 37 kJ/m^2. Compared to this research using denim waste in the form of nonwoven it can be concluded that a higher Youngs modulus, flexural strength and modulus were achieved in this research. The Charpy energy measured by Baghaei et al. resulted in a smaller interval between lowest and highest Charpy energy compared to the result from this research, where there was a higher interval between the lowest and highest measured Charpy energy. The difference in the results can depend on the different constructions of reinforcement, where Baghaei et al. used denim twill weave and a different matrix phase compared to this research where denim fibers in the form of nonwoven was tested. Since the reinforcement influences the mechanical properties it contributes to the difference in the results.

When comparing to mechanical properties of other ACCs found in literature it can be seen that similar results have been achieved (Figure 14). Gindl-Altmutter et al. (2012) produced ACCs by partial dissolution with flax and lyocell in the form of nonwoven. Where the flax ACC performed a Youngs modulus of 4.6 GPa. and tensile strength of 34 MPa, and the lyocell ACC performed a Youngs modulus of 7.2 GPa and tensile strength of 78 MPa. The researchers could conclude that the ACC produced from flax had a decrease in mechanical properties compared to a composite produced with flax and epoxy matrix. But the ACC produced with lyocell achieved similar strength compared to the composite with epoxy-matrix and lyocell. The researchers mean that the ionic liquid changes the structure of the cellulosic fibers in this case the flax fibers, to a degree where it weakens the mechanical properties of the flax ACC. (Gindl-Altmutter et al., 2012) It could be interesting to try the nonwoven laminated from discarded textiles with a bio-based resin to compare the mechanical properties.

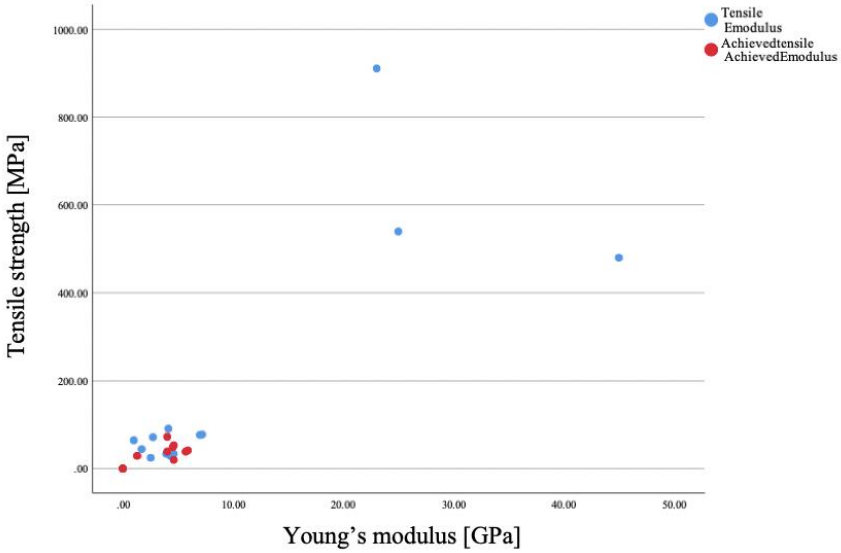

FIGURE 14. TENSILE PROPERTIES OF ACCS FROM LITERATURE PRESENTED IN TABLE 1 (BLUE DOTS) AND MEASURED TENSILE PROPERTIES FROM THIS STUDY (RED DOTS).

Huber et al., (2013) produced ACCs by using rayon twill and [BMIM][Ac] by partial dissolution. The achieved properties were - flexural modulus 3.73 GPa, flexural modulus 135.24 MPa and Charpy Energy 41.45 kJ/m^2. Compared to this research where highest flexural modulus was measured with RILCO 3.94 GPa and highest flexural strength was measured with BCO1 61.71 MPa and highest Charpy Energy was measured by BCO1 62.41 kJ/m^2. The results show similar flexural modulus, except Huber et al., measured a higher flexural strength. The result can depend on different matrix phases and material used in the ACCs. As mentioned earlier, where the construction of the reinforcement has influence on the mechanical properties. A higher impact resistance was measured in this research compared to Huber. It should be stated that different production methods and materials were used, which can influence the outcome in mechanical properties.

