A Modified Method of Cuticle Scale Height Determination for Animal Fibers

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Almost 16,000 tons of raw cashmere are produced globally each year.[1] Approximately 50% is lost to scouring and dehairing, leaving about 8,000 tons (or 1,600,000 pounds) for use in garments. The United States' definition of cashmere according to the American Association of Textile Chemists and Colorists (AATCC) was broadened in 2001, increasing the average diameter for cashmere from 15-16 μm to 15-18.5 μm.[2,3] This means that a wider range of specialty animal fibers, such as yak, could be substituted for cashmere with less risk of detection. Recently, the price for dehaired dark Afghan cashmere was U.S. $41/kg, whereas dehaired medium yak hair sold for U.S. $15/kg. Even greater price differences exist between white Chinese cashmere at U.S. $69/kg and 17 μm wool at U.S. $8.83/kg.[4] Numerous articles and court cases have pointed to the need for increased accuracy and objectivity in the quantitative analysis of intimate blends of cashmere with wool.[5-8] This need is similar to that currently seen in the forensic analysis of hair and other samples.[9]

Various technologies have recently been brought to bear on the problem of accurate, repeatable, and objective component analysis of cashmere blends. The problem is consistent with society's increased demand for standardized measurement techniques in commerce.[10] DNA probes, electrophoresis of extracted amino acids, high pressure liquid chromatography (HPLC) of treated fibers, Fourier transform infrared analysis (FTIR) both with and without neural nets, and image analysis with and without neural nets have all been applied to the problem of increasing the accuracy of cashmere intimate blend analysis.[11-15] However, published research indicates that all applied technologies to date, including transmitted light microscopy and scanning electron microscopy (SEM) are deficient in their ability to consistently and objectively differentiate between cashmere and other animal fibers.[16] AATCC Test Method 20-2005 indicates the risk of misidentification of cashmere/wool blends when it states, "The longitudinal/epidermal appearance of cashmere fiber, while generally fainter than that of sheep's wool, is not as faint as some specialty animal fibers such as camel and alpaca." The method currently does not provide any quantitative scale height measurements.

ABSTRACT

There is currently a need for a better method of cashmere/wool blend analysis. Vertical orientation of animal fibers and sequential scale height measurement using the scanning electron microscope provide previously unavailable objective statistics concerning cuticle scale height (CSH). Data for CSH of reactive-dyed Chinese cashmere, Merino wool, and alpaca fibers obtained by this method are supplied.

Key Terms

Cashmere
Cuticle Scale Height
SEM

TRADITIONAL LONGITUDINAL MEASUREMENT OF CSH

Cuticle scale height (CSH) measurement claims to objectively differentiate between wool and all specialty animal fibers, without exception.[17] The work performed by various researchers on quantitative blend analysis using SEM, particularly the German Wool Research Association, is embodied in International Wool Textile Organization (IWTO) Test Method 58. While IWTO 58 displays large amounts of data on CSH, it gives no specific instructions on exactly where to measure, how to measure, or the number of scales that should be measured to determine a CSH value.[18] The scale heights of fibers described in IWTO 58, and all other current documents concerning scale height, are obtained by measuring the scales while fibers are lying down longitudinally. Early animal textile fiber researchers realized the limitations of this method in that profile artifacts and limited scale views are often presented for measurement and uninterrupted, sequential scale height measurement is prevented.[19] Using this traditional method, scale height measurements are taken by focusing on a scale edge at 10,000¥ magnification, or higher, while the fiber snippet is lying longitudinally on an SEM stub. The SEM operator hunts along the fiber surface, subjectively judging that an appropriate scale has presented itself for measurement. When the first scale is measured, additional scales may be found, but no sample size is specified. The subjective process may be partly responsible for the lack of consistency in this technique.

A fiber prepared for traditional longitudinal CSH measurement is shown at both relatively low (Fig. 1) and high magnifications (Fig. 2). Only a few scales are visible at a time and not every scale along the fiber shaft can be measured.

