

Optical sorting technology for textile waste

Development of an identification method
with NIR spectroscopy

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ABSTRACT

This thesis deals with developing a material identification method for textile waste using near infrared (NIR) spectroscopy. The process involved collecting and validating reference samples, measuring and pre-treating NIR spectra, and setting up parameters for matching spectral patterns. The goal is to achieve a robust and reliable method for sorting unblended cotton, polyester and wool from a heterogeneous textile waste stream.

Sorting technology was developed as a part of a multidisciplinary initiative Telaketju, which aims to increase recycling rates and resource efficiency in the textile industry in Finland. The research was carried out in the Lahti University of Applied Sciences with a ProFOSS NIR process analyzer, which was equipped with Metrohm Vision™ software for spectral data management.

Based on the study, it can be stated that NIR spectroscopy is capable of identifying textile materials quickly and accurately based on their characteristic spectra. The identification method requires a standardized measurement distance, corrections for varying physical properties among the samples and identification tolerances that allow natural spectral variation but restrict impurities.

The research for this thesis advances the recycling possibilities of textile waste, but can also be applied to different material segments. This thesis offers theoretical background for spectroscopic measurements and guidelines for developing the identification method to cover a broad range of textile materials.

Key words:

NIR, spectroscopy, textile waste, data pre-treatment, material identification, recycling

Lahden ammattikorkeakoulu
Materiaalitekniikan koulutusohjelma

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TIIVISTELMÄ

Opinnäytetyön tarkoituksena oli tutkia tekstiilimateriaalien koneellisen tunnistuksen mahdollisuuksia ja kehittää lajittelutekniikkaa lähi-infrapunaspektroskopian (NIR) avulla. Kehitysprosessi koostuu referenssinäytteiden valikoimisesta ja validoinnista, näytteiden mittauksesta ja spektrinkäsittelystä sekä spektrien muodontunnistuksen parametrien määrittämisestä. Tavoitteena oli kehittää kattava ja luotettava metodi puuvillan, polyesterin ja villan erottelemiseen heterogeenisestä poistotekstiilivirrasta.

Lajittelutekniikkaa kehitettiin osana monialaista Telaketju-hanketta, joka pyrki edistämään tekstiilien kierrätysastetta ja tekstiiliteollisuuden materiaalitehokkuutta Suomessa. Tutkimus suoritettiin Lahden ammattikorkeakoulussa ProFOSS NIR-prosessianalysointilaitteella sekä spektrinkäsittelyyn ja datan hallintaan soveltuvalle Metrohm Vision™-tietokoneohjelmistolla.

Työssä todistetaan NIR-spektroskopian soveltuvuus tekstiilimateriaalien nopeaan ja luotettavaan tunnistukseen. Tunnistusmetodin kehitys vaatii mittausetäisyyden vakioimisen, näytteiden fyysisten ominaisuuksien matemaattisen korjauksen sekä tunnistustoleranssin, joka sallii luontaista vaihtelua spektriarvoissa mutta hylkää epäpuhtaudet tehokkaasti.

Tehdyn tutkimuksen avulla voidaan edistää tekstiilien kierrätettävyyttä, minkä lisäksi tekniikka on sovellettavissa myös muiden materiaalien tunnistukseen. Opinnäytetyö tarjoaa teoreettisen pohjan spektroskooppisiin mittauksiin sekä opastuksen automaattisen materiaalintunnistuksen kehittämiseen.

Asiasanat:

NIR, spektroskopia, tekstiilijäte, spektrinkäsittely, materiaalintunnistus, kierrätys

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1 INTRODUCTION

As the world's population growth and global standards of living increase, there is a well argued concern on the sufficiency of the world's material resources. Although there has been significant improvement in recycling technologies and resource efficiency, many complex material constructions lack the standards and methods for efficient recycling. One such material segment is textiles, which exhibit a large variety of chemical and physical constructions, and thus present a challenge in recycling.

While there are already viable recycling methods (mechanical, thermal and chemical) for many types of textiles, there is a lack of market demand and manufacturers utilizing recycled post-consumer textile fibers. This is partly because of deficiency of reliable and cost-efficient practices for collecting and sorting a heterogeneous mass of textile waste. (VTT 2017.)

According to a research by the Finnish Environment Institute (2013-2015), 71.200 tons of textiles are discarded annually in Finland alone. Only 30% of this amount is channelled to re-use and recycling, while 70% is mainly incinerated. After a landfill ban in Finland for organic material in 2016, the need for alternative solutions for textile recycling has increased significantly. (SYKE 2015.)

TELAKETJU-initiative is a multidisciplinary consortium, which aims to develop a nationwide network to support circular economy of textiles in Finland. The network should cover all stages of the recycling value chain from the collection of textile waste from consumers, to sorting and processing, and eventually to the market with new recycled textile products. As a part of the initiative, this thesis aims to develop a method for sorting textile waste using near infrared (NIR) spectroscopy.

1.1 Scope

This thesis demonstrates the process of developing a material identification model with NIR spectroscopy. The process involves:

1. Selection and evaluation of reference samples
2. NIR measurements and pre-processing of spectra
3. Development and testing of the identification model.

Confirmed sample materials for library construction are evaluated by their spectral features and corresponding physical properties. Possible spectral outliers are noted and ruled out of the identification method. The experimental section of the thesis deals with optimizing spectral pre-treatment methods and the mathematical pattern recognition features for reliable material identification. The focus of the thesis is in qualitative analysis of textile materials, but a concise introduction on quantitative analysis (determining textile blend percentage) is given for future development purposes.

1.2 Contents

Theoretical background on the fundamentals of vibrational spectroscopy and different measurement technologies is included, as well as a description of the NIR analysis instrument and spectral processing software used in developing the model. The possibilities and limitations of NIR-identification are evaluated in terms of accuracy and efficiency.

Final results for data pre-processing and identification method parameters are excluded from the public version of the thesis. However, all the available mathematical tools included in the Vision™ software and their effects are presented.

2 TELAKETJU-INITIATIVE

The Telaketju-initiative works to improve sustainable practices for collection, sorting and utilization of discarded textiles in Finland. Telaketju brings together research facilities and industrial companies to share views and know-how in the textile recycling industry. The project is co-managed by the Finnish national technological research institute VTT and a communal waste management company LSJH. Notable collaborative partners include waste collection and sorting companies (Fida, Remeo, Suez), research partners (Turku UAS, Lahti UAS) and industrial companies - ranging from clothing and other fiber products (Pure Waste Textiles, Soften, Finlayson) to IT-systems and robotics (MJV-Sähkö Oy, ZenRobotics). (VTT 2017.)

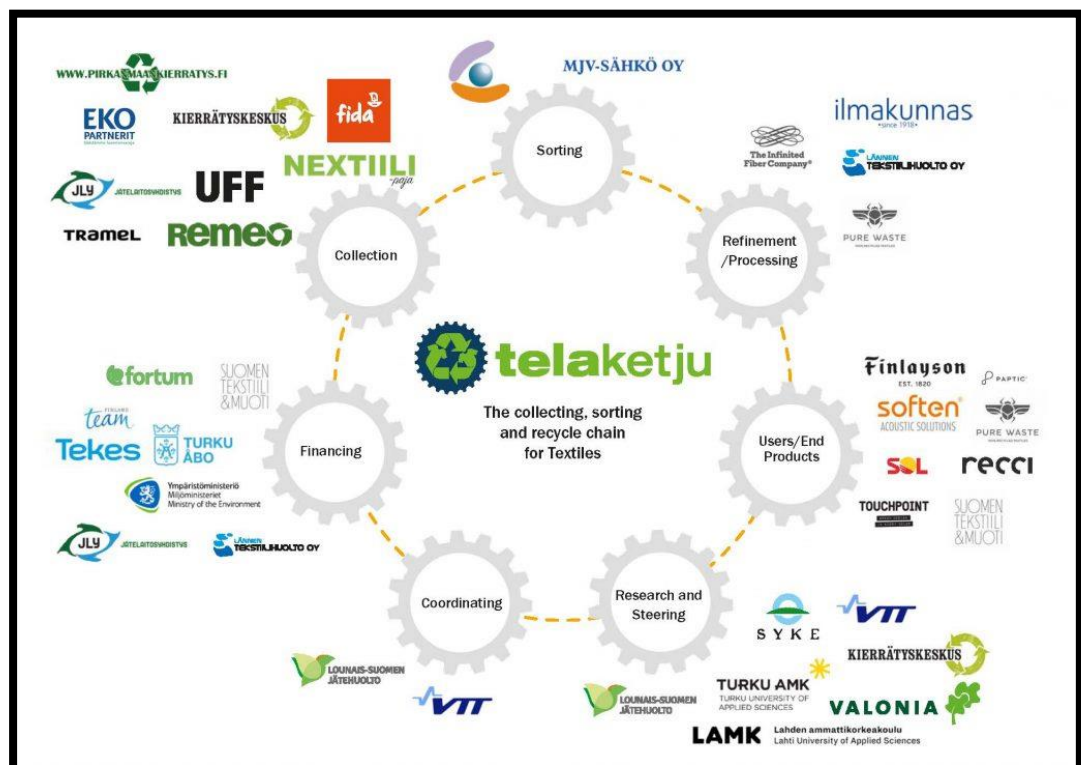


FIGURE 1. Collaborative partners of Telaketju-initiative. (VTT Extranet)

Telaketju aims to build an efficient infrastructure around the whole value-chain in the textile industry. As seen from Figure 2, the objective includes development in collection and logistics, material processing and refinement, innovative new practices and new business models involving fibrous products. Examples of such (already achieved) development are

e.g. chemical recycling of cotton using a carbamate solution (VTT) and utilization of denim production scraps as a raw material for t-shirts (Pure Waste Textiles). The Telaketju-initiative is set to continue until September 2018. (VTT 2017.)

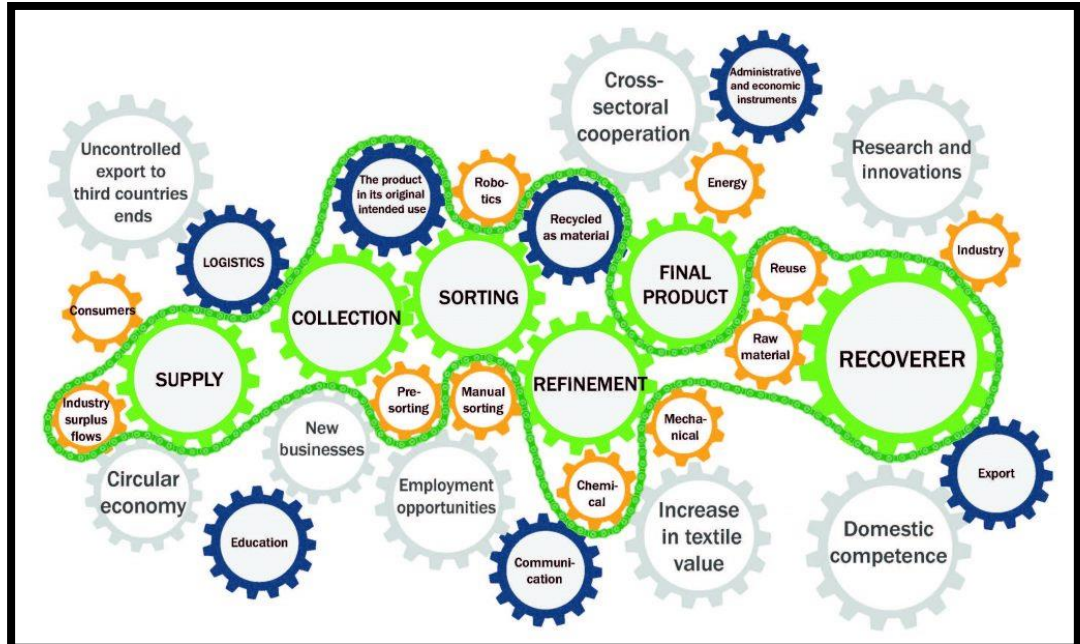


FIGURE 2. Functionality model and “building blocks” of Telaketju-initiative (VTT extranet)

2.1 Automated sorting

Discarded textile waste can be re-used in its original form or converted to other products using mechanical or chemical recycling. Both methods require accurate knowledge on the chemical and structural composition, to ensure efficient recycling processes and desirable properties for the end products. Clothing products usually lack adequate or reliable information on the material composition, so it is important to develop methods for determining constituents in the textile waste stream. Lahti University of Applied Sciences, the commissioner of this thesis, is responsible for evaluating the feasibility of NIR spectroscopy as a fast and reliable sorting method.

2.1.1 REISKAtex® sorting unit

Lahti University of Applied sciences has built a pilot phase sorting unit to develop an automated sorting method for discarded textiles. The unit (Image 1) includes a conveyor belt line, an NIR process analyzer and pressurized air deflectors to sort recognized materials to assigned bins. As the articles are fed onto the conveyor belt, the online NIR spectrometer acquires a spectrum, which is then evaluated to a reference library to determine if it fits the pre-established criteria. Each article is tracked by a positioning sensor, which enables deflection to the correct material bin. The sorting unit is equipped with three separate bins for different materials, while the unrecognized stream goes through the line without deflection.