4.3 MICROSCOPIC ANALYSIS

In Figure 15, it was possible to visually realize the difference between the different materials and production methods - partial dissolution, two-step method and two-step method with DMSO. It was possible to imply that the BCO1 sample had a more dissolved cross-section compared to the CO1 and BCO2 samples. Furthermore, it was difficult to differentiate between the fibers and layers in the cross-section of the CO3 and BCO3 samples compared to the other samples. In the CO1 and BCO1 samples it was possible to detect voids, showed with the white circles.

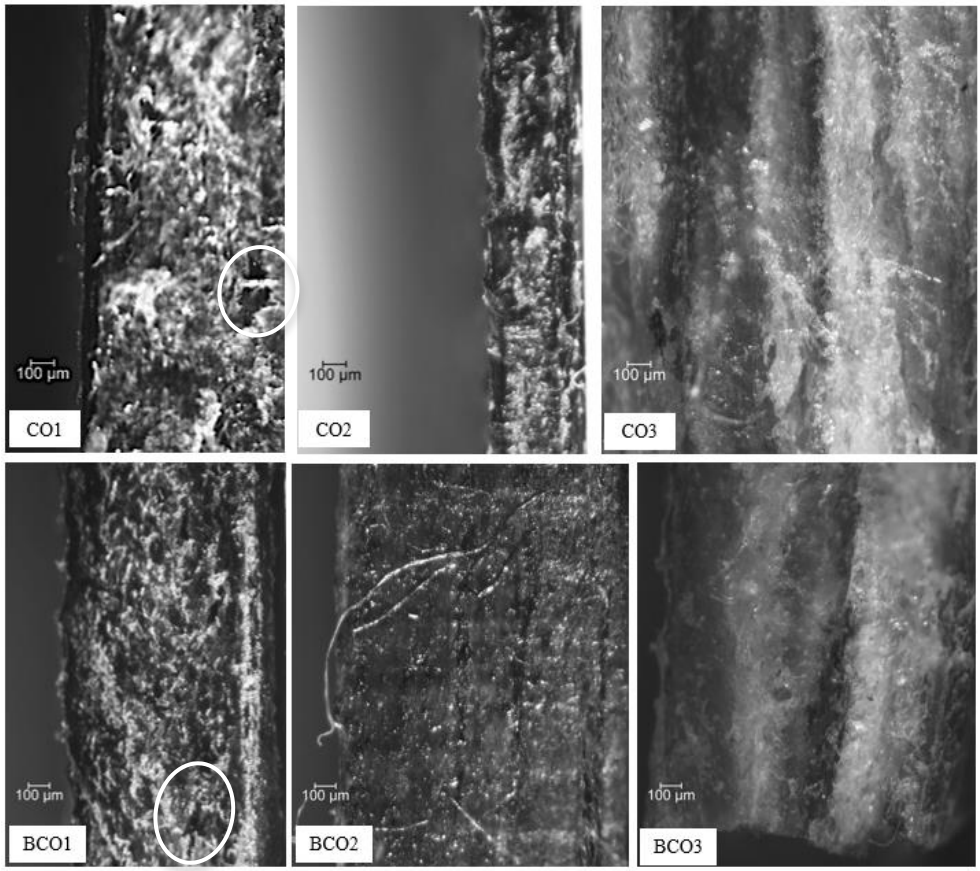

FIGURE 15. MICROSCOPIC COMPARISON (X4) BETWEEN COTTON WITH PARTIAL DISSOLUTION (CO1); TWO-STEP METHOD (CO2); TWO-STEP METHOD WITH DIMETHYL SULFOXIDE (CO3). AND BLEND COTTON WITH PARTIAL DISSOLUTION (BCO1); TWO-STEP METHOD (BCO2) AND TWO-STEP METHOD WITH DIMETHYL SULFOXIDE (BCO3).

Figure 16 shows the samples produced with partial dissolution - CO1, RILCO and PCO. The cross-section of the CO1 sample shows a uniform surface where fiber ends sticking up. In the RILCO sample it was possible to distinguish layers of nonwoven, seen in the white circle. Although, it can be seen that some dissolution has occurred. Even though the lack of focus in the image of the PCO sample, it was possible to detect parts of dissolved cellulose and the different layers of woven fabrics in the sample. The result can depend on the polyester content in the reinforcement, since it cannot be dissolved by the [BMIM][Ac].

