

1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
44
45
46
47
48
49
50

Identification and Quantitative Determination of Virgin and Recycled Cashmere: a Near Infrared Spectroscopy Study

Anastasia Anceschi[†], Marina Zoccola[†], Raffaella Mossotti[†], Parag Bhavsar[†], Giulia Dalla Fontana[†] and Alessia Patrucco^{†*}

[†]CNR-STIIMA, Italian National Research Council, Institute of Intelligent Industrial Technologies and Systems for Advanced Manufacturing, Corso G. Pella 16, 13900, Biella (BI), Italy

[*alessia.patrucco@stiima.cnr.it](mailto:alessia.patrucco@stiima.cnr.it)

This is a not peer-reviewed version of the manuscript published on ACS-Sustainable Chemistry and Engineering

<https://pubs.acs.org/doi/full/10.1021/acssuschemeng.1c05289>

Abstract

Cashmere plays an important role in luxury fashion due to its characteristic of fineness, warmth and softness. In frame of “green economy”, many companies start to produce and market recycled cashmere textiles. Recycled cashmere derives from post-factory and post-consumer waste. The mechanical action involved in recycling cashmere causes several damages of fibers affecting their morphological and mechanical properties. In order to safeguard the consumers and the companies to possible adulteration, a correct identification of fibers is required. NIR spectroscopy was applied to analyze and distinguish virgin and recycled cashmere fibers. Since no significant differences can be seen in the recorded spectra, the principal component analysis (PCA) combined with the soft independent modelling class analogies (SIMCA) method allows the classification of fibers in two distinct classes leading to have a sure identification of recycled and virgin cashmere. A calibration curve was also performed in order to quantify recycled cashmere fibers in a blend with virgin ones. The interclass distance in SIMCA method was found to be 249,77, whereas results from quantitative calibration shows a standard error of prediction (SEP) value of 5,8 % w/w (for a mean value of 50 % w/w). Microscopy analysis was also carried out to confirm the origin of recycled cashmere. These preliminary results confirmed that the NIR coupled with chemometric methods might be a useful tool for a rapid screening of recycled cashmere fibers and their raw quantification in a blend.

Keywords Recycling, cashmere fiber, NIR, chemometric analysis, quantitative analysis, SIMCA modelling, PCA analysis

Introduction

Cashmere is regarded as a luxury and rare animal fiber [1]. It is characterized by fineness, softness, and warmth and it derives from the fine dehaired fiber of goats undercoat [2]. Cashmere fibers are used to make a large variety of woolen products such as scarves, pull-overs and cardigans and it also used as raw materials for-high value adding by the textile industries [3]. Thus, cashmere has a high natural value related in part for its intrinsic properties and in part for the tradition associate with its production [4]. At the present, some European countries produce and import cashmere for the production of luxury garments that leads to a multi-billion-dollar global trade [5]. Globally, most of the world cashmere production belongs to Asian countries, such as China, Mongolia, Afghanistan, Kazakhstan, Iran and India which are able to produce different grade of cashmere. Relatively small quantities of cashmere are also produced by Britain, Australia and New Zealand [6]. Anyway, the global production of cashmere is negligible respect to other products in whole industrial textile, but it is a luxury component used by major fashion brands [4]. Due to its high value and in frame of “circular economy”, many brands start to produce and sell recycled cashmere products. “Recycled cashmere” is a term used to indicate fibers that derives from post-factory scraps and post-consumer textile wastes. Recycled cashmere product is a relatively evolving concept, and in literature little attention is paid on it, due to the fact that it is questionable to recycle cashmere fiber to produce luxury products without affecting the quality [7]. This kind of fibers have a lower quality than the virgin ones due to the mechanical recycling processes that they have been undergone. Current U.S. legislation requires to indicate the presence of recycled fibers in textile products, but false declaration and adulteration of virgin cashmere manufactures are not uncommon [4]. Thus, the identification and recognition of virgin and recycled cashmere fibers is becoming fundamental.

Throughout the literature, the most common methods used for distinguishing fine animal fibers are light microscopy (LM), scanning electron microscopy (SEM), MALDI-TOF and DNA analysis [8]–[10]. Light and SEM microscopies are

1 the most well-assessed methods due to their low cost and high efficiency. However, the accuracy of these techniques
2 depends mainly on the skills of the operator.

3 MALDI-TOF spectroscopy is a biochemical method for fiber identification of closely related species after an enzyme
4 digestion [11]. In this case, peptide mass finger printing (PMF) has been successfully used to identify the species origin.
5 Regarding the DNA analysis, it is based on the isolation of the DNA in cells of different kind of fibers. The DNA profiling
6 can identify fibers of different species if a suitable and unique DNA probe can be recognized [12]. However, this technique
7 has many drawbacks, for instance, it is difficult to differentiate DNA of closely species, it requires time for setting the
8 instrumentation and the analysis of DNA can be affected by different extraction yields [13]. The proposed methods, which
9 are able to discriminate from different animal hair origin, are not suitable in the case of virgin and recycled cashmere
10 identification.