NIR spectroscopy was chosen as the primary analysis method because of its established position in quality control in process industry. NIR technology has applications in the textile industry, ranging from fiber identification, moisture analysis and quality control. NIRS is utilized by manufacturers, retailers, testing facilities and customs officials. (Rodgers & Beck 2009.) Details on the advantages and limitations for using NIR are given in Chapter 4. Inspiration for the automated sorting unit ReiskaTex has come from a Dutch enterprise Valvan Baling System, which uses a similar NIR instrument to sort fabrics by fiber type for recycling. The Fibersort-unit by Valvan can identify one piece per second (Valvan 2017) and REISKAtex® aims for at least the same efficiency. The mechanical and electrical design, logical programming, hardware construction and analytical features have been included in educational projects within the Lahti University of Applied Sciences.



IMAGE 1. Textile sorting unit, REISKAtex® (Photo by Jaakko Zitting)

3 VIBRATIONAL SPECTROSCOPY

Spectroscopy uses light, or more specifically electromagnetic radiation, to analyze materials based on their interaction and the transfer of energy. EM radiation can be depicted as a continuous wave propagation of fluctuating electric and magnetic charges, perpendicular to each other. The energy of EM radiation correlates with its wavelength and frequency, which are inversely proportional. Since the speed of light in vacuum is constant (approx. $2,997 \cdot 10^8$ m/s), an increase in wavelength causes a decrease in frequency. Light can also be viewed as a stream of energetic particles, photons. The energy of a photon is proportional to frequency:

$$E = h\nu \quad (1)$$

Where E = Energy [J]
 h = Planck constant, $6.626 \cdot 10^{-34}$ J·s
 ν = Frequency (1/s)

(Jaarinen & Niiranen 2009, 46-48.)

At temperatures above 0 K, atomic bonds vibrate with different intensity and coordination depending on the difference in mass and electronegativity between the atoms, as well as the molecular structure. Many organic molecular bonds absorb radiation energy at the same frequency as their characteristic vibrational state. This phenomenon can be used to identify materials by their characteristic absorption spectrum, which is obtained by measuring the absorption of radiation at different vibrational levels. As radiation energy in the IR-region is absorbed, the changes in molecular vibration and their dipole moment can be observed through spectroscopic instruments. Possible molecular vibrations consist of stretching and bending (Figure 3), and the number of possible vibrational patterns depend on the number and orientation of atoms in a molecule. Stretching vibrations cause internuclear distance to alter and bending/wagging vibrations cause angular rotation and the atoms to deflect from the planar normal state. (Stuart 2004, 2-8.)

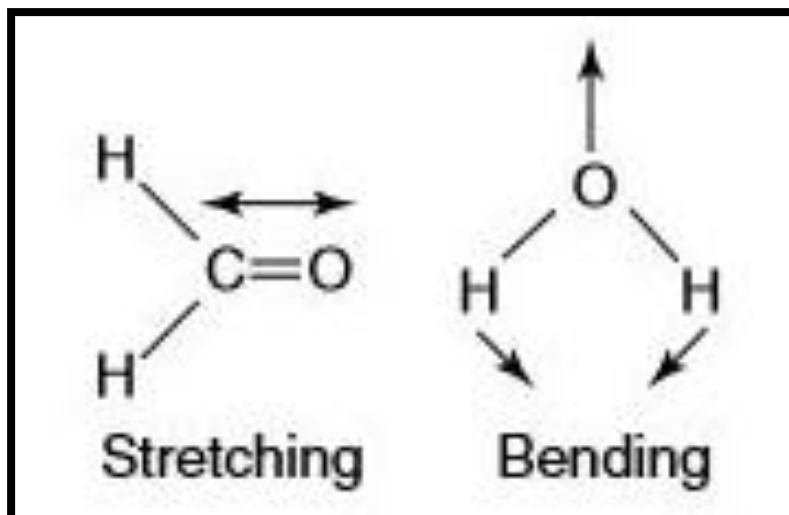


FIGURE 3. Two main types of molecular vibrations (Stuart 2004)

3.1 EM spectrum and IR frequencies

Visible light constitutes only a small fraction of the electromagnetic spectrum (Figure 4). Radiation bands with greater frequency, and thus higher energy than visible light, are divided into ultraviolet-, x-ray- and gamma-radiation. Lower frequencies consist of infrared-, microwave- and radio wave -bands. In analytical chemistry, the most valuable spectroscopic phenomena are electronic excitation in the UV-region (190-400 nm), nuclear magnetic resonance in the low-energy radio wave -region, and vibrational absorptions in the IR-region (800-25000 nm). The IR-region is further divided into near-IR (800-2500 nm) and mid-IR (2500-25000 nm), each with different characteristic features. (Jaarinen & Niiranen 2009, 46-49.)

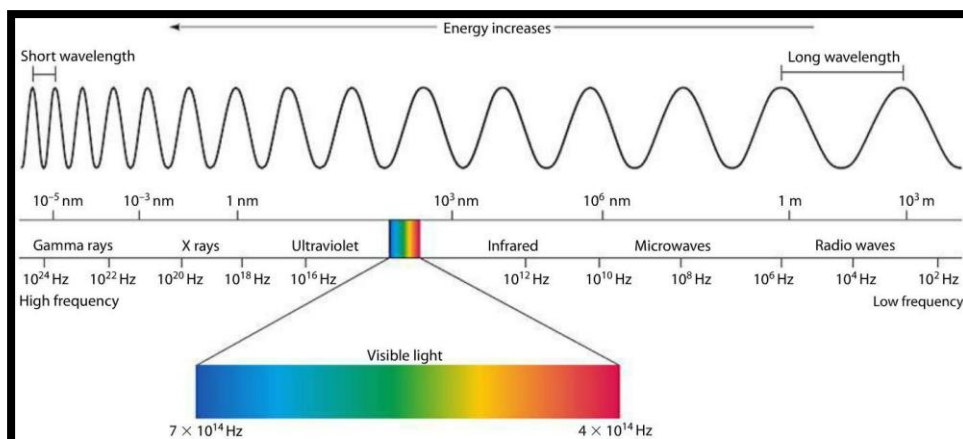


FIGURE 4. Regions of EM-spectrum

(https://www.miniphysics.com/electromagnetic-spectrum_25.html)

The infrared region of the EM spectrum, and its lower frequency range at 7000-20000 nm, is sometimes called the “fingerprint”-region, because it produces unique spectra from a vast variety of materials in different phases. The mid-IR region exhibits strong absorption peaks, which correspond to bonds and functional groups usually present in organic molecules. According to a *selection rule* for infrared spectroscopy, a bond must show variance in its dipole moment when vibrating between two extreme states. Most functional groups in organic molecules are not centrosymmetric and can thus produce an absorption spectrum. (Williams & Fleming 1989, 29-32.)

3.1.1 Fundamentals and overtones

Molecular vibrations interact not only with one specific frequency, but with the multiples of that frequency. The strongest absorptions occur at the so called *fundamental* frequencies at longer wavelengths, but bands called *overtone*s are also visible at higher frequencies. Due to anharmonic nature of molecular vibrations, the exact overtone bands of specific bonds are hard to determine, but multiplying frequencies with integers yields a good approximation for lower overtones. The intensity of absorptions at overtone bands decreases substantially, which makes higher overtones difficult to detect. (Nilsson 2017.) An example of absorption bands and

their relative intensities on a simple C-H stretching vibration is given in Table 1:

TABLE 1. Absorption bands of C-H stretch (adapted from: Workman 2014)

Absorption band	Wavelength region [nm]	Relative intensity
Fundamental (n)	3380-3510	100
1st overtone (2n)	1690-1755	1
2nd overtone (3n)	1127-1117	0.1
3rd overtone (4n)	845-848	0.01

Infrared-active, vibrating molecules can be viewed as a spring connecting two masses with a dipole moment. The normal state vibrational frequency, which corresponds to the bond elasticity and atomic electronegativity, can be calculated by applying the Hooke's law:

$$\nu = \frac{1}{2\pi} \sqrt{\frac{K}{\left(\frac{1}{m_1} + \frac{1}{m_2}\right)}} \quad (2)$$

Where ν = characteristic vibration frequency of a specific bond
 K = force constant of a specific bond
 m_1 and m_2 = masses of bonded atoms

(Workman & Weyer 2008, 12-14.)

Overtone bands are not exact multiples of fundamental vibration bands because the intermolecular forces cause changes in the force constant. As a result of intermolecular forces and resonances between adjacent vibration bands, overtone bands can overlap and shift in position. (Reich 2005, 2.) This phenomenon is strongly present in the NIR region and is further described in Chapters 4.1 and 4.2.

3.2 Components in the IR spectrum

An IR-spectrum is commonly visualized as a graph with wavelength [λ , nm] on the x-axis, and absorbance on the y-axis. Another popular format in spectroscopy is to use wavenumber [$\bar{\nu}$, cm^{-1}] (instead of wavelength in nm), which is the reciprocal of a distance unit. It represents the number of wavelengths within one centimeter distance. Wavenumbers are linear with radiation energy and frequency, and are therefore intuitive units for spectroscopy. Mid-infrared spectroscopy uses mainly wavenumbers while near-infrared uses commonly wavelength in nanometers. (Workman 2014)

IR measurements can be made either in reflectance or transmission modes. Absorption at each wavelength band is measured by comparing the intensity of light reflected from the sample, against the initial intensity from the radiation source. The ratio of reflected and initial light intensity is then converted to a unitless absorption value (Formula 3), which is then plotted in the absorption spectrum. The absorption value in transmission measurements can also be calculated if the material-specific *absorption coefficient* and analyte concentration is known, or vice versa. This equation (Formula 4) is known as the Beer-Lambert law, and can be used in quantitative analysis. (Jaarinen & Niiranen 2009, 50-54.)

$$A = \log\left(\frac{1}{I/I_0}\right) \quad (3)$$

Where A = absorbance [unitless value]
 I = measured light intensity reflected from the sample
 I_0 = initial intensity of light from the radiation source

$$A = \varepsilon \cdot c \cdot b \quad (4)$$

Where A = absorbance [unitless value]
 ε = material-specific absorption coefficient [$\text{l} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$]
 c = concentration of analyte [mol/l]
 b = sample thickness in transmission measurements [cm]

3.3 Qualitative analysis

Qualitative analysis of IR spectra is based on pattern recognition techniques. One method is to look for a limited amount a known absorption bands corresponding to a material of interest. This method can be used in mid-IR spectroscopy, where the absorption bands are strong and well resolved. Matching specific absorption peaks and their amplitudes can give a close approximate identification on the analyzed material but can miss certain characteristics and thus yield a misidentification. Pattern recognition of the whole spectral range is done by comparing the spectra to known samples in an identification library. Chemometric tools for pattern recognition range from simple correlation- and distance-based methods to a more complex but statistically robust principal component analysis (PCA), linear discriminant analysis and factorial discriminant analysis. (Manley et. al. 2008, 77-79.)

A closer description on the qualitative tools available in Vision™ software (PCA, spectral correlation analysis and amplitude distance restrictions) are given in Chapter 5.4. Figure 5 illustrates the principle and tolerances set for spectral pattern analysis.

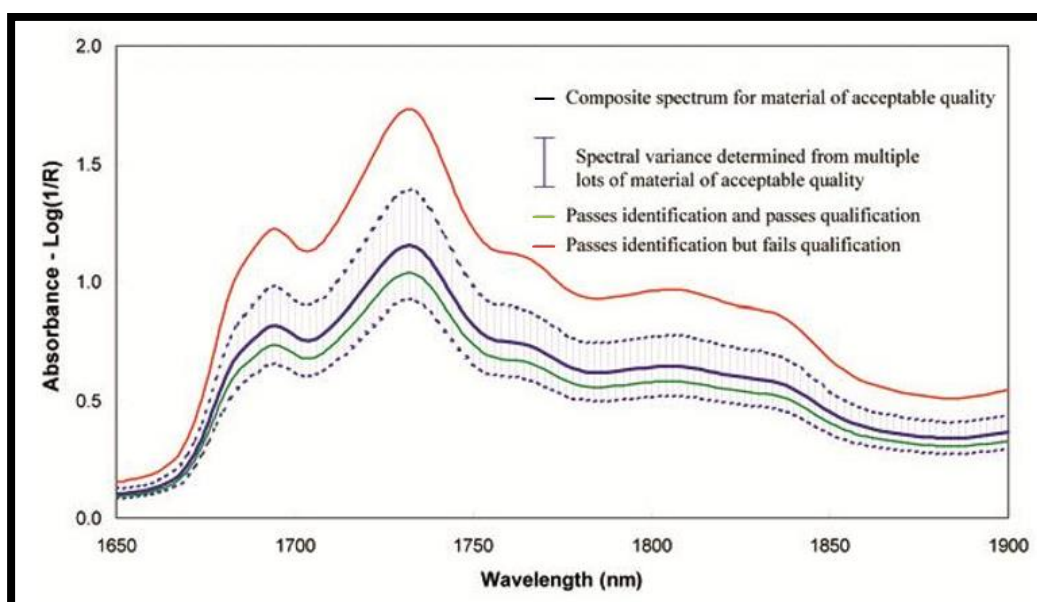


FIGURE 5. Visual portrayal of the principle in qualitative analysis (Metrohm 2013a)

3.4 Quantitative analysis

The percentages of an analyte (constituent) of interest in a sample can be determined by quantitative analysis of IR spectra. Most common methods include principal component regression (PCR) and partial least-squares (PLS) regression. Quantitative model development requires reference samples with a range of known percentages of constituents. Matching correlations in spectral patterns with constituent values enables calibration for a regression model, which can predict values of unknown samples. (Reich 2005, 7.)

