

NIRS IDENTIFICATION OF BLACK TEXTILES

Improvements for waste textiles sorting

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Abstract

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Title of publication NIRS IDENTIFICATION OF BLACK TEXTILES Improvements for waste textiles sorting		
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Abstract <p>Near infrared spectrometry (NIRS) can be harnessed to identify organic compounds by building libraries of reference spectra with validated samples.</p> <p>In Lahti University of Applied Sciences (LAMK), NIRS is utilized in a pilot-sized textile sorting line to identify the composition of samples. Comparing sample spectra to spectral libraries, the sample material may be identified. The libraries currently cover homogeneous wool, polyester, cotton and viscose, with more under construction.</p> <p>NIRS generally ignores colorants, but black textiles have proven to occasionally share spectral anomalies that make their material identification impossible with the current algorithm.</p> <p>The purpose of this thesis was to find improvements for the current pilot-sized textile sorting line of LAMK. The focus was on black textiles and how to potentially improve sorting capabilities of such materials.</p> <p>A test run with a limited supply of black textiles was set up. It seems that with the use of NIRS, it would be possible to improve the detection of black samples in the sorting line. However, further testing with more verifiable samples is required to increase identification accuracy. The result of the test is an identification method that due to its inaccuracy claims small percentile blends to be pure materials.</p> <p>Other means to deal with small percentile blends were studied in the thesis. With high enough resolution, a camera capable of shape recognition could potentially detect the weave of textiles, expanding sorting potential by possibly filtering high risk textile weaves out of the stream.</p>		
Keywords black waste textile streams, near infrared, spectrometry, machine vision		

Tiivistelmä

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Työn nimi MUSTIEN TEKSTIILIEN TUNNISTUS NIR-SPEKTROMETRILLÄ Tekstiilien tunnistusmetodin kehittäminen		
Tutkinto Materiaalitekniikan insinööritutkinto (AMK) Polymeeri- ja kuitutekniikka		
Tiivistelmä <p>Lähi-infrapunaspektrometriaa voidaan hyödyntää orgaanisten yhdisteiden tunnistamiseen käyttäen validoituja näytteitä käyttäen vertailukelpoisia spektrikirjastoja.</p> <p>Lahden ammattikorkeakoulussa oleva, pilotti-kokoluokan, tekstiilien lajittelulinjasto hyödyntää lähi-infrapunaspektrometriä näytteiden kemometriseen tutkimukseen, jossa näytteestä saatuja spektrejä verrataan villalle, polyesterille, puuvillalle sekä viskoosille luotuihin spektrikirjastoihin. Vertaamalla näytteen spektriä kirjastoon voidaan tuntemattoman näytteen materiaali tunnistaa. Laitteiston spektrikirjastoja kehitetään edelleen.</p> <p>Värit eivät merkittävästi vaikuta lähi-infrapunaa hyödyntävässä tunnistuksessa, mutta mustien tekstiilien kohdalla on havaittu spektreissä keskenään yhteneväisiä poikkeavuuksia muihin homogeenisiin tekstiilimateriaaleihin verrattuna. Poikkeavuudet spektrissä tekevät materiaalin tunnistamisesta mahdotonta nykyisellä algoritmilla.</p> <p>Opinnäytetyön tarkoitus oli kehittää Lahden Ammattikorkeakoulun lajittelulinjastoa. Mustien tekstiilien tuomien ongelmiin pyritään etsimään keinoja tutkimalla vaihtoehtoisia ratkaisuja.</p> <p>Pienellä näytemäärällä erilaatuisia tekstiiliartikkeleita kyettiin osoittamaan, että mustien tekstiilien materiaalitunnistus olisi mahdollista algoritmia säätämällä, vaikkakin saavutettu tunnistustarkkuus jää heikommaksi nykyiseen verrattuna. Menetelmän parantamiseksi tulisi hankkia suurempi määrä validoituja näytteitä.</p> <p>Opinnäytetyön aikana tutkittiin myös tapoja, joilla pienien prosentiosuuksien sekoitetekstiilien erottelu voisi olla mahdollista. Korkean resoluution konenäkökameraa voitaisiin esimerkiksi hyödyntää tekstiilien rakenteiden havainnointiin ja kenties siten erotella näytteitä, joilla on suurempi riski kätkeä seoskuituja.</p>		
Avainsanat: mustat tekstiilijätevirrat, lähi-infrapuna, spektrometria, konenäkö		

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KEYWORDS & ABBREVIATIONS

chemometrics

Interdisciplinary science of information extraction from chemical systems

FT-IR ATR

Fourier transformed infrared, Attenuated total reflection (-spectrometer)

homogeneous

Mass consisting of one type-material, texture, mixture-for example
distilled water is homogeneous liquid

LAMK

Lahden ammattikorkeakoulu, Lahti University of Applied Sciences

NIRS

Near infrared spectrometry,-meter

purity

in this thesis used to describe homogeneousness of textiles/materials

WO/PA 80/20

Wool 80% / Polyamide 20%-blend

1 INTRODUCTION

Waste streams contain a lot of usable materials and in the current climate of booming circular economy, waste can be seen as a resource. Therefore, it is important to find a reliable way of sorting value out of waste.

According to the European Commission, textile industries in Europe employ 1.7 million people and produce a turnover of 166 billion EUR annually. Competitiveness of the producers has been increasing as the manufactures turn from mass production of simple products to products with higher value, quality and design content. (European Commission 2019.) To continue the trend of added value in textiles, recycled materials need to be of high quality if they are to be used.

Using a fast and reliable way to identify the materials in the waste streams would be extremely beneficial in terms of cost-efficient collection of recyclable raw materials. Absolute purity of raw materials sifted from waste streams would increase their value. By focusing on sorting materials from waste streams headed for incineration, both the amount of wasted resources and carbon emissions caused by their incineration can be lowered. Capturing more material from these streams also increases the potential raw material for upcycling processes, both current and under development.

Additionally, European Commission has issued directives to begin separate collection of waste materials. The aim is to increase circular economy by reducing landfilling and wasted resources by capturing re-usable material from waste streams.

In Lahti University of Applied Sciences, a pilot-sized machinery that sorts textiles using a near infrared (NIR) detector has been designed. The textiles sorting line was named REISKATex. The aim was to develop a method to identify homogeneous textile materials out of a stream of unidentified samples. Based on tests and studies done with the textile sorting line, it can be said that NIR technology is a method capable of fast and reliable material identification of end-of-life textile streams. (Zitting 2017, 7-9, 21-26, 39-40.)

Further testing has brought to attention the possibility that at the very least in some older textile materials, there is either chemical degradation, certain colorants or something else, that makes it so that certain spectral characteristics appear in the observed infrared region observed with some black textile samples.

Thesis and scope

Improvements in the REISKATex pilot-sized textile sorting line could potentially be applied to a large-scale facility. Improving the capabilities of the sorting line by improving its ability to identify materials is one step towards lower wasted resources.

Thus, improvements to the NIR material identification method, and to the sorting line in general, to improve separation of valuable raw materials from waste streams would be beneficial.

Finding solutions to circumvent the technological limitations of the REISKAtex textile sorting line is the focus of this thesis. The NIR identification method cannot currently identify all black textile materials reliably.

Enhancing of capabilities is researched from the realm of machine vision. As detection of elastane, for example, in small quantities in textile weaves has proven to be difficult with NIRS, perhaps finding other ways to bypass this problem could be possible.

Figure 10 (page 22) shows the spectra of a few black or dark wool samples. Similar spectral phenomena where most of the dispersion is focused on wavelengths lower than 1350 nm, provide a working theory that the current algorithm could be modified to ignore this portion of the spectra. To test this possibility, a small test is set up.

Contents

There is a short overview of polymers and their recycling to point out why end-of-life materials need identification methods such as those in the textile sorting line. A general look at spectrometry is provided as it is fundamental for the sorters' ability to identify the material of samples. A short test to determine whether the hypothesis holds true is carried out.

2 RECYCLING OF MATERIALS

Fibers of textiles are formed of hydrocarbon chains either by naturally or synthetically. Their chemistry affects how they can be identified with spectrometric means.

Polymers are macromolecules formed of smaller molecule units bound together with covalent bonds (Napari 2012, 31-32, 313-320.) Hydrocarbon monomers can be simple, like ethylene C=C, or more complex, like methylenediphenyl di-isocyanate (Figure 1) or cellulose. Polymerization reactions connects one end of the hydrocarbon chain to another monomer and continue to add these building blocks until the reaction is interrupted.

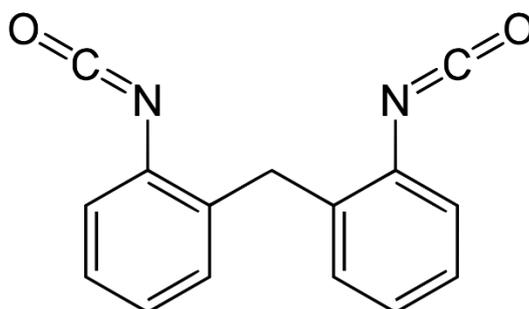


Figure 1. Methylenediphenyl di-isocyanate (Wikimedia 2007)

Organic polymers are hydrocarbon molecules. Inorganic polymers can be formed with elements other than carbon, like silicon in the case of polydimethylsiloxane, i.e. silicone rubber.

Organic polymers are categorized in two types: synthetic and natural polymers, such as proteins and saccharides. Synthetic polymers are the basic materials for most plastics, rubbers, paints and some glues. (Napari 2012, 311.)

Theoretically, as long as the polymer does not become chemically inert, it could be chemically broken into pieces, possibly to re-usable monomers. Chemical processes are developed for de-and repolymerization of certain textile polymers (polyester, cellulose). Cellulose, cotton for example, can be processed fully into pulp and back into usable fibers. (Infinited Fiber Company 2019.)