11 Recently, a fast, efficient and objective detection technology has become an imperative scientific and a technological
12 demand. NIR technology has made significant progresses in many fields, such as food analysis [14], agrochemical quality
13 control [15] and in cognitive neuroscience research [16]. Actually, NIR technology has found out different applications
14 in quality control in order to protect the consumer from possible products adulteration. For instance, Prieto *et al.* used the
15 NIR and the multivariate analysis for quantitative and qualitative purposes in meat sector, whereas Schmutzler and co-
16 workers employ the NIR for the effective quality control of apples [17], [18]. Currently, NIR spectroscopy is becoming
17 a routine analytical technique in textile field, and actually in fiber recognition. The success of the NIR spectroscopy is
18 mainly due to the non-destructive detection and rapidity. NIR is a part of electromagnetic spectrum ranging from 13000
19 to 4000 cm^{-1} [19]. All the absorption bands in this region are the results of overtones and combinations originating in the
20 fundamental mid-range (4000-600 cm^{-1}) infrared region of the spectrum. The bonds involved are usually R-H (C-H, O-
21 H, N-H, S-H) [18]. After a calibration based on the chemometric methods, the NIR allows a simple and fast test for
22 samples. Generally, in the NIR approach, the principal component analysis (PCA) is performed. In PCA, multiple original
23 variables are replaced with a lower number of variables in order to eliminate the overlap spectroscopy data without losing
24 the main information. Actually, to characterize many raw materials or to control textile products and processes, the PCA
25 is coupled with soft independent modelling class analogies (SIMCA). The SIMCA method is based on modelling each
26 class by a separate model [20]. The scope of this statistical tool is to reduce dimensionality of the multivariate data set
27 and to find classes of samples in data set [21]. With the help of SIMCA model, many research groups successfully carried
28 out the correct identification and classification of samples in the field of biology, chemistry and medicine [21]–[23].
29 Using NIR spectroscopy, it is also possible to find quantitative relationships between the NIR spectra and some
30 characteristics of the samples, in general obtained by expensive or time-consuming tests. Those expensive measurements
31 can be predicted rapidly and with high accuracy using a NIR calibration. The multivariate regression methods, most
32 frequently used in NIR spectroscopy, are principal component regression (PCR) and partial least-squares (PLS)
33 regression.

34 NIR spectroscopy can be applied to various kinds of materials such as bulk, thin or thick polymers, tablets, human bodies
35 and to various fields, including polymer science, on-line monitoring, inorganic material research, medical diagnosis, and
36 NIR imaging [24]–[26]. NIR spectroscopy is particularly powerful in non-invasive, non-destructive and *in situ* analysis.
37 In the textile field NIR spectroscopy in combination with chemometric methods is an emerging quantitative and
38 qualitative technique for identification of textiles, adulteration control, textile wastes sorting for recycling, moisture
39 measurements, process control, forensic and cultural heritage textile analyses [7], [27], [28].

40 For instance, Wu *et al.* [29] proposed a quick and non-destructive method to identify some textile fibers as sorting
41 technique for textile wastes recycling. A fast and accurate identification model was built using SIMCA method and a
42 prediction accuracy of 100% for polyester, polyamide, acrylic, silk and wool was found. Ishfaq [30] discusses the
43 availability of NIR in recycling textile waste concluding that it will be possible a large commercial scale for sorting textile
44 automatically using NIR equipment. Lately, Zoccola *et al.*, in field of fine animal fibers, have successfully used the NIR
45 spectroscopy to classify and discriminate wool, cashmere, yak and angora fibers in raw state and quantify wool and
46 cashmere in blends, concluding that NIR spectroscopy coupled with multivariate analysis can be used as a tool for a first
47 and rapid screening of unknown animal fiber samples in the raw or combed sliver state and useful to obtain a fast and
48 coarse estimate of the amount of wool and cashmere fibers in a blend [8]. Concerning the cashmere, Wu *et al.* applied the
49 NIR spectroscopy combined with PCA to build an analysis model to discriminate 5 varieties of cashmere [31]. On the
50 field of recycled materials, Xin *et al.* developed a NIR spectroscopic method employing partial least squares discriminate
51 analysis able to distinguish between tissue papers made from virgin and recycled fibers in blends [32].

52 The aim of this work is the development of a novel, fast quantitative analysis for the identification of virgin and recycled
53 cashmere that might be important for developing portable or in-line devices for testing in real-time the quality or the

1 possible adulteration of commercial cashmere textiles. Herein, it is reported the qualitative and quantitative identification
2 of virgin and recycled cashmere fibers, by using NIR technology.

3

4

5 **Materials and methods**

6 In this study, 74 different virgin and recycled cashmere samples in the raw or combed states were analyzed. The recycled
7 cashmeres were provided by different textile companies in Biella, whereas for the analysis of the virgin fibers, samples
8 from CCMi (Camel Hair Manufacturers Institute) were used.

9 - *Moisture uptake*

10 Moisture uptake versus time of virgin and recycled cashmere samples, previously dried to constant mass at 105 °C, was
11 determined by weighting samples over time in a conditioned room with standard conditions (20 °C, 65% RH).

12 - *Sample preparation*

13 Before NIR spectra acquisition, all the recycled and virgin cashmere samples were dried in an oven at 105° C until
14 constant weight and then conditioned for 24 h under standard atmosphere (20 °C, 65% R.H.). For the quantitative analysis,
15 18 blends were prepared. More in detail, known amounts of recycled and virgin cashmere were mixed by hand cardes in
16 order to obtain 18 blends with a quantity of recycled cashmere ranging from 0% to 100%.

17 - *NIR spectra acquisition*

18 An FT-NIR spectrometer (PerkinElmer-Spectrum IdentiCheck) equipped with a reflectance sphere was used for the
19 analysis of the samples. For each sample, 32 scans were recorded between 10000 and 3700 cm⁻¹ with a resolution of 4
20 cm⁻¹. All the spectra were collected using 1g of cashmere sample.

