

Review

Analytical Methods for the Identification and Quantitative Determination of Wool and Fine Animal Fibers: A Review

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Abstract: The identification and quantitative determination of wool and fine animal fibers are of great interest in the textile field because of the significant price differences between them and common impurities in raw and processed textiles. Since animal fibers have remarkable similarities in their chemical and physical characteristics, specific identification methods have been studied and proposed following advances in analytical technologies. The identification methods of wool and fine animal fibers are reviewed in this paper, and the results of relevant studies are listed and summarized, starting from classical microscopy methods, which are still used today not only in small to medium enterprises but also in large industries, research studies and quality control laboratories. Particular attention has been paid to image analysis, Nir spectroscopy and proteomics, which constitute the most promising technologies of quality control in the manufacturing and trading of luxury textiles and can find application in forensic science and archeology.

Keywords: wool; cashmere; fine animal fibers; analytical methods; identification



Citation: Zoccola, M.; Bhavsar, P.; Anceschi, A.; Patrucco, A. Analytical Methods for the Identification and Quantitative Determination of Wool and Fine Animal Fibers: A Review. *Fibers* **2023**, *11*, 67. <https://doi.org/10.3390/fib11080067>

Academic Editor: Damien Soulat

Received: 3 May 2023

Revised: 21 July 2023

Accepted: 27 July 2023

Published: 2 August 2023



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1. Introduction

Fine animal fibers, also known as specialty or luxury fibers or hair, derive from animal species other than sheep and have been selected according to their characteristics and performances and their possibility of being spun with traditional systems. These fibers are generally employed to obtain valuable and luxury textile items due to their characteristics of finesses, softness, gloss, luster, color, wear comfort properties and even rarity. The limited production quantities and sometimes the difficulties of supply make their price relatively high compared to wool [1]. The relatively non-damaging production of animal fibers in comparison with synthetic fibers and their biodegradability instead of microplastics pollution production make them a partial replacement for synthetic fibers, even if in small amounts in terms of quantity [2]. Moreover, the production and commercialization of some animal fibers like cashmere, alpaca, camel and cashgora have a great impact on the rural economy, preventing migration to cities and protecting mountain areas in remote pastoral regions [3,4]

Labeling textiles to show their composition necessitates the use of analytical control methods not only for the finished product but also for the raw materials and the material throughout all stages of production. Aside from the legal labeling problems, the price difference between the constituents of many popular fiber blends is a primary motivator for developing precise analytical processes.

Other fields of interest are forensic science, textile care and laundry services, archeology and other investigative sectors [5–9].

Following Annex I (list of the textile fibers names) of EU Regulation No 1007/2011 of 27 September 2011 and the consolidated version of 15 February 2018, fine animal hair is classified in the number 2 category as alpaca, llama, camel, cashmere, mohair, angora, vicuña, yak, guanaco, cashgora, beaver and otter, followed or not by the word ‘wool’

or 'hair'. Additions must be made to this list to identify some species that must be killed to obtain their fine hair, such as shatoosh, which was identified as an endangered species and was categorized as a category I animal under state protection and whose hair commercialization is forbidden [10].

Wool is a fiber derived from the fleeces of sheep or lambs (*Ovis aries*). The most used wool in the textile field is produced by the Merinos breed from Australia, selected for the production of fine, high quality and quantity wool (about 4–5 kg of raw wool per year per sheep) [11]. Fine animal hair comes from goats (cashmere goat (*Capra hircus laniger*), mohair or angora goat (*Capra hircus aegagrus*), and angora goats are crossed with feral Australian or New Zealand goats to produce cashgora), camels (camel (*Camelus bactrianus*) and South American camelids, lama (*Lama glama*), alpaca (*Vicugna pacos*), vicuña (*Vicugna vicugna*), guanaco (*Lama guanicoe*), bovines (yak (*Bos grunniens*)), and rabbit (Angora rabbit (*Oryctolagus cuniculus*)) (see Figure 1).

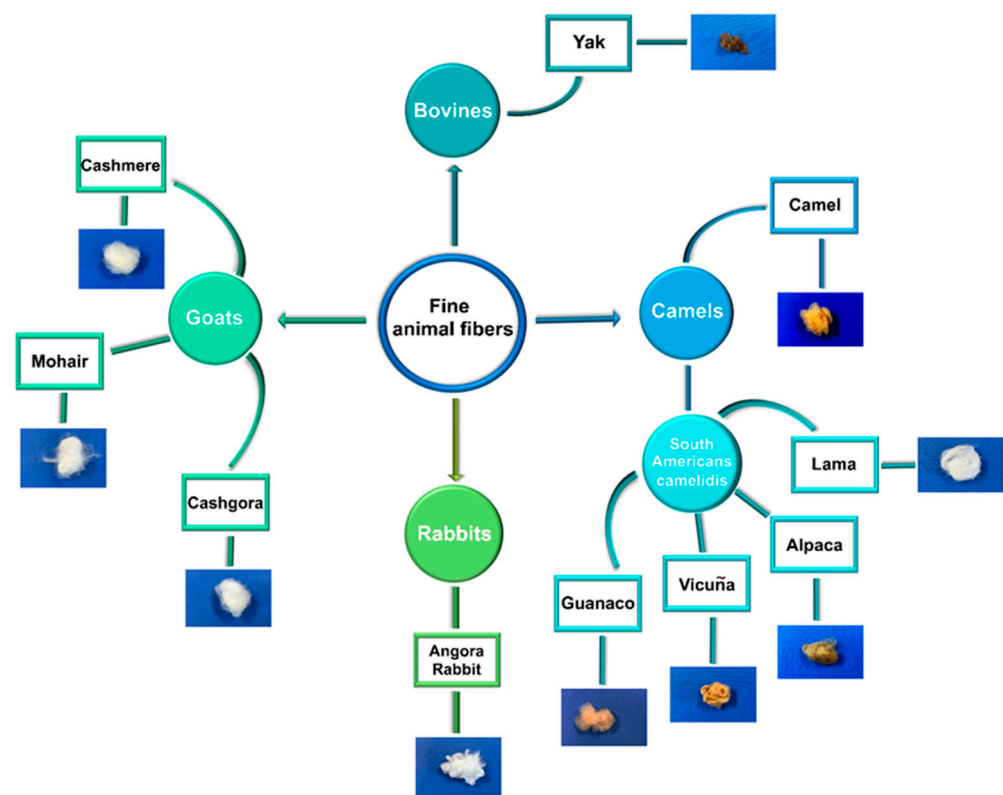


Figure 1. Fine animal fibers.

The main animal breeding countries and principal characteristics of fine animal fibers are shown in Table 1. The fibers can originate from the whole fleeces or, in general, the finest ones, from the smooth and soft undercoat of animals bred at high altitudes, while the long and coarse hair from the upper coat had to be removed with a process named dehairing [12]. Colors are due to the presence of melanin pigments, divided into eumelanin, responsible for brown and black colors, and pheomelanin, for yellow and reddish colors [13].

Table 1. Fine animal fibers: main breeding countries or areas and characteristics.

Fiber	Main Breeding Countries	Coat or Undercoat	Fineness	Natural Color	Reference
cashmere	China, Mongolia, Afghanistan and Iran	undercoat	15–19 μm	white, gray and brown	[14]
mohair	South Africa and the U.S.A.	coat	25–55 μm	white and glossy	[15]
cashgora	Australia and New Zealand	coat	18 to 23 μm	white	[16]
camel	China, Mongolia, Iran, Afghanistan, Russia, New Zealand and Australia	undercoat	5–20 μm	golden tan	[14]
lama	South America	coat	10–44 μm	various colors, sometimes brown	[17–19]
alpaca	South America	coat	20–40 μm	Grey, fawn white, black, café, etc.	[17–19]
vicuña	Perù, Bolivia and Argentina	undercoat	13–14 μm	from golden to cinnamon	[17,18]
guanaco	South America	undercoat	16.5–24 μm	light brown	[17,18]
yak	China, Afghanistan, Nepal, and other Asian countries	undercoat	15–20 μm	dark brown	[20]
angora	China	coat	14–16 μm	white	[21]

Wool and fine animal fibers have similar chemical, physical and histological characteristics, which is why their mixtures cannot be mechanically or chemically separated through solubility in selective solvents. They are composed of the protein keratin, which has a high sulfur content and strong disulfide bonds that render it insoluble in water and resistant to a variety of chemical agents [22].

