Chapter - 3
3.1 EXPERIMENTAL

RAW MATERIALS

One sample of pure Angora hairs (pure Angora heavy type) and three varieties of cross-breed Angora goat fibres (pashmina type, ringlet and flat-lock) were procured from Mahatma Phule Agricultural University, Rahuri, Maharashtra. A detailed description of these samples is as follows:

- Source of Mohair: Cross-breed Angora
- Genetic group: 3/4 Angora and 7/8 Angora
- Sex: Female
- Age: Adult goats (3 to 5 yrs)
- Month of shearing: Winter Session/Jan.
- Climatic conditions: 10-40°C temp and 20-95% RH

Besides these adult mohair fibres, a kid mohair fibre (Autum session/oct.) from M.P.A.U., Rahuri, Maharashtra was also collected and subjected for its basic fibre characteristics.

A Russian Angora rabbit hair sample was obtained from Department of Fur Animal Breeding, Garsa (H.P.). Further, few more samples of pure and cross-breeds of Angora rabbits, two of German breed from Almora Hills
(A), Nanital Hills (B) and one German X Russian cross-breed sample from Nanital Hills (summer session/March) were collected and evaluated for their characteristic properties.

Also, different varieties of wool (Autum session/Oct.) such as ramb. x chokla, chokla and Indian merino, provided by Central Sheep and wool Research Institute, Avikanagar (Rajasthan), along with one merino sample from Amritsar (Punjab) were collected to undertake the studies of their effective use in blends with varieties of mohair and rabbit hairs, based on their inherent fibre characteristics and with respect to different end-applications.

3.2
3.2.1

**FIBRE CHARACTERISTICS**

**Physico - Chemical**

Considering the importance of these properties, it was planned to undertake the evaluation of physical and chemical characteristics of speciality hairs such as mohair and Angora rabbit hairs. So far a very limited systematic data has been produced on physico-chemical properties of these speciality hairs, produced under Indian agro-climatic conditions. The studies would help in grading these fibres according to their qualities and subsequently in producing yarn
and fabric of better performance. In view of the significance of fibre fineness, fibre length and allied physical properties and their ultimate effect on yarn processing and fabric performance, a thorough analysis of these properties was undertaken with both wool and speciality hairs. Besides, grease content, suint content, vegetable matter, fibre yield and alkali solubility are some of the commercially significant properties which have also been investigated. These studies will also help to decide about scouring, carbonising, blending, carding, combing and other processing techniques of these hairs in subsequent experiments. The analytical problem of determining the amino-acids in a protein depends on the extent to which the amino-acid mixture is separated into individual amino-acids before the actual quantitative determinations are made. The qualitative and quantitative analysis of different amino-acids in hydrolysates of mohair, rabbit hair and wool have also been undertaken during these studies by using the thin layer chromatography, high voltage electrophoresis and auto-aminio-acid analyser.

**Fibre Diameter**

Representative samples from the lot were withdrawn from various sections. Diameter determination was performed for both raw and scoured fibres. For scour-
ing of these samples, four tub-scouring system with a liquor containing 3 gpl sodium carbonate and 1 gpl of lissapol-D paste at temperature 52°C for 3 minutes with 1:60 material to liquor ratio, was applied. The sample was centrifuged for 5 minutes followed by oven-drying at 105 ± 2°C. These fibres were first straightened and cut into small pieces with the help of microtome and then mounted in cedarwood oil. Fibre diameter was recorded under a projection microscope HEICHERT, AUSTRIA at 500 magnification as per IS-744-1977. About 300 observations were recorded for each sample of fibres to calculate average fibre diameter, standard deviation, coefficient of variation and confidence limit.

For estimating fibre diameter variation within a single fibre, 20 fibres of each variety were selected and mounted in cedarwood oil. Ten diameter readings were recorded along with the entire length of each fibre. Average diameter, standard deviation and coefficient of variation were calculated which represented average variation within a single fibre (CVw%).

**Medullation Percentage**

Fibre samples were selected from the lot randomly. These fibres were first combed to make a bunch of
parallel fibres and then cut into small pieces of 0.1mm length by the use of microtome. Cedarwood oil was used as a mounting fluid. Fibres were examined under a projection microscope HEICHERT, AUSTRIA at 500 magnification in accordance with IS-2899-1965. Since medullation in mohair is of not much significance, only total number of medullated fibres were counted to calculate percentage and no distinction was made between partially medullated and coarsely medullated fibres.