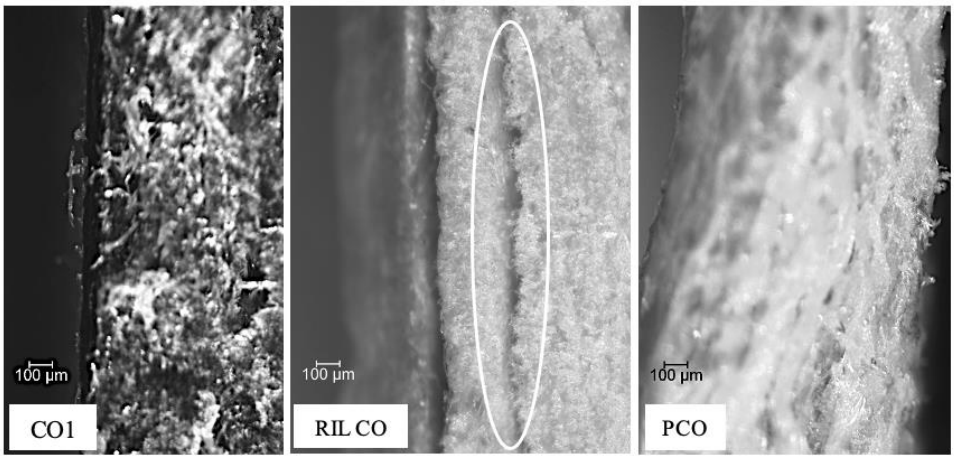

FIGURE 16. MICROSCOPIC COMPARISON (X4) BETWEEN SAMPLES DONE BY PARTIAL DISSOLUTION WITH PURE [BMIM][AC] AND COTTON (CO1); RECYCLED [BMIM][AC] AND COTTON (RILCO) AND PURE [BMIM][AC] WITH POLYESTERCOTTON (PCO).

The detected and unobserved voids can depend on uneven dissolution of the material, since the impregnation of [BMIM][Ac] on the nonwoven laminates might be uneven.

4.4 VOID CONTENT

Table 7 below shows the measured density and void content of each sample. Lowest void content was achieved by the samples with only cotton – CO1 and CO2. The composites that contain a mix of materials resulted in a slightly higher void content – BCO1, BCO2 AND BCO3. At last, the PCO sample had the highest void content. There was a statistic significance between the PCO and CO1 sample (p-value < 0.001). This result can depend on the higher amount of polyester in the sample, since the polyester cannot be dissolved. Statistical analysis shows that there was a difference between the composite produced with recycled [BMIM][Ac] and pure [BMIM][Ac] (p-value < 0.05). There was no significance difference (p-value > 0.05) for the samples produced with partial dissolution, two-step method, 100% cotton and blend cotton which indicates that the method and material in this case does not influence the void content.

TABLE 7. OVERVIEW OF DENSITY OF COMPOSITES AND VOID CONTENT

Sample	Density at 25°C [g/cm^3]	Measured density [g/cm^3]	Void content [%]
Ethanol	0.79		
Cellulose	1.54		
CO1		1.43	7.45
CO2		1.43	7.00
CO3		1.40	8.89
BCO1		1.40	9.15

BCO2	1.40	8.79
BCO3	1.41	8.37
PCO	1.20	22.10
RILCO	1.36	11.46

4.5 SUSTAINABILITY ISSUES

As mentioned earlier in section *2.6 Sustainability*, sustainability includes three aspects - social, economic and ecological sustainability. It is necessary to consider all three aspect when discussing sustainability issues.