From a morphological perspective, the cuticle, the cortex, and the cell membrane complex are the three main structural components of wool and other animal fibers. A thin layer of flat, overlapping “cuticle cells” that surrounds the cortex makes up the cystine-rich cuticle, and it is strongly cross-linked. The cortex is made up of extended “cortical cells” that are parallel to the fiber axis and include micro-fibrils of α -helix crystalline proteins with low sulfur contained within an amorphous matrix of high sulfur and glycine/tyrosine-rich proteins. The cell membrane complex serves to bind cortical and cuticle cells together and is also known as intercellular cement [23]. In the fibers with larger diameters, or in some fine animal fibers (e.g., angora rabbit), an inner channel named medulla, both continuous or interrupted or fragmental, can be present [24].

In this review, the results of relevant research from morphological, chemical and biotechnological methods of wool and animal fibers identification and quantification are shown and discussed (Figure 2).

Each group of methods moved from general or subjective analysis to modern techniques following technological innovations and targeted approaches as technology and animal fiber studies have progressed. Regarding the morphological analysis of the fibers, many studies are now focusing on image analysis to try to overcome the problems related to subjective and time-consuming classic techniques of recognition of the fibers using optical or electron microscopy performed by expert operators. As far as chemical techniques are concerned, analysis moved from the more dated techniques related to the chemical components of the fibers, i.e., amino acids and internal lipids, to much faster spectroscopic analyses, which take advantage of modern chemometric techniques of spectra evaluation. Finally, biotechnological techniques have passed from simple one- or two-dimensional electrophoresis to DNA analysis and finally to proteomics as proteins with attributes like persistence, quantity and DNA derivation make up the majority of animal fibers. Among the different fibers, the majority of examined papers concern the distinction between wool and cashmere, with cashmere being the most produced and marketed animal fiber in the world. The global market for cashmere clothing was estimated to be worth USD 3015.98

million in 2021 and is anticipated to grow to USD 4105.41 million by 2029, showing a CAGR of 3.93% from 2022 to 2029 [25].

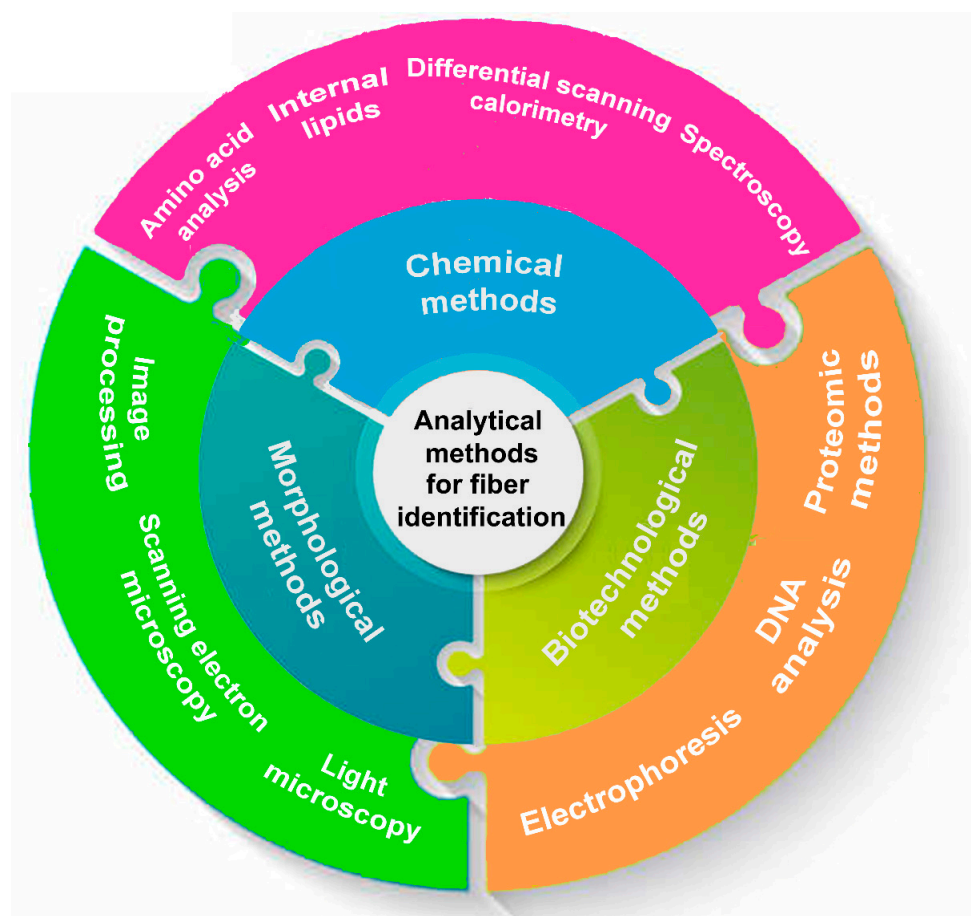


Figure 2. Analytical methods for wool and fine animal fibers identification.

2. Analytical Methods

2.1. Morphological Methods to Identify Wool and Fine Animal Fibers

The identification of fine animal fibers is an essential task in many activities ranging from research studies and quality control laboratories to large industries and small to medium enterprises (SMEs). Classical and extensively used methods for the identification of wool and fine animal fibers are morphological methods using Light- (LM) and Scanning Electron Microscopies (SEM). Although new instrumental identification techniques that originated with technical advancements are now available, these traditional methods are prevalent in small industries as they are the most affordable alternative.

2.1.1. Light- and Scanning Electron Microscopy

LM and SEM are the old and classical methods to identify wool and fine animal fibers. Using LM, fiber snippets of fixed length are cut and dispersed in a mounting medium with an appropriate refractive index, e.g., glycerine. Morphological characteristics that allow distinguishing wool and different fine animal fibers using LM are based on cuticular cell morphology, pigment distribution and fiber medulla, as described in great detail by Wildman [15] and specified in the ISO 17751-1:2016 standard [26], providing in-depth information about the sampling and statistics to be used. The simplicity of sample preparation and the ability to see both surface and internal fiber morphology, including medulla and pigment distribution, are benefits of LM as a method for animal fiber identification. The limitations are due to the poor resolution of the instrumentation and the interference with dark dyes and pigments.

Using SEM analysis, fibers are cut in snippets of determined length, made to adhere to specimen stub and coated to a thin layer of gold prior to SEM observation, following the ISO 17751-2:2016 standard [27].

Compared to LM, the advantages of SEM are related to high magnification and resolution, which allow for measuring the thickness of cuticular cells greater than 0.6 μm for wool and less than 0.5 μm for fine animal fibers (Figure 3).

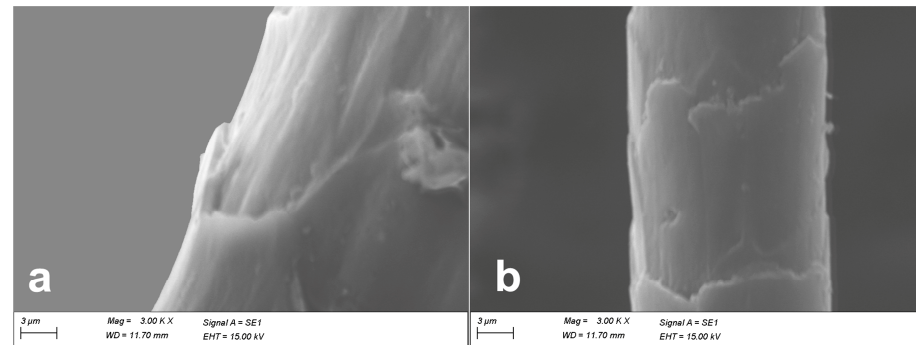


Figure 3. Example of a scale from (a) wool and (b) cashmere (3000 \times).

The main disadvantage consists of the possibility of examining only the surface characteristics of the fibers without investigating the medulla and pigment distribution [28].

Figures 4 and 5 show the images of wool and fine animal fibers obtained by LM and SEM, respectively, and principal morphological characteristics useful for wool and fine animal fibers identification are summarized in Table 2.