VERTICALLY-ORIENTED SEQUENTIAL MEASUREMENT OF CSH

Vertical Orientation

Vertically-oriented sequential CSH measurement provides a new and useful technique for determining CSH of wool and specialty animal fibers in a less subjective manner. Six fibers from each

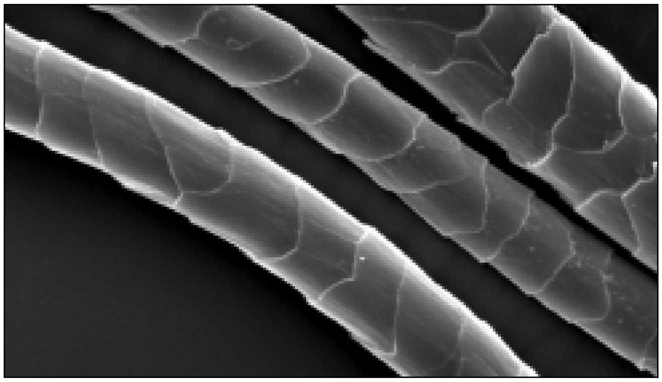

Fig. 1. Fiber prepared for longitudinal CSH measurement (1,370x magnification).

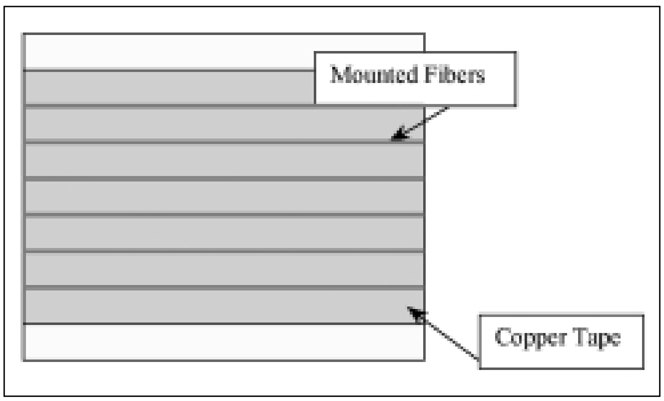

Fig. 3. Copper tape with fibers, bent 180º.

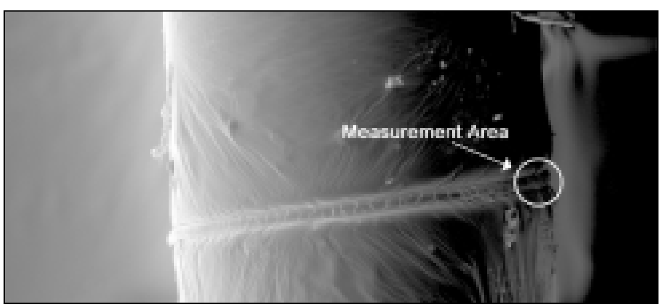

Fig. 5. Measurement of a single scale edge (12,930x magnification). t = 780.423 nm, 611.947 nm, 769.100 nm, 671.523 nm.

sample were selected at random for measurement. The fibers were placed on the sticky side of a 0.25-in. wide conductive copper tape (3M), parallel and equidistant from each other. Distal and proximal ends are determined under magnification.

An aluminum SEM stub with a 0.5-in. slotted head and a 0.125-in. pin was prepared for receipt of the copper tape by placing a double-sided adhesive carbon conductive tab with a 12-mm diameter on the aluminum mounting surface of the aluminum stub.

The tape holding the six fibers was bent 180°, and transferred to the prepared stub. The fibers were bent back on themselves in the middle of the stub. The bent section of the copper tape rises 0.125-0.25 in. above the surface of the stub (Fig. 3). Once mounted, the sample was coated with gold, using a Cressington 108 Sputter Coater.