Color of matter is formed when some of the white polychromatic light is absorbed into it as the electrons in atoms become charged. To get this result with visible light, the energy difference of the ground and primed state must not be too large and therefore chromophores are needed. Chromophores and color attachment groups add to the molecular structure of the polymers. (Napari 2012, 309-310.) NIRS detects some of the

fingerprints of molecular structures and therefore chromophores could affect gained spectra.

2.1 Plastics

Synthetic textile polymers hold few similarities with plastics. Similar trends are seen in both fields and solutions from one could have potential applications for the other also.

Recycled plastics are most often then mixed with virgin materials to keep physical properties as good as possible. The biggest problems with recycled plastics are the colorants and additives (like softeners and flame-retardants) in plastic product compositions (Lehtonen & Lehtonen 2008, 224, 228). Combining different colors results in homogeneous colors or grey / black if no color sorting is done beforehand. Additives also limit usable end products of the recycled plastics as for example the European food packaging industry has its own set of directives limiting what the plastic composition may include. If one cannot be certain of the plastic composition of the raw material, they cannot be certain of the composition of the end-product either.

Current pressures for environmentally light and recyclable materials have turned some packaging producers to look towards monomaterial solutions. New materials are under development and monomaterial solutions increase the recyclability of packages and containers. (Reifenhäuser 2019.)

There is a great number of different plastics, each with their own additives and structures. This makes identification and recycling some plastics difficult. Monomaterial solutions would also make it easier to identify plastics out of the waste streams. Currently, most waste plastics end up in incineration, unless they are exported to countries where their handling is questionable.

In waste recycling facilities plastics are sorted using NIR spectrometry. In the Fortum waste incineration facility in Riihimäki, Finland, they originally had complications with black plastics, which were found to be hard to identify reliably. (Ripatti 2019.) The root cause for this seems to be carbon black, a colorant used for plastics. In industries there are benefits in moving towards middle infrared (MIR) detectors to bypass this problem (Specim 2019). However, MIR equipment is currently a more expensive option compared to NIR equipment.

2.2 Textiles

Textiles are formed by combining fibers into thread and further woven into textiles. There are multiple ways to produce both threads and textiles, increasing the variety of textile properties. For example, weave structures can allow stretching or can be made rigid. Producing blended textiles can be done by combining different materials as individual threads crossing each other at varying intervals or by making the thread of multiple materials.

Additives are used to manipulate the properties of the material further. These can be colorants, softeners or fire-retardants, which can be integrated into the synthetic polymer structure. (Boncamper 2011, 69, 76-77, 280-282.)

With population increase, the need for clothing increases as well. Using natural resources to produce an increasing amount of textiles is taxing for the environment. Production of virgin cotton material uses clean water extensively. Use of cotton has been surpassed by the use of synthetic fibers in textiles (Boncamper 2011, 21).

Textiles with long fibers have a benefit of being highly recyclable. Downcycling textiles into new products is commonplace. Mechanical recycling processes the textiles into fiber, which can be remade into thread and textile products. The processes shorten the fibers and has a negative effect on the mechanical properties of the products manufactured from the recycled material. Mechanical recycling can handle blended materials well, with elastane being one of the biggest issues for shredding processes. Elastane stretches and tangles into the machinery, blocking it.

Cotton and some other cellulose-based fibers can be chemically de-and repolymerized. Viscose can be polymerized from wood pulp and reused in the same process. For chemical processes the material fed to the system needs to be unblended, because the chemicals used are material specific. According to the VTT Technical Research Centre of Finland, development of bio-and chemical processes is an ongoing endeavor in Finland. (VTT Technical Research Centre of Finland 2019.)

Reliable identification and sorting of the materials used in textiles is therefore important for improving resource efficiency. Unblended materials could be used as raw material for new products and chemical processes increase the quality of the recycled material.

2.3 Waste as an energy source

Circular economy challenges the previous, or in some cases current, way of doing things in industries and in the world in general. The linear economy extracts profit from materials

via a few steps, from cradle to the grave, where the “grave” is un-usability. The realization of the fact that landfilling is not sustainable brought attention to the value of waste. The implementation of the waste hierarchy (Figure 2), into the thinking serves as a kickstart to turn linear economy into a circular one. The hierarchy Figure can be seen in multiple sources, all of which talk about the value of waste and circular economy.

Circular economy is the outcome where the material extracted is formed into a product which firstly does not use as much material, secondly can be reused as is, thirdly can be reused as raw material and as fuel for energy production and only ultimately is disposed of if the previous is not possible. The result is the circular economy model where new potential for industries to harvest profits arises at every turn the material/product makes another loop, as demonstrated in Figure 3. The European Commission has adopted this and are basing some directives, such as EU Directive 2018/850, on it.

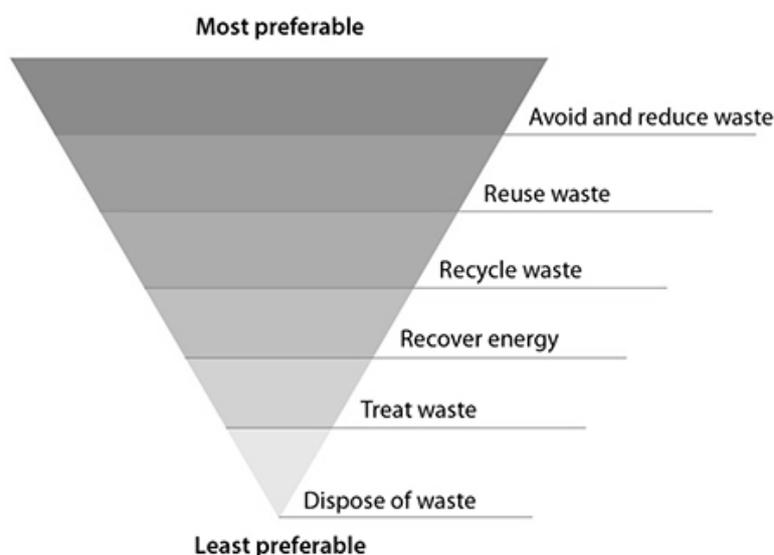


Figure 1. Waste Hierarchy (New South Wales Environment Protection Authority 2019)

Instead of losing the value of the material, new cradles are found for it. This way of thinking is used to aim for sustainable growth. The realization of the limitations of natural resources is emerging and some industry giants, such as Vattenfall, are aiming to turn to greener pastures of renewable sources. Vattenfall’s roadmap aims to become fossil free in one generation and has spread its branches to surrounding industries, essentially helping them reach similar goals. While Vattenfall seeks to reduce burning of carbon-based materials, they also try to help their partners reduce CO₂ emissions. (Vattenfall 2019.)

The sustainable growth of circular economy is achieved with new designs of reusable products, helped with additive production methods and the regeneration of natural systems. (Ellen MacArthur Foundation 2019a.)

OUTLINE OF A CIRCULAR ECONOMY

PRINCIPLE

1

Preserve and enhance natural capital by controlling finite stocks and balancing renewable resource flows
ReSOLVE levers: regenerate, virtualise, exchange

Renewables    Finite materials

Regenerate Substitute materials Virtualise Restore

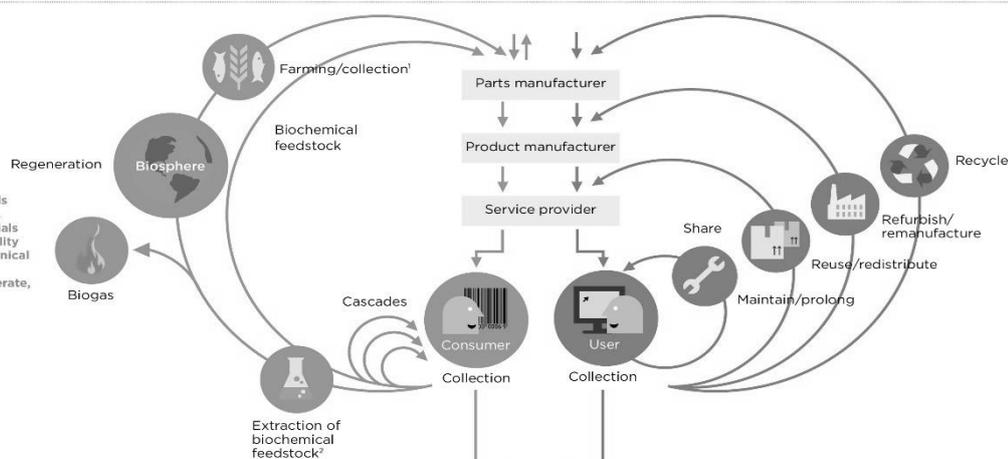
Renewables flow management

Stock management

PRINCIPLE

2

Optimise resource yields by circulating products, components and materials in use at the highest utility at all times in both technical and biological cycles
ReSOLVE levers: regenerate, share, optimise, loop



PRINCIPLE

3

Foster system effectiveness by revealing and designing out negative externalities
All ReSOLVE levers

Minimise systematic leakage and negative externalities

1. Hunting and fishing
2. Can take both post-harvest and post-consumer waste as an input
Source: Ellen MacArthur Foundation, SUN, and McKinsey Center for Business and Environment; Drawing from Braungart & McDonough, Cradle to Cradle (C2C).

Figure 2. Circular Economy diagram (Ellen MacArthur Foundation 2019b)

This ties into the textile waste streams, which are seen as a source for raw materials for new materials and products under development. These include materials based on recycled cellulose. The VTT Technical Research Centre of Finland is involved in many of these developments. Essentially, chemical reduction and regeneration of fibers allows for, in some cases, infinite recycling of materials. All the motivation for new methods to extract raw materials from waste streams stem from the circular economic way of thinking and realizing the value in waste.