**Kemp Content**

To evaluate the kemp content in a lot, fibre samples were selected randomly so as to be representative of the lot. Thus, the test specimen weighing about one gram was withdrawn. Fibres were first degreased by extraction with petroleum ether. Solvent was evaporated and then fibres were treated with sodium oleate solution at 40°C for 3 minutes and washed with water. Fibres were gently pressed between two pads of filter paper to remove water and then dried at 60°C. Test specimen was conditioned to moisture equilibrium at 65 ± 2% RH and 27 ± 2°C temperature and measure correctly to constant weight. Kemp fibres were separated out following IS-1348-1971. Kemp free fibres were again conditioned to moisture equilibrium before
weighing. Kemp content was calculated as percent by weight.

3.2.1.4 **Staple Length**

Staples were withdrawn from various sections of the samples. Each staple was spread on a black velvet board and its length was recorded manually in accordance with IS-6653-1972. About 100 observations were recorded for each sample. Average staple length, standard deviation and coefficient of variation were calculated.

3.2.1.5 **Fibre Length**

Sample of animal fibres was taken out from different parts of the lot. Fibres were straightened and fixed in the fibre length tester. Each fibre was stretched with the help of forceps and the length was measured for both raw and commercially scoured fibre in accordance with IS-1377-1971. Overall 500 to 600 observations for each sample were taken and the average length, standard deviation and coefficient of variation were calculated.

3.2.1.6 **Crimp Percentage**

The fibres were withdrawn from different parts of the lot of samples. Their stretched and unstretched lengths were recorded manually by spreading on a velvet board. The percentage difference between
stretched and unstretched lengths gives crimp percentage. IS-6124-1971 was followed to evaluate crimp frequency for both raw and commercially scoured fibres. For each sample 300 observations were recorded to calculate average crimp percentage, standard deviation and coefficient of variation.

**3.2.1.7 Moisture Regain**

Pre-conditioning of the specimen was performed by keeping it in a desiccator having saturated solution of sodium nitrite i.e. relative humidity of 65 ± 2% for about 48 hrs and a temperature of 20 ± 2°C. Moisture regain of each raw and commercially scoured sample was estimated using standard procedure, IS-6637-1972, and averages of five observations are reported.

**3.2.1.8 Wax Content**

About 10 gm. of oven dried raw animal hairs of a sample was taken and washed in 400 ml. of distilled water to remove suint from the surface. Subsequently, the specimen was dried and extracted in a soxhlet apparatus with absolute alcohol for about 20 siphon cycles. Heating of the bath was so adjusted that the total time taken for 20 siphon cycles was about 3 hrs. The extract was filtered, the alcohol was evaporated and the residue was dried at 105 ± 2°C to
constant weight. About five experiments were performed with each sample of keratinous hairs to calculate percentage wax content on dry weight basis.

**Suint and its pH**

Two specimens were taken randomly from the unconditioned sample and oven dried each weighing 10 ± 0.1 gm. The fibres were cut into lengths of about 5 m.m. Each specimen was then transferred into a separate flask. 400 ml of distilled water having a pH of 6.5 was poured into the flask at room temperature (21°C). Subsequently, stopper the flask, shake vigorously for about 30 sec. to wet the specimen thoroughly. It was agitated mechanically for about 2 hrs at a rate that the temperature of the water in the flask should not exceed above 28°C. The sample was filtered, dried and weighed to determine suint on the dry weight basis of the fibres. The pH of the extract was measured electrometrically with a glass electrode and then the average value was calculated for each sample.

**Fibre Content and Laboratory Scoured Yield**

The clean fibre content and laboratory scoured yield along with alcohol extractable matter, ash content and vegetable matter of keratin fibres were determined according to IS-1349-1964.
Weighed amount \((W_1\text{ gm})\) of test specimen was taken and opened it by hand to remove impurities such as dirt, dung, strings etc. \(\text{(except vegetable matter)}\). The samples were scoured using \(3\text{ gm. of anhyd. sodium carbonate and 1 gm. of lissapol-D (neutral soap) per litre of water.}\) Scouring solution was heated to \(52 \pm 5^\circ\text{C}\) and transferred into three tubs in equal proportions. Ratio of keratinous fibre weight to volume of solution used for scouring bath was maintained below 15 gm. per lit. Specimen enclosed in a 40 mesh net bag was agitated in scouring solution for about 3 to 5 minutes. Sample was taken out from the mesh bag and squeezed. It was subjected similarly in the rest of the two tubs of scouring solution. Now the specimen was washed with hot \((52 \pm 5^\circ\text{C})\) distilled water so as to remove remaining sand and scouring solution. Finally, the animal hairs sample was squeezed and centrifuged to remove the excess of water, dried it in oven at \(105 \pm 2^\circ\text{C}\) to constant weight to get oven-dry weight \((W_2)\) of the sample.