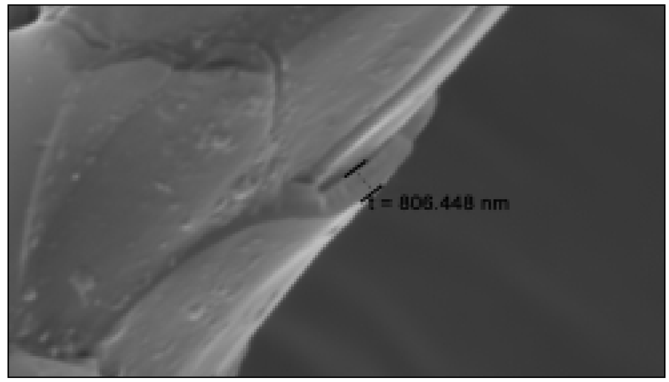

Fig. 2. Fiber prepared for longitudinal CSH measurement (10,000x magnification).

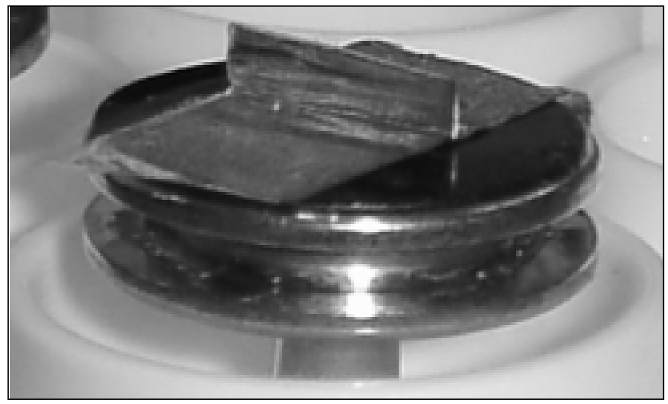

Fig. 4. Animal fiber bent over copper tape showing areas where sequential vertical scale height can be measured (223x magnification).

Measurement Technique

The gold-coated fibers and stub were placed on the motorized SEM (Tescan Vega TS5130MM) stage. The fibers were brought into focus at low magnification (~100x). Only the fiber portion on one side of the bent copper tape (with the distal end toward the operator) can be measured. When this portion of the vertically-oriented fiber was brought into focus at about 1000x, a sequence of measurable scales presented themselves to the operator (Fig. 4). These scales were then measured in sequence by focusing on each one at about 10,000x magnification and using the image analysis feature of the SEM software to measure the CSH (Fig. 5).

Of particular importance in reducing the subjectivity in fiber examination is the ability to standardize the selection of scales for measurement. With sequential vertical analysis, the operator works with a group of contiguous scales. No subjective hunting for additional scale specimens is required.

ANALYSIS

Fiber Sample Selection

Cashmere, wool, and alpaca fibers were selected for analysis. Cashmere and wool fibers were taken off the animals to be sure of their origin. Cashmere was obtained on May 10, 2004 from a two-year-old doe in E Tuo Ke Qi, Erdos City, Inner Mongolia

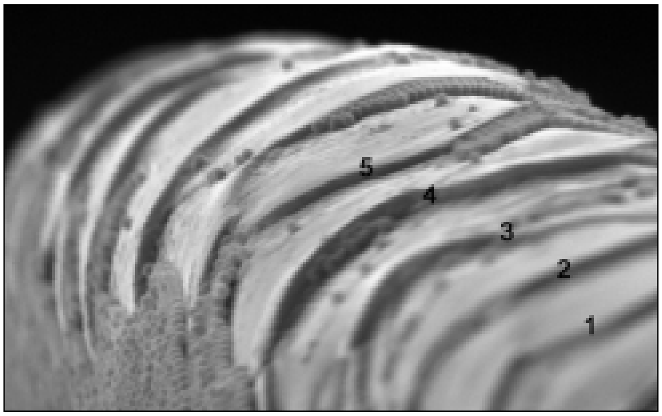

Fig. 6. Cashmere fiber with NIST internal standard and scale sequence (6,490x magnification).