21 - *Classification of the sample*

22 For the classification of the two different classes of cashmere, the SIMCA method was used. In this work, 54 samples (27
23 of virgin cashmere and 27 of recycled cashmere) were applied to build the model, and other 20 samples (10 of virgin and
24 10 of recycled cashmere) was used to test the reliability of the model. The number of principal component selected was
25 two. The method was built and calibrated using the PCR algorithm. The number of evaluated components are 2. The
26 spectra were processed in the range of 7500-3700 cm⁻¹ in order to avoid interferences caused by the pigmentation of the
27 cashmere. Spectral pre-processing was performed to eliminate noise and scattering. 8 cm⁻¹ resolution, standard normal
28 variate transformation (SNV) algorithms followed by Savitzky-Golay smoothing (5 smoothing points) was applied to
29 correct the baseline shift and multiplicative scatter effects and ~~the autoscaling function were also applied~~. In order to
30 provide a sample selection for the calibration and validation set as representative as possible and to ensure uniformity of
31 dataset, the X and Y distances were taken into account simultaneously, by applying the Kennard–Stone (KS) algorithm.
32 Predictive capabilities of this calibration model and its validation was evaluated by using several statistical parameters,
33 i.e. the coefficient of determination (R²), standard error of estimate (SEE) and standard error of prediction (SEP)

34 In SIMCA, the PCA was performed for each class. The PCA is a useful chemometric method for spectral data compression
35 and information extraction [33]. It allows to describe with a small number of principal components (PC) the most
36 important information contained in a spectrum. Thus, SIMCA model creates a multidimensional box for the two classes
37 and it allows to classify an unknown sample in accordance with its fitting to one of the two classes constructed [7].

38 - *Quantitative analysis*

39 The 18 samples of virgin and recycled blend fibers were obtained as previously described and used for the calibration.
40 The weight percentage of virgin cashmere in each sample was associated with the respective spectrum. The data were
41 processed by means of Spectrum Quant+ (PerkinElmer) software package and the PCR algorithm were used for the
42 calibration in the range of spectra acquisition 7500-3700 cm⁻¹. The principal statistics used to assess the quality of the
43 results were the standard error of prediction (SEP), which is the magnitude of error expected when independent samples
44 are predicted using the model, the standard error of estimate (SEE), and the coefficient of determination of the calibration
45 (R²).

46 - *SEM analysis*

1 The morphological investigation of the recycled and virgin cashmere was carried out by Zeiss EVO 10 SEM. SEM
 2 operated with an acceleration voltage of 15 kV, 50 pA probe and 20 mm working distance. Samples were mounted on
 3 aluminum stubs and coated with a gold layer 20-30 nm thick in argon, using an Emitech K 550 sputter coater with a
 4 current of 20 mA for 180 s.

5

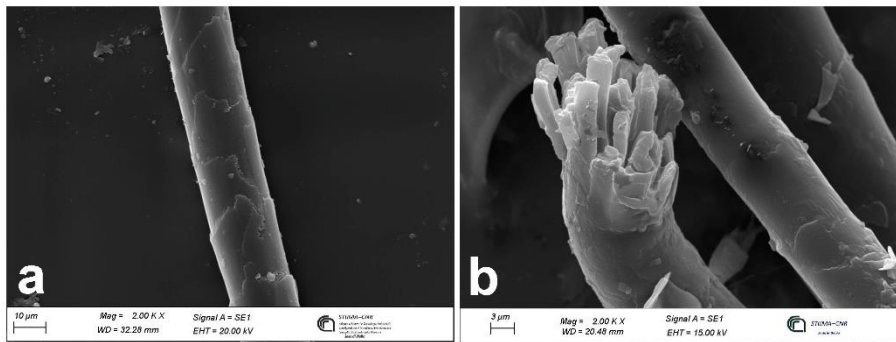
6 - *Light microscopy*

7 The microscopic investigation was carried out on virgin and recycled cashmere with a DM-L Light Microscope (Leica)
 8 in the Transmitted Light mode. The specimens were prepared dripping some drops of glycerin onto the sample placed on
 9 a glass microscope slides.

10 **Results**

11 *Virgin and recycled cashmere characterizations and spectral analysis*

12 Virgin and recycled cashmere samples were examined with a scanning electron microscopy (SEM), in order to evaluate
 13 differences in morphology of fibers.



14

15 **Figure 1:** SEM analysis of virgin (a) and recycled (b) cashmere

16 Figure 1a shows the typical morphology of a virgin cashmere fiber [34]. Fiber has a cylindrical shape with an even
 17 diameter. The surface scale displays the typical overlapped patterns and the scales are relatively distant and smooth. The
 18 mean scale frequency is 6-7 per 100 μm and the thickness of the cuticle cells at the distal edge is less than 0.5 μm . In
 19 Figure 1b recycled cashmere fiber with typical breakings due to mechanical re-processing is shown and a typical partial
 20 cleavage (semi-rupture) called “brushes” is reported. The SEM analysis is often used for recycled animal fiber
 21 identification, but this method has some drawbacks due to the intrinsic subjectivity of the technique.

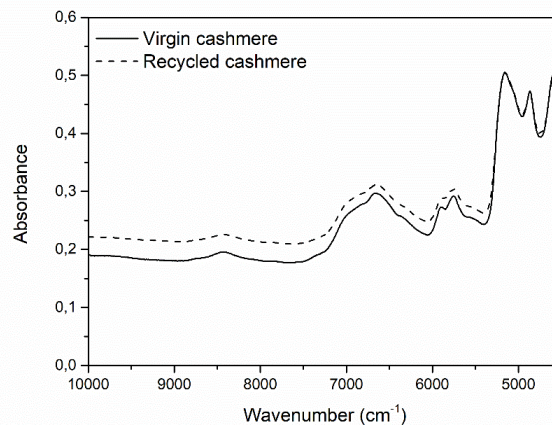
22 In this work, NIR spectroscopy analysis has been used as an objective method to identify recycled and virgin cashmere
 23 fibers. Since specific regions in the NIR spectra are sensitive to the concentration of water absorbed, the samples were
 24 dried and conditioned in order to evaluate possible differences in moisture uptake. The obtained results for virgin and
 25 recycled cashmere samples are listed in Table 1.