In the case of blended textiles, a study by Rodgers & Beck (2009, 7-11) proved a possibility to determine the cotton content in CO/PES-fabrics. Finding a correlation in absorbance values to cotton content enabled them to build a regression model based on least-squares regression. The calibration yields a regression coefficient of 0.977 with standard error of prediction at just 2.9%.

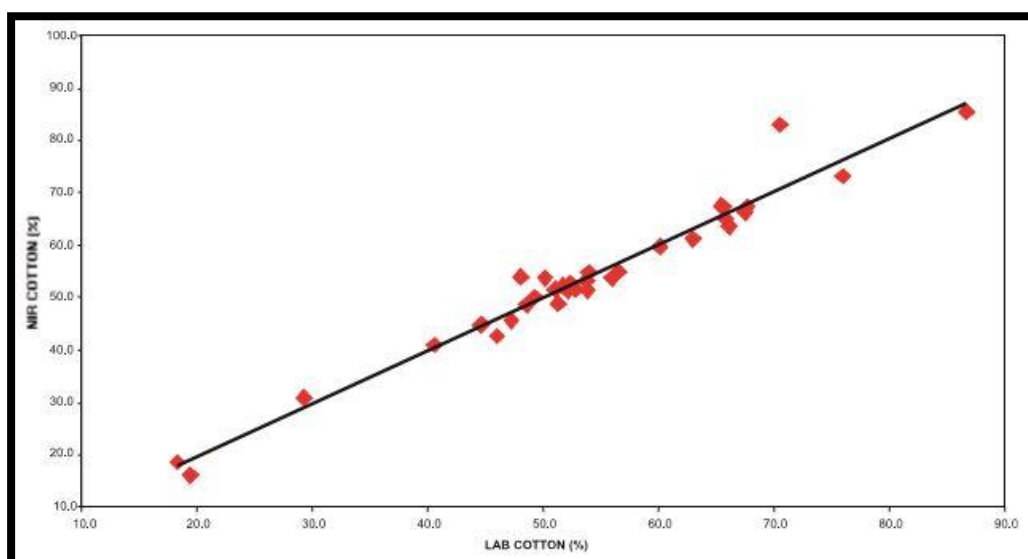


FIGURE 6. Correlation between known cotton content and calibrated prediction values in qualitative NIR analysis (Rodgers & Beck 2009)

4 NEAR-INFRARED SPECTROSCOPY

For its extensive advantages as a fast and non-destructive analytical technique, NIR spectroscopy has been utilized in many industrial applications. As there usually is no need for sample preparation, it is optimal for continuous quality control. Notable applications for NIR can be found within petrochemical and polymer manufacturers, pharmaceutical and cosmetics companies, border control and health inspection officials, and environmental research facilities. (Pasquini 2003; 2-3, 17.)

4.1 Fundamentals of NIR spectroscopy

Despite the significant advantages from being simple, reproducible and chemical-free, NIR spectroscopy is considered a secondary method compared to traditional analytical techniques, such as gas chromatography (GC) or nuclear magnetic resonance (NMR). This means that NIRS is dependent on primary reference methods in order to build identification models or quantitative calibrations. However, once these models and calibrations are done, NIRS can be used as a primary analysis method in the intended field of application. (Manley 2014, 2-3.)

The near-infrared region extends from 800 to 2500 nm ($12500\text{-}4000\text{cm}^{-1}$), adjacent to the visible light region of the EM spectrum. As seen from the Figure 7, the NIR region is sensitive to X-H bonds (e.g. C-H, O-H, N-H), as hydrogen produces always a significant dipole moment. NIR spectra contain a lot of information because many absorption bands occur up to six times within the spectral region. NIR is efficient in analyzing a variety of organic materials, such as proteins, carbohydrates and oils. Water is strongly absorbing in the NIR region, which sometimes hides other spectral features. The effect of strong O-H absorptions can be removed from the spectra by mathematical manipulation, or it can be utilized in moisture analysis of e.g. agricultural products. (Eremina 2017.)

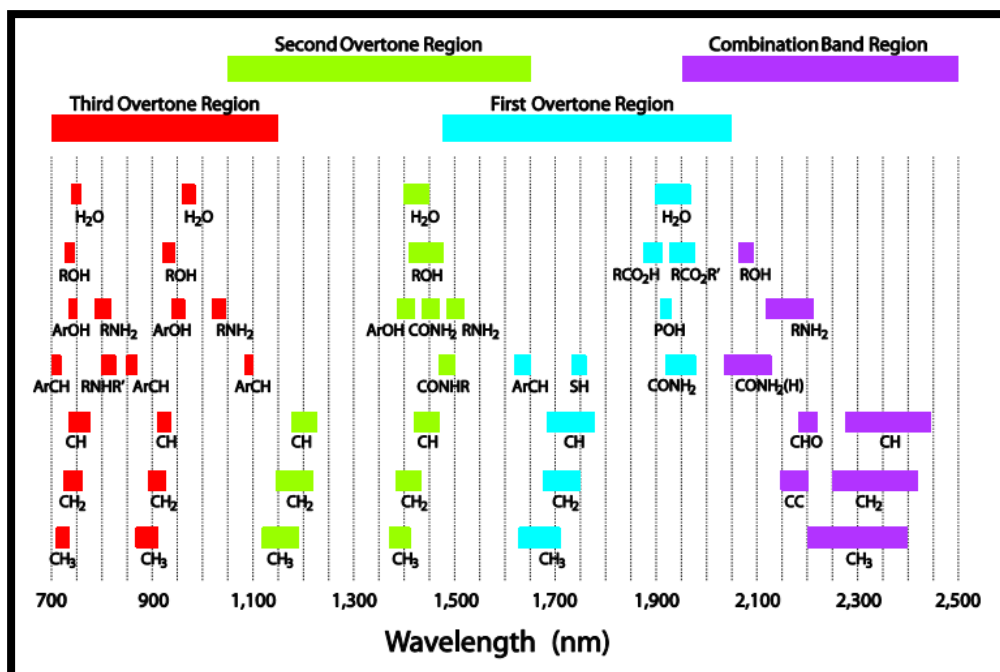


FIGURE 7. Absorption bands in the NIR -region (Metrohm 2013a)

4.2 Common features of NIR spectra

While the mid-infrared region contains fundamental bond vibrations in most organic substances, the NIR region comprises of overtones and combinations of these frequencies. NIR absorption bands are typically broad, overlapping and up to 10-100 times weaker than the fundamental absorptions in mid-IR. The weaker signals in NIR measurements require chemometric data processing to extract information from the spectra. However, weaker absorption allows for greater measurement depth and distance, as well as analysis of highly absorbing or scattering materials without sample preparation. (Reich 2005, 3-4.)

Differences in typical pure cotton and wool spectra from MIR and NIR instruments can be seen in Figures 8 & 9. It is clear that the well-defined peaks in MIR enable visual identification of bonds and functional groups, while NIR spectra must be analyzed with chemometric tools. NIR is not optimal for detecting individual molecular bonds, but has proven efficient in qualitative and quantitative analysis based on pattern recognition.

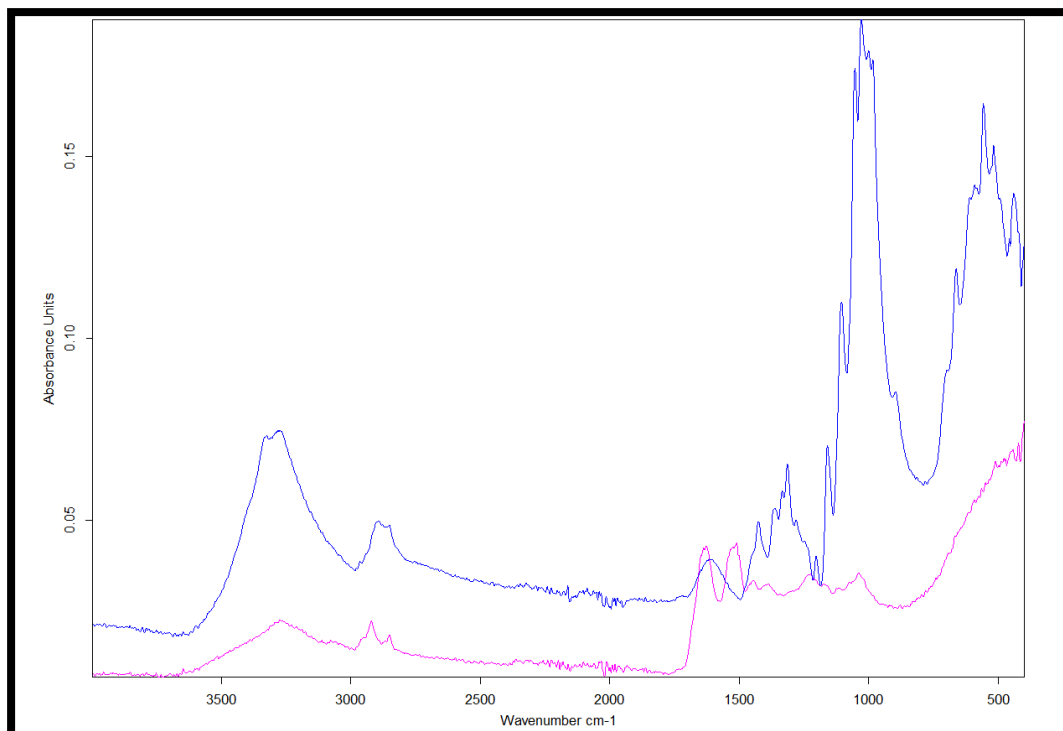


FIGURE 8. MIR spectra of 100% cotton (blue) and 100% wool (purple).
(Capture from Bruker OPUS™)

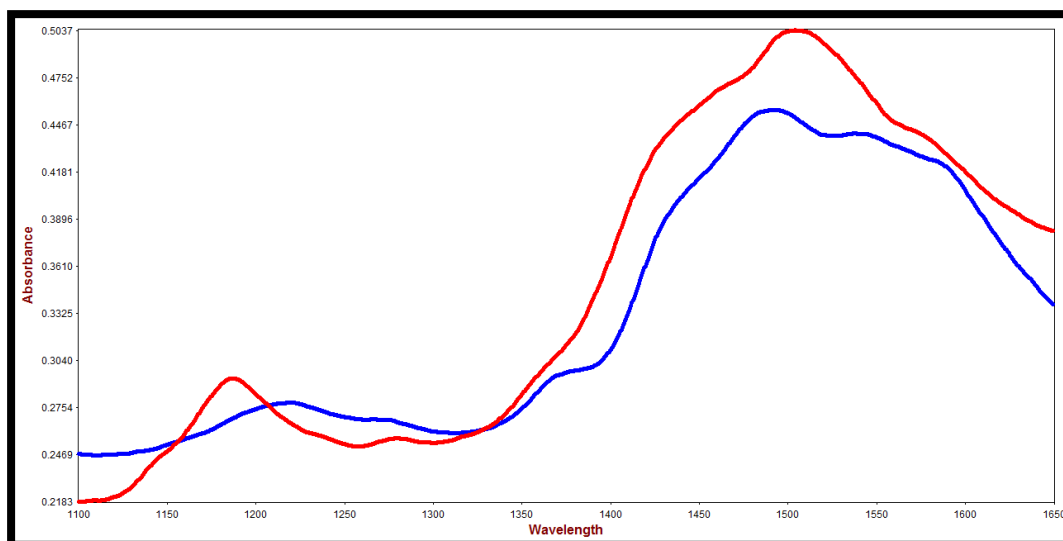


FIGURE 9. NIR spectra of 100% cotton (blue) and 100% wool (red).
(Capture from Vision™)

4.3 Instruments and software

Research for this thesis was conducted on an NIR process analyzer by FOSS Analytix, which was equipped with a spectral manipulation software Vision by Metrohm. Danish FOSS analytix specializes in

providing advanced analytical tools for the agricultural industry, including a wide range of spectroscopic instruments for both laboratory and process conditions (FOSS 2017). Swiss manufacturer of analytical tools and laboratory systems Metrohm is collaborating with FOSS by offering its spectral processing software Vision™ along with an extensive marketing network.

4.3.1 NIRS Analyzer Pro

The NIR process analyzer (Image 2) used in REISKAtex™ sorting unit is equipped with a high-resolution InGaAs diode array and provides non-destructive spectral analysis of solids, liquids, granules or pastes. The model uses a spectral range of 1100-1650 nm, which includes most first and second overtones of common organic molecular bonds. The measurement time ranges from 5-50 ms depending on the assigned accuracy and measurement range. The analyzer can be mounted on a production line or fitted into a pipeline. The instrument is designed to withstand temperatures up to 150 °C and the sapphire lens endures pressures from near vacuum to 200 bar. The process instrument model variants include a possibility to take measurements with a hand-held fiber optic probe. (Metrohm 2013b.)