One of the end points for all matter is incineration, which extracts the last drops of the energy content of the material. In accordance with the waste hierarchy, waste can be seen as a resource even at the end of its lifecycle, though the endpoint should generally be moved further down the line.

At this point, waste incineration is possibly the better solution for the waste we already have and still produce, because landfilling produces more greenhouse gasses than the incineration facilities. European facilities are under the scrutiny of the EU directives and a subject to fines should their emissions of harmful compounds exceed set limits. As an example, in Sweden NO_x emissions over the limit would cost approximately €4/kg (Naturvårds Verket 2006), and as the emissions generally revolve around tens to hundreds of kilotons, there is pressure to develop technology for better filtration. These directives and the requirement that the facilities need to observe and report their emission annually have pushed many facilities and energy companies to improve their facilities. Generally, energy production companies of the Scandinavian region are motivated to turn toward lowering their carbon footprints. For example, Fortum already has 96% share of the CO₂ free energy productions of Europe (Fortum 2019) and Vattenfall aims to become fossil free in their operations by the year 2030 (Vattenfall 2019).

2.4 Value in textile waste

The current ideological shift towards more circular economy has sprung EU directives that are slowly driving more and more industries to look for more ecological methods to handle their operations. Much like the collection and recycling of plastics is going to help sustainable growth, so too will doing the same for textiles.

At the end of their life textiles are mostly burned for energy, but some are still discarded into landfills. While incineration grants some benefits to society and its CO₂ emissions are dropping down as technologies for flue gas cleaning are advancing, energy production is not the optimal way to recycle materials.

This is to be changed, as the EU directives (2018/850; 2018/851 and 2018/852) require member states to reduce landfilled waste significantly and by 2030 to stop landfilling of waste suitable for recycling or other recovery altogether. To help this, separate collection of materials, including textiles, is to begin by the year 2025. (EU Directives 2018/850-852.)

In Finland, approximately 72 kilotons of textiles are recycled annually (Dahlbo, Aalto, Eskelinen & Salmenperä 2017). Figure 4 shows estimates of the distribution of textile waste streams in Finland. The data was gathered as a part of the REISKA-project between 2015 and 2017. (Reiska 2017.)

From the 72 kilotons, only 20% is separated initially and some of that is given new life; 70% is given to charities or as donations or otherwise exported out of the country. In total, nearly 85% of textile waste streams are directed into incineration facilities, as landfilling of organic materials is banned in Finland. In the future, the aim is to cut down incineration of

textile waste according to the stipulations of the previously mentioned directives. A few of the materials under development are aiming to gain their raw materials from textile waste streams.

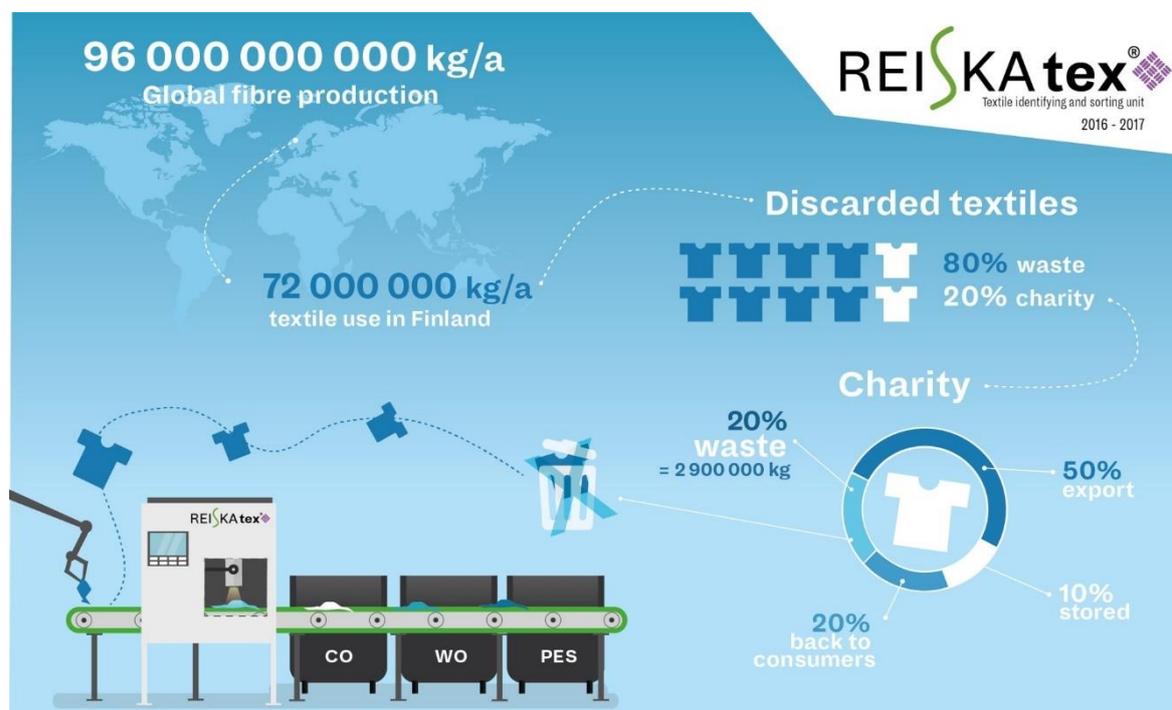


Figure 4. Textile recycling in Finland (Rouhiainen 2017)

Admittedly, lowering these incineration amounts would result in loss of energy, but as the drive and pressure from the European Union's directives push the world towards more sustainable economies, redirection of these streams is going to be necessary.

Additionally, the total impact of textile incineration for energy production in Finland is minimal, as waste incineration produces only 0.6% of the annual total of 68.8 TWh (EU Commission, June 2018.)

Lowering the amounts of waste and incineration are both on the agenda as methods to reach the EU 2050 goals of CO₂ reductions. While there are ways to reduce emissions by better filtering, going for the source is far more effective and so identifying usable material from waste streams is a way towards cleaner air. Gaining usable raw material from waste cuts growing, harvesting and manufacturing of virgin materials out of the equation efficiently, if not completely.

Difficulty lies in the identification of raw materials. Fast and reliable identification methods usable for industrial volumes are limited. There is no realistic way manual sorting of waste would be efficient enough in the long run. All the sorting of material from waste streams

must be automated and therefore the identification of the usable materials must also be based on automation.

The fastest way to identify anything is via visual confirmation. The human eye is limited to a narrow spectrum of electro-magnetic radiation and cannot differentiate between actual materials based on vision alone. Near infrared equipment is an established way for analysis and quality control. While machines can be harnessed with equipment to achieve a great number of different results, they too have their limits. (Rodgers & Beck 2017.)

NIR can be used to identify materials easily, but its development has had complications. As previously mentioned, the identification of black materials has been an issue. This problem emerged when a test run of a prototype textile sorting line in Lahti University of Applied Sciences was carried out; here black natural fibers occasionally gave spectra that had common features across different textile materials.

3 SPECTROMETRY

Spectrometry is an analytical science based on electro-magnetic radiation, specifically what is classified as light, and its effects on molecules and their atomic bindings. Infrared (IR) spectrometry is an extension of spectrometry that focuses on a specific region of electromagnetic wavelengths, namely the infrared region. (Lehtonen & Sihvonen 2009, 219.)

Infrared radiation is more specifically heat radiation. Heat results in movement (vibrational, rotational, bending) in the atomic bindings. (Lehtonen, Jaarinen, Jansson, Pohjakallio, & Repo 2006, 122, 145; Jaarinen & Niiranen 2008, 90). When atomic bindings are excited with energy projected unto them, their movement takes a portion of that energy and when this difference between projected and reflected energy is observed, we are talking about absorbance. Most atoms and functional groups of a molecule are affected by different wavelengths of electro-magnetic radiation; each binding is in the same phase with certain wavelengths, making them identifiable. (Lehtonen & Sihvonen 2009, 219; Jaarinen & Niiranen 2008, 90.) When a functional group, for example –OH, is present, we see an increase in absorbance at wavenumbers 2700 – 3700 cm^{-1} , as seen in Table 1, and depending on the position of the group (“Addendum”) it affects the spectrum, giving it certain visual characteristics listed in the “Appearance” column. These are commonly referred to as “fingerprints”.

Table 1. IR spectrum table & chart (Sigma-Aldrich 2019)

Wavenumber (cm^{-1})	Appearance	Group	Compound Class	Addendum
3700-3580	medium, sharp	O-H ; stretch	Alcohol	Free
3550-3200	strong, broad	O-H ; stretch	Alcohol	Intermolecular bonded
3300-2500	strong, broad	O-H ; stretch	Carboxylic acid	Usually centered on 3000 cm^{-1}
3200-2700	weak, broad	O-H ; stretch	Alcohol	Intramolecular bonded

With enough repetition and verified samples, we can build charts such as Table 1. These can then be used to identify functional groups on the molecular level of unknown samples.

Spectrometry mostly operates by comparing an unknown sample to a known, verified, sample. In many verified laboratories, when detecting elements in a sample, comparison is done with references built specifically for that purpose, for example determining iron content by focusing on the amount of ferrous ions in a sample. (Lehtonen & Sihvonen 2009, 217.) Comparison can also be done easily with spectral libraries, where usually the spectrometer or material producer has compiled sample spectra from their verified samples. The sample spectrum is then referred to the spectral library and mathematically compared.

Other spectrometric methods include breaking down the components into atoms and gaining spectrum data from those. Another method is emission spectral analysis, where the atoms are loaded with extra energy and their inherent projected spectrum is captured by observing the radiation released. (Jaarinen & Niiranen 2008, 48-50, 133.)