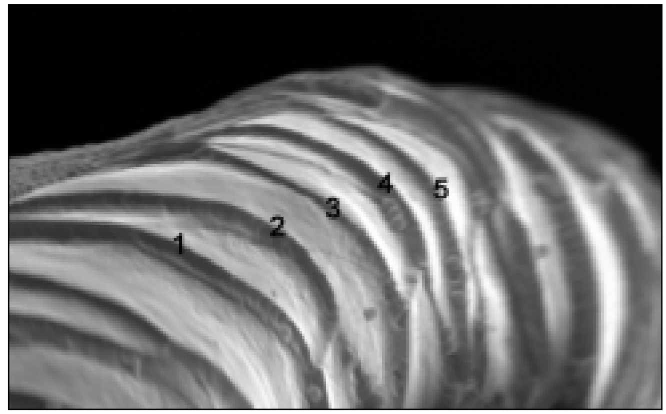

Fig. 7. Wool fiber image with NIST internal standard and scale sequence (6,840x magnification).

Autonomous Region of China. Merino wool was obtained from a 31/32 pure, 17 µm Merino flock from Catskill Merino, New York, on July 10, 2004. Scoured, undyed alpaca fibers were taken from a fiber library (Vartest Labs, Inc.) rather than directly from the animal.

Raw Fiber Scouring and Dyeing

The E Tuo Ke cashmere and Merino wool fibers were dyed using common commercial scouring and reactive dyeing formulas to produce samples typical of dyed and finished fabric.

The samples were scoured separately using laboratory scale equipment. The cashmere and wool were scoured in a series of beakers with water temperature between 40C and 45C. In the first beaker, soda ash (Na_2CO_3) was added to the water, adjusting the pH to 8. The second and third beakers, each contained sodium tripolyphosphate 3/1000 (v/v) solution and Triton X-100 detergent 3/1000 (v/v) solution. The pH was adjusted to 6.5-7.5. Beakers four and five were used for water rinses.

The cashmere and Merino samples were then pretreated by rinsing in water at 60C for 30 min, and adding Cibaflow CIR deaerator (0.5 g/L). Samples were dyed at liquor ratio 1:100, for 30 min at 100C. The dyebath contained Lanasol Blue 3G (2.00% owf), ammonium sulfate $(NH_4)_2SO_4$ (4%), and Albegal B dye leveler (2%). The pH was adjusted to 5.4 with acetic acid.

After scouring and dyeing, all samples were air-dried under ambient lab conditions and mounted on copper tape as previously described. Undyed fiber measurements were not taken in this study, but vertically-oriented sequential scale height measurement could be applied to undyed fibers in the future.

Measurement of Vertically-Oriented Specimens

A sample size of five scales per fiber and two measurements (maximum and minimum height) per scale was used on the first of the mounted fibers. The other five fibers on the stub were reserved for future use. To verify the calibration of the SEM and confirm the veracity of the generated data, an internal size standard, in the form of NIST Traceable Nanosphere Size Standards, Lot Number 21270 (Duke Scientific) with a mean diameter of 503 ±4 nm, a standard deviation of 6.3 nm, and a coefficient of variation of 1.3%, was introduced to the surface of the fibers, which were then sputter-coated as previously described.

TABLE I.

Scale Height of Reactive-Dyed Cashmere, Wool, and Alpaca Fibers

Fiber Type	Source	Scale	Height (nm) Average	Minimum	Maximum
Cashmere	2-Year-Old Doe	1	510	462	558
	E Tuo Ke Qi, Erdos,	2	595	534	656
	China	3	569	494	644
	(15 µm diam.)	4	740	560	921
		5	405	335	475
		Average	563		
Merino Wool	Catskill Merino	1	735	627	842
	(~17 µm diam.)	2	637	635	639
		3	600	489	711
		4	893	766	1019
		5	838	754	922
		Average	740		
Alpaca	Vartest Fiber Library	1	278	250	302
		2	258	224	293
		3	200	171	218
		4	291	271	291
		5	343	310	380
		Average	274		