The thesis can clearly be related to the environmental sustainability, since materials are taken care of and recycled. It can therefor reduce the number of textiles ending up on landfill or incineration, which prevents already produced energy, consumed chemicals and water going to waste and also a decrease in land pollution. Even if the main idea when designing jeans is not to recycle it into composites, it's a way to handle the waste problem. By choosing to shred the material and produce nonwoven out of it, it can enable the possibility to recycle torn fabrics. Where fibers with poor quality can be upcycled. This is an opportunity to operate towards a circular economy (Lerpold & Nordbrand 2017, p.191). The environmental sustainability can also concern possibility to recycle the composite. Composites produced with only one material can be compared to conventional composite using petroleum-based polymers and mixing of different materials. If the composite consists of only one material it could simplify the recycling of the composite (Nishino et al., 2004). The choice of chemical can likewise influence the ecological sustainability. For example, the ionic liquid used in this thesis, which can be recovered and reused. However, it is needed to consider if the ecological cost in carbon footprint[14] when recycling ionic liquid is higher than producing the chemicals from the beginning. Furthermore, it is important to consider if it is necessary to add any other solvents since it can complicate the recycling of the ionic liquid.
Regarding the economic sustainability, it can concern the circular economy. By reusing and recycling produced material it can reduce the costs when producing new materials but also contribute to the long-term growth of economy. Where there is a possibility to achieve economic growth with less impact on the environment.

Concerning social sustainability, the choice of using ionic liquids can be discussed. Ionic liquids are considered to be beneficial to work with compared to other hazardous solvents, since ionic liquids have for example

[14] The carbon footprint is all the emissions of greenhouse gases produced by among others, humans and/or organizations (Gröndahl & Svanström 2011, p.83).

low flammability (Pringle et al. 2017). This can reduce the health risks for the operator working with the solvent.

5. CONCLUSIONS

The research in this thesis aims to investigate if it is possible to use the technique of ACCs as a method to recycle textiles into composites. It has shown to be possible to use discarded textiles in the form of nonwoven sheets to produce composites with both partial dissolution and two-step method with and without DMSO. It was also possible to use plain weave hospital sheets with partial dissolution to produce composites. By evaporating the water content in the [BMIM][Ac] solution, it was possible to recover and recycle the [BMIM][Ac] used to produce the ACCs. The achieved results show the possibility to work towards a circular economy, where no material goes to waste.

The achieved mechanical properties of the produced composites has shown to have similar properties compared to ACCs found in literature. This indicates that the produced composites have comparable properties and possibility to use in appropriate applications. Hence, the results show the opportunity to actually recycle discarded textiles into composites with successful results that can be united with the right applications.

The performed tensile properties of the polycotton sample resulted in the highest tensile strength. The continuous polyester thread in the composite could contribute to this result, since polyester have a higher elasticity and strength compared to cotton. Nevertheless, the choice of partial dissolution with recycled [BMIM][Ac] (RILCO), pure [BMIM][Ac] (CO1) or with blend cotton (BCO1) had no influence on the tensile properties. Instead, the result showed that two-step method, with and without DMSO will influence the Young's modulus but not the tensile strength. There is a difference in flexural properties between partial dissolution and two-step method. Hence, the two-step method weakens the ACC compared to partial dissolution. For the other combinations of ionic liquid, material or method there was no influence on the flexural properties. Regarding the impact properties of the samples the only factor likely to influence the impact energy was partial dissolution with cotton (CO1) and blend cotton (BCO1). Between the other samples there was no significance difference between material and method. The reinforcement is an important factor for the mechanical properties which has been shown in this research.

A higher void content was found in the polycotton sample, this can depend on the polyester content in the sample and the different fabric constructions. The significance difference in void content between the samples with

recycled [BMIM][Ac] and pure [BMIM][Ac] could indicate lack of dissolution in the RILCO sample. Whereas the samples consisting of cotton and blend cotton showed a lower void content and no significance difference between the different materials and methods. The microscopic analysis only displays parts of the cross-section of each composite, where a few voids could be detected.

While the society continues to buy and throw their textiles, research is ongoing to find solutions where to use the discarded textiles. This to avoid unnecessary disposal of already produced chemicals, water, energy and material etc. This research has shown the possibility to use discarded textiles to produce durable composites.

6. FUTURE WORK

To continue with the research, it would be necessary to study the structural composition of the produced ACCs, this by using Scanning Electron Microscopy (SEM). By using SEM, it can be possible to study e.g., the fracture surface from mechanical testing, hence it can be possible to study the interfacial bonding (Baghaei et al., 2020). Future research should perform crystallinity measurement on the produced ACCs. Since it is wanted to determine the crystallinity and crystalline structure of the cellulose in the produced ACCs (Baghaei et al. 2020).