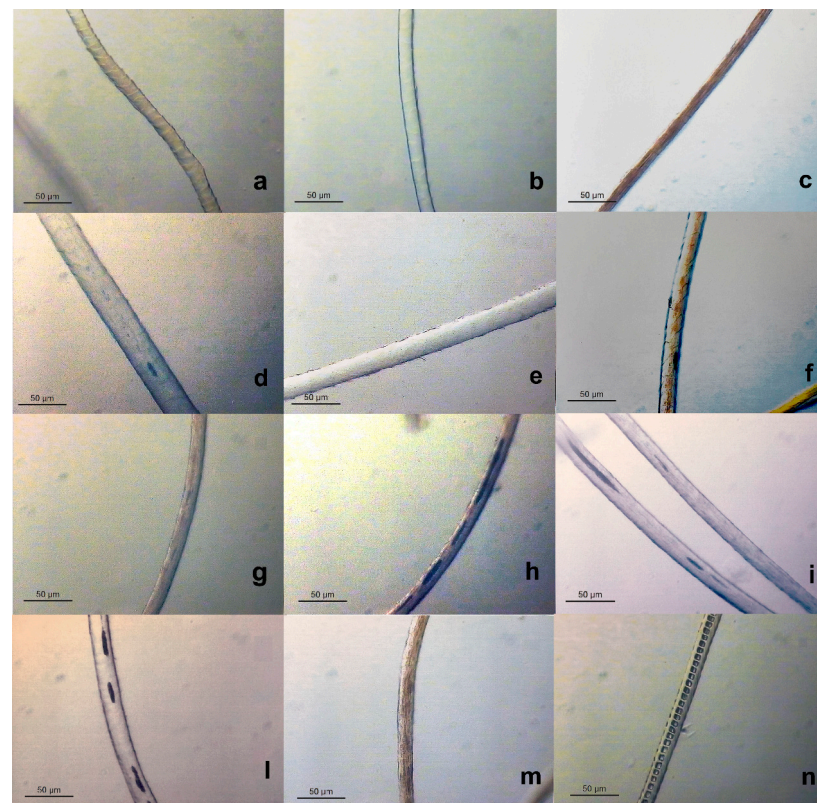


Figure 4. LM pictures (200 \times) of wool and fine animal fibers: (a) wool; (b) cashmere; (c) pigmented cashmere; (d) mohair; (e) cashgora; (f) camel; (g) vicuña; (h) guanaco; (i) lama; (l) alpaca; (m) yak; (n) angora rabbit.

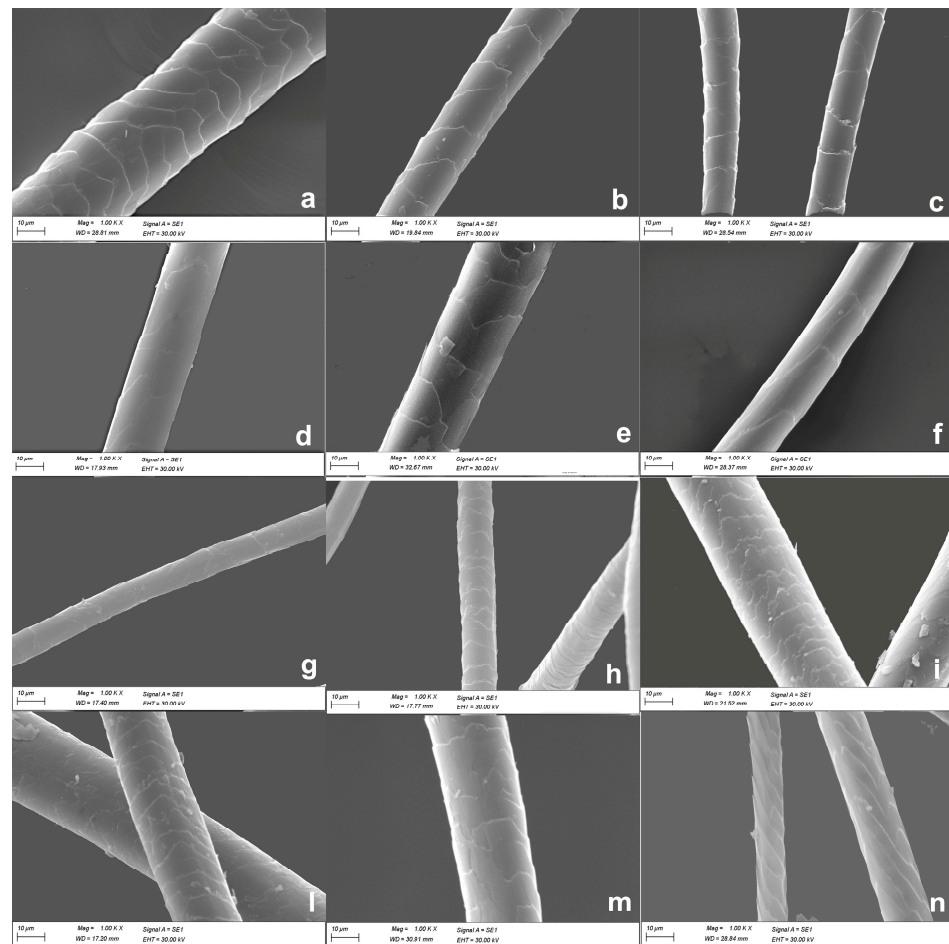


Figure 5. SEM pictures (1000×) of wool and fine animal fibers: (a) wool; (b) cashmere; (c) pigmented cashmere; (d) mohair; (e) cashgora; (f) camel; (g) vicuña; (h) guanaco; (i) lama; (l) alpaca; (m) yak; (n) angora rabbit.

Table 2. Wool and fine animal fibers morphological characteristics.

Fiber	Cuticular Cells Thickness	Cuticular Cells Morphology	Medulla	Pigments	Reference
wool	$\geq 0.6 \mu\text{m}$	cuticular cells quite close along the fiber axis	absent in fine wool	usually absent	[29–31]
cashmere	$\leq 0.5 \mu\text{m}$	distant and smooth cuticular cells margins	usually absent	sparsely distributed when present	[13,28,32]
mohair	$\leq 0.5 \mu\text{m}$	distant cuticular cells margins	absent	absent	[31]
cashgora	$\leq 0.5 \mu\text{m}$	distant cuticular cells margins	absent	absent	[33]
camel	$\leq 0.5 \mu\text{m}$	high cuticular cell margins slope	usually absent	present	[15]
lama	$\leq 0.5 \mu\text{m}$	smooth cuticular cells margins	fragmental medulla	present	[15]
alpaca	$\leq 0.5 \mu\text{m}$	smooth cuticular cells margins	fragmental medulla	present	[15]
vicuña	$\leq 0.5 \mu\text{m}$	smooth cuticular cells margins	fragmental medulla	present	[15]
guanaco	$\leq 0.5 \mu\text{m}$	smooth cuticular cells margins	fragmental medulla	present	[15]
yak	$\leq 0.5 \mu\text{m}$	distant and smooth cuticular cells margins	usually absent	distributed in string	[33]
angora	$\leq 0.5 \mu\text{m}$	chevron cuticular cells patterns	Ladder type of medulla	absent	[15]

The identification methods based on LM or SEM were often criticized because they lack objectivity and require operators with a high level of expertise and skill, mainly for LM [34]. An additional problem arises from superficial treatments hiding the fiber's surface (e.g., antifelting treatments) [35].

However, LM and SEM are still primarily employed in many laboratories, SMEs and bigger companies for animal fibers identification and quality control. Moreover, LM and SEM are the classic identification methods to quantify animal fibers and compare obtained

amounts with quantities obtained with new analytical methods, where the exact amounts of fibers in samples like yarns or fabrics are not available [36,37].

Different morphological approaches for fiber identification were tried to overcome the lack of objectivity of LM and SEM methods. McGregor et al. measured the cuticular and cortical cell dimensions of different fine animal fibers, including cashmere, alpaca, vicuña and mohair, but this study has not led to any sure conclusions being these measurements not enough standardized and affected by fiber diameters and animal age and productivity [38]. Similarly, the investigation carried out by Tian et al. [39] on yak, cashmere and wool fibers led to the detection of differences in cuticular cell scale thickness and frequencies between these fibers, but a standardized application of measured parameters in fiber identification was not obtained.

On the contrary, many studies using image analysis are obtaining excellent results.

2.1.2. Image Processing

In recent years, image processing has been developing rapidly. For more accurate wool and fine animal fiber (mainly cashmere) recognition, several researchers apply related algorithms to examine the texture or morphological characteristics of it. Improvements include automation and batch fiber identification, which significantly increase work efficiency. Accurate fiber identification is also improved, avoiding subjective identification. Many studies have been carried out on image processing for animal fiber identification, and many of them have been in the last few years, as shown in Table 3.

