Physical and morphological characteristics of wool fibres

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Fibres of Chokla and Merino wools have been examined for physical and morphological characteristics. Merino fibres show ortho/para cortex in 2:1 proportion with a clear bilateral cortical structure whereas Chokla fibres appear to be para dominant. The relatively poor thermal stability of Merino wool has been correlated to its cortical morphology.

Keywords: Chokla wool, Merino wool, Morphological characteristics, Thermal stability, Wool fibre

1 Introduction
Optical and electron microscopic techniques have been widely used to elucidate the morphology and internal structure of wool fibre. On the basis of the arrangement and ultrastructure of fibrils in the cells, three types of cortical cells have been identified in wool fibres, viz. ortho, para and meso cells. The fine structures of these three cortical cells are as follows:

Ortho cells: Microfibrils are grouped in fingerprint (whorl) pattern of discrete and regular macrofibrils with a high microfibril : matrix ratio.

Para cells: Microfibril arrangement is random or, occasionally, hexagonal. Macrofibrils are poorly defined and extensively fused and tend to form nuclear remanants in the middle of the cell. The microfibril : matrix ratio is relatively low.

Meso cells: The macrofibrils are similar to those in para cells but have more ordered microfibrils. The microfibril : matrix ratio is intermediate.

Wools of different origins have a wide structure and property spectrum. Each wool has its own specific cortical cell characteristics. The association between the properties of different cortical cells and fibre properties have been reported for different wools. Indian wools are broadly classified as carpet wools; however, only very limited studies have been made on property-structure relationships in these wools. Compared to the widely studied wool fibres like Merino, Indian wools are known, in general, to have higher stiffness, lower wet strength, poorer settability, lower directional frictional effect and poorer dye uptake characteristics. The present study was aimed at gaining an understanding of property-structure correlations in wool fibres obtained from two different breeds of sheep, including an Indian breed. Some physical and morphological characteristics of the selected samples are presented as the starting point. Out of the wide range of Indian wools, Chokla is perhaps one of the most important varieties and was, therefore, selected for the present study. Merino fibre was selected as the second wool since it has been well studied and there is considerable published information on it.

2 Materials and Methods
2.1 Origin and Preparation of Wool Samples
2.1.1 Sample Selection
The samples were selected at random from the greasy fleeces of the Chokla and Merino wools. Chokla sample was obtained from the Central Sheep and Wool Research Institute, Avikanagar, and the Australian Merino sample was obtained from the Raymond Woollen Mills Ltd, Bombay, from a commercial lot imported through the Australian Wool Marketing Board.

2.1.2 Cleaning
Grease and other extraneous matter were removed by extraction in petroleum ether followed by
thorough rinsing in distilled water. The samples were then left to dry under normal room conditions for several days.

2.1.3 Preparation of Set Fibres
Clean samples in the form of parallel bundles (described as control) were used as the starting material. The samples were stretched by 20% and 40% in distilled water at 25, 50, 75, 100, 125 and 135°C, held in the taut condition for about 60 min at the respective temperatures and then allowed to remain in the same environment for further 60 min in the free-to-relax condition. Some fibres were set without any stretch by subjecting them to a similar thermal treatment in the unstretched state as for the stretch-set case.

2.2 Moisture Regain
Moisture regain was measured on samples equilibrated at different relative humidities over respective saturated salt solutions. For both wools, three samples (each weighing about 500 mg) were tested at all the relative humidities beginning with 0% RH; at each humidity, the result being an average of the three observations.

2.3 Dyeing of Fibres for Optical Microscopy
To observe bilateral structure using optical microscope, the samples were oxidized and stained in basic dye; para cortex of oxidized section dyed more than the ortho cortex. Bulk samples were oxidized in performic acid, i.e. in freshly prepared solution of formic acid (98%, 25 ml), distilled water (65 ml) and hydrogen peroxide (30% wt/vol., 10 ml) for 1 h followed by washing in water. Cross-sections of a 100-200 fibre bundle were obtained using Hardy microtome with collodion as embedding medium. The sections were then dyed by floating on a drop of 0.1% methylene blue solution at pH 2.5 for 1 min at room temperature. The sections were then washed, dried and examined on a Carl-Zeiss microscope. Quantitative estimation of the proportion of dyed and undyed regions in these fibres was made from area measurement of photomicrographic enlargements of the stained cross-sections. For each wool, nearly 50 or more fibres were measured.