3.1 Electro-magnetic radiation

While radiation as a word brings up thoughts of nuclear mishaps, the range of electromagnetic radiation is so vast that, aside from the intensive γ - and x- radiations, most of it is harmless; in fact, television programming is channeled to households using electromagnetic radiation. Basic breakdown of EM radiation is represented in Figure 5, where high frequency radiation is at the top and low at the bottom.

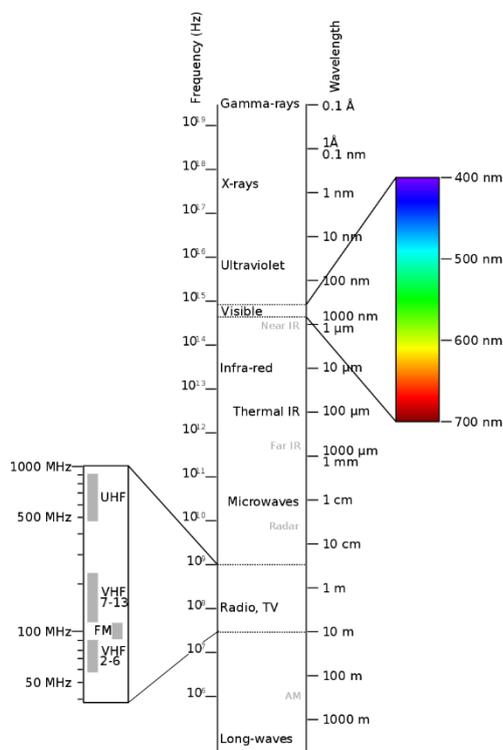


Figure 3. EM spectrum (Blacus 2012)

The longer the wavelength, the less energy a pulse projects onto an object, and in turn, the higher the frequency of the waves, the more it affects materials. This is why UV radiation damages skin, x-rays are not recommended too often, and gamma rays are harmful, even deadly. (Jaarinen & Niiranen 2008, 47-50; Lehtonen et al. 2006, 143.)

The energy contained in waves can be calculated with Planck's constant with Equation 1:

$$E = \frac{hc}{\lambda} \quad (1)$$

where h = Planck's constant; c = speed of light and λ = wavelength

The energy of ultraviolet light breaks down skin by killing the cells, and some of the penetrating radiation may even affect DNA strands in the cells, which may result in skin cancer. In the same manner, all high frequency radiation when projected onto organic material becomes harmful to it. High frequency radiation penetrates further into matter and so in a human γ -rays will affect internal organs, causing irreversible damage quickly by affecting the nuclei of atoms. Enough energy projected onto the nucleus causes it to destabilize leading to nuclear fission reactions, where α -, β -and γ -radiations are released, the former two of which are particles and the latter purely energy. Properly handled high frequency radiation is used in the medical field as a tool to try to identify endodermic problems. X-rays are used to scan the density of matter and is useful to detect anomalies in bone structures, revealing for example osteoporosis. (Pisani, Renna, Conversano, Casciaro, Muratore, Quarta, Di Paola, & Casciaro 2013.)

Visible and UV light affect the outermost electrons of the atoms, whereas gamma rays penetrate to the core of the atom. Infrared radiation has enough energy to excite movement in atomic bonds, but not enough to cause them to break, and is thus considered harmless. The absorbed energy results in heat, which is why any surface feels warm after enough light has been shone on it. (Lehtonen, et al. 2006, 122.)

3.2 Infrared spectrum

When infrared wavelengths are projected on a sample, it causes movement in the molecules. Stretches, bends and rotations in the binds of elemental atoms absorb the energy of the EM radiation. How much radiation and at what specific wavelengths is absorbed into the material is the very basis of IR-spectrometry. Any material consisting of multiple elements has absorbance peaks at characteristic wavelengths based on its composition, as each of its elements is in phase with different wavelengths. The intensity of absorbance is determined by the amount of the same-phase bonds. When the observed absorbance is linearized as a function of wavelength, we get a visualized spectrum (an example in Figure 6).

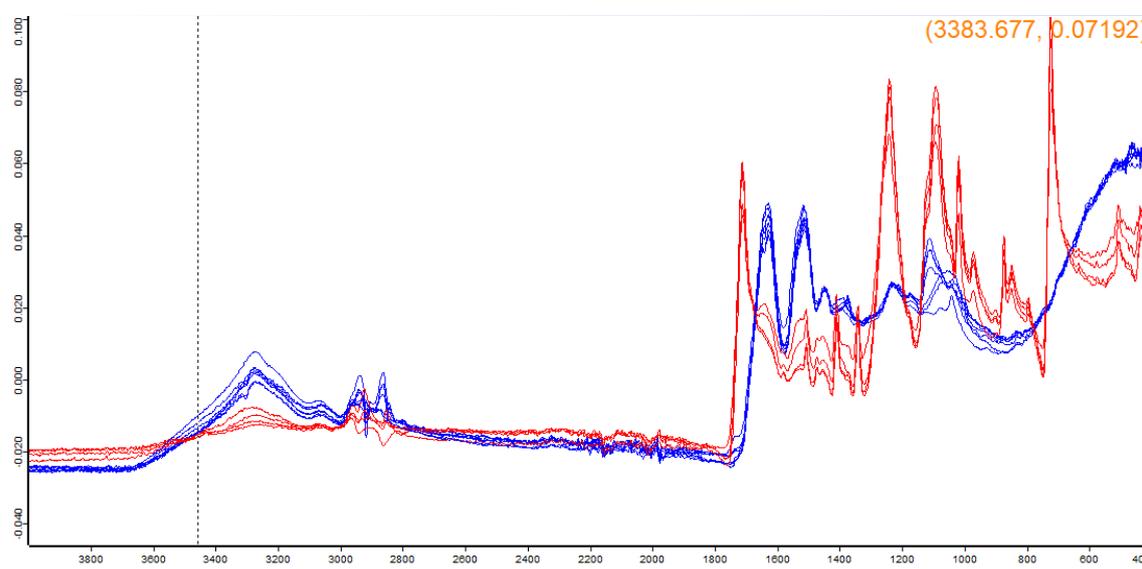


Figure 4. Example of FT-IR spectra. Wool spectra in blue, wool / polyester blend in red

With the help of IR-spectrum tables one can determine that these samples (Figure 6) probably contain-NH as both aliphatic primary amine and secondary amine, highly likely-OH alcohol, some C=C bonds and C-O and so on. The more clues one gets, the easier it becomes to estimate the compound's composition. The spectra in Figure 6 are of wool (in blue) and wool / polyester blend (red). The clues can be compared to tables, similar to Table 1, which can be used to estimate the chemical formula of the compound. Such tables have been built by measuring large numbers of samples of pure elements and expanding from that by adding known chemical compounds to the mix.

IR-spectrometry is often divided into three categories: near-infrared spectrometry (NIRS), mid-infrared spectrometry (MIRS) and far-infrared spectrometry (FIRS). Named as such based on how 'far' from visible light spectrum their wavelength ranges are. Where the previous ends, the next begins; NIRS (700 nm – 2500 nm), MIRS (>25 000 nm) and FIRS

(-300 000 nm) (Theophanide 2012). The general “fingerprint” range falls under MIR, where absorption is the most intense for functional groups present in organic molecules.

Most IR spectrometers function in the same manner. A wide spectrum of wavelengths are produced and directed to the sample and then the difference of detected energy is compared to the source light intensity. What happens between the source and the detector is dependent on the machinery used. The detection is done either of reflected or transmitted spectra.

Spectrometers are divided into two main categories: monochromatic and Fourier-transform (FT) spectrometers. Where the FT-IR makes use of the full IR spectrum from the start, monochromatic spectrometers use either mirrors or other means, such as gratings, to ‘break’ the light into a prism and filter the used wavelengths one at a time onto the sample. The Fourier transformation refers to the method with which the interferogram is mathematically unfolded into a readable spectrum. (Lehtonen & Sihvonen 2009, 220-221.)

Among the more effective spectrometers are FT-IR-spectrometers that benefit from attenuated total reflection method, where the light is bounced onto the sample surface multiple times before the actual detection. This increases the total absorbance of energy into the sample giving a clearer picture of the intensity.

3.3 NIR

NIR equipment is used for the analyzing of compounds mostly formed of C-H-, O-H-or-N-H bindings. Absorption levels in the NIR region are generally low and a comprehensive compositional fingerprint analysis is impossible. NIR can be used to determine moisture or alcohol, saccharide or fiber content and more. (Lehtonen et al. 2006, 146.)

Building libraries of spectra to which one may compare the sample spectrum allows NIR to be used to identify unknown samples. Since organic polymers are mostly formed of previously mentioned elements, their “fingerprints” may be detectable in the NIR region. However, many similarities in the molecular structure may give off enough similar fingerprints to have mathematical comparison fooled.

Cellulose-based fibers have a similar monomer base, with alternating binds, both within the monomer and polymer structure. Using a NIR detector for textile analysis is a proven method for some materials and demonstrably some cellulose polymers can be differentiated despite their similarities. (Zitting 2017, 38, 41)

4 IMPROVEMENTS FOR TEXTILE SORTING

Identification of materials is very important if we are trying to find recyclable materials from post-consumer products. Recycling methods for materials are quite specific for each and impurities might halt the systems. As an example, while mechanical textile recycling is not much affected by heterogeneous blends, the inclusion of elastane in the mix will obstruct some machinery. Identification of homogeneous materials is one step towards better use of resources, the next step would be to be able to differentiate blended materials.

There are different methods for material identification. One can test their mechanical or chemical properties and, based on the findings, try to compare the results with existing tables. Testing the material is slow; with chemical dissolution identification may take up to days for blended textile materials or heterogeneous batch identification. In industrial-sized facilities where the material flows are measured in tonnes per hour, such methods are too slow.