Table 3. The literature overview for animal fibers identification and quantification by imaging analysis. Abbreviations: SVM = Support Vector Machine, CNN = Convolutional Neural Network, GLCM = Gray-Level Co-Occurrence Matrix, HOG = Histogram of Oriented Gradient, SURF = Speeded-Up Robust Features, MLP = Multi-Layer Perceptron, RPS = Region Proposal Strategy, KRR = Kernel Ridge Regression, RQA = Recurrence Quantification Analysis, DGD = Direct Geometrical Description, DWT = Discrete Wavelet Transform, BP = Back Propagation, GA = Genetic Algorithm, LVQ = Learning Vector Quantization, ANN = Artificial Neural Network, 2DDTCWT = Two-Dimensional Dual-Tree Complex Wavelet Transform.

Animal Fibers	Accuracy (%)	Fiber Processing Stage	Imaging Type	Techniques	References	Year
wool, cashmere	94.39	fiber	SEM	Local binary pattern, gray level co-occurrence matrix algorithm	[40]	2023
wool, cashmere	98.95	fiber	SEM	Improved Xception network	[41]	2022
wool, cashmere	up to 91	fiber	SEM and LM	Local binary pattern, Sparse dictionary learning	[42]	2022
wool, cashmere	95.2	fiber	SEM	Feature fusion method, multi-scale decomposition of wavelet analysis, maximum inter-class variance, SVM	[43]	2022
wool, cashmere	96.67	fiber	LM	Texture feature selection method-local binary pattern, the gray level co-occurrence matrix algorithm; SVM	[44]	2022
wool, cashmere, yellow wool, goat hair	99.15	fiber	LM	CCN and deep learning-AlexNet, VGG-16, VGG-19, GoogLeNet	[45]	2022
wool, cashmere	90	fiber	SEM	Gray-gradient co-occurrence matrix model; feature selection algorithm; random forest model	[46]	2021
wool, cashmere	98.7	fiber	LM	Multi-focus image fusion and CNN	[47]	2021
wool, cashmere	97.1	fiber	SEM	GLCM, HOG	[48]	2021
wool, cashmere	up to 90	fiber	LM	LC-KSVD algorithm—A label-consistent clustering singular value decomposition	[49]	2021
wool, cashmere	97.1	fiber	LM	CNN	[50]	2021

Table 3. Cont.

Animal Fibers	Accuracy (%)	Fiber Processing Stage	Imaging Type	Techniques	References	Year
wool, cashmere	93.33	fiber	SEM	GLCM and Gabor wavelet transform	[51]	2021
wool, cashmere	94.2	fiber	LM	Image processing: Hessian matrix, Frangi filter edge detection; Bayesian classification model	[52]	2020
wool, mohair	99.8	fiber	LM	Image processing: feature extraction process; CNN	[53]	2020
wool, cashmere and wool cashmere blends	recognition higher than 93	fiber	SEM	Image processing: original image, image binarization, dilation, filling margin, removing noise and removing background; SURF feature extraction	[54]	2019
wool, cashmere	94.29	fiber	LM	Image processing: GLCM algorithm, interactive measurement algorithm and k-means clustering algorithm	[55]	2019
wool, cashmere	90.07	fiber	LM	Image processing: morphological processing algorithm, contrast stretching algorithm, Otsu algorithm; Analysis: wavelet multi-scale analysis, texture feature extraction, SVM	[56]	2019
wool, cashmere	95.25	fiber	LM	Image processing: co-occurrence matrix algorithm, central axis algorithm; multidimensional and clustering analysis: K-means algorithm	[57]	2019
wool, cashmere	92.5	fiber	LM	image processing: HOG descriptor; SVM	[58]	2019
wool, cashmere	96	fiber	SEM	Image processing: Hough Transform and Feature Extraction; MLP	[59]	2019
wool, cashmere and wool cashmere blends	around 90	fiber	LM	CNN method with RPS	[60]	2019
wool, cashmere and wool cashmere blends	97.47	fiber	LM	Image preprocessing: contrast stretching algorithm, digital analysis methods: fractal algorithm, parallel-line algorithm and K-means clustering algorithm	[61]	2019
wool, cashmere and wool cashmere blends	more than 90	fiber from top	LM	Image preprocessing: highpass filtering, contrast stretching, binarizing, removing small connected components, filling margin, segmenting from the background; bag of word model; SVM	[62]	2018
wool, cashmere and wool cashmere blends	up to 95.2	fiber	LM	CNN and fine-grained method	[63]	2018
wool, cashmere	90	fiber	LM	Image analysis: pairwise rotation Invariant co-occurrence local binary patterns; SVM	[64]	2018
wool cashmere blends	around 90	fiber from top	LM	Image processing: projection curve; neural network with MLP, SVM, and KRR/classification; data training and testing, RQA, DGD, and DWT	[65]	2017
wool, cashmere	81.17	fiber	LM	Image processing: Tamura texture feature method; BP neural network	[66]	2015
wool, cashmere	87.35	fiber	LM	Digital image, SVM	[67]	2014
wool, cashmere	above 83	fiber	SEM	Extraction scale density, SVM and image processing using filtering method and high frequency emphasized filter	[68]	2012
wool, cashmere	over 92	fiber	xxxxxxx	GA- SVM	[69]	2011
wool, cashmere	higher than 93	fiber	LM	Image processing and LVQ model, ANN	[70]	2011
wool, cashmere and stretch wool, cashmere	99 and 81.06	fiber	xxxxxxx	Digital image processing: character parameter extract using sub-measurement to measure the diameter set up the Bayesian model	[71]	2010

Table 3. Cont.

Animal Fibers	Accuracy (%)	Fiber Processing Stage	Imaging Type	Techniques	References	Year
wool, cashmere	xxxxxxx	fiber	SEM	Image analysis: 2DDTCWT texture analysis	[72]	2010
wool, cashmere blends	xxxxxxx	yarn	LM	Image processing: SVM	[73]	2010
wool, cashmere	until 98.75	fiber	LM	Image processing and LVQ model neural network classifier based on scale pattern	[74]	2008
wool, mohair	xxxxxxx	fiber	LM	Image processing: Model I: feature extraction with image processing, Model II: feature extraction with MLP and unsupervised ANN	[75]	2002
wool, mohair	88	fiber	LM	Image processing: filtering, contrast stretching, thresholding, interactive operations, rotating, and morphological operations. ANN	[76]	2001
wool, cashmere	until 97.5	fiber	SEM	Image analysis, scale pattern data: automatic image scanning by means of a boundary tracking algorithm; transforming the image data from the spatial domain to the frequency domain and analyzing the resultant power spectral image	[77]	2000
wool, cashmere	xxxxxxx	fiber	SEM	Semi-automated imaging techniques for characteristic scale pattern data	[78]	1997

As shown in Table 3, the proposed methods related to the extraction of features from images are utilized for training supervised classification algorithms, which either follow deep learning [45,53,63] or, more often, a machine learning approach.

In the literature, both features extracted from scale patterns [52,74–76,78] and height [77] and texture features [61,65] have been employed with success. For the classification, different algorithms such as linear discriminant analysis [78], Multi-Layer Perceptron [75,79] and Support Vector Machine [54,58,62,64] have been employed.

From Table 3, it can be seen that most of the studies focus on the distinction between wool and cashmere, sharing these fibers the majority of the market [80], and only a few papers deal with the distinction between other fibers like wool and mohair [53,75,76]; in this case, optimal discrimination was obtained.

Imaging types are, in most cases, obtained by LM as the easiest and cheapest way to obtain images from fibers. This partially contradicts many works demonstrating that manual identification of wool and cashmere is mainly carried out by measuring the thickness of cuticular cells, which can only be determined by means of SEM [31]. However, in most cases, good accuracies have been obtained, often exceeding 90% and even much higher up to 98–99% [41,45,47,53], with the highest accuracies being obtained in more recent studies.

Undoubtedly, image analysis is one of the most promising techniques for the identification of wool and fine animal fibers, but some problems are still open.