2.4 Scanning Electron Microscopy (SEM)
Gold-palladium coated specimens were examined on a Cambridge Stereoscan 150 SEM at an operating potential of 10 kV for the scale structure and the cross-sectional profiles.

2.5 Transmission Electron Microscopy (TEM)
TEM studies were intended to be made only on Chokla fibres. However, limited studies on some Merino fibres were also made for comparison. Specimens were examined in a Philips Electron Microscope (model TEM-300) operating at an accelerating potential of 80 kV.

2.5.1 Cross-sections for Studying Fine Structure
Cross-sections of wool fibres were prepared following Roger's method. Clean samples reduced in 0.5 M thioglycollic acid at pH 5.5 for 24 h at 20°C were fixed in 1% osmium tetroxide at pH 7.5 for 5 days at 4°C in dark condition. The dried fibres were embedded in epoxy resin (Araldite) in the usual way. The matrix hardened by crosslinking at 48°C for two days and thin cross-sections, silver-grey in colour, were cut on a Reichert ultramicrotome using diamond knife. Sections were collected on copper coated grids (300 mesh) and post-stained sequentially in 1% uranyl acetate and 1% lead citrate for 15 min each at room temperature. The sections were then thoroughly rinsed in distilled water, dried and carbon coated in vacuum.

2.5.2 Cross-sections for Studying Gross Histological Morphology
For studying gross histological details, a simpler method reported by Swift was followed in which fibres stained in 0.05 M silver nitrate solution were embedded and cut in the usual way using glass knife. Cross-sections collected on coated copper grids were directly examined.

3 Results and Discussion
3.1 Physical Characteristics
3.1.1 Fineness Distribution

The diameters of the fibres in the three wools were determined with the help of a projection microscope. The data are presented in Fig. 1 in the form
of fineness distribution curves for the two wool types. It may be stated that whereas the Merino fibres are free of medullation, the Chokla fibres contain 20% medullated fibres. The average diameter of the Merino and Chokla wools was found to be 24 $\mu$m and 35 $\mu$m respectively. The average fineness quality, calculated on the basis of fibre diameter and coefficient of variation$^{20}$, was 64s and 44s for Merino and Chokla wools respectively.

### 3.1.2 Cross-sectional Profiles

Scanning electron micrographs showing cross-sectional profiles of the two wool fibres are presented in Fig. 2. In general, the circularity of the fibre depends on the thickness of the individual fibre; the finer the fibre, the more circular its cross-section. Average circularity of the samples can be obtained from the shape factor ($\varepsilon$) defined as

$$\varepsilon = 4\pi A/P^2$$

where $A$ and $P$ are the area and perimeter of the fibre cross-section and can be calculated from the cross-sectional photographs. The average shape factor for the Merino and Chokla wool samples are 0.92 and 0.86 respectively. Although the finer fibres of Chokla have near circular cross-section, the elliptical shape of the coarser fibres, which are greater in number, gives rise to its low shape factor.

#### 3.1.3 Moisture Regain

Table 1 shows the moisture sorption of water for samples equilibrated at different relative humidities over respective saturated salt solutions. The differences in the moisture regain between the two wool samples are less than the scatter within a sample, which is around 1% at saturation regain and, therefore, not significant.

### 3.2 Morphological Characteristics

#### 3.2.1 Scale Structure

The scale structures for the two wool types, as revealed by the scanning electron microscope, are shown in Fig. 3 and the characteristics of the scales are given in Table 2. The scale structures are coronal or annular in Merino and flattened and reticulate type in Chokla. The latter is a common feature of most of the Asiatic wools$^{21}$. The scale index (ratio of scale height to fibre diameter) is about 0.5 in Chokla fibres whereas it is nearly 0.85 in Merino fibres. Since friction is related to this index$^{20}$, low index in Chokla fibres accounts for the low directional frictional coefficient reported for this wool$^{11}$.

### Table 1—Moisture regain during absorption cycle

<table>
<thead>
<tr>
<th>Relative humidity %</th>
<th>Merino wool</th>
<th>Chokla wool</th>
</tr>
</thead>
<tbody>
<tr>
<td>9</td>
<td>5.6</td>
<td>5.7</td>
</tr>
<tr>
<td>20</td>
<td>7.2</td>
<td>7.1</td>
</tr>
<tr>
<td>33</td>
<td>10.3</td>
<td>10.6</td>
</tr>
<tr>
<td>43</td>
<td>12.6</td>
<td>12.7</td>
</tr>
<tr>
<td>65</td>
<td>15.9</td>
<td>16.1</td>
</tr>
<tr>
<td>70</td>
<td>19.9</td>
<td>20.3</td>
</tr>
<tr>
<td>98</td>
<td>28.7</td>
<td>29.2</td>
</tr>
<tr>
<td>100</td>
<td>32.6</td>
<td>33.5</td>
</tr>
</tbody>
</table>