With default settings, the instrument measures the absorbance values at 0.5 nm wavelength intervals covering the whole measurement range. Optionally, the user may limit the measurement range to a specified wavelength band of interest. (Metrohm 2015a.) NIR spectra are plotted with the absorption values as a function of wavelength in nanometers. The resulting absorbance spectrum is displayed and analyzed in the Vision™ software. The absorbance spectrum can also be converted into a standard format excel-sheet for closer inspection.



IMAGE 2. NIR Analyzer Pro (Photo by Jaakko Zitting)

4.3.1.1 Calibration

Before acquiring the data to be used in model development, the instrument must be calibrated for internal error corrections. *Reference standardization* creates a virtual correction file to account for variations in internal instrument responses. Without standardization, the identification method may not be fully transferable to other NIR measurement units. The calibration is done by measuring a photometric standard of known reflectivity and comparing that to the instruments measurement. Foss Analyzer PRO is equipped with a non-absorbing Spectralon® -disk (Image 3) as a reference standard. The difference in measurement response is used to correct all measurements on different NIR instruments. (Metrohm 2015b, 24.)

Performance test assures reliable functionality of the instrument. Testing includes measurements of max/min background noise, errors of

communication (EOC), linear bias of interference peaks and the root-mean-square (RMS) of peak-to-peak noise (Metrohm 2013a, 53-54). Proper instrument response is verified on the performance test report (Attachment 1).



IMAGE 3. Spectralon®-disk for reference standardization. (Photo by Jaakko Zitting)

4.3.2 Metrohm Vision™

Vision™ by Metrohm is a spectral processing software that offers tools for measuring and managing data, as well as qualitative and quantitative analysis for spectroscopic instruments. It is built around four fundamental modes:

1. Data acquisition mode:

Combines all functions related to spectral measurements, data pre-processing and data management. Spectra can be viewed as a graphical XY-plot or converted to an excel-sheet. Instrument diagnostics and system settings are also done in data acquisition mode.

2. Qualitative analysis mode

Includes functions for sample selection, identification method development and library validation. Parameters for spectral pattern recognition are done in qualitative analysis mode.

3. Quantitative analysis mode

Enables quantitative regression model development for predicting constituent values and percentages.

4. Routine analysis mode

Identification and quantification of unknown samples are done in routine analysis mode. This mode enables analysis of stored data or real-time measurements. Analysis reports are created in routine analysis, and can be saved to results storage database.

(Metrohm 2015a, 11-12.)

The user can store spectra in separate *projects*, which act as a fundamental storing unit. Identification libraries are built around a specific project and changes in other data sets do not alter the models created with other projects. Different materials and their spectra are divided into *products*, which determine the final identification result. Calculations for average spectrum or standard deviation within a product are possible in data acquisition mode.

Vision™ recognizes different user profiles, which can be given access to different functionalities. The *system manager* –profile has authority to all settings and model development tools while the *system user* –profile can be restricted to just routine analysis operations. (Buytaert 2016.)

5 SAMPLE LIBRARY CONSTRUCTION

An automated sorting unit with an NIR analyzer must be equipped with a sample library. Once a reliable reference library with desired materials is established, the system can identify unknown materials and sort them for further recycling processes. Pure cotton (CO) and polyester (PES) were chosen for library development because of their large volumes and widespread use in textile industry. Wool (WO) was also included because of its value and potential in recycling applications.

5.1 Choosing reference samples

5.1.1 Initial sample selection

Textile samples for library development were gathered from the inventories of Lahti University of Applied Sciences and sorted garments from recycling centers (Patina, Fida, LSJH). Sample sets included both commercial samples from textile manufactures as well as post-consumer textiles (Image 4). This was to assure sufficient representability, as samples only from manufacturers would lack the real-life characteristics of textiles with contaminants from e.g. grease, dirt and washing detergents. Sample sets should contain natural variations in textile properties, such as color, texture and fabric composition. However, selected samples must be relatively pure as to not interfere with spectral measurements.



IMAGE 4. Pre-consumer and post-consumer samples. From left to right: Wool, Cotton, Polyester. (Photo by Jaakko Zitting)

5.1.2 Verification of sample purity

All samples were verified with an FT-IR laboratory spectrometer (Bruker Alpha [ATR], Image 5) to rule out impure samples and textile blends. Commercially available identification libraries can easily compare known materials to the measured spectra (EssentialFTIR 2017). Should this library search not provide unambiguous results, the spectra were cross-checked with raw fiber samples or standard fabrics (e.g. James Heal, polyester adjacent fabric, as specified in ISO 105-F04). Only verified textile samples of known material composition were accepted as reference samples for identification model development.



IMAGE 5. FT-IR measurements for purity verification. (Photo by Jaakko Zitting)

5.2 Measurements and manual sample selection

No amount of data pre-treatments or chemometrics can compensate for bad data. Therefore, the measurement method and resulting spectra has to be carefully analyzed for errors before spectral manipulation. (Nilsson 2017.)

All reference samples were measured twice (both sides) to eliminate random outliers from measurement conditions. Maximum measuring distance was defined by evaluating the increasing spectral noise from a single sample, along with increasing distance at approx. 10 mm intervals. Normalizing spectral baseline shifts with measurements at under 30 mm distance resulted in insignificant variations in noise levels. This distance can thus be regarded as a maximum measuring distance with the analyzer in use. The differences in the noise levels can be seen in Figure 10. Samples were also measured flat and wrinkled, with no noticeable difference in absorption levels.

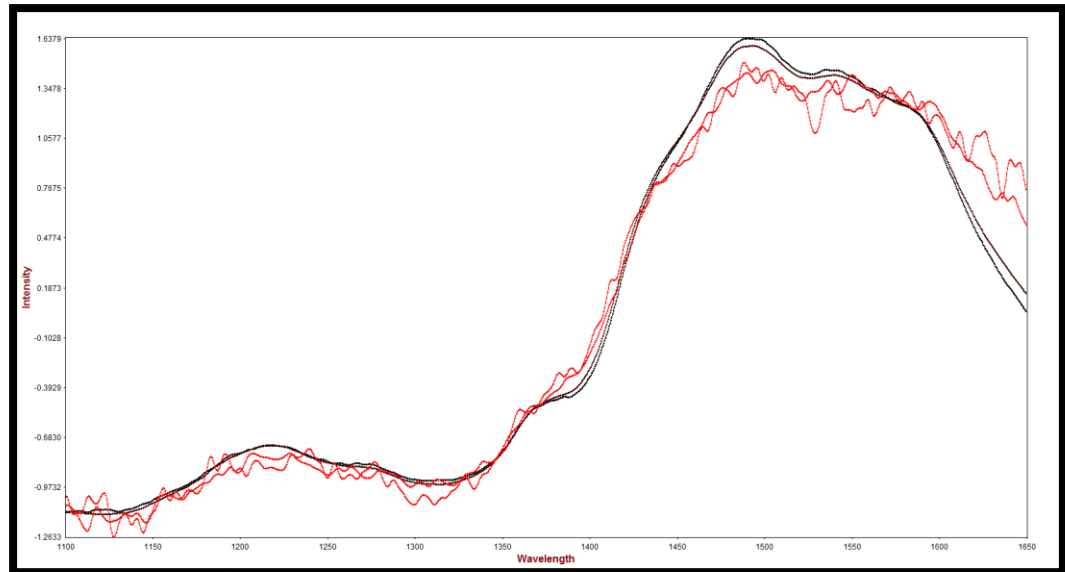


FIGURE 10. Measuring distance vs. noise. Black spectra measured at 30mm and in contact, red spectra at 60 mm and 100 mm. Cotton 100%. (Capture from Vision™)

5.2.1 Outliers and accepted variations

Spectra from sample sets was first evaluated visually to rule out clear outliers. All product sets showed three types of spectra within a single material. Along with clear material spectra and noticeable outliers, some noisy/shifted spectra were visible from the sample sets. Calculations of standard deviation, with and without the variate spectra, were compared to the peak-to-peak scale of the corresponding sample set. The results (Table 2) show 35-71% improvement in the score given to the products. Clear spectra were denoted with reference “a” and sets with slight variates as “b”. Despite the significant improvements in the sample set score, the shifted spectra were not excluded from identifications sets. This was to ensure natural spectral variation within the identification library product. The spectra denoted with “b”-mark, and the calculated deviation levels, were used to define correct tolerances for the identification method. While the denoted variate spectra could be excluded from the identification set, it is simpler to adjust threshold values later on if needed.

TABLE 2. Scores for sets of spectra

Material	Max deviation	P-P	Score	Difference
CO100-a	0.059	2.862	0.020	-34.7 %
CO100-b	0.095	3.038	0.031	
PES100-a	0.247	8.849	0.028	-48.2 %
PES100-b	0.515	9.554	0.054	
WO100-a	0.107	3.152	0.034	-70.8 %
WO100-b	0.379	3.266	0.116	

5.3 Pre-treatment of spectra

The objective of spectral pre-treatments is usually to remove effects of physical phenomena from the data set in order to make chemical analysis more reliable. The two main types of mathematical pre-treatment are scatter correction and spectral derivatives. (Rinnan et al 2009, 1.)

NIR reflectance measurements exhibit both diffuse and specular reflections. Specular reflections do not contain any chemical information, but can often be excluded with optimal instrument design and measurement conditions. Diffuse reflections carry information on the absorbances in the sample, but are also affected by scattering effects relative to the particle size and other microstructures. Differences in sample thickness and density exhibit variations in baseline level, and thus produce shifts in absorbance values. These scattering effects and shifts must be corrected for reliable spectral analysis. (Huang et al. 2010.)

The broad overlapping features in NIR spectrum hamper discrimination of adjacent absorbance bands and contain few characteristic features for spectral pattern recognition. Derivative functions on finite data sets can

help identify underlying responses in absorption levels, as the slopes become more resolved. (Owen, 1995.) Figures 11 & 12 illustrate the effect of mathematical pre-treatments on the measured spectra (same data set on both plots).

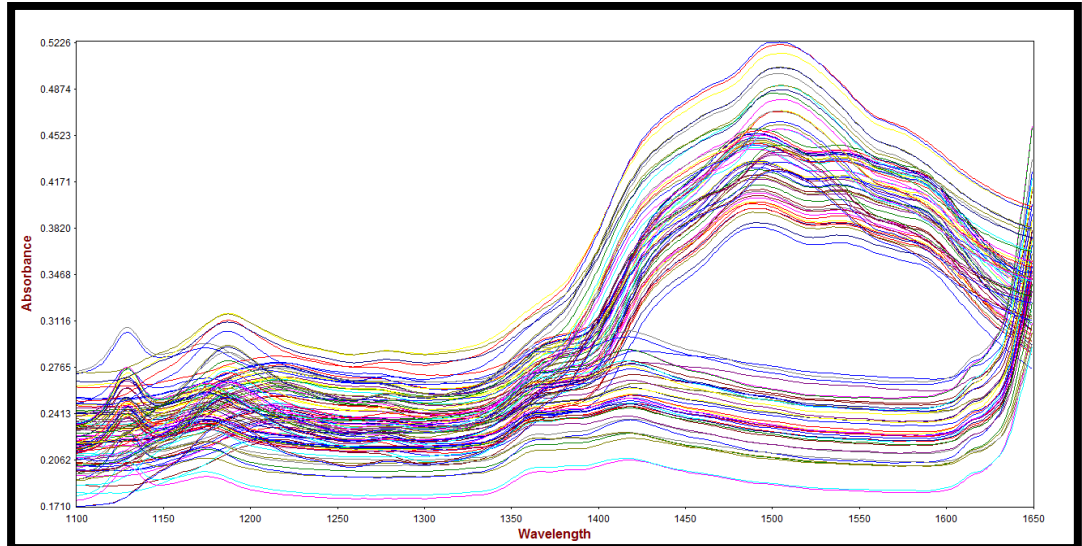


FIGURE 11. Raw spectra without mathematical corrections. (Capture from Vision™)

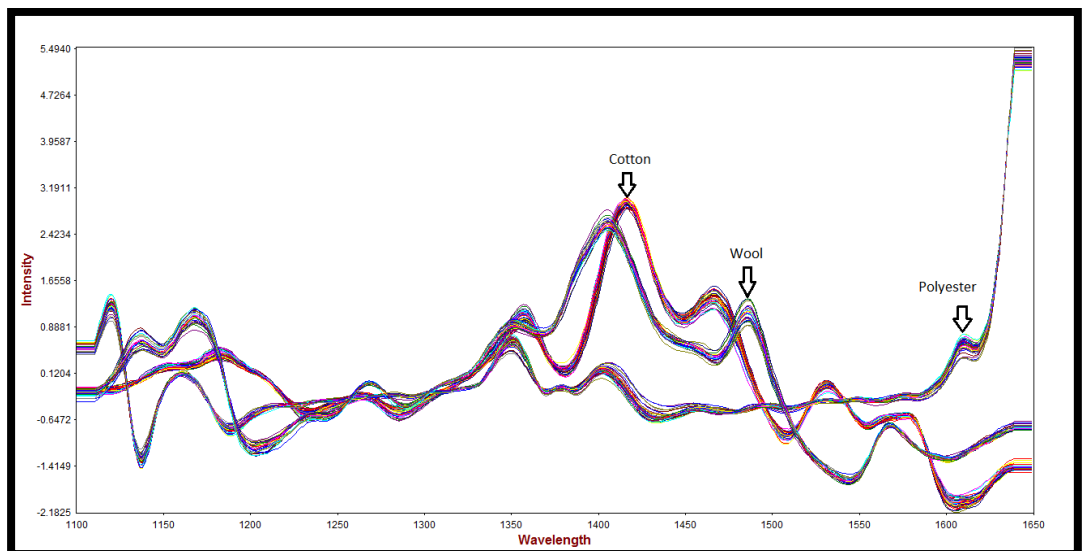


FIGURE 12. Normalized derivative spectra. (Capture from Vision™)

Vision™ offers multiple mathematical tools to extract relevant information from the raw IR spectrum. The mathematical basis and effects on NIR spectra of these pre-treatment functions are presented through Chapters 5.3.1 and 5.3.2.