Firstly, even if the fibers to be recognized for commercial purposes are 11 (10 fine animal fibers and wool), research typically focuses on binary classification with the exception of a few works. Indeed, Xing et al. [45] reported a unique deep learning and transfer learning-based fiber identification approach for distinguishing between four types of fiber images: goat hair, yellow wool, sheep wool, and cashmere. Rippel et al. [81] used SEM images of four animal fiber types (wool, cashmere, yak, and silk) from ten different sources by applying out-of-distribution-detection techniques to assess the efficacy of natural fiber identification algorithms under the open set condition. Moreover, in the reviewed literature, with few exceptions [73], images originate from raw fibers or combed slivers; therefore, in general, they are from unprocessed fibers, not from real samples on the market or fibers at different processing stages. As an example, problems for the identifications can arise from

treatments that mask the surface morphology of the fibers, such as widespread treatment to impart felt resistance, which includes chlorination and polymer adhesion [35]. Finally, some problems in fiber identification can occur from marketed recycled wool and cashmere textiles derived from post-industrial and post-consumer waste, currently produced in the frame of a green economy. Although it is not possible to use completely regenerated cashmere yarns due to poor mechanical characteristics, the presence of damaged fibers with classic brush breaking can prevent their recognition [82]. In a similar way, problems in fiber identification can be found in archaeological textiles where the recognizable structural information of hair has not survived [83].

2.2. Chemical Methods

2.2.1. Amino Acids and Internal Lipids Analysis

Wool and fine animal fibers consist mainly of protein and a small amount of internal lipids. The first chemical attempts to identify wool and fine animal fibers focused on their main composition, i.e., protein and their main components, amino acids. Wool and fine animal fibers are made up of eighteen amino acids and characterized by the abundance of the amino acids cysteine, which forms disulfur intra and inter-molecular chain bonds that confer the protein named keratin, a high chemical resistance. Cystine can be oxidized by the cleavage of disulfur bonds until the production of cysteic acid by the effect of solar light on the fleece [84]. It was found that lama, vicuña, alpaca and guanaco have much higher cystine levels than yak, cashmere, cashgora and wool [85]. Moreover, the cysteic acid levels of lama, vicuña, yak and camel were higher than cashmere, cashgora and wool [86]; however, in this case, samples of South American camelids are the results of more than one year of fiber growth, and hence they are subjected to great photodegradation. Despite these differences, amino acid composition depends on animal species and environmental conditions, such as the changes in diet and textile processing conditions in yarns and fabrics, so the amino acid composition can not be considered a strong enough discriminant between different animal fibers.

Internal lipids, one of the components of the cell membrane complex in the fibers, were also investigated to discriminate between wool and different animal fibers. They consist mainly of ceramides, sterols, and free fatty acids for a percentage of about 1.5% of fiber weight [87]. Some authors concluded that it is possible to use sterol analysis of fiber extracts and Gas Chromatography (GC) fatty acids analysis as an addition to conventional procedures to aid in fiber identification [88,89]. However, it was found that lipid analysis as a criterion for fine animal fiber discrimination should be confined to untreated samples because the textile process can affect the fibers' internal lipids fatty acids composition [90]. In any case, no fiber quantification was tried using internal lipid analysis.

2.2.2. Thermal Analysis

Different attempts were made to identify fine animal fibers using modern analytical techniques such as Differential Scanning Calorimetry (DSC), which was studied as an alternative qualitative method to identify different textile animal hair fibers. DSC has well-known applications for studying the thermal properties of materials, including melting, glass transition, crystallization, evaporation, thermal decomposition, denaturation, specific heat capacity and thermal history. The thermograms in Figure 6 show DSC traces of wool and different animal fibers consisting of a first endothermic peak due to water evaporation and a second peak around 230 °C due to denaturation of the α -helix keratin crystallites of cortical cells [91].

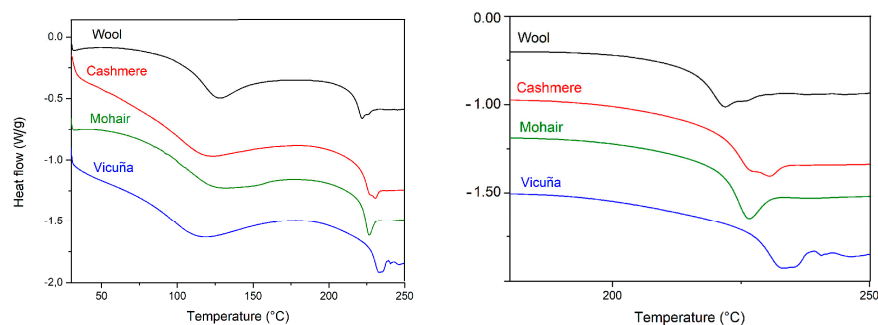


Figure 6. DSC traces of wool, cashmere, mohair and vicuña fibers (left) a detail from DSC traces (right).

Wortmann et al. [92] found that denaturation temperature is positively correlated with cystine content in keratin. The double-peak found in wool and other fine animal fiber thermograms originates from ortho and para cortical cells, whose difference in sulfur amount is sufficient to allow endotherm separation. In the mohair fibers, a single endothermic peak appears because they consist of ortho-cortical cells only (see Figure 6).

Vineis et al. [33] used DSC traces to distinguish between animal fibers from domestic livestock (merino wool, yak, alpaca, mohair, cashmere, camel, angora) and wild and hybrid livestock (yangir, cashgora, vicuña, shatoosh) based on different DSC traces of crystalline proteins in the ortho and para cortex.

They stated that hair tends to develop a higher amount of cysteine-rich paracortex when animals are exposed to thermal and nutritional stresses. However, changes in DSC traces due to the cortical cell transition from α -helix to the β -sheet conformation or rearrangements in the matrix can be caused by industrial treatments such as stretching or steaming [93]. In conclusion, DSC can be used on various animal fibers without previous long classification studies, but it remains a fast method of qualitative analysis to confirm animal fiber origin or study thermal modification in different fiber processing stages.

2.2.3. Spectroscopy

Spectroscopies in the near-infrared field (NIR), in the mid-infrared field (IR) and Raman have been proposed by many authors as a tool to identify fine animal hair and for quantitatively determining wool and cashmere in a blend (See Table 4).

Table 4. The literature overview for animal fibers identification and quantification by spectroscopies. Abbreviations: PET: polyethylene terephthalate, PLA: polylactic acid; PP: polypropylene; PA: polyamide, PU: polyurethane, RMSEP: root mean standard error of prediction; SEP: standard error of prediction.

Fibers	Analytical Method	Identification or Quantification	Accuracy	Fiber Processing Stage	References	Year
wool, mohair	Raman spectroscopy and ratiometric analysis	identification	xxxxxxx	fiber	[94]	2022
shahtoosh, cashmere, angora rabbit	FTIR and chemometry	identification	100%	xxxxxxx	[6]	2022
wool, cashmere, wool/cashmere blend	NIR spectroscopy	identification	93.33% for cashmere and 96.60 for cashmere wool blend	textiles from market	[95]	2019
cotton, Tencel, wool, cashmere, PET, PLA, PP	NIR spectroscopy	identification	100% identification	fiber sliver by carding	[96]	2019
wool, cashmere, rabbit, camel	NIR spectroscopy	identification	100% sensitivity and 100% specificity	fiber	[97]	2019

Table 4. Cont.

Fibers	Analytical Method	Identification or Quantification	Accuracy	Fiber Processing Stage	References	Year
wool, cashmere, qiviut, bison, vicuña	FTIR	identification	xxxxxxx	fiber	[98]	2018
wool cashmere blends	NIR spectroscopy	quantification	SEP of cashmere content 0.5%	fiber	[99]	2017
wool/cotton, wool/mohair, wool/spandex, wool/silk and wool/cashmere blends	NIR spectroscopy	blend identification	from 100% to 85%	fabric	[100]	2016
wool cashmere blend	NIR spectroscopy	quantification	RMSEP: 2.8% percentages of recognition and rejection of 98–100%. SEP: 13.10 for wool/cashmere blend	fiber	[101]	2014
wool, cashmere, yak, angora rabbit and wool–cashmere blends	NIR spectroscopy	identification and quantification	100% discrimination between wool and cashmere	combed sliver	[102]	2013
wool, cashmere, PET, PA, PU, silk, flax, linen, cotton, viscose, cotton-flax blending, PET-cotton blending, and wool–cashmere blending	NIR spectroscopy	identification	SEP: 1.2061	fiber, yarn, fabric	[103]	2010
wool, cashmere and wool/cashmere blend	NIR spectroscopy	identification and quantification		fiber	[104]	2010

Among them, NIR spectroscopy is the most studied and the most promising one. Absorption in the NIR field is associated with overtone and combinations of vibrations of the chemical bonds, mainly R-H, and physical characteristics of a material, such as sample size and surface scattering [105]. In Figure 7, NIR spectra of wool and some fine animal hair are shown in the wavenumber range from 10,000 to 3700 cm^{-1} .