RESULTS

Vertical orientation and measurement reconfirmed the scale structure outlined by Dobb, et al.—the distal cuticle scale lies loosely on the fiber shaft somewhat like a pocket flap.[19] The presence of detritus under and in front of the scale echoed the

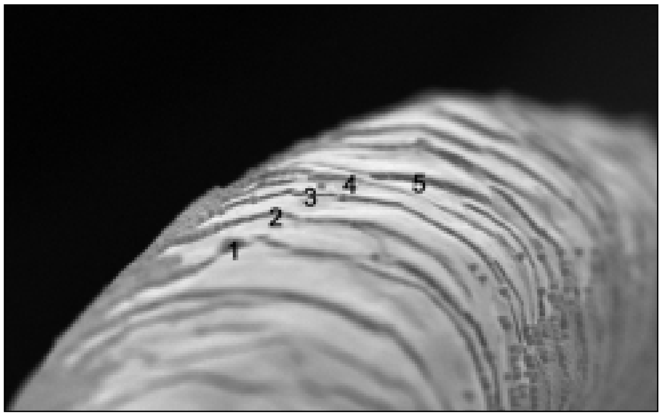

Fig. 8. Alpaca fiber with NIST internal standard and scale sequence (6,140x magnification).

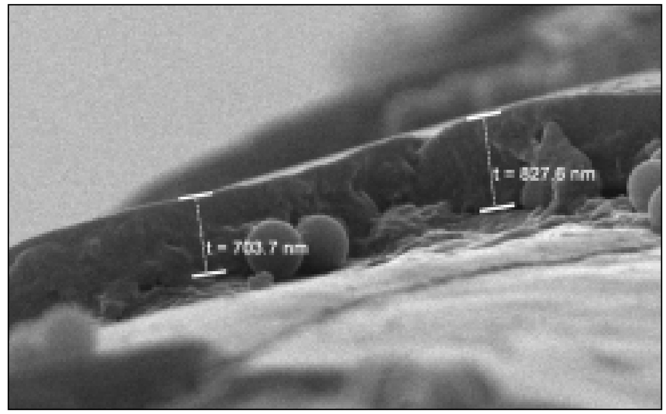

Fig. 9. Reactive-dyed white Chinese cashmere showing scale structure with an average scale height of 746 nm (40,229x magnification).

work of Wortmann.[20] In all cases, portions of cuticle scales were visible for measurement free of the shaft and any obstructing detritus.

Sequential vertically-oriented scale height measurements taken from the dyed cashmere, wool, and alpaca samples are shown in Table I. Figs. 7-9 show the sequential scales to be measured at a relatively low magnification. Of particular interest was the presence of scales displaying heights significantly higher than the statistical parameters given by previous investigators (Fig. 9).[16,17] These scales contradict the findings of previous researchers, such as Arns, that "there was no overlap" in the CSH of wool and specialty animal fibers[21] and Wortmann that cashmere samples "showed scales heights between 0.39 ±0.02 μm and 0.35 ±0.01 μm."[17] Vertically-oriented sequential scale height analysis showed the presence of scales double the height reported by previous researchers, implying that analysis using the previous scale height criteria could well cause cashmere fibers to be misidentified as wool. While previous research took advantage of the SEM's ability to measure scale features that are not quantifiable with transmitted light, vertically-oriented sequential scale measurement provides a more objective, accurate, and precise way of using the SEM for CSH analysis.

CONCLUSION

Vertical, sequential CSH measurement of cashmere, wool, and alpaca fibers reveals that there is greater overlap in CSH properties of cashmere and wool than has been previously published. However, this new technique also provides the ability to obtain average scale height measurements in a more objective manner than was previously possible. The subjective component of testing based on the measurement of scale height is reduced.

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