Time (h)	Water adsorbed by virgin cashmere (%)	Water adsorbed by recycled cashmere (%)
1	0	0
2	8.4	8.7
3	9.4	10.4
4	9.6	10.6
5	9.8	10.7
6	9.8	10.7
7	9.9	10.7
15	11.1	11.2
24	11.3	11.6

26 **Table 1:** Moisture uptake (%) by virgin and recycled cashmere recorded at different times

1 As shown in table 1, after 3 h, the recycled cashmere absorbed a higher amount of water than the virgin sample and this
2 behavior can be related with the presence of breakages along the fibers that allows the diffusion of water molecules into
3 the structure. Indeed, the disruption of hydrophobic external cuticle layer leads the recycled cashmere to be faster in
4 water adsorption, but after 15 h, water absorption slightly increases for both the samples until the achievement of
5 equilibrium conditions leading to the same moisture uptake. The initial differences in moisture absorption could be
6 emphasized and exploited in NIR spectroscopy, but the equilibrium states are easier to manage in analytic conditions,
7 which are more reproducible among laboratories, without affecting the speed and simplicity offered by NIR spectroscopy.
8 For this reason, all samples were dried at 105°C until constant weight and then conditioned for 24 h before being analyzed.

9 The conditioned samples were analyzed using the NIR spectrophotometer. All the NIR spectra collected show similar
10 patterns and for brevity, in Figure 2, one spectrum for a virgin cashmere sample (black solid line) and one for a recycled
11 one (black dotted line) are reported. For both the samples, the peak positions and overall shape are very similar due to the
12 same chemical composition. The shoulder at 7000 cm⁻¹ is assigned to the first overtone of the O-H stretching vibration of
13 the adsorbed water whereas the bands at 5200 cm⁻¹ is related with the combination of O-H stretching and H-O-H bending
14 vibration of water. The overtone at 5800 cm⁻¹ is ascribed to C-H stretching of the protein side chains [8].

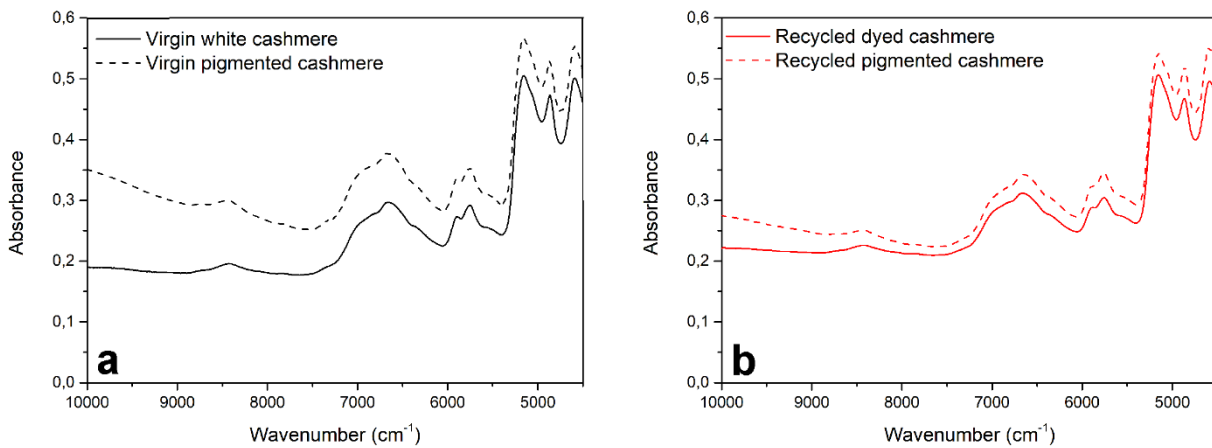


15

16 **Figure 2:** Virgin cashmere (black solid line) and recycled (black dotted line)

17

18 However, a significant discrepancy in the baseline absorption intensity can be observed. This spectral difference should
19 be associated with the physical differences between the two kinds of fibers. These variations can be related with different
20 physical characteristics, such as sample packing, surface roughness and fiber diameter and they cause the light scattering
21 effect [31]. Another characteristic that alters the NIR spectra is the present of pigments in cashmere fibers. Indeed,
22 cashmere is not only white, but it can be found in different shade of brown. These difference are mainly due to the
23 presence of pigments, mainly, melanin. Thus, the virgin and the recycled cashmere fibers can be pigmented or not, but a
24 visual comparison between the two kinds of cashmere is difficult because recycled samples are usually found out dyed in
25 different colors. Since the presence of color used for dyeing cashmere does not affect the spectral baseline, the presence
26 of pigments can be detected by NIR analysis [7] [35]. In Figure 3a the spectra of virgin (black solid line) and virgin
27 pigmented cashmere (black dotted line) are reported, whereas in Figure 3b the spectra of recycled (black solid line) and
28 recycled pigmented cashmere (red dotted line) are shown.