Spectra differ from each other mainly for the tail in the range 10,000–7300 cm^{-1} due to absorptions of eumelanin pigments present in pigmented fibers and correlated with eumelanin amount in the sample [106]. Moreover, spectra absorption intensity at different wavenumbers is another difference, and it is imputable to a different scattering of the NIR radiation with is correlated with physico-morphological characteristics of samples such as the fiber diameter, the presence or absence of medulla, and the shape and distribution of cuticular cells [102].

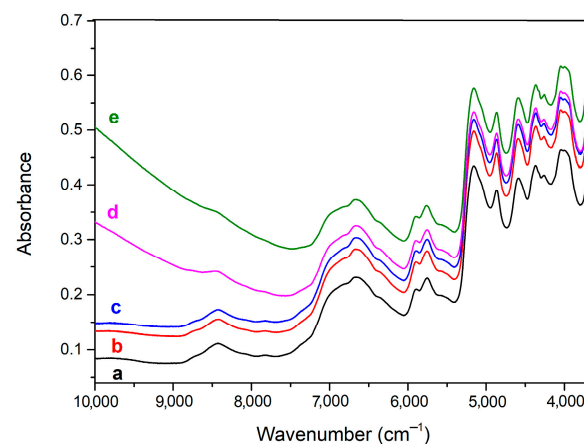


Figure 7. NIR spectra of fibers from (a) angora rabbit, (b) white cashmere, (c) wool, (d) pigmented cashmere, and (e) pigmented yak.

NIR spectroscopy has the advantage of being a fast and non-destructive technique, able to be employed directly on the production line or using portable instruments. The

main disadvantage is the time-consuming calibration of the methods. Acquired spectra are then evaluated using modern chemometric methods [107].

From Table 4, we can see that identification of fibers is not restricted to wool and cashmere but includes angora rabbit, camel, yak, [97,102], and different natural and man-made fibers like cotton, tencel, PET, PLA, PP [96] and PET, PA, PU, silk, flax, cotton, viscose and their blends [103], or a wool blend with cotton, mohair, silk, cashmere and spandex [100]. For quantitative analysis, wool/cashmere blends have usually been tested. In general, fibers are in raw state or as combed slivers, but yarn, fabrics and textiles from markets have also been tested, obtaining good discrimination accuracy [103]. The most popular statistics used for identification purposes was SIMCA (Soft Independent Modelling by Class Analogy) [97,102,103] and for quantitative analysis algorithms such as PCR (Principal Component Regression) [102], Partial Least Squares regression (PLS) [101] and Multiple Linear Regression (MLR) [99,104] were applied. In qualitative studies for the identification of textile materials, where the accuracy achieved is often 100%, even when the distinction occurs between chemically different fibers and similar fibers (wool and cashmere), more specific algorithms were used for wool cashmere discrimination [96].

Quantitative tests to assess the amount of wool and cashmere in a blend gave discordant results with standard error of prediction (SEP) ranging from 13.10 [102] to 1.2061 [104] and 0.5 [99], depending on the sampling and algorithm used for calibration. Good results were also obtained by Sun et al. [95], who tested NIR on real samples in the market and achieved an accuracy of 93.33% for cashmere textiles and 96.60% for cashmere–wool blended textiles using a portable NIR-based textile analyzer.

Even more in detail, NIR spectroscopy was proposed to discriminate among varieties of cashmere material [108] and to distinguish between virgin and recycled cashmere fibers [109]. In conclusion, NIR spectroscopy is a fast and non-destructive analysis that needs long and accurate calibration work, and it is valuable to sectors where a large number of textile samples must be tested, such as quality control in large enterprises and in import/export business.

Alternative methods of fiber identification using spectroscopies, such as Fourier Transform Infrared Spectroscopy (FTIR), sensible to amino acids variation correlated with animal species, and Raman spectroscopy, were investigated. Although some works have not produced satisfactory results [94,98], positive results were obtained when FTIR analysis was coupled with chemometric tools. Indeed, in recent work as a proof-of-concept study, illustrating the potential of ATR FT-IR spectroscopy in animal fibers identification, Sharma et al. [6] obtained a complete differentiation between cashmere, angora and shahtoosh using FT-IR spectroscopy coupled with Partial Least Squares Discriminant Analysis (PLS DA).

2.3. Biotechnological Methods

2.3.1. Electrophoresis

Keratin synthesis is under genetic control and is species-specific for this reason. The first attempt to distinguish between wool and different animal fibers focused on protein separation by one- or two-dimensional polyacrylamide gel electrophoresis analysis. In one-dimensional gel electrophoresis, the proteins extracted from fibers by reducing the disulfide bonds are separated according to their molecular mass, while in two-dimensional gel electrophoresis, proteins are separated according to their isoelectric point and in the other dimension according to their molecular mass [83]. In Figure 8, one-dimensional electrophoresis patterns of wool and different animal fibers are shown. We can see two bands at about 50 kDa corresponding to the low sulfur proteins from intermediate filaments in cortical cells, different bands in the range 28–11 kDa corresponding to high sulfur proteins extracted from cuticular cells, and bands at molecular weight below 10 kDa corresponding to high glycine and tyrosine proteins from the matrix from cortical and cuticular cells and embedding cortical cells [22].

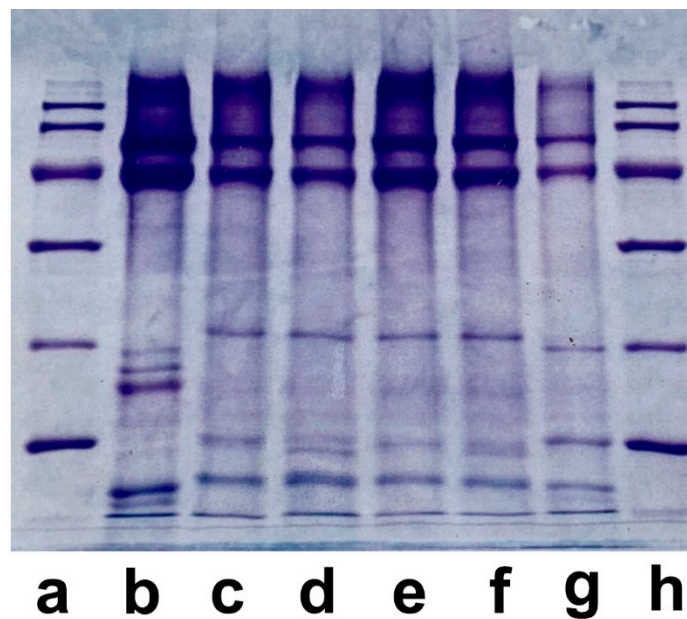


Figure 8. Electrophoretic separation patterns (SDS-PAGE): Lane a: MW standard; lane b: wool; lane c: guanaco; lane d: vicuña; lane e: lama; lane f: alpaca; lane g: camel; lane h: MW standard.

Marshal et al. [110] demonstrated that by using two-dimensional electrophoresis, it is possible to distinguish between wool, mohair, camel and alpaca, mainly according to differences in high sulfur protein separation patterns. Tucker et al. [111] applied two-dimensional electrophoresis using either acidic or alkaline gels to distinguish among cashmere, mohair, cashgora and wool, concluding that this technique is able to differentiate between goat and sheep fibers but not unequivocally between cashmere, mohair and cashgora. The relatively simple method of one-dimensional electrophoresis was applied by Wortmann et al. [32] to distinguish between yak and cashmere and between lama and mohair fibers, as well as between their blends. Despite the positive judgment of the gel electrophoresis to differentiate between fine animal fibers, the main problems arise from the low protein extraction yields of many hair samples following industrial textile processes or extreme weathering, which seriously affect their quantitative determination.

2.3.2. DNA Analysis

In the late 1980s, it was demonstrated that DNA (deoxyribonucleic acid), the molecule that carries hereditary and genetic information, can be extracted from the hair shafts and not only from the hair roots, opening new paths for the identification of animal fibers. For the first time, Kalbé et al. [112] isolated DNA from whole fiber and cuticular cells of animal hair (i.e., alpaca, angora rabbit, cashmere, cashgora, mohair, merino wool and yak). The extracted DNA was then hybridized with selected DNA fragments appositely prepared from rabbit, bovine livers and sheep. The results from dot blot hybridization showed that yak and angora were recognized by bovine and rabbit DNA probes, but goat and sheep may be distinguished only gradually using these probes. However, these first results allowed new possibilities to identify animal fibers employing analytical methods from molecular biology.