5.3.1 Noise reduction and scatter correction

Internal signal interference, scattering from specular light reflectance, varying measurement distance and impurities in the sample material contribute to noise (see Figure 10), which occurs in practically all spectroscopic measurements. These minor inconsistencies in the IR-spectrum interfere with pattern recognition functions and may even hide valuable information in the absorbance spectrum. Correct measurement conditions and corrective instrument calibration improve the signal to noise ratio (S/N) but mathematical smoothing of spectra is often required. Baseline shifts (due to e.g. density variations) should also be corrected for reliable pattern recognition. (Eremina 2017.)

Vision™ software includes several functions for improving S/N and scatter corrections:

5.3.1.1 NPS – N-Point Smoothing

An average absorbance value of a predefined wavelength segment, S , is calculated and placed in the center point of the segment. Calculations are swept through the entire spectrum at half S intervals. This smoothing treatment removes noise effectively but may lose relevant absorbance data. The main parameter, segment size, must be set experimentally. The default value in Vision™ is 10nm. Due to averaging, half the number of data points in S is lost by NPS at each end of the data set. (Metrohm 2015c, 9.)

5.3.1.2 SNV – Standard Normal Variate

SNV is used to normalize multiple spectra within a data set when differences in e.g. particle size or effective wavelength path cause baseline shifts. Scatter correction is always recommended when measuring solid, granulate or powder samples. The spectra are normalized to a uniform base level by mean centering the spectra and dividing with its standard deviation:

$$S_i^{SNV} = \frac{s_i - \bar{s}}{\sqrt{\frac{\sum_{i=1}^n (s_i - \bar{s})^2}{n-1}}} \quad (5)$$

Where s_i = single absorbance value at wavelength point i
 \bar{s} = average value from each spectrum at i
 n = number of data points in the treated spectrum

(Metrohm 2015c, 12.)

SNV converts the y-axis values to arbitrary units of signal intensity. While they are not directly comparable to initial absorbance values, they enable efficient pattern recognition when applied to all spectra. The effects of SNV on the same data set (cotton) are illustrated in Figures 13 & 14.

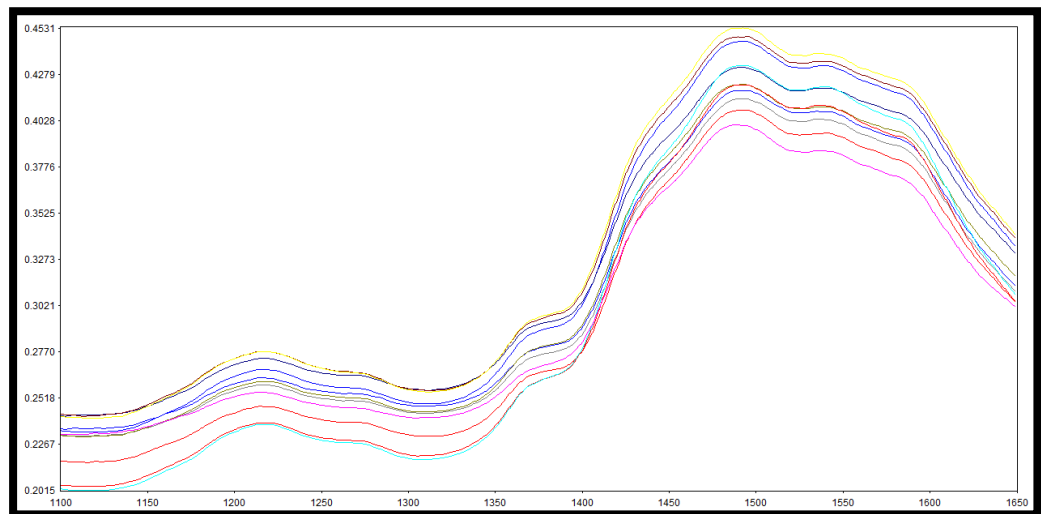


FIGURE 13. Raw spectra of 100% cotton (Capture from Vision™)

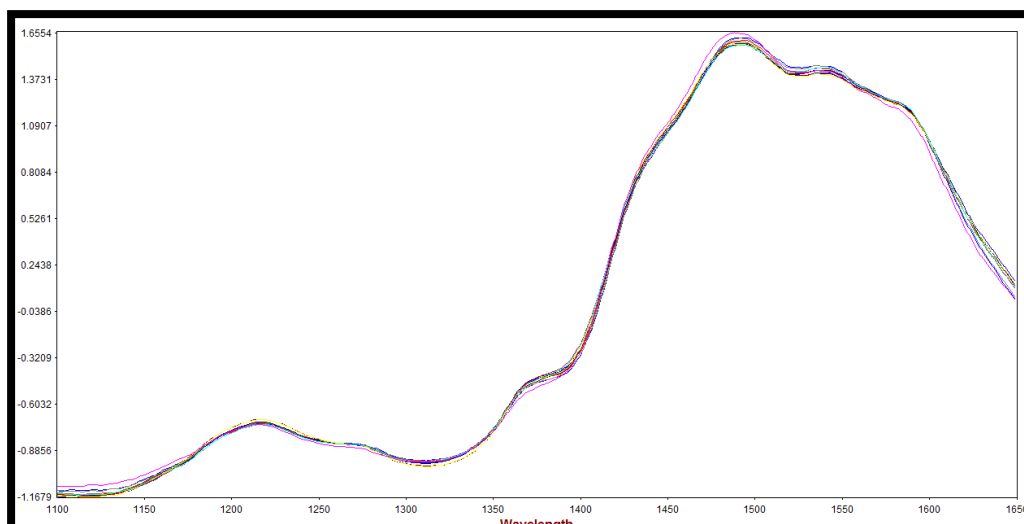


FIGURE 14. SNV treated spectra of 100% cotton (Capture from Vision™)

5.3.1.3 Detrend

Detrend is used to eliminate baseline offset, slope and curvature from a spectrum. A baseline polynomial function is fitted to the spectra by a least squares fit, and then subtracted from the spectrum. The polynomial degree determines the correction effect. First order polynomial removes offset and slope, while second order polynomial removes also parabolic curvature. (Metrohm 2015c, 12-13.)

According to Eremina (2017), slope and curvature are present when measuring spectra at a wide range ($\Delta\lambda > 1500$ nm). Therefore, with most NIR process analyzers with limited spectral range, there usually is no need for slope/curvature correction by Detrend.

5.3.1.4 Baseline correction

Manual baseline correction is done by subtracting either a spectral value at a specified wavelength point or a fixed value defined by user (Metrohm 2015c, 12).

5.3.2 Feature enhancement

Derivative functions are especially efficient with NIR spectra with broad features, since they can enhance slight changes in curvature.

Emphasizing slope can separate overlapping peaks and give relevant information on different absorption bands. Feature enhancement by derivatives can act also as a scatter correction since slight variations in baseline level have negligible effect on the resulting derivative spectrum. (ASDinc 2017.)

Derivative functions are efficient in pointing out single characteristic absorbance bands as the surrounding background features are suppressed. This is due to the fact that the amplitude of a Gaussian band in the n^{th} derivative is inversely proportional to the original bandwidth, as illustrated in Figure 15. Even if a spectral feature with strong slope has a low amplitude in the raw spectrum, it can become dominant in the derivative spectrum. (Owen 1995, 5-6.) These sudden peaks in absorbance usually carry the most relevant spectroscopic information, and should therefore be enhanced.

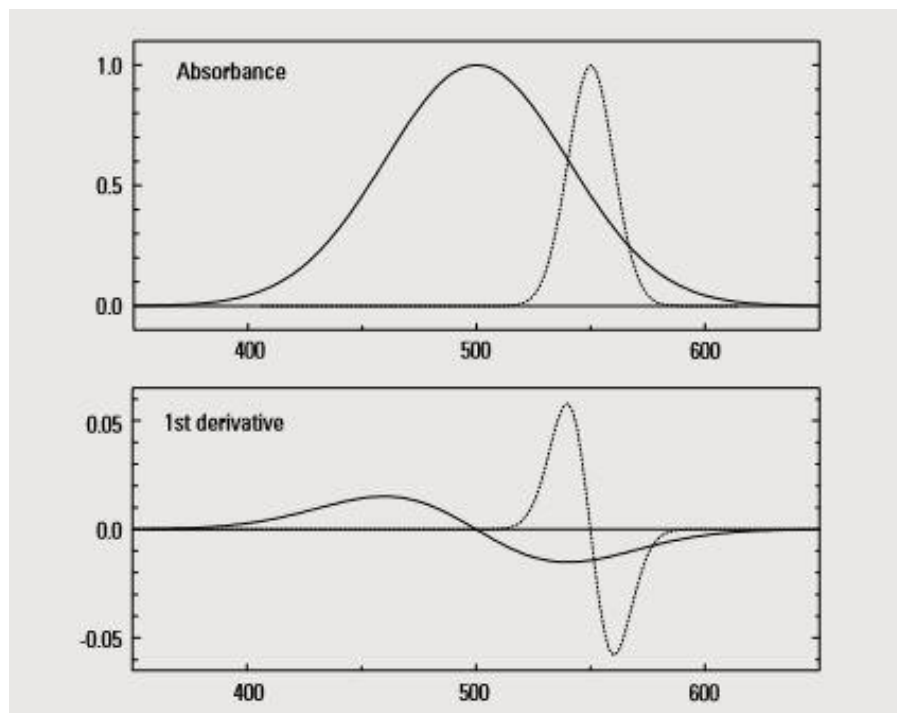


FIGURE 15. Peaks with narrow initial bandwidth are dominant in the derivative spectrum. (Owen 1995)

Vision software includes the following options for derivative functions:

5.3.2.1 Finite-difference derivatives

First and second order derivatives enhance sudden changes in absorbance levels. First order derivative removes baseline offset and converts the steepest slopes into distinctive peaks. Second order derivative eliminates also possible slope while exhibiting artificial intensity peaks derived from the 1st order derivative.

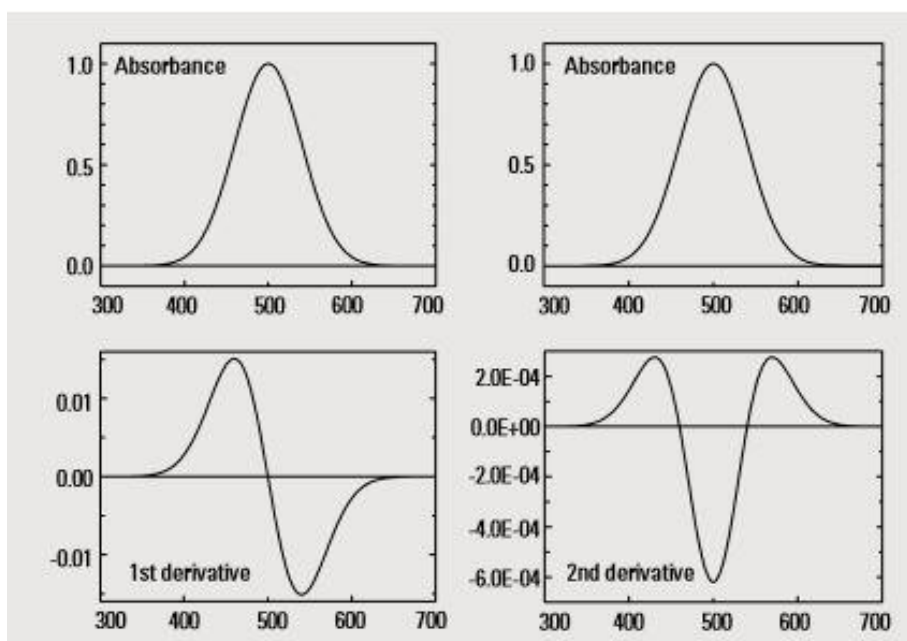


FIGURE 16. Illustration of 1st and 2nd order derivatives on a Gaussian band. (Owen 1995)

The first order derivative is calculated by averaging absorbance values at two adjacent segments (A and B). The resulting value of $A-B$ is placed in the middle of the gap between the segments. This procedure is repeated by shifting the whole sequence one data point at a time and placing derivative values between segments. Second order derivative accounts for adjacent segments A, B and C, and is calculated as $A-2B+C$. The resulting value is placed in the midpoint of the second segment B, and the calculations are repeated through the spectrum, shifting at one data point intervals. Both functions require two parameters to be defined: the length

of a wavelength segment used for derivation and the gap between those segments. (Metrohm 2015c, 9-12.)