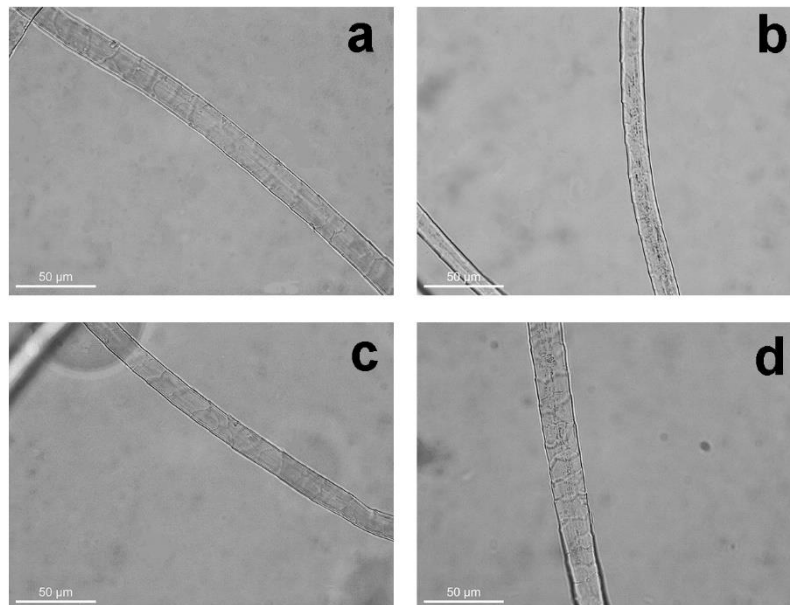
1

2 **Figure 3:** a) Spectra of virgin cashmere (black solid line) and virgin pigmented cashmere (red solid line); b) Spectra of
 3 recycled cashmere (black dotted line) and recycled pigmented cashmere (red dotted line)

4

5 As shown in Figure 3, due to the presence of pigments on the surface of the fibers, both the pigmented cashmere presents
 6 a heavy drift of the spectral baseline in wave region from 10000 to 7500 cm^{-1} .

7 The presence of pigments in the cashmere samples can be also evaluated using light microscopy, as shown in Figure 4:



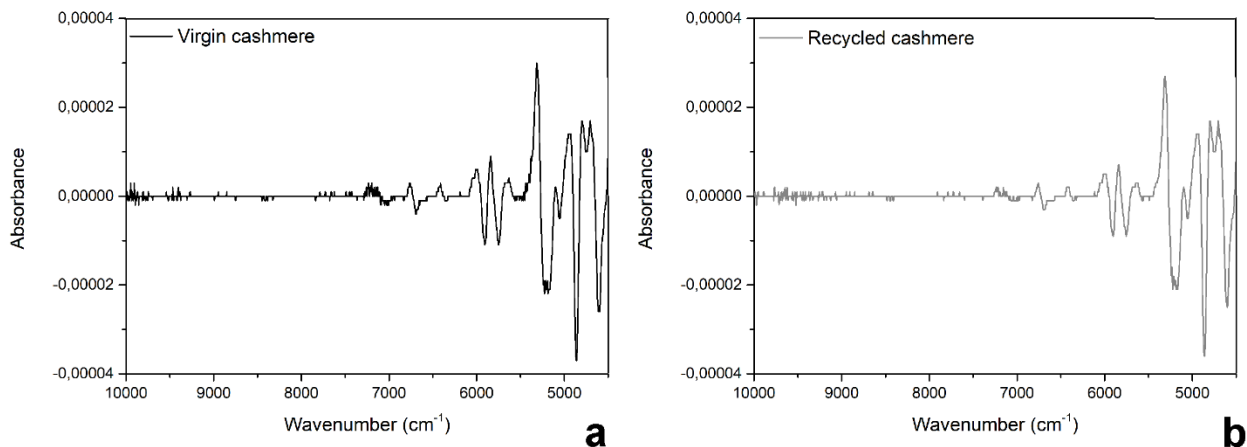
8

9 **Figure 4:** Light microscopy images of virgin cashmere (a), pigmented virgin cashmere (b), recycled cashmere (c) and
 10 recycled pigmented cashmere (d)

11 Figure 4a depicts a white virgin cashmere with typical regular scale shape and frequency and similar surface features can
 12 be observed in recycled cashmere (Figure 4c). Figure 4b displays a pigmented virgin cashmere in which are clearly visible
 13 pigment granules unevenly distributed on the surface of the fiber [36]. The same pigment deposition in the fibers can be
 14 observed in recycled cashmere (Figure 4d), supporting the considerations already pointed out by the NIR analysis.

15 The baseline shift makes difficult a visual inspection of the raw NIR spectra and hard to discriminate virgin and recycled
 16 cashmere. To eliminate the influences and to enhance the small changes in the spectra, the 2nd derivative was applied to

1 all the spectra collected, but for brevity Figure 5 shows a spectrum for virgin cashmere samples (a) and one for recycled
2 ones (b).



3
4 **Figure 5:** Second derivative spectra of virgin (a) and recycled (b) cashmere

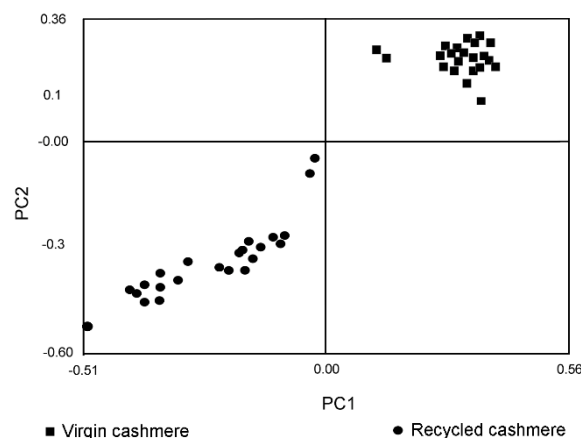
5

6 After spectral processing, the signal resolution has been significantly improved and the baseline shift has been also
7 reduced. Furthermore, the 2nd derivative spectra of virgin and recycled cashmere are very similar, showing the same
8 characteristic absorption peaks. Actually, no differences can be detected in the spectral range of 5000 and 3700 cm⁻¹,
9 region referred to the protein type structure. Indeed, the prevalence of α -helix secondary structure is clearly demonstrated
10 by the presence of the peaks at 4615 cm⁻¹ and by the absence of the peak at 4525 cm⁻¹ attributed to the β -sheet structure
11 for both the virgin and recycled cashmere [8]. Thus, it can be concluded that the spectral differences are mainly originated
12 from the physical state of the virgin and recycled cashmere.