Fastest solutions for the identification can be found from visual determinations. Machine vision is capable of, aside from maintenance breaks, around the clock observations and with the wider spectrum vision they can access information beyond human capabilities. Machine vision is also capable of handling hundreds and even thousands of pictures per second and so, theoretically, the industrial line speeds can be increased to the limit of data processing speeds.

Material identification might be the next important step toward better recycling, but there are benefits gained from machine vision for improving the textile sorting line further.

4.1 Machine vision

With set algorithms and possibly even neural network learning, a machine can interpret what is put in front of its receptor. A camera is used to capture an image and the data is processed with a computer.

Similarly to many other methods involving computers, machine vision also needs to be taught. First, it must be taught what it is seeing, and all this preliminary work must be done manually for now.

The main benefits of machine vision compared to its human equal is that machines will not tire and are capable of seeing a much wider range of the light spectrum. By attaching certain cameras or detectors, one can improve a computer's ability to process visual data, unlike a human who would be stuck with the narrow visible light spectrum. All this and the capacity to process hundreds of pictures per second allows faster and far more precise

detection of whichever properties are wanted. The main uses for machine vision can be found in processing units and lines, places where the speed of assessment is key or where assessing is required continuously. (Sonka, et al. 2015, 5-9, 12-27)

In essence, machine vision is just a camera attached to a processing unit, a computer. The processor is taught to differentiate between variations of shades of grey, colors or just spectrum data. The gathered data is then used to formulate a command to operate the attached machinery. This allows fully automated production with the assistance of robotics. (Prince 2012, 1-5, 55-73.)

There are detectors capable of detecting up to 4096 shades of grey. Generally, machine vision is built around the detection of shades of grey, but of course there are detectors capable of detecting colors. With light manipulation, the sample surface can be made more easily inspectable by the grey detector. (Sonka, et al. 2015, 14-27, 118-120.)

Many machine vision systems operate by observing either a line or a matrix and gathering varying data from that area. The line can be as small as one pixel wide and the whole production line at length. This way the detector operates much like the common household scanner, where the light moves from side to side. When a sample passes the light line, the camera captures images at every pixel interval as the sample moves past. The processor unit then compiles all the pixel-wide pictures into one cohesive vision.

Detection matrix is an area of certain dimensions in a 2D-plane. The camera is set to capture a frame at intervals synchronized with a tachometer for example, essentially capturing wide lines as the sample passes the area at the set constant speed.

A picture forms on the computer showing whatever sample passed through the line or matrix. Depending on the type of detector, the picture is either grey with 256 – 4096 shades or colored with RGB space if a color camera is used. With a sample put on a conveyor belt of uniform color, a greyscale camera with properly set lighting can make up the shape of the sample. The lighting is used to project shadow on the belt and the camera detects the difference in shades, the image is processed and a picture forms. (Soini, 1996; Sonka, et al. 2015, 14-27, 120-121, 156; Eskelinen, Tirronen & Rossi 2017, 78-79.)

Greyscale camera setups are used in wood industries to detect surface flaws. (Reini 2013, 14-15 ; Soini 1996) The same idea can be used to identify for example pockets on textiles. Perhaps, with good enough resolution, the weave structure of textiles could be identifiable. Such capabilities could be explored with the REISKATex -sorting line. Some

weave structures may hide elastane better than others and identifying those could be beneficial for the recycling processes.

4.2 Near infrared textile sorting line

LAMK has developed a textile material identification and sorting line named REISKATex (Figures 7 & 8). The sorting line's conveyor belt automatically drives the sample to the NIR detector, which in tandem with the computer-operated identification library determines the sample material and sorts the samples into material specific containers with air pressure.



Figure 5. Textile sorting line

The material identification libraries have been built using validated samples to identify 100% wool, 100% cotton and 100% polyester. Additional capability to identify 100% viscose was developed later. Currently the identification libraries are being expanded.

The textile sorting line was developed as a part of a three-year REISKA -project aiming to improve resource efficiency in Päijät-Häme, Finland. The project involved mapping of waste streams, consultation for waste handling and planning for resource efficiency of events and more. (Reiska 2017.)

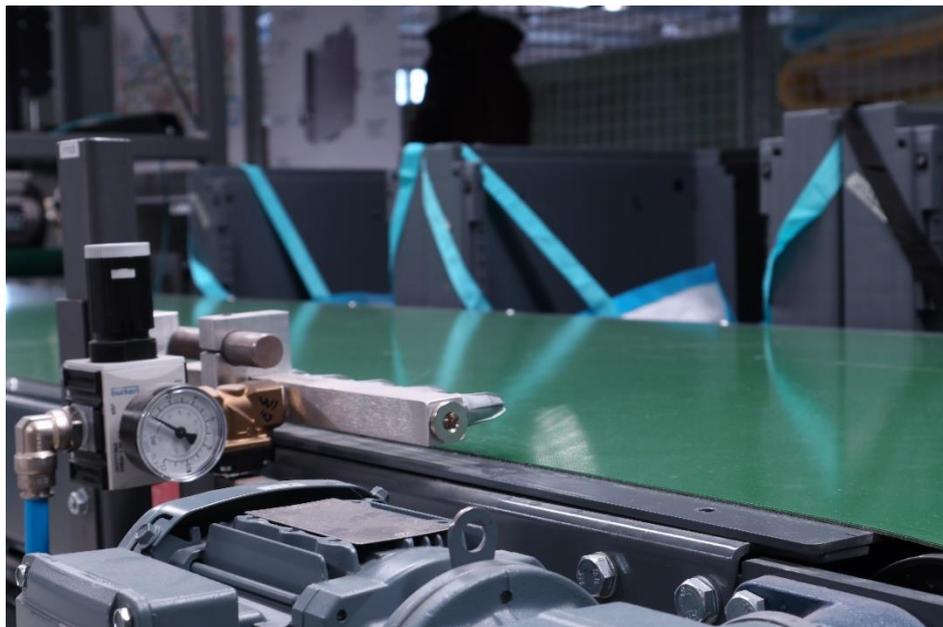


Figure 6. Sorting line-air blower & containers

More in depth look on the development of the identification method of the sorting line can be read in thesis “Optical sorting technology for textile waste” (Zitting 2017). The thesis describes how the identification libraries and method were built and what mathematical operations govern it and why.

One problem with NIR detectors across materials has been black-colored materials. As the near infrared range is closer to the visible light spectrum, colorants may affect the spectra gained from samples.

With the textile sorting line, in woolen samples the black color was found to affect the identification more often than in other materials, suggesting that the root cause could be a certain colorant that is used with organic fibers. Later similar spectral characteristics were observed in polyester samples.

The spectral phenomena are nearly identical across textile materials, yet colorants used may vary in different materials. Could there be a way to isolate the cause and improve the developed material identification method?

5 BLACK MATERIAL IDENTIFICATION WITH A NIR DETECTOR

Black materials absorb more of the visible light and energy compared to colored materials. Being organic compounds, colorants could also affect what NIR range wavelengths show peaks in absorbance.

The NIR spectrometer of the textile sorting line in LAMK has no trouble identifying the sample material in four categories: wool, cotton, polyester and viscose. The weave and color of materials hadn't been an issue when developing the identification method.

During a test run of a few hundred kilograms of post-consumer collected samples the identification method had trouble with some black or dark samples. Among the materials were mostly cotton, wool and polyester and some blended materials; rarely there were some more uncommon materials.

To test the capabilities of the NIRS and find a possible way to circumvent the issues with black materials, a small-scale test was set up. In order to find out if there is a commonality with the spectral data across textile materials, black samples had to be collected and inspected with the current equipment. Similarities were found and the algorithm was modified and tested.

5.1 Equipment and settings

The sorting line is equipped with NIRS Analyzer Pro by Metrohm. Equipped with a high-resolution InGaAs diode array, the unit operates at a spectral range of 1100-1650 nm. The sample spectrum is drawn with absorbance as a function of wavelength and displayed and manipulated in the Vision™ software by Metrohm. The equipment is controlled with the Vision™ software.

The sample spectra were collected with 64 scans as raw data and separately saved before and after the mathematical operations. Standard Normal Variate (SNV) and 2nd order derivation with 10 nm segments were applied to the spectra. SNV converts absorbance to absorption intensity and derivation enhances the spectral features.

The reference libraries have been built previously and consist of validated samples. The libraries used are for pure cotton, polyester, wool and viscose (CO, PES, WO & VIS) and are not commercially available. Each has been built with between 50 to 100 validated samples. Additionally, an incomplete library built of blended wool and polyamide was used for confirmation of a few samples.

For verification of samples, a Bruker Alpha FT-IR ATR laboratory spectrometer with Bruker OPUS software was used. Commercially available identification libraries were used in addition to a textile specific library of LAMK's own making. Samples were cross-referred to further reduce misidentifications.

5.2 Samples

Black samples are listed in attachments 1, 2 and 3.

Samples were collected from the sample sets used to build the reference libraries. Regular (colored) samples for each material type were used to provide information on whether the changed algorithm could still identify them properly. These were samples used to build the reference libraries.

Black samples were collected for three material categories (CO, PES, WO). None were available for viscose. 20 woolen, 10 cotton and 10 polyester samples were collected from sample sets originally planned to be used to build the reference libraries. Approximately half of the samples are "feelers" and contain the producer's markings of their material. Samples used for reference libraries had been validated both by a third party and at LAMK with a FT-IR ATR spectrometer.

Using post-consumer collected waste textiles, 10 black unknown material samples were added to the mix. Textile products' labeling is not considered a reliable way to determine the material of the product as some may be incorrectly marked. Still, labeling was considered a guide as to what result should be expected. In case of positive id of material, results were then verified with the ATR spectrometer. Selection of samples can be seen in Figure 9.



Figure 7. A few samples: cotton (black and colored), wool (black/purple and colored), polyester and viscose

5.3 Test

Samples were fed to the sorting line for material identification with the original algorithm. The algorithm compares wavelength range 1100-1650 nm of the sample to reference libraries and mathematically determines whether the differences between the sample and references fall under a set threshold.