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![Fig. 2—Scanning electron micrographs of the cross-section of fibre bundles of (a) Merino wool and (b) Chokla wool](image-url)
Table 2—Morphological details of scales of wool fibres

<table>
<thead>
<tr>
<th>Wool type</th>
<th>Scale outline</th>
<th>No. of scales per cm</th>
<th>Scale height $\mu$m</th>
<th>Scale index</th>
<th>Scale length mm</th>
<th>Fibre diam. $\mu$m</th>
</tr>
</thead>
<tbody>
<tr>
<td>Merino</td>
<td>Annular</td>
<td>860</td>
<td>17.0</td>
<td>0.85</td>
<td>8.0</td>
<td>25</td>
</tr>
<tr>
<td>Chokla</td>
<td>Reticulate</td>
<td>1340</td>
<td>17.5</td>
<td>0.50</td>
<td>15.0</td>
<td>35</td>
</tr>
</tbody>
</table>

3.2.2 Thermal Stability

Scale structures of set fibres reveal the set level and longitudinal stability of the fibre. The scale pattern is unaffected for set treatments in water up to a temperature of 100°C. Melted fibres (i.e. heated in water above 100°C) show severe shrinkage (Fig. 4). In a relative comparison, Merino fibres show severe shrinkage when heated in water at 125°C whereas Chokla fibres are stable at this temperature and show similar melting effect for temperature above 125°C. In general, the degree of shrinkage in stretch-set and heated samples is more in Merino than in Chokla wool, indicating relatively low thermal stability of this wool. These observations are in good agreement with the mechanical properties, optical orientation, and other properties shown by these wools i.e. in set fibres, the stability progressively decreases with increase in temperature and set strain; set at higher temperature leads to melting and low molecular orientation in the ordered regions and hence a relatively poor stability.

3.2.3 Bilateral Structure

When the bulk dyed fibres were viewed longitudinally, differential dyeing was observed in Merino fibres only and when the cross-sections of the dyed fibres were examined under optical microscope, the dye uptake profiles of the two wools were found to be distinctly different (Figs 5 and 6). Fig. 5 shows...
bilateral asymmetry of dyeing with a good contrast between ortho and para regions for Merino fibres. The para cortex is heavily stained, the ortho cortex less intensely stained and the boundary between the stained and the unstained cortical regions sharp; this contrast in dye uptake is found to remain even when the ortho regions are stained by allowing the dyeing to proceed for longer time. The average unstained area (ortho fraction) is found to be about 65% of the total fibre cross-sectional area (Table 3). The dyeing behaviour for Merino wool reported here is in good agreement with that reported by Horio and Kondo and others. The effectiveness of the staining treatment was further confirmed by examining the cross-sections of para rich Lincoln fibres, which showed uniform staining of the entire cross-section.

Fig. 6 shows two-phase staining of a different kind for Chokla fibres; the dyeing is non-uniform, irregular and typical of para rich fibres. These fibres show preferential dye uptake on the outer annular region, i.e., they enclose a small unstained region in the centre. This ring effect becomes more pronounced in coarser fibres. In hairy fibres, the cortex in the immediate vicinity of the medulla does not stain and appear to contain ortho cells. The boundary between stained and unstained regions is not as sharp as in the case of Merino fibres. On the other hand, the chemical composition and reactivity of these wools are known to be different. Merino is chemically more reactive than Chokla and hence the difference in the dye uptake. Hence, the bilateral staining in these fibres is not as well defined as in Merino fibres and the unstained region (ortho fraction) is about 15% of the total fibre cross-sectional area (Table 3). The examination of a large number of cross-sections of Chokla fibres revealed variation in stain uptake of para cortical cells (Fig. 6b).

