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 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

Key Terms
Cashmere
Cuticle Scale Height
SEM

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.

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
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.

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.
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 (Na₂CO₃) 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 Lanosol Blue 3G (2.00% owf), ammonium sulfate (NH₄)₂SO₄ (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.

**RESULTS**

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

**References**


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