Some years later, Hamlyn et al. [113] described the advantages and limits of DNA analysis used to distinguish between wool, cashmere and yak. DNA hybridization analysis using a classical dot blot technique is usually carried out on fibers in their raw state, while in processed materials and finished textile products, the amount of DNA present in the fibers is so reduced that DNA must be amplified *in vitro* with an analytical method known as polymerase chain reaction (PCR) before the quantitative determination. The authors affirmed that even if the DNA analysis with PCR amplification is able to detect

fraudulent substitution of small amounts of fibers, the analysis is not quantitative. A major challenge could be identifying DNA in situ directly on the fiber shafts, but this technology was not developed yet because DNA is encapsulated in a waterproof environment of the keratinized cells of the fibers. Kerkhoff et al. [114] studied a DNA analytical method with PCR amplification to identify cashmere/cashgora, fine wool, yak and camel hair in untreated and treated (washed, bleached, dyed) fibers samples (Table 5). The authors concluded that by using this method, it is possible to differentiate between fine wool–cashmere and cashmere–yak hair, which are the most difficult fibers to distinguish by SEM methods. However, the main problems arise from the differentiation between breeds or varieties of the same species (cashmere, cashgora and mohair) and from obtaining quantitative results.

Table 5. The literature overview for animal fibers identification and quantification by DNA analysis.

Animal Fiber	Identification or Quantification	Accuracy	Fiber Processing Stage	References	Year
wool/cashmere blend	quantification	results of DNA analysis and LM in fabrics were quite close	fiber, yarn, dyed and finished fabrics	[36]	2015
rabbit, wool, cashmere, yak, alpaca, duck down	identification of rabbit	good accuracy	fiber	[115]	2015
wool/cashmere blend	identification	minimum amount of wool detectable in cashmere 9.09%	fiber	[116]	2015
wool, cashmere	identification	minimum amount of wool detectable in cashmere 11.1%	fiber	[117]	2015
wool, cashmere	quantification in blend	xxxxxxx	fiber and fabric	[118]	2014
shahtoosh, cashmere	identification	minimum amount of shahtoosh detectable in cashmere: 1%	fiber and processed product	[119]	2014
wool, cashmere and wool/cashmere blend	identification and quantification in blend	more precise and accurate than traditional microscopic examination	fabric	[120]	2013
wool, cashmere	identification and quantification in blend	minimum amount of wool detectable in cashmere and vice versa: 11.1%	fiber	[121]	2012
wool, cashmere and wool/cashmere blend	identification and quantification in blend	minimum amount of wool detectable in cashmere: 1%	fiber	[122]	2011
cashmere/cashgora, fine wool, yak and camel	identification and quantification in blend	detection limit of about 3% for fine wool/cashmere and yak/cashmere blend	untreated and treated (dyed, bleached) samples	[114]	2009
wool and goat (cashmere, cashgora, mohair)	distinguishing between sheep and goat fiber	xxxxxxx	fiber	[123]	1992

In general, even if many studies are focused on the distinction between wool and cashmere and on identifying the presence of wool in cashmere-labeled products, the studies concern different fibers ranging from shatoosh to alpaca, yak, camel and rabbit. Particular attention was paid to the distinction between yak and cashmere, which are two fibers that are particularly difficult to distinguish under microscopy, while DNA analysis makes identification easier as they belong to genetically distant species [114]. Some problems have been found in distinguishing between genetically similar species, such as mohair, cashgora and cashmere goat [123], while no literature was found about the distinction between fibers of South American camelids. The studies were carried out both on raw fibers [116,117] and on finished products on the market [119], with particular attention to

dyed products [36,118], as dyeing has been demonstrated to be the main process damaging the DNA present in the fibers.

DNA analysis is basically a qualitative analysis, able to identify fibers very similar in the microscopic analysis, while the quantitative analysis presents some problems. The quantitative result generally consists in determining the minimum amount of foreign fiber that can be detected in a sample, and this ranges from about 10% [116,117] to 1% [119,122].

Although DNA analysis to identify animal fibers is currently still used in some laboratories following the ISO 18074 standard [124], there have been no studies in the recent literature on DNA analysis, probably because they have been replaced by proteomics studies.

The main problem of DNA analysis is its low and probably uneven amount in animal fibers. In contrast, keratins and keratin-associated proteins in animal fibers are abundant, persistent and derived from DNA, making them an ideal target for distinguishing animal fibers.

2.3.3. Proteomic Analysis

Proteomic methods are able to distinguish one species from another by MS (Mass Spectrometry) approaches applied in protein or peptide identification. Usually, the “bottom-up” or “shotgun” proteomic approach is employed, consisting in detecting only peptides and identifying the unique peptides to confirm the presence of proteins in the sample. Proteins are extracted from animal fibers using a buffer solution containing a reducing agent, usually dithiothreitol [7,125,126], able to cleave the disulfide bonds between cysteine’s side chains. In some cases, mercaptoethanol [127,128] has been used instead of DTT. Extracted proteins are usually digested by trypsin, a proteolytic enzyme able to cleave proteins at the C-terminal side of arginine and lysine, obtaining short peptide fragments of up to 20–30 residues [37,129]. In one case, double digestion was carried out with trypsin–chymotrypsin (sensitive to Asp/Glu) or trypsin–Glu-C (sensitive to Phe/Tyr/Trp/Leu) [130] in order to improve the identification of the species-specific peptide in similar species.

Digestion can be performed either in solution or after protein separation by gel electrophoresis from the bands (one dimension—SDS Page) [131] or spots (two dimensions) formed on the gels. Peptides are then analyzed in MS mode, where they are identified by their mass, or MS/MS mode, where the amino acid sequence of the peptide can be obtained and then compared with protein sequences in databases. To be detected in MS mode, peptides are ionized or by matrix-assisted laser desorption ionization (MALDI) or electrospray ionization (ESI). The first is often coupled with a time-of-flight mass spectrometer (TOF-MS), where the ions are accelerated through a fixed electric field, and their time of flight to reach the detector determines their mass-to-charge ratio; the second is the interface between a separation system where the sample is injected (high-performance liquid chromatography (HPLC), ultra-performance liquid chromatography (UPLC)) and the MS detector.

As shown in Table 6, common approaches used are UPLC/ESI-MS [126]; UPLC/ESI-MS/MS [37] in order to identify peptidic species-specific markers able to differentiate between wool, cashmere and yak fibers; MALDI-TOF MS [128]; and MALDI TOF MS/MS [132].

Table 6. The literature overview for animal fibers identification and quantification by proteomic analysis. Abbreviations: RMSE: root mean squared error.

Animal Fibers	Protein Extraction	Peptide Production	Analytical Method	Identification or Quantification	Accuracy	Fiber Processing Stage	References	Year
cashmere, shahtoosh	DTT	sds page and trypsin	Maldi TOF-MS	quantification	minimum amount of shahtoosh detectable in cashmere: 5%	raw fiber and fabric	[10]	2022
vicuña, alpaca, guanaco, lama	DTT	trypsin	UHPLC MS/MS and chemometry	Identification of guanaco, vicuña, alpaca	100% discrimination guanaco, vicuña, alpaca	fiber and ancient textiles	[125]	2021

Table 6. Cont.