The length of the shredded fibers could not be selected in this thesis, which lead to very short length of the fibers. It would therefore be interesting to study if longer fibers from the shredded textiles, in nonwovens, affects the properties of the ACCs. Longer fibers can contribute to better entanglements in the nonwoven and then make is possible to avoid a brittle nonwoven, which could improve the mechanical properties.

7. REFERENCES

Albertsson, A.-C., Edlund, U. & Odelius, K. (2012). Naturliga polymerer. *Polymerteknologi, Makromolekylär design.* Stockholm: Albertsson, A-C, pp. 100-101.

Amde, M., Liu, J. & Pang, L. (2015). Environmental Application, Fate, Effects, and Concerns of Ionic Liquids: A Review. *Environmental Science & Technology,* 49(21), pp. 12611-12627. doi:10.1021/acs.est.5b03123

Baghaei, B., Compiet, S. & Skrifvars, M. (2020). Mechanical properties of all-cellulose composites from end-of-life textiles. *Journal of polymer research,* 27(9). doi:10.1007/s10965-020-02214-1

Baghaei, B. & Skrifvars, M. (2020). All-Cellulose Composites: A Review of Recent Studies on Structure, Properties and Applications. In.: Högskolan i Borås, Akademin för textil, teknik och ekonomi.

Cai, J. & Zhang, L. (2005). Rapid Dissolution of Cellulose in LiOH/Urea and NaOH/Urea Aqueous Solutions. *Macromolecular Bioscience,* 5(6), pp. 539-548. doi:10.1002/mabi.200400222

Caulfield, D. F., Feng, D., Prabawa, S., Young, R. A. & Sanadi, A. R. (1999). Interphase effects on the mechanical and physical aspects of natural fiber composites. *Angewandte Makromolekulare Chemie,* 272(1), pp. 57-64. doi:10.1002/(SICI)1522-9505(19991201)272:1<57::AID-APMC57>3.0.CO;2-W

Fan, Z., Chen, J., Guo, W., Ma, F., Sun, S. & Zhou, Q. (2017). Crystallinity of regenerated cellulose from [Bmim]Cl dependent on the hydrogen bond acidity/basicity of anti-solvents. *RSC Advances,* 7(65), pp. 41004-41010. doi:10.1039/c7ra08178b

Gröndahl, F. & Svanström, M. (2011). *Hållbar utveckling: en introduktion för ingenjörer och andra problemlösare.* . Stockholm: Liber.

Hatch, K. L. (1993). *Textile Science.* Opperman Drive: West Publishing Company.

Ho, M.-p., Wang, H., Lee, J.-H., Ho, C.-k., Lau, K.-t., Leng, J. & Hui, D. (2012). Critical factors on manufacturing processes of natural fibre composites. *Composites Part B: Engineering,* 43(8), pp. 3549-3562. doi:https://doi.org/10.1016/j.compositesb.2011.10.001

Huber, T., Bickerton, S., Müssig, J., Pang, S. & Staiger, M. P. (2012a). Solvent infusion processing of all-cellulose composite materials. *Carbohydrate Polymers,* 90(1), pp. 730-733. doi:https://doi.org/10.1016/j.carbpol.2012.05.047

Huber, T., Bickerton, S., Müssig, J., Pang, S. & Staiger, M. P. (2013). Flexural and impact properties of all-cellulose composite laminates. *Composites Science and Technology,* 88, pp. 92-98. doi:10.1016/j.compscitech.2013.08.040

Huber, T., Müssig, J., Curnow, O., Pang, S., Bickerton, S. & Staiger, M. (2012b). A critical review of all-cellulose composites. *Journal of Materials Science,* 47(3), pp. 1171-1186. doi:10.1007/s10853-011-5774-3

Leonas, K. K. (2017). The Use of Recycled Fibers in Fashion and Home Products. In Muthu, S. S. (ed.) *Textiles and Clothing Sustainability: Recycled and Upcycled Textiles and Fashion.* Singapore: Springer Singapore, pp. 55-77. doi:10.1007/978-981-10-2146-6_2

Lerpold, L. & Nordbrand, S. (2017). Kap 7 Hållbara värdekedjor. In Borglund, T., De Geer, H. & Sweet, S. (eds.) *CSR och hållbart företagande.* Stockholm: Sanoma utbildning, p. 191.