Figure 10 shows collected spectra of black or nearly black wool samples after standard normal variate and 2nd order derivation mathematical operations. The samples had been verified with the FT-IR spectrometer and its library to be wool. Most of the dispersion is focused on wavelengths lower than 1350 nm.

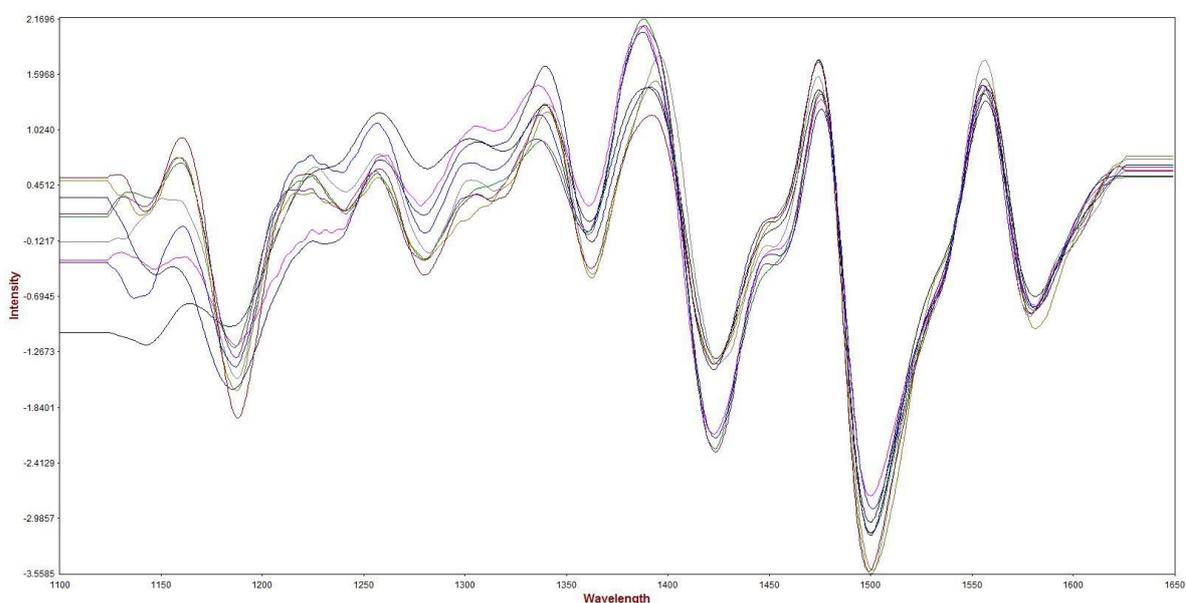


Figure 8. Spectra of black woolen samples listed in attachment 1

Comparison of black wool samples to pure wool spectra was done, resulting in identification failure of 95% of samples. Sample WO11 compared to four verifiably 100% woolen sample spectra depicted in Figure 11.

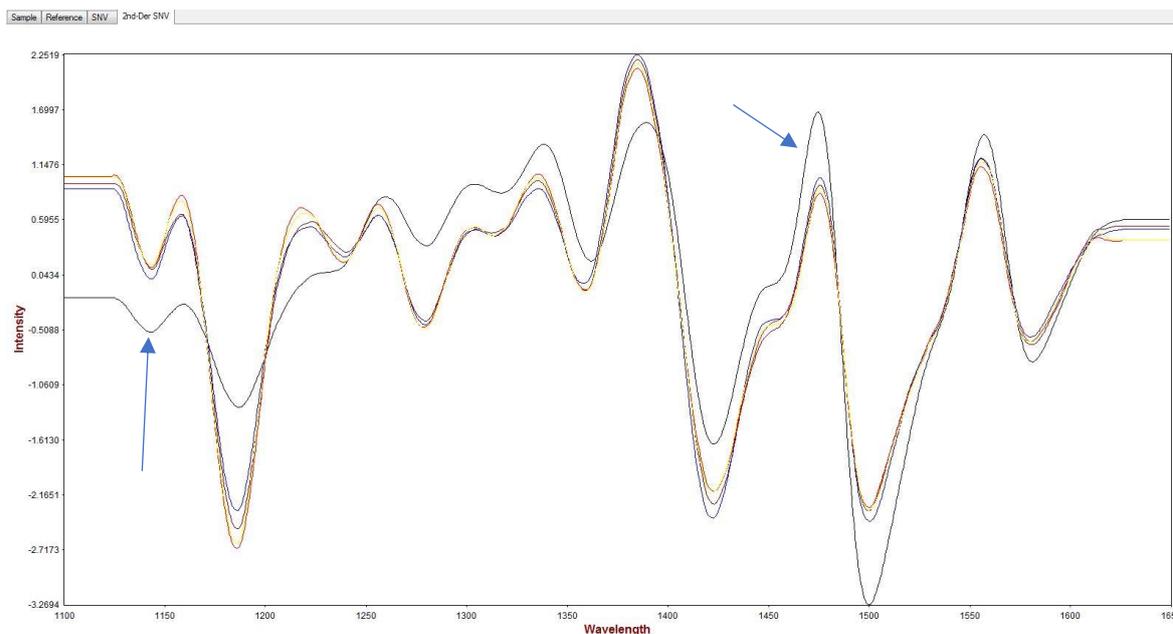


Figure 9. Sample WO11 spectrum (pointed) vs. 100 % wool spectra

Most of the variation seen in Figures 10 and 11 at wavelengths 1400 – 1450 nm could be within the threshold that is set up in the current identification algorithm. This would mean that the black samples could theoretically be identified as wool if, hypothetically speaking, the area before 1350 nm was not as scattered. Figure 12 shows the spectrum of sample WO5 compared to a verified wool spectrum.

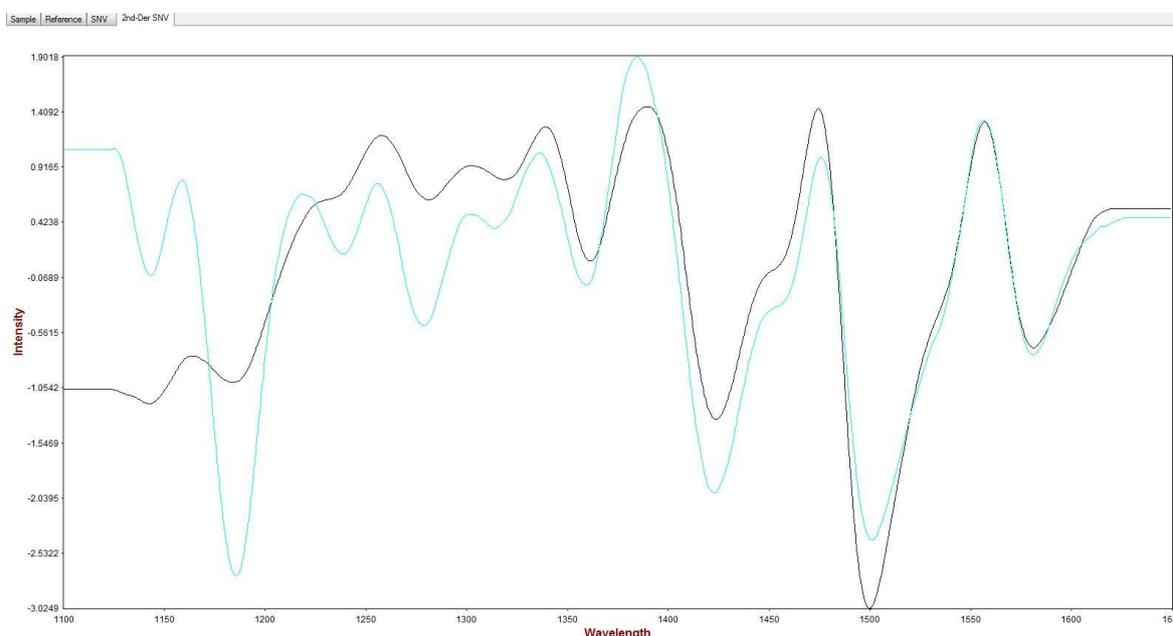


Figure 102. Sample WO5 (black line) compared to verified wool

It was found that some wool samples have enough similarities with wool 80%-polyamide 20% blends to merit verification of the sample. Using a library with small spectra quantity,

one unknown sample was found out to be WO/PA 80/20. Figure 13 shows a comparison of sample UK10 to WO/PA 80/20 samples.