Animal Fibers	Protein Extraction	Peptide Production	Analytical Method	Identification or Quantification	Accuracy	Fiber Processing Stage	References	Year
wool, goat, cattle, camel, human hair	DTT	trypsin	UHPLC-MS ESI-Q-TOF	species-specific marker list improvement	xxxxxxx	ancient raw fibers and ancient textiles	[7]	2019
wool, cashmere	DTT	trypsin, chymotrypsin, trypsin-GLU-C	NanoLC MS/MS	selection of species unique peptides	xxxxxxx	raw fibers and commercial textiles (for verification)	[130]	2018
wool, cashmere, yak	DTT	trypsin	UPLC/ESI-MS	quantification	average errors from -3% / -6% to 3% / 7% depending on the fiber	fiber, sliver, yarn, fabric	[126]	2017
wool, cashmere	DTT	trypsin	MALDI-TOF MS	marker identification marker	xxxxxxx	fiber	[133]	2016
wool, cashmere, yak	DTT	trypsin	nanoLC MS/MS triple TOF	identification, fiber identification and quantification	cashmere percentages are in good agreement with LM results very good linearity between the composition and the peak area ratio	fiber and fabric	[129]	2016
wool, cashmere, yak	DTT	sds page and trypsin	MALDI TOF/MS MS	quantification in blend	RMSE 0.365 for pure fiber RMSE 0.471 for blend	fiber and textile	[132]	2014
cashmere, wool, mohair, yak, camel, angora, alpaca	DTT	trypsin	MALDI-TOF MS and chemometric	identification	RMSE 0.365 for pure fiber RMSE 0.471 for blend	untreated and treated fibers and 50/50 blend	[134]	2013
cashmere, yak	mercaptoethanol	trypsin	MALDI TOF MS	identification	xxxxxxx	fiber and fabric	[127]	2013
wool, cashmere, yak	DTT	trypsin	UPLC/ESI MSUPLC/ESI MS MS	identification and quantification in blend	limit of detection: 5%	raw, bleached, depigmented, dyed fiber	[37]	2013
wool, cashmere, yak	DTT	sds page and trypsin	MALDI-TOF MS	specific marker identification for keratin I	xxxxxxx	fiber	[131]	2012
wool, yak, human, rabbit, dog, mohair, mink, fox	mercaptoethanol	trypsin	MALDI-TOF MS	identification and quantification	xxxxxxx	raw, dyed, bleached fibers	[128]	2002

In Table 6, the main literature about fine animal fiber identification using proteomic analyses is summarized.

Studies on the identification of animal fibers using proteomic methods concern, in many cases, wool, cashmere and yak, with the latter often being the last one used for the adulteration of cashmere products, and it is difficult to distinguish yak from cashmere using microscopic methods [37,131]. Moreover, some studies cover a wide range of fibers ranging from cashmere, wool, mohair, yak, camel, angora, alpaca, lama, mink, fox and dog [128,134]. The recognition of South American camelids (SACs) fibers has also been investigated on ancient textiles found in archeological sites [125], and the presence of shahtoosh fibers on cashmere fabrics has been investigated for fraud control to detect the illegal trade of shahtoosh [10]. Samples investigated range from raw fibers to yarn and fabrics and historical textiles. In most cases, raw fibers were used for species-specific marker screening and commercial textile fibers for marker verification [130]. Accuracy, when reported, is good, ranging from -3% / -6% to $+3\%$ / $+7\%$ [126], and the limit of detection is around 5% [10]; even if it is less sensitive than that PCR-based DNA analysis method where the limitation of detection is 1% [119], in this case, the advantage lies in the fact that no false positives are detected.

Some studies focus on analysis for commercial purposes [126], while others focus on the identification of specific species markers to implement the existing databases [7,125] and allow the recognition of treated or damaged samples also in the field of palaeoproteomics and in the case where the surface fibers morphology does not allow fibers recognition.

In some cases, analyses are particularly challenging due to the extensive hybridization between the species, e.g., domestics SACs lama and alpaca identification [125]. In conclusion, the proteomic approach is a long and complex process, useful for the discrimination among fibers or materials difficult to distinguish with other methods. It is also important in

revealing information about relationships between close species or sub-species, evaluating morphological characteristics in fibers related to the expression and quantitation of proteins (e.g., fineness of wool), and studying the degradation of proteins following industrial processes in commercial fabrics or aging, in historical textiles.

3. Comparison between the Principal Analytical Methods

The most employed and promising analytical methods are compared in Table 7. These methods are image analysis, NIR spectroscopy and proteomic methods, alongside the well-consolidated microscopical methods using both LM and SEM.

Table 7. Comparison between analytical methods.

Methods	Instrument Depreciation Cost	Chemicals and Consumables Cost	Analysis Times	Pros	Cons
LM and SEM	not high for LM, high for SEM	not high	long	consolidated analysis	lack objectivity; need of operators with a high degree of skill and experience; problems with fibers morphologically very similar or damaged most of the studies are limited to wool–cashmere classification and raw fibers; calibration using damaged fibers or fibers with very similar morphology
Image processing	not high	not high	short after an initial time-consuming calibration	high accuracy of fiber identification	
NIR spectroscopy	not high	not high	short after an initial time-consuming calibration	non-destructive analysis; availability of portable instruments; possibility to take measurements directly on the production line	discordant results in blend quantification
Proteomic analysis	high	high	long	results not influenced by very similar or altered surface fiber morphology	problems with fiber identification in very close or expensively hybridized species

Instrument, chemicals and consumables costs and analysis times are determining factors for the use of one technique rather than another, especially in SMEs and quality control laboratories. In this case, LM, NIR and image analysis have relatively low costs compared to SEM and proteomic techniques, which show high instrument and management costs. SEM, with higher amortization costs than LM, has the advantage of being a less subjective technique as the high resolution allows for measuring the thickness of the cuticular cells, which is greater than 0.6 μm in wool and less than 0.5 μm in fine animal fibers. As far as the analysis times are concerned, these are very long in the microscopical methods, which envisage the recognition of every single fiber (1050 fibers according to the ISO 17751-2:2016 standard) by an expert operator, while for image analysis and NIR analysis, time is definitely short after a time-consuming initial calibration. Proteomic techniques require long times for calibration, while analysis times depend on the automation of the method, but given the numerous analysis steps, they cannot be significantly reduced.

The pros and cons of the different techniques are shown in Table 7. In summary, it can be stated that the image analysis technique shows a high accuracy but has so far been focused only on wool and cashmere fibers in a raw state. It could be assumed that it will have to be refined in the identification of morphologically very similar fibers (cashmere and yak), and the identification of damaged, surface-treated or recycled fibers will be a problematic issue, as has already happened for LM and SEM. In these cases, valid alternative methods can be proteomic techniques, which are time-consuming and expensive, but the morphology of the fiber does not influence them. Finally, the NIR

technique has many advantages: it is a fast, non-destructive analysis with the possibility of being carried out directly on the production line, but its main problem is related to the accuracy of quantitative analysis.

4. Recommendations and Future Research Directions

The most promising recent identification methods of wool and fine animal fibers are image processing, NIR spectroscopy combined with chemometric analysis, and proteomic analysis, which have been developed following the evolution of technologies employed in different fields. These methods were tailored to the niche sector of animal fiber identification.

In the development of these methods, it is important to have an interdisciplinary approach involving experts in the respective analytical technique and experts in the textile field who know how to direct innovation towards the real needs of the sector without becoming lost in merely theoretical studies. This consideration is especially true for studies concerning image processing.

The future research direction will be largely influenced by the evolution of analytical methods taking into consideration the need to identify fibers on real samples, which may have undergone chemical or mechanical treatments or may derive from recycled fibers. Other issues to be considered are the identification of more than one fiber in a blend and the time and cost of routine analysis required in quality control and textile labeling.

New research directions consist of the investigation of new analytical techniques to be applied for the identification of wool and animal fibers that could also result from the coupling of techniques that are already applied in animal fiber identification, such as the example the hyperspectral imaging working in the NIR field [135].

5. Conclusions

The identification and quantitative determination of wool and animal fibers is a major challenge mainly for textile fraud control but also in fashion, forensics and archeological fields. Old methods, i.e., optical and electron microscopies, which are often criticized because they cause subjective results, still dominate in fiber identification and are the cheaper and more affordable ones and provide a range of information barely possible with other methods. However, many different methods are now available for techniques of identification and classification of fibers, which have evolved following advances in new technologies, especially in image processing and NIR spectroscopy coupled with chemometrics and proteomics. The prospects for expanding the use of these techniques depend on the application fields and on overcoming some critical issues. The automated analysis by means of image analysis techniques, as an obvious alternative to the expert-based analysis of fiber morphology, is one of the most promising techniques able to correctly identify animal fiber types, and ever-increasing classification performance is reported in many works. However, some gaps remain to be filled with regard to the enlargement from the wool–cashmere binary classification to different animal fibers and from raw fibers to commercial yarn and fabrics. NIR spectroscopy, as a fast and non-destructive technique, is valuable to areas where large numbers of samples have to be evaluated, while the proteomic approach is a long and complex analysis, useful for the discrimination among fibers or materials difficult to distinguish with other methods (e.g., cashmere–yak, cashmere–shahtoosh) for commercial purposes or fraud control. Additionally, it allows for the recognition of treated or damaged samples in the field of archeological textiles and in the case where the surface fibers morphology does not allow fibers recognition.

Author Contributions: Conceptualization, M.Z. and A.P.; data curation, M.Z., P.B. and A.A.; writing—original draft preparation, M.Z. and A.A.; writing—review and editing, P.B. and M.Z.; supervision, A.P. and P.B. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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