Luiken, A. & Bouwhuis, G. (2015). Recovery and recycling of denim waste. *Denim: Manufacture, Finishing and Applications.* pp. 528-540. doi:10.1016/B978-0-85709-843-6.00018-4

Lv, Y., Wu, J., Zhang, J., Niu, Y., Liu, C.-Y., He, J. & Zhang, J. (2012). Rheological properties of cellulose/ionic liquid/dimethylsulfoxide (DMSO) solutions. *Polymer (Guilford),* 53(12), pp. 2524-2531. doi:10.1016/j.polymer.2012.03.037

Mai, N. L., Ahn, K. & Koo, Y.-M. (2014). Methods for recovery of ionic liquids—A review. *Process Biochemistry,* 49(5), pp. 872-881. doi:https://doi.org/10.1016/j.procbio.2014.01.016

Mat Salleh, M., Magniez, K., Pang, S., Dormanns, J. W. & Staiger, M. P. (2017). Parametric optimization of the processing of all-cellulose composite laminae. *Advanced Manufacturing: Polymer & Composites Science,* 3(2), pp. 73-79. doi:10.1080/20550340.2017.1324351

McLoughlin, J., Haye, S. & Paul, R. (2015). Cotton fibre for denim manufacture. *Denim: Manufacture, Finishing and Applications.* Elsevier Ltd, pp. 15-36. doi:10.1016/B978-0-85709-843-6.00002-0

Meksi, N. & Moussa, A. (2017). A review of progress in the ecological application of ionic liquids in textile processes. *Journal of Cleaner Production,* 161, pp. 105-126. doi:https://doi.org/10.1016/j.jclepro.2017.05.066

Meng, X. et al. (2019). Recycling of denim fabric wastes into high-performance composites using the needle-punching nonwoven fabrication route. *Textile Research Journal*. doi:10.1177/0040517519870317

Nationalencyklopedin (n.y.a). Artefakt. http://www.ne.se.lib.costello.pub.hb.se/uppslagsverk/encyklopedi/lång/artefakt [2020-11-30]

Nationalencyklopedin (n.y.b). Denim. http://www.ne.se/uppslagsverk/encyklopedi/lång/denim [2020-10-07]

Nishino, T., Matsuda, I. & Hirao, K. (2004). All-Cellulose Composite. *Macromolecules,* 37(20), pp. 7683-7687. doi:10.1021/ma049300h

Periyasamy, A. P. & Militky, J. (2017). 10 - Denim and consumers' phase of life cycle. In Muthu, S. S. (ed.) *Sustainability in Denim.* Woodhead Publishing, pp. 257-282. doi:https://doi.org/10.1016/B978-0-08-102043-2.00010-1

Ponnusamy, S. K. & p.r, Y. (2018). Recycled Fibres. pp. 1-17. doi:10.1007/978-981-10-8515-4_1

Pringle, J. M., Annat, G., Kar, M. & MacFarlane, D. R. (2017). *Fundamentals of Ionic Liquids.* Newark, GERMANY: John Wiley & Sons, Incorporated.

Qin, C., Soykeabkaew, N., Xiuyuan, N. & Peijs, T. (2008). The effect of fibre volume fraction and mercerization on the properties of all-cellulose composites. *Carbohydrate Polymers,* 71(3), pp. 458-467. doi:https://doi.org/10.1016/j.carbpol.2007.06.019

Radhakrishnan, S. (2017). Denim Recycling. pp. 79-125. doi:10.1007/978-981-10-2146-6_3

Rebière, J., Heuls, M., Castignolles, P., Gaborieau, M., Rouilly, A., Violleau, F. & Durrieu, V. (2016). Structural modifications of cellulose samples after dissolution into various solvent systems. *Analytical and Bioanalytical Chemistry,* 408(29), pp. 8403-8414. doi:10.1007/s00216-016-9958-1

Rieland, J. M. & Love, B. J. (2020). Ionic liquids: A milestone on the pathway to greener recycling of cellulose from biomass. *Resources, Conservation and Recycling,* 155, p. 104678. doi:https://doi.org/10.1016/j.resconrec.2019.104678

Sandin, G. & Peters, G. M. (2018). Environmental impact of textile reuse and recycling – A review. *Journal of cleaner production,* 184, pp. 353-365. doi:10.1016/j.jclepro.2018.02.266

Scheiner, S. (1997). *Hydrogen bonding a theoretical perspective.* New York: Oxford University Press.