A drawback from the derivation process is the decrease in signal to noise ratio. Noise has always the sharpest features in IR measurements and exhibit unwanted features in the resulting derivative spectra. Prior to derivative pre-treatment, the data must be cleared of excess noise with good measurement conditions and, if needed, mathematical smoothing operations. Increasing the segment size parameter reduces noise but may lose valuable spectral information. (Owen 1995, 8.)

5.3.2.2 Savitzky-Golay

The S-G function combines smoothing, detrending and derivative effects on raw spectra. Similarly to the detrend function, S-G produces a least squares fit of a polynomial curve to a pre-defined segment on the original spectrum. Contrary to detrend, S-G fits multiple polynomials on limited wavelength bands and replaces the original absorbance values with the modeled polynomial. Parameters for S-G operation are the segment size and the order of the polynomial fitted to the spectrum. (Metrohm 2015c, 13.)

1st and 2nd order polynomials produce similar results as the simple finite-difference derivatives described previously, but the segment size has a stronger effect on the S/R ratio. Setting up parameters for S-G function requires insight on the dexterity of the raw spectrum and the overall noise level. Small segments with higher order polynomials result in excess noise, as the noise in raw data are incorrectly modeled in the correction process.

5.4 Identification method

Identifying materials based on NIR spectra requires a reference library for pattern recognition. The first step in library development in Vision™ software is *sample selection*, which can be done manually or by setting

tolerances for excluding outliers in a sample set. Sample selection divides spectra within a single product into three classes. Training set should contain the main spectral features that can be accepted for material identification. Outlier set includes samples that do not present the desirable material spectra and should be identified as *unknown*. Vision includes additionally a separate acceptance set that consists of redundant samples. While having multiple samples in a reference library product makes the identification more reliable and robust, it also increases the computational power needed in real-time sorting operations. Therefore, it is useful to use these redundant samples when defining correct tolerances for pattern recognition, but leaving them out in the operational computation process. The *acceptance set* is to include samples that are closest to the mean product spectrum, so the remaining operational *training set* contains the most variation within acceptable limits. (Buytaert 201.6)

Once the reference library products are chosen, pattern recognition parameters are set to differentiate between divergent spectra. This procedure is called *identification method development*, and is set globally to all products in a library. Vision supports two-step identification for highly similar spectra. In *qualification method development* it is possible to set additional product-specific restrictions to differentiate between otherwise conforming spectra. This procedure is optional, and can be omitted from library development if the library products exhibit clearly distinctive spectral patterns. Similar mathematical features are available for both *sample selection* and *identification method development*, and are presented in Chapters 5.4.1-5.4.3.

Final step for identification method development is *library validation*. Each sample spectrum is cross-checked internally against the pattern recognition functions and tolerances defined in identification method development. This procedure is useful when optimizing parameters and checking for possible misidentifications.

5.4.1 Mahalanobis distance in principal component space

Principal component analysis is a dimensionality reduction method. It enables conversion of multidimensional data to be presented by just a few *principal components*, which carry the most relevant data. For example, a multivariate graph with three distinctive peaks can be reduced to a single point in 3D-space (Figure 17), when applying the peak amplitudes as the primary coordinates for x-, y- and z-axes. When this method is repeated on multiple graphs, the resulting cluster in 3D-space (Figure 18) indicates whether the data are similar and shows outliers as singular points outside the cluster. A spectrum can be broken down to as many principal components as necessary to explain and reconstruct it to a predefined accuracy. (Bruker 2017.)

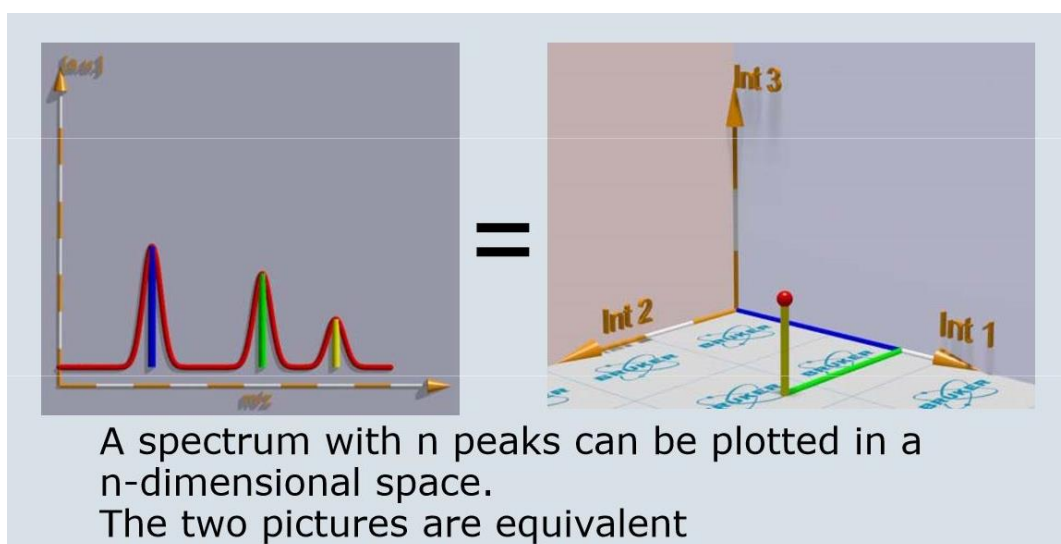


FIGURE 17. Simplified visual of dimensionality reduction by principal components. (Bruker 2017)

In Vision software the product set can be presented as a principal component cluster. When identifying unknown samples, the measured spectra is converted into principal components and placed in a multidimensional plot. The spectral similarity is evaluated by a metric called mahalanobis distance, which indicates a relative distance to the center of distribution in the PC cluster. Parameters for mahalanobis distance are unitless and have to be determined experimentally. (Metrohm 2015c, 25-26.)

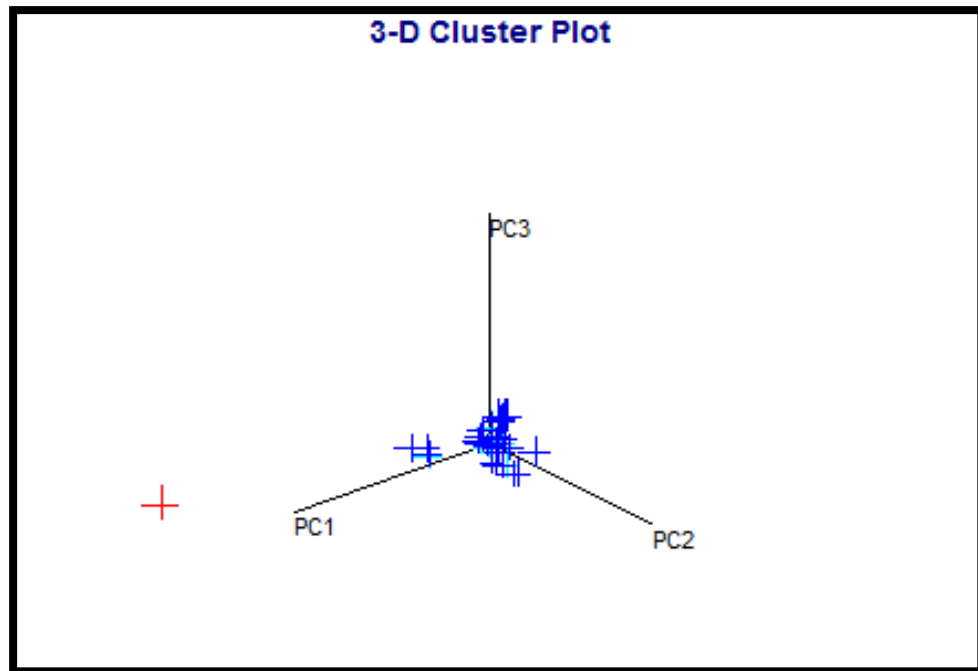


FIGURE 18. Principal component cluster in sample selection (wool 100%). Red score denotes a clear outlier in 3D principal component space. (Capture from Vision™)

5.4.2 Maximum distance in wavelength space

Identification by maximum distance in wavelength space is done by first inflating the mean product spectrum to cover a range of the standard deviation in a product set. An unknown spectrum is evaluated whether it fits within a predefined tolerance to the mean product spectrum. The defined distance tolerance is relative and wavelength-dependent, so regions with more variance allow for larger deviation from the mean spectrum. (Metrohm 2015c, 23.) This property is useful when spectral variations from e.g. moisture are an issue, and can't be fully eliminated. Similarly, it may pass spectra with small but narrow absorption peaks, indicating contaminated or otherwise unacceptable identification results.

5.4.3 Correlation in wavelength space

Correlation is a measure of spectral similarity. It is calculated by comparing absorbance values of an unknown spectrum to the corresponding values in the library product spectra, at each data point

(see Formula 6). Geometrically, correlation in wavelength space can be regarded as a cosine of an angle between two vectors representing spectra. The higher the correlation value, the higher the spectral similarity. Since this metric is scale invariant, it can be applied to spectra without normalization by standard normal variate.

$$D_c = \frac{\sum_i x_i y_i}{\sqrt{\sum_i x_i^2 \sum_i y_i^2}} \quad (6)$$

Where D_c = correlation coefficient between spectra
 x_i = absorption value of spectrum x at wavelength i
 y_i = absorption value of spectrum y at wavelength i

(Metrohm 2015c, 21-25.)

6 RESULTS

All mathematical tools for spectral pre-treatment and pattern recognition described in Chapter 5 were evaluated according to their performance and suitability in textile sorting technology. The best solutions for identification method development were determined by first ruling out redundant tools, and then running tests on Vision™ software. The resulting method was constructed to be universally viable. This means that the same treatments, procedures and parameters are expected to work on any kind of spectra without further optimization.

6.1 Optimized data pre-treatment

Vision™ includes many alternative pre-treatment methods for making spectral corrections. Considering the characteristic spectral features observed in cotton, polyester and wool samples, and taking into account the instrumental attributes (measurement range, internal noise levels, wavelength-dependent bias), a number of pre-treatment methods could be ruled out as unnecessary. For example, the limited measurement range does not generate a need for baseline curvature correction. After excluding redundant pre-treatment options, the remaining consideration included scatter and noise corrections, and feature enhancement by simple derivatives and Savitzky-Golay polynomial smoothing. Different parameters and combinations of data pre-treatments were compared in terms of smoothing performance, standard deviation of corrected spectra, and the ability to enhance identifiable features in the spectra.

6.1.1 Feature enhancement with derivatives

Derivative functions and S-G polynomial smoothing provide smoothing and scatter correction to some extent, as well as enhancing significant absorption bands. Therefore, they were first evaluated individually to see if there was need for additional corrections. Derivative functions were set with segment sizes of 10 nm (system default), 15 nm, 20 nm and 40 nm to evaluate the smoothing effect with different parameters. Gap between

segments was set to zero in order to maintain all relevant features in derivation. The smallest segment size showed visually significant noise levels that would affect correlation analysis later on. Even if the spectra from cotton, wool and polyester could be separated with smaller amount of detail, it was assumed that further library development with e.g. other cellulose-based fabrics (viscose, linen etc.) would require more detailed derivative spectra. Also, the size of lost wavelength bands at each end of the spectrum increases with larger segments.

Savitzky-Golay polynomial fitting was examined with different order polynomials, first and second order derivation and multiple segment sizes. Contrary to simple first order derivative, S-G provided different results when applied to different materials. This is presumably because the polynomial that is fitted to the spectrum should match the complexity of each spectra. The segment size into which the polynomial is fitted may also contain broad bands with little to none distinctive features except for a single, relatively narrow peak. Therefore, the segment size should be kept at a reasonable minimum.

6.1.2 Smoothing and scatter correction

Properly adjusted measurement distance and setting the data collection method to plot averages of multiple scans eliminated most of the noise in sample measurements. The need for smoothing operations depends on the resulting noise levels after feature enhancement. NPS alone is suitable for visual analysis of measured spectra and rough smoothing for high-tolerance correlation analysis. After derivation, there usually is still significant variation in amplitudes of positive and negative peak extremes in derivated spectra. This is common with solid samples and should be corrected, because large deviations in amplitude would require broader tolerances for identification.

6.2 Identification method

The product sets for cotton, wool and polyester were deliberately equipped with at least one clear outlier and a few slightly variate spectra (marked with affix “b”) to help determine correct parameters and tolerances for identification. Sample selection was conducted so that outliers are marked as reject, but the b-noted variate spectra passes identification. This set-up enabled adjusting identification parameters right outside the accepted variance in spectra. The software’s sample selection phase is not critical to the method development as it does not affect the rules of identification. Principal component analysis, maximum amplitude restrictions or spectral correlation can be used to set guidelines for accepted spectra in the data sets, and to verify that outliers are marked correctly.