After scouring, weighed amount \((a\text{ gm.})\) of oven-dry specimen was extracted with absolute alcohol in a soxhlet extractor for about 20 extractions. Extraction flask was cooled and disconnected from soxhlet assembly and alcohol was distilled over. The residue was evaporated and dried to a constant weight at
105 ± 2°C. The alcohol extractable matter was calculated as

\[
X = \frac{100 \times a}{b}
\]

Where \( X \) = alcohol extractable matter, percent, by weight;
\( a \) = weight of residue, in gm; and
\( b \) = weight of oven-dry specimen, in gm.

For ash content, scoured oven-dry specimen was placed in silica dish and then cooled in desiccator and weighed. Slowly char the sample in a dish over Bunsen burner till it ceases to produce volatile matters. Now the charred sample was kept in muffle furnace at 700 ± 20°C for one hour or more till the constant weight. The ash content was estimated as

\[
Z = \frac{100 \times a}{b}
\]

Where \( Z \) = percentage of ash content, by weight;
\( a \) = weight of ash, in gm; and
\( b \) = weight of oven-dry specimen, in gm.

For vegetable matter, 40 gm. of scoured oven-dry animal hair sample was subjected to 400 ml. of boiled 10% sodium hydroxide solution for about 3 minutes with vigorous stirring. The residual vegetable matter was first settled down and the alkali solution
was decanted through a sintered glass crucible. About 100 ml of cold water was added and the residual vegetable matter was transferred to the sintered crucible. The residual vegetable matter was washed with distilled water and dilute acetic acid to remove the left out traces of alkali. Finally, it was washed with distilled water until the filtrate becomes neutral. Vegetable fibres and tag material was removed with the help of forecep. Subsequently, the remaining vegetable matter was transferred into a porcelain crucible dried at 105 ± 2°C until constant weight. The vegetable matter was calculated as

\[
V = \frac{a \times F \times 100}{b}
\]

Where \(V\) = percent of vegetable matter;
\(a\) = oven - dry weight of the residue, in gm;
\(F\) = correlation factor taken to be equal to 1.1; and
\(b\) = oven - dry weight of the specimen, in gm.

From these above calculated values of alcohol extractable matter, ash content and vegetable matter the clean fibre content was calculated by following equation.

\[
F = \frac{W_2 \times [100 - (X + Z + V)]}{W_1}
\]

Where \(F\) = percent of clean fibre content;
$W_2 = \text{oven-dry weight of the specimen after scouring, in gm;}$

$X = \text{percent of alcohol extractable matter;}$

$Z = \text{percent of ash content;}$

$V = \text{percent of the vegetable matter content;}$

and

$W_1 = \text{initial weight of the specimen, in gm.}$

The laboratory scoured yield was calculated by the following formula

$$L = F + V_1$$

Where $L = \text{percent laboratory scoured yield of the sample;}$

$F = \text{percent of clean fibre content;}$ and

$V = \text{percent of vegetable matter adjusted to standard conditions of individual sample.}$

3.2.1.11

**Alkali Solubility**

The sample of speciality hairs was first separated and cut into shorter lengths of one centimeter. The fatty material of sample was extracted with light petroleum in a soxhlet apparatus for about six extractions in an hour. Further, the vegetable matter and other foreign matter was removed by hand picking. Petroleum extracted sample was then dried at 105 ±
2°C for about three hours, cooled it in a desiccator, 1.0 gm of it was taken in a flask. 100 ml of sodium hydroxide solution with a varying concentration from 0.01 N to 0.15 N was poured into the flask. Stopper it loosely and immersed into the water bath at 65±5°C for about 60 minutes with shaking after 15 minutes interval. The time of shaking did not exceed 5 minutes. The left out hair content of the flask was filtered through already weighed filtering crucible. The residual fibres were first washed with distilled water for about six times and then twice with acetic acid solution in water (10 ml. in 1 lit. of water). Again the fibres were washed six times with distilled water. Finally, the filtering crucible containing residual speciality hairs was dried at 105 ± 2°C for about three hours, cooled it in a desiccator and weighed. Alkali solubility was calculated in terms of percentage weight loss on dry weight basis following IS-3429-1966.

**Sulphur Content**

Sulphur content of these animal fibres was estimated by using a semi-micro method of high accuracy as reported by Ballard et al.282 Sample was cleaned by soxhlet extraction for 6 hrs. with petroleum ether followed by absolute alcohol for a similar period. The fibres were then washed with distilled water till
no turbidity in washings was observed. The sample was dried at 105°C and 0.2 gm of oven dry sample was accurately weighed for analysis and placed in a Kjeldhal flask. The acid oxidation mixture was placed by dissolving 60 mg. K₂Cr₂O₇ in 120 ml. concentrated nitric acid and adding 60% perchloric acid. With this mixture oxidative hydrolysis of fibres was carried out for 20 min. This oxidatively hydrolysed product was washed, diluted and sulphate was estimated gravimetrically in G-4 sintered crucible. For each variety of sample five observations were made and average of these is being presented.