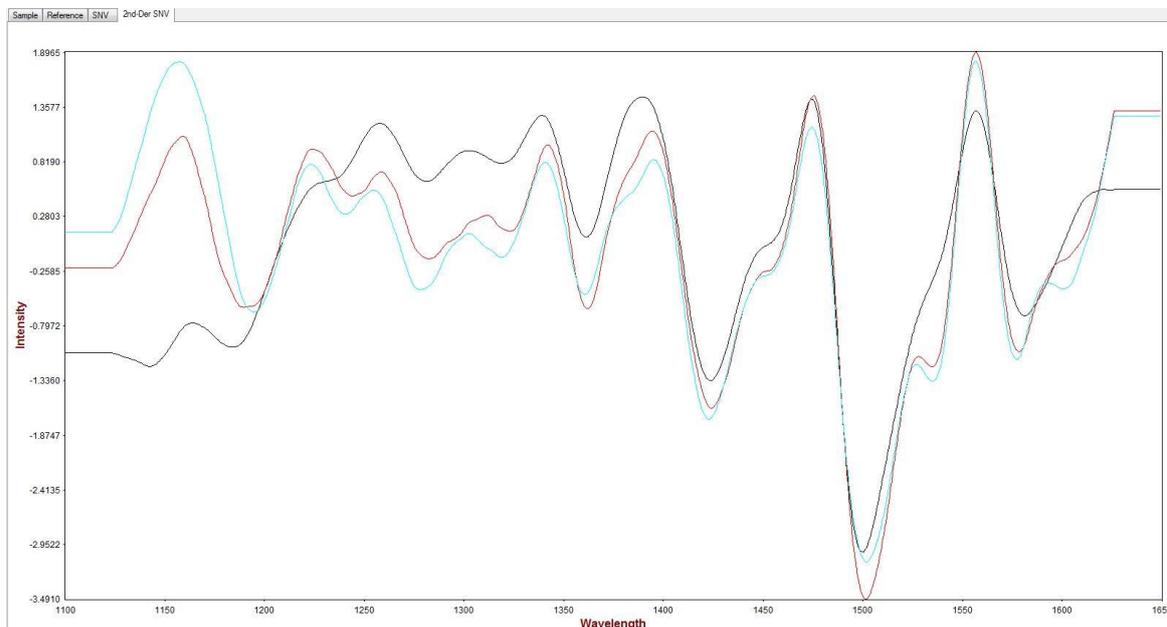


Figure 113. Sample UK10 compared to Wool 80 / Polyamide 20 blend samples

Figures 12 and 13 demonstrate that while the 1100 – 1350 nm area has enough difference in spectra to help identification, the rest of the spectra could fall under the threshold. This means that cutting the 1100 – 1350 nm range could produce problems with the differentiation of WO/PA blends and pure wool.

Figure 14 shows sample UK3, a black sleeve, (seen in Figure 9) compared to 100% cotton spectra. The scattering can be seen before wavelength 1350 nm. The same can be seen across materials. One of the unknown samples, UK1, gives a spectrum seen in Figures 15 and 16, in which the sample is compared to other materials. According to FT-IR ATR, both the UK1 and UK3 are pure cotton.

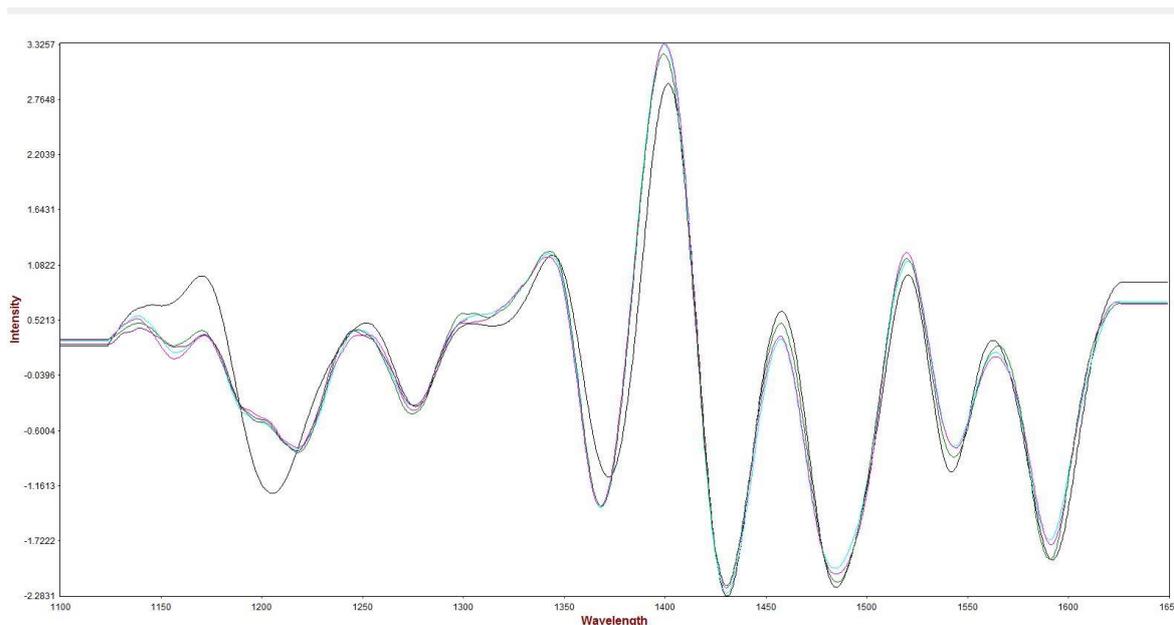


Figure 124. Sample UK3 (black line) compared to 100% cotton

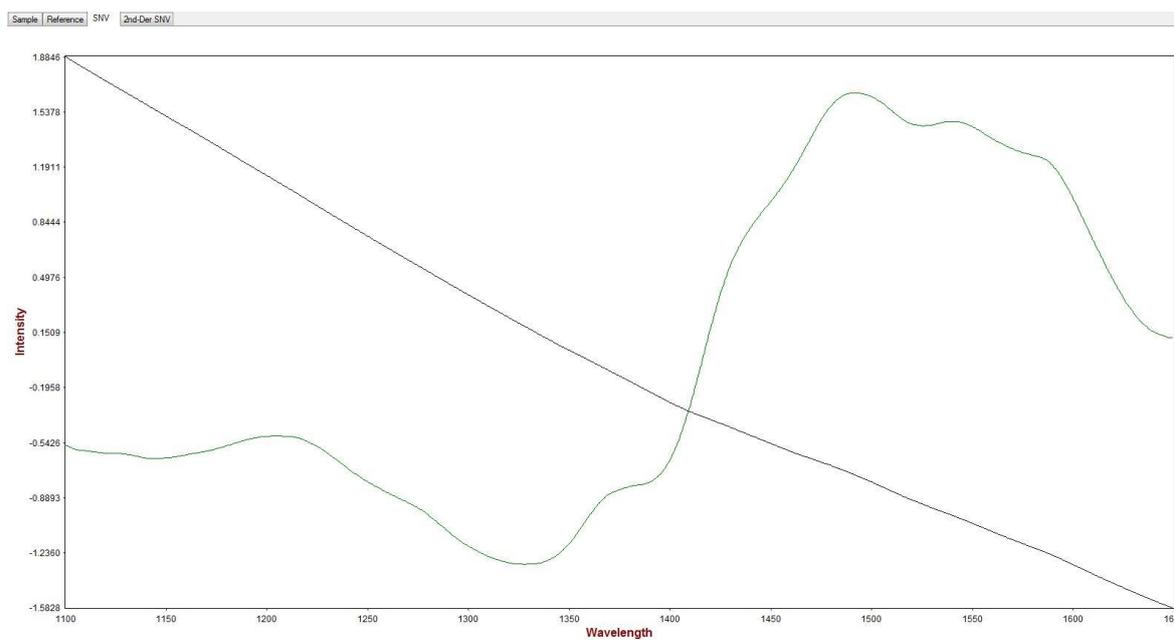


Figure 135. Sample UK1 compared to cotton spectrum. Standard normal variate spectra

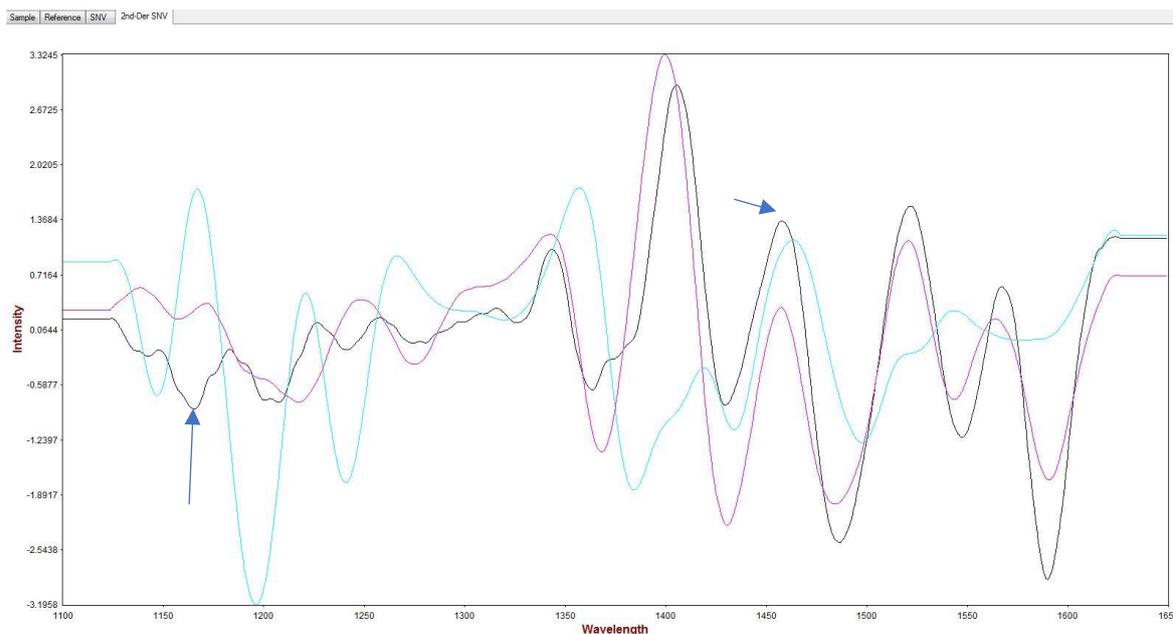


Figure 146. Sample UK1 (pointed) compared to cotton (purple) and elastane (blue). SNV + 2nd order derivation spectra

Further testing confirmed that in most instances the black color gives spectra that have scattering between wavelengths 1100 nm and 1350 nm. Based on this, the algorithm was modified to compare only the 1350-1600 nm range for the wool, cotton and polyester spectra.

To build a new identification method, the same spectral data that was used to build the current one was used. The threshold for sample disqualification was kept the same as in the original algorithm. The Vision software's identification method validation shows that the three categories do not misidentify, indicating that differentiating unblended materials is possible with the new set range.