### Table 3—Ortho content in wool fibres

<table>
<thead>
<tr>
<th>Wool type</th>
<th>Ortho fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Merino</td>
<td>Low  50</td>
</tr>
<tr>
<td>Chokla</td>
<td>Low  5</td>
</tr>
</tbody>
</table>

The examination of a large number of cross-sections of Chokla fibres revealed variation in stain uptake of para cortical cells (Fig. 6b).
areas consist of bilaterally distributed heavily and lightly dyed regions, often joined, adjacent to each other. Since staining of meso cortical cells resembles that of ortho cortical cells, though their fibril morphology resembles that of para cells, the lightly stained regions seen in Chokla fibres may represent meso cells. Although it is difficult to judge the exact type of cortical cell type from optical micrographs alone, the depth and clarity of the stained regions in the micrographs suggest that the heavily and lightly dyed regions in the cortex of Chokla fibres correspond to the predominance of para and meso cells respectively. An approximate estimation of the proportion of the lightly stained region (meso) shows that in most of the fibres it occupies about 50-70% of the total stained area (para + meso).

### 3.2.4 Gross Morphology

The cross-section of Merino fibre shows a distinct ortho-para structure, as illustrated by a typical electron micrograph (Fig. 7). In Chokla fibres, such a distinctive division is not observed. Fig. 8 depicts

![Fig. 8](image1)

Fig. 8—Transmission electron micrographs of transverse sections of Chokla fibres: (a) fine fibre, and (b) coarse fibre. Sections were stained with 0.05 M silver nitrate solution.

![Fig. 9](image2)

Fig. 9—Transmission electron micrographs of (a) Merino fibre and (b) Chokla fibre showing the difference in the cortical segmentation. Sections were stained with 0.05 M silver nitrate solution.
the full cross-section of a fine Chokla fibre and a part of the cross-section of a coarse Chokla fibre. The cross-section of fine Chokla fibre shows stained and unstained regions whereas the cross-section of coarse Chokla fibre appears to be relatively more uniformly stained. The optical micrographs of oxidized and dyed fibres, described earlier, too show a similar result. This possibly relates to the presence of meso cortical cells in Chokla fibres.

3.2.4.1 Nature of Wall Boundary
The transmission electron micrographs of Merino and Chokla fibres (Fig. 9) show that the cortical cell walls of Chokla fibres differ markedly from those of the Merino fibres and show extensive interdigitation i.e. one cell penetrates into the neighbouring cell, giving rise to small protrusions at many places along the cell wall boundary. Such a pattern is absent in Merino fibres.

3.2.4.2 Size and Shape of Cortical Cells
Cortical cells in the two wool fibres show large variation in size ranging from 0.5 μm to 8 μm. Fig. 9 illustrates the difference between these two wool types. The cortical cells are defined by the cell wall boundaries. The maximum size in Merino fibres rarely exceeds 5 μm whereas coarse fibres of Chokla show cortical cell width up to about 8 μm. Chokla fibres, having elliptical or bean-shaped cross-section, show a larger number of such degenerated elliptical shaped (ribbon-like) cortical cells. The average ellipticity factor, defined as the ratio of cell widths in two perpendicular directions, is less than 3 in Merino fibres whereas it is nearly 5 in Chokla fibres. In general, the cells in the vicinity of the cuticle are smaller in size and are usually elongated, and such a pattern is seen in both the wools.

3.2.4.3 Fine Structure
The ultrafine structure of the cortical cells of Chokla fibres is shown in Fig. 10. The microfibrils appear as whorls having an annular pattern. The annular lines appear to be continuous but in some micrographs they are like a beaded necklace, each bead representing microfibrils which are arranged in near hexagonal or irregular form. In most of the macrofibrils, the microfibrils are arranged in near hexagonal pattern in the centre whereas they are continuous, but form an irregular contour, on the outer regions. These characteristics are typical of meso and para cortical cells described previously and it is these cells which make up the greater part of the cortex in this wool. These fibrillar patterns deviate from the more generally accepted description of hexagonal or pseudo hexagonal packing in para regions of Merino fibres.1,2.

In view of the limited studies made, these inferences can only be taken as indicative. In the absence of detailed chemical analysis and electron microscopic studies in each of the cell types, it is difficult to compare filament packing arrangement and quantify para/meso content in Chokla fibres.

4 Conclusions
4.1 Bilateral cortical asymmetry is seen in Merino fibres, which contain ortho and para regions in 2:1
proportion. Some fine Chokla fibres also show a cortical structure with two types of cells, but with much poorer contrast as compared to that in Merino. Coarse Chokla fibres show radial staining and contain a significant para-like component. The average proportion of ortho cortex in this wool is about 15% of the total cross-sectional area.

4.2 Oxidized and stained Chokla fibres show some evidence for different cell types in the dyed region; the heavily stained region corresponds to para cortex and the lightly stained region corresponds to meso cortex.

4.3 The low thermal stability of Merino fibres is apparently related to their bilateral structure, the ortho cortex being less stable than the para cortex.

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