13 *Classification of virgin and recycled cashmere*

14 In order to create a calibration model, 54 samples were randomly selected and the dataset composed of 20 samples (10 of
15 virgin cashmere and 10 of recycled cashmere) were used for external validation. PCR was performed in the range from
16 7500-3700 cm⁻¹, to cover the whole spectral variation. The pretreated data with SNV methods were used to develop
17 regression models with PCR. The PCR shows a R^2 of 0.97, SEE of 0.061 and a SEP of 0.081.

18 All the recorded spectra for the two types of cashmere were analyzed using the principal component analysis, PCA. The
19 PC is a linear combination of spectra and it requires no knowledge of the data set structure and acts to reduce the dimension
20 of the multivariate data. Thus, PC describes the source of variation between the two sets of cashmere. The first principal
21 component explained 91% of variation between the virgin and recycled cashmere whereas the second component is 8.5%.
22 The interpretation of the results of a principal component analysis is usually carried out by visualization of the component
23 scores. Figure 6 shows the score plot of PC1 vs PC2.



24
25 **Figure 6:** Score plot PC1 vs PC2

1 A clustering tendency related with the fiber origin, virgin or recycled, is clearly displayed, with a distribution from the
2 left to the right. Essentially, the recycled cashmere is located in the quadrant where PC1 and PC2 were negative.
3 Contrarily, the virgin sample is situated where the PC1 and PC2 value were positive. In other words, a good discrimination
4 between virgin and recycled cashmere was obtained. Thus, the PC analysis revealed a clustering tendency that can be
5 related with differences in physical and morphology characteristics of the fibers [7]. The differences between the fibers,
6 mainly due to the recycling process, creates changes in the physical structure of the cashmere. These morphological
7 differences between the two cashmeres can be stressed by the chemometric analysis leading to a creation of two different
8 classes and allowing a discrimination of chemically similar samples.

9 *SIMCA modelling and identification*

10 The SIMCA recognition technology was applied for the NIR model of virgin and recycled cashmere. It is essential to
11 perform mathematical pre-treatments to reduce the systematic noise, such as baseline variation, light scattering and path
12 length differences. In this study, a data pre-processing method was applied which was the Standard Normal Variate
13 transformation (SNV) [33]. Generally, the SNV process requires the de-trending to remove the effect of the baseline shift
14 and the curvilinearity, but, in this case, was not used. Thus, the SIMCA technology allows to evaluate the interclass
15 distance from the clustering tendency of the virgin and recycled cashmere when subjected to PC analysis. The value of
16 the interclass distance represents the differences between the recycled and virgin cashmere. Actually, if this value is high,
17 the diversity between the samples is great, whereas a low value indicates high similarity between the samples. Therefore,
18 a high interclass distance leads to an easy classification and identification. On the contrary, a low value indicates that the
19 two samples are difficult to discriminate [20]. The interclass distance evaluated after modelling with SIMCA is 249.77,
20 consequently well above the limit of 3.0 regarded as significant to identify two groups of samples as different classes,
21 hence a correct classification of fibers has been obtained.

22 The prediction capacity of the SIMCA model was tested, adding to the 54 samples previously analyzed, 20 samples (10
23 of recycled and 10 of virgin cashmere fibers) in order to make a validation of the model. The obtained results are listed
24 in Table 2.

25

Sample	Recognition rate (%)	Rejection rate (%)
Virgin cashmere	98% (36/37)	100% (37/37)
Recycled cashmere	100% (37/37)	100% (37/37)

26 **Table 2:** Percentages of recognition and rejection from SIMCA modelling

27 As shown in Table 2, the recycled cashmere has been recognized as belonging to the respective class, giving 100%
28 recognition. Furthermore, the recycled sample has been rejected from virgin cashmere class with a 100% of rejection.
29 Regarding the virgin cashmere, the recognition rate is 98%. This incorrect classification can be related with the very dark
30 pigmentation of the sample. Despite of this result, an accurate classification can be obtained. Furthermore, all samples
31 were rejected from other class, giving 100% of rejection. In the achieved validation, all the samples were correctly
32 identified as belonging to the proper class and no false positive results were found out. This result clearly highlights the
33 powerful of the evaluated method. The technique can be successfully applied for a correct discrimination between virgin
34 and recycled cashmere resulting in a fast and non-destructive method. Therefore, NIR spectroscopy can be easily applied
35 in industrial field for fiber discrimination as a fast screening method to avoid frauds.

36 *Quantitative analysis of virgin and recycled cashmere*

37 In this perspective, it is necessary to recognize the quality of cashmere fibers also when they are in blend. Recycled
38 cashmere fibers are often mixed with different amounts of virgin fibers to obtain yarns with suitable mechanical properties
39 for fabrics re-manufacturing.

40 Therefore, the identification of recycled cashmere in blends with virgin one it has been studied. With a view to prevent
41 possible counterfeiting of samples, the evaluation of the amount of recycled fibers has been carried out using a calibration
42 curve. The calibration was performed using 18 samples produced by carefully mixing known amounts of virgin and
43 recycled cashmere. The obtained result is depicted in figure 7:

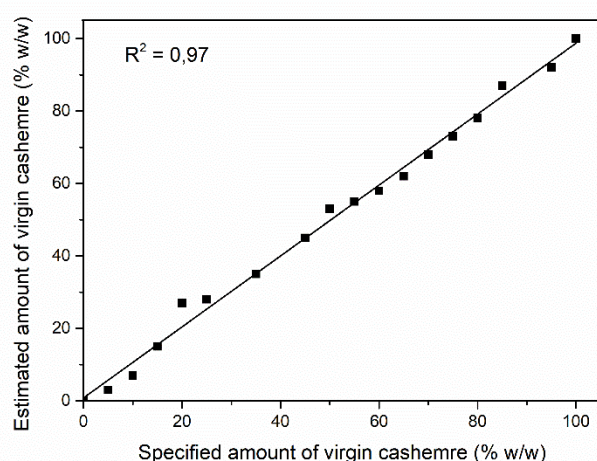


Figure 7: Specified values (virgin cashmere % w/w) vs. estimated values

Figure 7 compares the % w/w of virgin cashmere amount with the amount predicted by NIR spectral evaluation. The Standard Error of the Estimate (SEE) which is the square root of the residual variance divided by the degrees of freedom, was found out to be 5.2. The magnitude of the expected when independent sample were predicted using the model were calculated as the Standard Error of Prediction (SEP) and it was about 5.8 for a mean value of 50. Also, the R^2 was evaluated and a value of 0.97 was obtained from the calibration. From the calibration curve, it is possible to deduce that up to 5% of recycled cashmere can be detected within a virgin cashmere blend [8]. Thus, this method can be successfully applied to identify even small quantity of recycled cashmere, preventing possible adulteration.

Conclusion

In this work, NIR spectroscopy coupled with chemometric method has been successfully used for the identification of virgin and recycled cashmere and for the quantitative analysis of cashmere blends. The differences between fibers were evaluated throughout the elaboration of the NIR spectra and correlated with physical and morphological characteristics rather than chemical features of the cashmere fibers. The SIMCA method was used for the classification of samples and the interclass distance found is large enough to allow a sure identification of the samples. The prediction test carried out gave no false positive results allowing a correct identification of unknown samples. Furthermore, the results of the quantitative calibration of cashmere blends show an SEP value of 5.8 w/w indicates that it is possible to correctly identify the amount of recycled cashmere in blend with virgin fibers, even if the amount of recycled fibers is only 5% (w/w). Thus, the NIR spectroscopy coupled with chemometric analysis is proved to be a suitable tool for a fast and sure identification of recycled and virgin cashmere fibers and to be successfully applied in industrial fields for preventing possible adulteration of textile luxury product.

Bibliography

- [1] Y. Zhong, K. Lu, J. Tian, and H. Zhu, "Wool/cashmere identification based on projection curves," *Text. Res. J.*, vol. 87, no. 14, pp. 1730–1741, 2017.
- [2] B. A. McGregor, "Variation in the softness and fibre curvature of cashmere, alpaca, mohair and other rare animal fibres," *J. Text. Inst.*, vol. 105, no. 6, pp. 597–608, Jun. 2014.
- [3] B. . Mcgregor, "Recent advances in marketing and product development of mohair and cashmere," in *7th International Conference on Goats*, 2000, pp. 631–636.
- [4] S. I. Ishrat, N. P. Grigg, N. Jayamaha, and V. Pulakanam, *Sustainability in Luxury Fashion Business*, no. August. Springer Singapore, 2018.
- [5] S. Waldron, C. Brown, and A. M. Komarek, "The Chinese Cashmere Industry: A Global Value Chain Analysis," *Dev. Policy Rev.*, vol. 32, no. 5, pp. 589–610, 2014.
- [6] D. B. Shakyawar, A. S. M. Raja, A. Kumar, P. K. Pareek, and S. A. Wani, "Pashmina fibre -Production, characteristics and utilization," *Indian J. Fibre Text. Res.*, vol. 38, no. 2, pp. 207–214, 2013.