Physical properties must be taken into consideration to achieve best results in identification method development. Textile samples with relatively much spectral variation due to physical properties, such as texture and density, can be incorrectly rejected. Especially dark and dense wool fabrics exhibited higher absorption values at shorter wavelengths. Narrow spectral regions with large variations due to physical phenomena can be excluded from the qualitative analysis. If no regions are to be excluded, the identification parameters must be set to adjust for variations in wavelength-dependent absorbance levels within acceptable samples.

The chosen pattern recognition function was able to adjust relative tolerances based on the uniformity level of certain spectral regions. One outlier in a larger data set does not affect the mean absorbance values enough to distort the reliability of acceptable spectral patterns.

6.3 Library validation

The developed identification library was validated by running internal cross-checking with all measured spectra as unknown. The routine analysis report (Attachment 2) showed that there was no misidentifications and outliers were correctly labeled as “no match”. All the unidentified

samples are denoted with an affix “c”, which indicates a spectral outlier. The validation was supported by running analysis on new samples from a set with fire resistant PES100% and CO100%. The success rate of 83% and 85%, for CO and PES respectively, showed that even with the same underlying material matrix, the identification method is capable of ruling out contaminants and unwanted additives in textile samples. Finally, a set of viscose samples was tested to see if a material with similar chemical composition (cellulose) as cotton would pass identification. The system was able to disqualify viscose from the cotton product set.

7 CONCLUSIONS

This thesis has demonstrated the process of developing an identification method for textiles using NIR spectroscopy. The sorting technology has been proven to be capable of differentiating between unblended cotton, wool and polyester. It was also clear that with correct feature enhancement and identification parameters, NIR can separate even fabrics with a substantially similar chemical composition. Viscose and cotton were identified correctly despite both being purely cellulose-based fibers, and unblended cotton and polyester with fire resistant finishes (assumed phosphorus/chlorine -based additives) were discarded with a good percentage.

The divergence in NIR spectra between cotton, wool and polyester would make it possible to identify these materials with just a simple scatter correction, smoothing operation and a broad-tolerance correlation analysis. However, for future development of a more complex identification library, it is beneficial to have a more meticulous identification method to differentiate between pure and blended textiles. The optimization of pre-treatment parameters and library development were conducted with future expansion in mind to ensure high accuracy.

7.1 Future development

A full, real-time test run to determine the systems reliability needs further development in logical programming for the sorting line, and a motorized guide rail to keep measurement distance uniform for every sample. Once the measuring distance is standardized and the logical commands from the software to the sorting line's deflectors is timed correctly, the system can begin routine identification analysis of waste textiles.

The system's performance is not limited by the spectroscopic hardware or the software's calculation processes. When conducting identification tests without the sorting line in operation, the result can be obtained in 50 ms. The hindering factor for the performance of the system is expected to be

the speed of the conveyor belt and the pressure line's response in deflecting samples to correct bins. The sorting line is not yet equipped with an automated feeding system, which is also needed for larger scale operations.

7.2 Viability for waste reduction

With the developed textile sorting technology, it is possible to reduce the amount of textile waste going to incineration. This will improve resource efficiency, as the valuable materials can be kept in circulation for a longer period of time. Having an efficient and reliable sorting method for textile waste can generate new possibilities for textile recycling, as it increases the availability of pure recycled material. A heterogeneous stream of waste textiles have usually been downcycled for insulation, padding and industrial wipes. A sorting line for pure unblended textile materials opens potential for developing upcycling practices with more added value.

REFERENCES

Printed sources

Jaarinen, S. Niiranen, J. 2005. Laboratorion analyysitekniikka. Helsinki. Edita Publishing.

Stuart, B. 2004. Infrared spectroscopy: fundamentals and applications. USA. Wiley & Sons publishing.

Williams, D.H. Fleming, I. 1989. Spectroscopic methods in organic chemistry. London. McGraw-Hill Publishing Co.

Workman, J. Weyer, L. 2008. Practical guide to interpretive near-infrared spectroscopy. USA. Taylor & Francis Group.

Online publications

Manley, M. 2014. Near-infrared spectroscopy and hyperspectral imaging: Non-destructive analysis of biological materials. Chemical Society Reviews, Vol 43 Issue 24 [accessed 26.10.2017]. Available at: <http://pubs.rsc.org/en/Content/ArticleLanding/2014/CS/C4CS00062E#!divAbstract>

Manley, M., Downey, G. & Baeten, V. 2008. Spectroscopic technique: Near-infrared (NIR) spectroscopy. Modern techniques for food authentication [accessed 2.11.2017]. National University of Ireland. Available at: http://197.14.51.10:81/pmb/BIOLOGIE/0123740851%20%20Modern_Techniques_for_Food.pdf#page=90

Pasquini, C. 2003. Near infrared spectroscopy: Fundamentals, practical aspects and analytical applications. Journal of the Brazilian chemical society, Vol 14 Issue 2 [accessed 22.10.2017]. Available at: http://www.scielo.br/scielo.php?pid=S0103-50532003000200006&script=sci_arttext

Reich, G. 2005 Near infrared spectroscopy and imaging: Basic principles and pharmaceutical applications. *Advanced drug delivery reviews*, Vol 57 Issue 8 [accessed 2.11.2017]. Available at:

<http://www.sciencedirect.com/science/article/pii/S0169409X05000578>

Rinnan, Å., van der Berg, F., Balling Engelsen S. 2009. Review of the most common pre-processing techniques for near-infrared spectra. *Trends in analytical chemistry*, Vol 28 Issue 10 [accessed 17.10.2017]. Available at: <http://www.sciencedirect.com/science/article/pii/S0165993609001629>

Rodgers, J., Beck, K. 2009. NIR characterization and measurement of the cotton content of dyed blend fabrics. *Textile research journal*, Vol 79 Issue 8 [accessed 12.10.2017]. Available at:

<http://journals.sagepub.com/doi/abs/10.1177/0040517508090884>

Digital sources

ASDinc. 2017. What is a derivative spectrum. [accessed 27.10.2017]

Available at <https://www.asdi.com/learn/faqs/what-is-a-derivative-spectrum>

Bruker. 2017. Principal component analysis. Educational presentation [accessed 2.11.2017]. Available at:

http://research.med.helsinki.fi/corefacilities/proteinchem/pca_introduction_basics.pdf

EssentialFTIR. 2017. Spectral library search. [accessed 06.10.2017]

Available at: <http://www.essentialftir.com/librarySearch.html>

Huang, J. Romero-Torres, S. Moshgbar, M. 2010. Practical considerations in data pre-treatment for NIR and Raman spectroscopy. *American pharmaceutical review*. [accessed 30.10.2017] Available at:

<http://www.americanpharmaceuticalreview.com/Featured-Articles/116330-Practical-Considerations-in-Data-Pre-treatment-for-NIR-and-Raman-Spectroscopy/>

Metrohm. 2013a. NIR Spectroscopy: A guide to near-infrared spectroscopic analysis of industrial manufacturing processes. Monograph

[accessed 12.10.2017]. Available at: http://www.mep.net.au/wpmep/wp-content/uploads/2013/05/MEP_Monograph_NIRS_81085026EN.pdf

Owen, A. 1995. Derivative spectroscopy. Agilent technologies. Application note [accessed 20.10.2017]. Available at: http://www.who.edu/cms/files/derivative_spectroscopy_59633940_175744.pdf

Syke. 2015 Tekstiilijätteen kierrätyksen mahdollisuudet ja esteet (TEXJÄTE). Online article. 22.01.2015. [accessed 21.10.2017] Available at: <http://www.syke.fi/hankkeet/textjate>

Valvan Baling Systems. 2017. Fibersort. [accessed 17.10.2017] Available at: <http://www.valvan.com/products/equipment-for-used-clothing-wipers/sorting-equipment/fibersort/>

VTT. 2017. Tekstiilikierron ekosysteemi vie kierrätyksen uudelle tasolle. Online article. 4.4.2017 [accessed 21.10.2017] Available at: <http://www.vtt.fi/medialle/uutiset/tekstiilikierron-ekosysteemi-vie-kierrätyksen-uudelle-tasolle>

Workman, J. 2014. An introduction to near-infrared spectroscopy. SpectroscopyNow [accessed 16.10.2017] Available at: <http://www.spectroscopynow.com/details/education/sepspec1881education/an-introduction-to-near-infrared-spectroscopy.html?tzcheck=1&tzcheck=1>

Other

Buytaert, H. 2016. Metrohm. NIRS & Vision system software training course. Lahti. November 2016.

Eremina, N. 2017. NIR Spectroscopy. Training seminar by Bruker Optics. Helsinki. 27.-28.09.2017.

Metrohm. 2013b. NIRS Analyzer Pro. PDF-manual for instrument set-up and configuration. Unpublished

Metrohm. 2015a. Vision manual – reference. Software tutorial.
Unpublished

Metrohm. 2015b. Vision diagnostics. PDF-manual for instrument
calibration and diagnostics. Unpublished

Metrohm 2015c. Vision theory. PDF-manual for underlying mathematical
software. Features. Unpublished

Nilsson, A. 2017. Vibrational Spectroscopy and FT-IR. Training seminar by
Bruker Optics. Helsinki. 27.-28.09.2017

APPENDIX

Attachment 1: Performance test report – Sample channel

Attachment 2: Full library validation report

Attachment 3: Identification report – CO100% and PES100% with fire retardant finishing

Attachment 1: Performance test report – Sample channel

XDS Instrument Performance Test

Lamp time:
Date: 2.10.2017 Time: 12:15:57
Instrument Model: NIRS Analyzer Pro
Serial number: 5939898
EPROM Version: 0
Wavelength Rev: 5
Motherboard S/N: 5939898
Sampling System: NIRS Analyzer Pro Window Reflection
Amplifier: Reflectance
Sampl. System S/N: N/A Fiber Type:
Amplifier S/N: Tip Type:
Test Type: Performance Test Fiber Length:
Multiplexer Channel: Sample
Scan Range: 1100 - 1650
Author/Operator: NIRSystems Default User

Sample Fiber

Noise Test (mA) Scan Range: 1100 - 1650
seg 1 1100 - 1650

Scan	EOC	P-P	Minimum	Wavelength	Maximum	Wavelength	Bias	RMS	Gain
1	0	0,474	-0,270	1645	0,205	1110	-0,013	0,061	N/A
2	0	0,304	-0,140	1480	0,165	1107	-0,001	0,058	N/A
3	0	0,375	-0,093	1599	0,282	1108	0,048	0,058	N/A
4	0	0,594	-0,250	1139	0,344	1100	-0,017	0,096	N/A
5	0	0,437	-0,171	1644	0,266	1129	0,041	0,071	N/A
6	0	0,268	-0,124	1355	0,143	1106	0,016	0,053	N/A
7	0	0,376	-0,186	1641	0,190	1182	-0,021	0,067	N/A
8	0	0,378	-0,179	1133	0,199	1570	0,009	0,063	N/A
9	0	0,368	-0,230	1647	0,138	1610	0,003	0,051	N/A
10	0	0,400	-0,207	1621	0,193	1125	-0,012	0,067	N/A

Summary of Noise

Date: 2.10.2017 Time: 12:15:57
Instrument Model: NIRS Analyzer Pro
Serial number: 5939898
EPROM Version: 0
Wavelength Rev: 5
Motherboard S/N: 5939898
Sampling System: NIRS Analyzer Pro Window Reflection
Amplifier: Reflectance
Sampl. System S/N: N/A Fiber Type:
Amplifier S/N: Tip Type:
Test Type: Performance Test Fiber Length:
Multiplexer Channel: Sample
Scan Range: 1100 - 1650
Author/Operator: NIRSystems Default User

seg 1 1100 - 1650

Summary of Peak-to-Peak (mA)

	Overall
Minimum	0,268
Average	0,397
Maximum	0,594

Summary of Bias (mA)

	Overall
Minimum	-0,021
Average	0,005
Maximum	0,048

Summary of RMS (mA)

	Overall
Minimum	0,051
Average	0,065
Maximum	0,096

Operation Qualification

Instrument Model: NIRS Analyzer Pro
Serial number: 5939898
PowerPC Version: N/A
Wavelength Rev:
Motherboard S/N: N/A
Sampling System: NIRS Analyzer Pro Window Reflection
Amplifier: Reflectance
Sampl. System S/N: N/A
Amplifier S/N:
Test Type: Performance Test
Multiplexer Channel: Sample
Scan Range: 1100 - 1650
Author/Operator: NIRSystems Default User

Fiber Type:
Tip Type:
Fiber Length:

10 samples taken at 32 scans/sample

Noise Test (mA) Scan Range: 1100 - 1650

Test	Specification	Actual	Valid
P-P	1,0000	0,3974	Yes
RMS	0,2000	0,0646	Yes
Bias	0,2000	0,0052	Yes

Date: 2.10.2017

Time: 12:15:57

Attachment 2. Full library validation report

Routine Analysis (Analyze Stored Data)

Acquired:

Date: 04/10/2017
Time: 13:49:58
Author/Operator: NIRSystems Default User
Instrument Model: NIRSystems NIRS Analyzer Pro
Serial number: 5939

Re-analyzed:

Date: 03/11/2017
Time: 16:36:48
Author/Operator: NIRSystems Default User

Library: telaketju_lib
Output Project: ident_test

Sample ID	Selected	ID as	ID Result	P/F
co001-1	unknown	Cotton 100	2.566	Pass
co001-2	unknown	Cotton 100	1.859	Pass
co002-1	unknown	Cotton 100	1.218	Pass
co002-2	unknown	Cotton 100	2.258	Pass
co003-1b	unknown	Cotton 100	1.932	Pass
co003-2b	unknown	Cotton 100	1.972	Pass
co004-1c	unknown	No Match	12.480	Fail
co004-2cc	unknown	No Match	21.670	Fail
co005-1	unknown	Cotton 100	1.664	Pass
co005-2b	unknown	Cotton 100	2.785	Pass
co006-1	unknown	Cotton 100	1.210	Pass
co006-2	unknown	Cotton 100	1.305	Pass
co007-1b	unknown	Cotton 100	2.480	Pass
co007-2b	unknown	Cotton 100	2.589	Pass
co008-1	unknown	Cotton 100	1.156	Pass
co008-2	unknown	Cotton 100	1.155	Pass
co009-1b	unknown	Cotton 100	3.062	Pass
co009-2b	unknown	Cotton 100	3.464	Pass
co010-1b	unknown	Cotton 100	1.075	Pass
co010-2b	unknown	Cotton 100	1.602	Pass
co011-1b	unknown	Cotton 100	2.544	Pass
co011-2b	unknown	Cotton 100	2.954	Pass
co012-1	unknown	Cotton 100	2.405	Pass
co012-2	unknown	Cotton 100	1.487	Pass
co013-1	unknown	Cotton 100	1.320	Pass
co013-2	unknown	Cotton 100	1.493	Pass
co014-1	unknown	Cotton 100	2.327	Pass
co014-2	unknown	Cotton 100	2.222	Pass
co015-1bc	unknown	No Match	11.836	Fail
co015-2bc	unknown	No Match	13.113	Fail
co016-1	unknown	Cotton 100	1.142	Pass
co016-2	unknown	Cotton 100	1.195	Pass
co017-1	unknown	Cotton 100	1.588	Pass
co017-2	unknown	Cotton 100	1.599	Pass
co018-1	unknown	Cotton 100	1.177	Pass
co018-2	unknown	Cotton 100	1.261	Pass
co019-1	unknown	Cotton 100	1.617	Pass

co019-2	unknown	Cotton 100	2.126	Pass
co020-1	unknown	Cotton 100	1.708	Pass
co020-2	unknown	Cotton 100	1.500	Pass
co021-1	unknown	Cotton 100	1.564	Pass
co021-2	unknown	Cotton 100	1.467	Pass
co022-1cc	unknown	No Match	30.397	Fail
co022-2cc	unknown	No Match	25.965	Fail
co023-1	unknown	Cotton 100	1.785	Pass
co023-2	unknown	Cotton 100	1.246	Pass
co024-1	unknown	Cotton 100	1.677	Pass
co024-2	unknown	Cotton 100	1.926	Pass
co025-1	unknown	Cotton 100	1.995	Pass
co025-2	unknown	Cotton 100	1.465	Pass
co026-1	unknown	Cotton 100	1.198	Pass
co026-2	unknown	Cotton 100	1.655	Pass
co027-1	unknown	Cotton 100	1.244	Pass
co027-2	unknown	Cotton 100	1.131	Pass
co028-1cc	unknown	No Match	8.488	Fail
co028-2cc	unknown	No Match	7.624	Fail
co029-1b	unknown	Cotton 100	1.617	Pass
co029-2b	unknown	Cotton 100	1.298	Pass
co030-1	unknown	Cotton 100	1.785	Pass
co030-2	unknown	Cotton 100	3.074	Pass
pes001-1b	unknown	Polyester 100	2.710	Pass
pes001-2b	unknown	Polyester 100	2.452	Pass
pes002-1b	unknown	Polyester 100	2.983	Pass
pes002-2b	unknown	Polyester 100	1.999	Pass
pes003-1b	unknown	Polyester 100	2.165	Pass
pes003-2b	unknown	Polyester 100	2.900	Pass
pes004-1b	unknown	Polyester 100	2.334	Pass
pes004-2b	unknown	Polyester 100	2.583	Pass
pes005-1	unknown	Polyester 100	2.584	Pass
pes005-2	unknown	Polyester 100	1.765	Pass
pes006-1	unknown	Polyester 100	2.161	Pass
pes006-2	unknown	Polyester 100	1.831	Pass
pes007-1	unknown	Polyester 100	1.967	Pass
pes007-2	unknown	Polyester 100	1.877	Pass
pes008-1b	unknown	Polyester 100	2.857	Pass
pes008-2b	unknown	Polyester 100	2.841	Pass
pes009-1	unknown	Polyester 100	0.922	Pass
pes009-2	unknown	Polyester 100	1.242	Pass
pes010-1	unknown	Polyester 100	1.220	Pass
pes010-2	unknown	Polyester 100	0.965	Pass
pes011-1	unknown	Polyester 100	1.696	Pass
pes011-2	unknown	Polyester 100	1.404	Pass
pes012-1	unknown	Polyester 100	0.769	Pass
pes012-2	unknown	Polyester 100	1.126	Pass
pes013-1	unknown	Polyester 100	1.221	Pass
pes013-2	unknown	Polyester 100	2.032	Pass
pes014-1	unknown	Polyester 100	0.918	Pass
pes014-2	unknown	Polyester 100	1.177	Pass
pes015-1b	unknown	Polyester 100	2.762	Pass
pes015-2b	unknown	Polyester 100	3.697	Pass
pes016-1	unknown	Polyester 100	1.431	Pass

pes016-2	unknown	Polyester 100	1.316	Pass
pes017-1	unknown	Polyester 100	2.019	Pass
pes017-2	unknown	Polyester 100	1.729	Pass
pes018-1	unknown	Polyester 100	1.365	Pass
pes018-2	unknown	Polyester 100	1.212	Pass
pes019-1b	unknown	Polyester 100	2.211	Pass
pes + paperilappu	unknown	No Match	25.958	Fail
pes019-2b	unknown	Polyester 100	1.617	Pass
pes020-1	unknown	Polyester 100	2.791	Pass
pes020-2	unknown	Polyester 100	3.296	Pass
pes021-1	unknown	Polyester 100	2.035	Pass
pes021-2	unknown	Polyester 100	1.899	Pass
pes022-1	unknown	Polyester 100	1.279	Pass
pes022-2	unknown	Polyester 100	1.328	Pass
pes023-1	unknown	Polyester 100	1.315	Pass
pes023-2	unknown	Polyester 100	1.607	Pass
pes harso	unknown	No Match	4.107	Fail
pes024-1	unknown	Polyester 100	1.084	Pass
pes024-2	unknown	Polyester 100	2.017	Pass
pes025-1	unknown	Polyester 100	0.874	Pass
pes025-2	unknown	Polyester 100	1.088	Pass
pes026-1b	unknown	Polyester 100	2.371	Pass
pes026-2	unknown	Polyester 100	1.588	Pass
pes027-1	unknown	Polyester 100	1.945	Pass
pes027-2	unknown	Polyester 100	1.722	Pass
pes028-1c	unknown	No Match	18.212	Fail
pes028-2c	unknown	No Match	16.677	Fail
pes029-1c	unknown	No Match	7.597	Fail
pes029-2c	unknown	No Match	10.499	Fail
wo001-1cc	unknown	No Match	18.376	Fail
wo001-2cc	unknown	No Match	17.892	Fail
wo002-1	unknown	Wool 100	1.477	Pass
wo002-2	unknown	Wool 100	1.329	Pass
wo003-1	unknown	Wool 100	1.217	Pass
wo003-2	unknown	Wool 100	1.198	Pass
wo004-1	unknown	Wool 100	0.913	Pass
wo004-2	unknown	Wool 100	0.933	Pass
wo005-1b	unknown	Wool 100	1.326	Pass
wo005-2b	unknown	Wool 100	1.457	Pass
wo006-1	unknown	Wool 100	1.989	Pass
wo006-2	unknown	Wool 100	0.824	Pass
wo007-1b	unknown	Wool 100	1.956	Pass
wo007-2b	unknown	Wool 100	2.058	Pass
wo008-1cc	unknown	No Match	6.137	Fail
wo008-2cc	unknown	No Match	6.066	Fail
wo009-1cc	unknown	No Match	21.062	Fail
wo009-2cc	unknown	No Match	21.170	Fail
wo010-1c	unknown	No Match	17.643	Fail
wo010-2cc	unknown	No Match	20.051	Fail
wo011-1	unknown	Wool 100	1.429	Pass
wo011-2	unknown	Wool 100	2.063	Pass
wo012-1	unknown	Wool 100	1.196	Pass
wo012-2	unknown	Wool 100	1.189	Pass
wo013-1	unknown	Wool 100	1.575	Pass

wo013-2	unknown	Wool 100	1.591	Pass
wo014-1b	unknown	Wool 100	2.603	Pass
wo014-2b	unknown	Wool 100	1.782	Pass
wo015-1	unknown	Wool 100	0.777	Pass
wo015-2	unknown	Wool 100	1.037	Pass
wo016-1b	unknown	Wool 100	3.513	Pass
wo016-2b	unknown	Wool 100	3.603	Pass
wo017-1b	unknown	Wool 100	2.138	Pass
wo017-2b	unknown	Wool 100	2.315	Pass
wo018-1	unknown	Wool 100	0.735	Pass
wo018-2	unknown	Wool 100	0.877	Pass
wo019-1b	unknown	Wool 100	2.797	Pass
wo019-2b	unknown	Wool 100	2.234	Pass
wo020-1	unknown	Wool 100	1.422	Pass
wo020-2cc	unknown	No Match	13.489	Fail
wo021-1	unknown	Wool 100	0.817	Pass
wo021-2	unknown	Wool 100	1.203	Pass
wo022-1	unknown	Wool 100	1.033	Pass
wo022-2	unknown	Wool 100	1.523	Pass
wo023-1cc	unknown	No Match	8.000	Fail
wo023-2cc	unknown	No Match	8.088	Fail
wo024-1	unknown	Wool 100	1.446	Pass
wo024-2	unknown	Wool 100	1.886	Pass
wo025-1	unknown	Wool 100	1.246	Pass
wo025-2	unknown	Wool 100	1.426	Pass

Attachment 3. Identification report – CO100% and PES100% with fire retardant finishing

Routine Analysis (Analyze Stored Data)

Acquired:

Date: 20.10.2017
Time: 12:38:37
Author/Operator: NIRSystems Default User
Instrument Model: NIRSystems NIRs Analyzer Pro
Serial number: 5939

Re-analyzed:

Date: 2.11.2017
Time: 12:11:24
Author/Operator: NIRSystems Default User

Library: telaketju_lib
Output Project: ident_test

Time	Sample ID	Selected	ID as	ID Result	P/F
13:59:08	frco007-1	unknown	No Match	11.791	Fail
13:52:17	frco001-2	unknown	No Match	5.505	Fail
13:53:00	frco002-1	unknown	No Match	6.263	Fail
13:53:55	frco002-1	unknown	No Match	5.043	Fail
13:54:46	frco003-1	unknown	No Match	12.682	Fail
13:55:12	frco003-2	unknown	No Match	12.881	Fail
13:55:47	frco004-1	unknown	Cotton 100	2.535	Pass
13:56:06	frco004-2	unknown	Cotton 100	2.319	Pass
13:56:30	frco005-1	unknown	No Match	10.125	Fail
13:56:50	frco005-2	unknown	No Match	9.812	Fail
13:57:49	frco006-1	unknown	No Match	9.984	Fail
13:58:13	frco006-2	unknown	No Match	10.104	Fail
13:41:18	trevira001-1	unknown	No Match	7.533	Fail
13:42:21	trevira001-2	unknown	Polyester 100	2.791	Pass
13:43:14	trevira002-1	unknown	No Match	4.621	Fail
13:43:39	trevira002-2	unknown	Polyester 100	2.214	Pass
13:44:23	trevira003-1	unknown	No Match	5.121	Fail
13:44:52	trevira003-2	unknown	No Match	5.150	Fail
13:45:27	trevira004-1	unknown	No Match	5.023	Fail
13:46:05	trevira004-2	unknown	No Match	5.372	Fail
13:46:54	trevira005-1	unknown	No Match	5.629	Fail
13:47:16	trevira005-2	unknown	No Match	5.543	Fail
12:32:07	trevira006-2	unknown	No Match	5.775	Fail
12:31:27	trevira006-1	unknown	No Match	5.366	Fail
12:33:56	pesfr001-1	unknown	No Match	28.811	Fail
12:34:48	pesfr001-2	unknown	No Match	49.877	Fail
12:36:15	pesfr002-1	unknown	Polyester 100	3.120	Pass
12:36:43	pesfr002-2	unknown	No Match	23.816	Fail
12:37:13	pesfr003-1	unknown	No Match	23.246	Fail
12:37:39	pesfr003-2	unknown	No Match	25.369	Fail
12:38:06	trevira007-1	unknown	No Match	5.714	Fail
12:38:37	trevira007-2	unknown	No Match	5.405	Fail