An initiation test was done to see if the method is still usable. Testing the new method with a few samples gave results shown in Table 3, where "Sample ID" is the description of the sample, "ID as" tells what the algorithm claims the sample to be, "ID Result" is an average difference between the sample spectrum and library spectra and "P/F" is pass / fail. "CO" means cotton, "PES" polyester, and "EA" elastane.

Testing was done with the disqualification threshold set to 4.2, meaning that samples with "ID Result" over that would fail identification. This threshold had been found to be efficient in filtering monomaterials with the full range of the used NIR detector, with approximately 0.8% error margin.

The test run showed that neither wool, cotton, polyester nor viscose were misidentified across. However, blends with small quantities of another fiber showed an increased risk of misidentification.

Table 2. Sample test run

Time	Sample ID	Selected	ID as	ID Result	P/F
14:33:28	test0010	unknown	No Match	23.064	Fail
14:33:51	viscose100	unknown	Viscose 100	3.331	Pass
14:34:23	wool100	unknown	Wool 100	1.849	Pass
14:34:45	cotton100	unknown	Cotton 100	1.827	Pass
14:35:09	polyester100	unknown	Polyester 100	2.219	Pass
14:35:31	polyester100_2	unknown	Polyester 100	2.636	Pass
14:36:01	blackcottonsamp	unknown	No Match	4.725	Fail
14:36:37	blackwoolsamp1	unknown	Wool 100	1.427	Pass
14:39:33	co92Pes6EA2	unknown	Cotton 100	2.236	Pass
14:40:33	COPE80/20	unknown	No Match	9.634	Fail
14:41:12	COPE80/20_2	unknown	No Match	9.185	Fail
14:42:05	COblanket color	unknown	Cotton 100	2.355	Pass
14:43:46	blackunknown	unknown	Cotton 100	2.629	Pass

Samples labelled with "...100" in table 3 are samples that had been used for library building and are verifiably pure. Other sample materials are based on sample labeling.

5.4 Summary

Following the initial test, identification of black samples was tested. In total, 42 of the 50 black samples could be identified with the new algorithm.

Black samples that exceeded the disqualification threshold were: UK 4-8, WO16, WO18, CO6 and CO10.

Additionally, feelers other than black, used for reference library building were used to test the method further. Among these were CO 80 / PES 20, CO90/PES10, WO80/PA20 and 100% pure samples.

The tests show that the three main materials (wool, polyester, cotton) do not mix when using unblended samples. There is indication that at 80 / 20 percent blend proportions the method can reliably fail samples, but at 90 / 10 blend proportions the method itself begins to fail.

5.5 Discussion

Directly cutting a portion of the spectrum from the mathematical operations governing the identification seems not to be sufficient as the method becomes unreliable when fewer reference points are available. This results in small percentile blends passing the filter. Then again, detection of small quantities has been hard previously as well, as for example detection of elastane has proven difficult and highly dependent on the weave of the textile and/or the structure of the threads.

Some samples containing elastane need over 5% of elastane for it to have a visible impact on FT-IR identification and NIR might still not detect it. The elastane (EA) of one sample on table 3 could be hidden within the weave structure as no specifically stretching parts were found. As the sample contains 2 % of elastane, judging by the label on the sample, detection of it is unreliable.

Lowering the disqualification threshold will make the accuracy of the algorithm higher and raising it decreases accuracy. This is in direct correlation with the purity and homogeneousness of sorted materials and sorted yields of the system. High accuracy labels more samples with impurities, like elastane or blended materials, as failed and this decreases the amount of identified pure fractions.

This test was carried out in order to find out whether the material of black textiles could be identified with the current setup in LAMK. Having such a small quantity of samples cannot determine the actual validity of the method produced. In fact, even to properly prove if the method could work, the number of individual material samples should be increased.

Fine-tuning the identification threshold for optimal accuracy / yield rate is something to be done with a larger sample size. For future testing a proper set of samples is required. The samples need to be validated and contain both colored and black blended and unblended materials. With validated blended materials the threshold can be optimized, and tested, unblended materials are used to test mixed misidentification.

6 CONCLUSIONS

The objective of this thesis was to find improvements for the current pilot textile sorting line. Previously the sorting line could identify pure unblended wool, polyester and cotton textiles out of waste textile streams with a robust identification method, but it did have its limits. The focus was set on the issue of black textiles and how to potentially identify their materials.

Based on a few test runs with a limited sample size of black textiles with a NIR detector, it seems it would be possible to improve the detection of black samples in the sorting line. Most of the scattering caused by the colorants and black color is focused on shorter wavelengths. Demonstrably, NIR technology can differentiate pure cotton, polyester, viscose and wool textiles even at a cut-down wavelength range.

However, further testing is required to increase identification accuracy. There are textiles with small proportions of blended fibers that pass the filter. The method needs to be tested with more verifiable samples of both varying textile blends and monomaterial textiles to determine how much misidentification takes place and to optimize the method.

There is potential in identifying black textiles from textile waste streams even if small quantities of blended materials pass the filter as downcycling mechanical processes may use them. For chemical textile recycling, the homogeneousness of the material is more important and therefore simply cutting a portion of the examined spectra alone is not reliable enough; further development is needed to reach the required accuracy for the benefit of upcycling processes.

Other methods to improve textile identification of the sorting line would come from general machine vision; a camera that can detect enough shades of grey. With high enough resolution the camera could potentially detect the weave of textiles, expanding sorting potential. If, with further testing, it is found that certain weaves conceal blended materials more than others, the identification of it could be used to separate samples with a high risk of hidden fiber contaminants.

Additionally, machine vision can be harnessed to identify shapes of textiles. This opens another avenue for sorting. Sorting textiles into pants, shirts or other garments could be useful at an earlier stage of textiles' lifecycle. Shape recognition can be used to help recycling processes; certain portions of textiles could be identified and cut out. This would be beneficial as pockets, waistlines and collars could be cut out to increase the purity of the sorted material. Additionally, shiny objects and other anomalies in the surface of samples could be detected and their removal automated more accurately.

Dual identification with NIRS and grey-scale machine vision could improve any textile sorting line's accuracy and capabilities. Developing a sorting line capable of separating black samples and / or based on the weave structure for another identification would lower the amount of post-consumer waste streams directed to incineration. Black samples could be directed to their own conveyor belt where their material could be identified with the altered identification method, providing "second-grade" material usable for some recycling processes.

Detection of weaves and shapes of samples would require samples to be fed for the machinery flat and stretched to shape. Addition of such detection capabilities to a NIR material identification line at an earlier stage, for example at the post-consumer collection stage, could cut down labor costs and increase recyclable materials amount.

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ATTACHMENTS

Attachment 1. List of black samples. 100% pure with producer's identifiers.

Attachment 2. List of black samples verifiably 100% pure, but with no producer's identifiers.

Attachment 3. List of black unknown samples gathered from post-consumer waste.

Attachment 1. List of black samples. 100% pure with producer's identifiers.

Black wools samples				
Sample	Producer	Product	g/m2	Weave/Knit
WO1	Pontetorto	SIMPOSIO/B	560	Twill
WO2	Lanificio IL CALICE	APOLLO	570	Twill
WO3	Marcolana	SELLA/D/T	380	Plain
WO4	Marcolana	FUTA/D/T	500	Plain
WO5	Marcolana	BASHIV/D/T	560	Plain
WO6	Marcolana	CALAIS/D/T	600	Plain
WO7	Marcolana	SELLA/V/T	380	Nonwoven
WO8	LEPOUTRE	Gabardine super 100	290	Twill
WO9	LEPOUTRE	Twill aerostretch lavable	310	Twill
WO10	Gottstein fabrics	LA295	350	Nonwoven
Black cotton samples				
CO1	Fortex	Wild/D	480	custom
CO2	Fortex	Wonder	490	custom
CO3	Fortex	Wonder	480	custom
CO4	Texmantova	Terme G/skin	470	Jersey knit
Black polyester samples				
PES1	Deveaus s.a.	Marilor Bi-Stretch	190	custom
PES2	Deveaus s.a.	Marilor Bi-Stretch	195	custom
PES3	Linea 4	active	360	Fleece
PES4	Linea 4	active	380	Fleece
PES5	Linea 4	active	170	Rib knit
PES6	Linea 4	active	140	Rib knit

Attachment 2. List of black samples verifiably 100% pure, but with no producer's identifiers.

SAMPLES VERIFIABLY 100% PURE, NO LABELS / IDENTIFIERS		
Wool samples		
Sample	Weave/Knit	Additional remarks
WO11	Twill	
WO12	Twill	
WO13	Twill	
WO14	Twill	
WO15	Plain	Woolen jacket
WO16	Plain	
WO17	Twill	
WO18	Plain	Windowpane pattern
WO19	Twill	
WO20	Twill	
Cotton samples		
CO5	Tricot	
CO6	Rib knit	
CO7	Tricot	
CO8	Twill	
CO9	Rib knit	
CO10	Interlock	
Polyester samples		
PES7	Plain	
PES8	Plain	Floral pattern
PES9	Fleece	
PES10	Purl knit	

Attachment 3. Black unknown samples.

BLACK POST-CONSUMER "WASTE" TEXTILES			
Sample	Weave/Knit	Additional remarks	Label (if any)
UK1	Jersey	beanie	100% Cotton
UK2	Twill	Jeans	92% Cotton 2%EA 6% PES
UK3	Jersey	Sleeve of a hoodie	100% Cotton
UK4	Interlock	Turtleneck shirt	100% PES
UK5	Double knit	Sock	100% cotton
UK6	Plain	Bedsheet	100%PES
UK7	Tricot	Unknown cutout	
UK8	Plain	Unknown cutout	
UK9	Sateen	Unknown cutout	
UK10	Twill	Unknown cutout	