- 1 [7] X. Sun *et al.*, “A novel drying-free identification method of cashmere textiles by NIR spectroscopy combined
2 with an adaptive representation learning classification method,” *Microchem. J.*, vol. 149, p. 104018, 2019.
- 3 [8] M. Zoccola, N. Lu, R. Mossotti, R. Innocenti, and A. Montarsolo, “Identification of wool, cashmere, yak, and
4 angora rabbit fibers and quantitative determination of wool and cashmere in blend: A near infrared spectroscopy
5 study,” *Fibers Polym.*, vol. 14, no. 8, pp. 1283–1289, 2013.
- 6 [9] Y. Zhong, K. Lu, J. Tian, and H. Zhu, “Wool/cashmere identification based on projection curves,” *Text. Res. J.*,
7 vol. 87, no. 14, pp. 1730–1741, Aug. 2016.
- 8 [10] W. M. K. van Niekerk, S. Keva, M. Roets, and R. J. Coertze, “The accuracy of Video Image Analysis (VIA)
9 and Optical Fibre Diameter Analysis (OFDA) to measure fibre diameter of cashmere,” 2004.
- 10 [11] A. Koerner, S. Sareian, G. Elders, C. Henkel, and M. Moeller, “Identification of marker peptides in fine animal
11 hair for discrimination with regard to species and breed,” *Proc. 12th Int. Wool Res. Conf.*, vol. I, p. 28, 2010.
- 12 [12] G. Nelson, P. F. Hamlyn, L. Holden, and B. J. Mccarthy, “A Species-Specific DNA Probe for Goat Fiber
13 Identification,” *Text. Res. J.*, vol. 62, no. 10, pp. 590–595, 1992.
- 14 [13] M. Tang *et al.*, “A real-time PCR method for quantifying mixed cashmere and wool based on hair
15 mitochondrial DNA,” *Text. Res. J.*, vol. 84, no. 15, pp. 1612–1621, 2014.
- 16 [14] M. Zareef *et al.*, “An Overview on the Applications of Typical Non-linear Algorithms Coupled With NIR
17 Spectroscopy in Food Analysis,” *Food Eng. Rev.*, vol. 12, no. 2, pp. 173–190, 2020.
- 18 [15] S. Armenta, S. Garrigues, and M. Guardia, “Quality Control of Agrochemical Formulations by Diffuse
19 Reflectance Near Infrared Spectrometry,” *J. Near Infrared Spectrosc.*, vol. 16, Apr. 2008.
- 20 [16] J. Fuster *et al.*, “Near-infrared spectroscopy (NIRS) in cognitive neuroscience of the primate brain.,”
21 *Neuroimage*, vol. 26, no. 1, pp. 215–220, May 2005.
- 22 [17] M. Schmutzler, O. M. D. Lutz, and C. W. Huck, “Analytical pathway based on non-destructive nirs for quality
23 control of apples,” *Infrared Spectrosc. Theory, Dev. Appl.*, pp. 43–59, 2014.
- 24 [18] N. Prieto, O. Pawluczuk, M. E. R. Dugan, and J. L. Aalhus, “A Review of the Principles and Applications of
25 Near-Infrared Spectroscopy to Characterize Meat, Fat, and Meat Products,” *Appl. Spectrosc.*, vol. 71, no. 7, pp.
26 1403–1426, May 2017.
- 27 [19] S. Kawata and H. M. Heise, *Near-Infrared Spectroscopy*. .
- 28 [20] S. WOLD and M. SJÖSTRÖM, “SIMCA: A Method for Analyzing Chemical Data in Terms of Similarity and
29 Analogy,” pp. 243–282, 1977.
- 30 [21] N. Vogt and H. Knutsen, “SIMCA pattern recognition classification of five infauna taxonomic groups using
31 non-polar compounds analysed by high resolution gas chromatography,” *Mar. Ecol. Prog. Ser.*, vol. 26, pp.
32 145–156, 1985.
- 33 [22] E. Jellum, I. Bjpirnson, R. Nesbakken, E. Johansson, and S. Wold, “CLASSIFICATION OF HUMAN
34 CANCER CELLS BY MEANS OF CAPIL- LARY GAS CHROMATOGRAPHY AND PATTERN
35 RECOGNITION ANAL- The metabolic profiles of brain biopsies obtained at surgery were recorded using
36 capillary gas chromatography (GC). About 160 peaks were seen ,” vol. 217, pp. 231–237, 1981.
- 37 [23] W. J. Dunn and S. Wold, “Relationships between chemical structure and biological activity modeled by SIMCA
38 pattern recognition,” *Bioorg. Chem.*, vol. 9, no. 4, pp. 505–523, 1980.
- 39 [24] M. Manley, “Near-infrared spectroscopy and hyperspectral imaging: Non-destructive analysis of biological
40 materials,” *Chem. Soc. Rev.*, vol. 43, no. 24, pp. 8200–8214, 2014.
- 41 [25] S. Serranti and G. Bonifazi, *Techniques for separation of plastic wastes*, no. January 2018. Elsevier Ltd, 2019.
- 42 [26] Y. Ozaki, “Near-infrared spectroscopy--its versatility in analytical chemistry.,” *Anal. Sci. Int. J. Japan Soc.*
43 *Anal. Chem.*, vol. 28, no. 6, pp. 545–563, 2012.
- 44 [27] E. Cleve, E. Bach, and E. Schollmeyer, “Using chemometric methods and NIR spectrophotometry in the textile
45 industry,” *Anal. Chim. Acta*, vol. 420, no. 2, pp. 163–167, 2000.
- 46 [28] J. Yu and I. S. Butler, “Recent Applications of Infrared and Raman Spectroscopy in Art Forensics: A Brief
47 Overview,” *Appl. Spectrosc. Rev.*, vol. 50, no. 2, pp. 152–157, Feb. 2015.

- 1 [29] C. Zhou, G. Han, B. K. Via, Y. Song, S. Gao, and W. Jiang, "Rapid identification of fibers from different waste
2 fabrics using the near-infrared spectroscopy technique," *Text. Res. J.*, vol. 89, no. 17, pp. 3610–3616, Dec.
3 2018.
- 4 [30] M. Ishfaq, "Infrared technology and its applications in textile recycling technology : improving sustainability in
5 clothing industry," p. 86, 2015.
- 6 [31] G. F. Wu and Y. He, "Identification of varieties of cashmere by Vis/NIR spectroscopy technology based on
7 PCA-SVM," *Guang Pu Xue Yu Guang Pu Fen Xi/Spectroscopy Spectr. Anal.*, vol. 29, no. 6, pp. 1541–1544,
8 2009.
- 9 [32] L.-P. Xin, X.-S. Chai, D. Barnes, C.-X. Chen, and R.-Q. Chen, "Rapid Identification of Tissue Paper Made
10 from Blended Recycled Fibre by Fourier Transform near Infrared Spectroscopy," *J. Near Infrared Spectrosc.*,
11 vol. 22, no. 5, pp. 347–355, 2014.
- 12 [33] I. C. Yang *et al.*, "Integration of SIMCA and near-infrared spectroscopy for rapid and precise identification of
13 herbal medicines," *J. Food Drug Anal.*, vol. 21, no. 3, pp. 268–278, 2013.
- 14 [34] K. Lu, J. Luo, Y. Zhong, and X. Chai, "Identification of wool and cashmere SEM images based on SURF
15 features," *J. Eng. Fiber. Fabr.*, vol. 14, p. 1558925019866121, Jan. 2019.
- 16 [35] M. Zoccola, R. Mossotti, R. Innocenti, D. I. Loria, S. Rosso, and R. Zanetti, "Near infrared spectroscopy as a
17 tool for the determination of eumelanin in human hair.," *Pigment cell Res.*, vol. 17, no. 4, pp. 379–385, Aug.
18 2004.
- 19 [36] Z. Su, A. A. Deghani, L. Zhang, T. King, and B. Greenwood, "Vision system for auto-detection of cashmere
20 pigmented fibers," in *Proc.SPIE*, 2003, vol. 5011.

21