Schmidt, A., Watson, D., Roos, S., Askham, C. & Poulsen, P. B. (2016). *Gaining benefits from discarded textiles : LCA of different treatment pathways.* Copenhagen: Nordisk Ministerråd.

Schrott, W. & Paul, R. (2015). *20 - Environmental impacts of denim manufacture.*

Shirvanimoghaddam, K., Motamed, B., Ramakrishna, S. & Naebe, M. (2020). Death by waste: Fashion and textile circular economy case. *The Science of the total environment,* 718, pp. 137317-137317. doi:10.1016/j.scitotenv.2020.137317

Singh, P., Duarte, H., Alves, L., Antunes, F., Moigne, N., Dormanns, J., Duchemin, B., Staiger, M. & Medronho, B. (2015). From Cellulose Dissolution and Regeneration to Added Value Applications —

Synergism Between Molecular Understanding and Material Development. doi:10.5772/59889

Soykeabkaew, N., Nishino, T. & Peijs, T. (2009). All-cellulose composites of regenerated cellulose fibres by surface selective dissolution. *Composites Part A: Applied Science and Manufacturing*, 40(4), pp. 321-328. doi:https://doi.org/10.1016/j.compositesa.2008.10.021

Spörl, J. M., Batti, F., Vocht, M. P., Raab, R., Müller, A., Hermanutz, F. & Buchmeiser, M. R. (2018). Ionic Liquid Approach Toward Manufacture and Full Recycling of All-Cellulose Composites. *Macromolecular Materials and Engineering*, 303(1), pp. n/a-n/a. doi:10.1002/mame.201700335

Sustainability policies and commitments – Lifecycle assessment study. (2015). Levi Strauss & Co. https://www.levistrauss.com/wp-content/uploads/2015/03/Full-LCA-Results-Deck-FINAL.pdf [2020-08-30]

Sweet, S. (2017). Kap 3 Hållbarhet och företagande. In Borglund, T., De Geer, H. & Sweet, S. (eds.) *CSR och hållbart företagande.* Stockholm: Sanoma utbildning, p. 76.

Tan, X., Chen, L., Li, X. & Xie, F. (2019). Effect of anti-solvents on the characteristics of regenerated cellulose from 1-ethyl-3-methylimidazolium acetate ionic liquid. *International Journal of Biological Macromolecules*, 124, pp. 314-320. doi:10.1016/j.ijbiomac.2018.11.138

Temmink, R., Baghaei, B. & Skrifvars, M. (2018). Development of biocomposites from denim waste and thermoset bio-resins for structural applications. *Composites Part A: Applied Science and Manufacturing*, 106, pp. 59-69. doi:10.1016/j.compositesa.2017.12.011

Tervahartiala, T., Hildebrandt, N. C., Piltonen, P., Schabel, S. & Valkama, J. P. (2018). Potential of all-cellulose composites in corrugated board applications: Comparison of chemical pulp raw materials. *Packaging Technology and Science*, 31(4), pp. 173-183. doi:10.1002/pts.2365

Thomas, S., Joseph, K., Malhotra, S. K., Goda, K. & Sreekala, M. S. (2013). *Polymer Composites, Biocomposites.* Oxford, GERMANY: John Wiley & Sons, Incorporated.

UN (2015). *Transforming our world: the 2030 Agenda for Sustainable Development*. https://sustainabledevelopment.un.org/post2015/transformingourworld

Wei, X., Wei, W., Cui, Y.-H., Lu, T.-J., Jiang, M., Zhou, Z.-W. & Wang, Y. (2016). All-cellulose composites with ultra-high mechanical properties prepared through using straw cellulose fiber. *RSC Advances*, 6(96), pp. 93428-93435. doi:10.1039/c6ra20533j

Zhu, Y., Romain, C. & Williams, C. K. (2016). Sustainable polymers from renewable resources. *Nature*, 540(7633), pp. 354-362. doi:http://dx.doi.org/10.1038/nature21001

Östlund, Å., Wedin, H., Bolin, L., Berlin, J., Jönsson, C., Posner, S., Smuk, L., Eriksson, M. & Sandin, G. (2015). *Textilåtervinning: tekniska möjligheter och utmaningar*.