3.2.1.13 Amino-acid Analysis

3.2.1.13.1 Thin layer paper chromatography

a) Sample preparation for hydrolysis

Different types of fibres were separately scoured with an aq. solution of lissapol - D - paste, rinsed in water, dried and then extracted with petroleum ether for 12 hrs. Finally, the samples were dried and opened by hand to remove completely the extraneous matter remaining after scouring and extraction.

b) Hydrolysis

Nearly 100 mg each of mohair, wool and Angora rabbit hair sample was taken in a glass tube of
1/2" O.D. To this 8 ml of 6 N HCl was added, air was expelled by heating as far as possible and the tube was then sealed. The sealed tubes were kept in oven at 110°C for 24 hrs. to complete the hydrolysis. After that the hydrolysate was filtered, washed with distilled water to make the volume 10 ml and was stored in refrigerator.

c) **Preparation of standards**

Individual standards of aminoacids were prepared as 1 µg/µl solution in 6 N HCl solution. Hence, each standard was prepared by dissolving 10 mg. of an amino-acid in 10 ml of 6 N HCl solution.

d) **Layer**

Silica gel 1 B 2 sheets, used as received without pretreatment.

e) **Application of Initial Zones**

A line was drawn 2 cm. from the bottom of the sheet and a solvent front line 10 cm. away from the origin. 1 µl of the individual amino acids as a standard reference and 1 µl of hydrolysate of these samples on separate origin were placed.

f) **Mobile Phase**

n-butanol-aceticacid-water (40:10:50) (upper layer).
g) **Development**

The sheets were developed for a distance of 10 cm. from the origin in a saturated chamber containing 100 ml. of mobile phase. Since the mobile phase contains water, it took a long time i.e. 4 hrs. for development.

h) **Detection**

0.5% ninhydrin was incorporated into the mobile phase prior to development. After development the sheets were removed from the chamber, dry in air and then heated in an oven at 90°C for 5 min. Quantitative determination of these amino acids was performed by scanning chromatogram through HPTLC scanner.

3.2.1.13.2 High voltage electrophoresis

a) **Sample preparation for hydrolysis**

Each sample was separately scoured with an aq. solution of lissapol-D-paste, rinsed in water, dried and subsequently extracted with petroleum ether for 12 hrs. and finally dried in air. The samples were opened by hand to remove completely the extraneous matter remaining after scouring and extraction.
b) **Hydrolysis**

Nearly 200-300 mg. of sample was taken in a glass tube of 1/2" O.D. To this 8 ml. of 6 N HCl was added, air was expelled by heating as far as possible and the tube was then sealed. The sealed tubes were kept in oven at 110°C for 24 hrs. to complete the hydrolysis. After that the hydrolysate was filtered, washed with distilled water and excess acid was evaporated by heating on water bath at 35°C under vacuum. The dried residue was dissolved in 0.1 N HCl and the volume of each hydrolysate was made to 10 ml. and stored in refrigerator.

c) **Electrophoresis**

Electrophoresis apparatus of Systronix with Whatmann No. 4 paper (36 cm x 25 cm) was used. The samples were deposited on the meridien line at 18 cm.

The operating conditions are as follows.

- Saturation and conditioning of paper with a buffer solution mentioned below

**Buffer Solution**

- Pyridine (25 ml.)
- Acetic acid (4.5 ml.)
- Water (970.5 ml.)

Total vol. 1000 ml.
d) **Drying and developing of chromatogram**

The sheet of paper, after the electrophoresis, was dried in oven at 70°C for 15 minutes. Heilman's developer was used for developing, the constituents of which are as follows:

- Cadmium acetate 100 mg.
- Acetic acid 5 ml.
- Distilled water 10 ml.
- Ninhydrin 1 gm.
- Acetone 100 ml.

Again the paper sheet was dried in an oven at 70°C for 30 minutes.

3.2.1.13.3 **Auto-amino acid analyser**

a) **Sample preparation for hydrolysis**

One sample each of kid mohair, adult mohair (ringlet type), Angora rabbit hair (Russian breed) and cross-breed wool (ramb. x chokla) was scoured separately with an aq. solution of lissapol-D paste, rinsed in water, dried and was subsequently extracted with petroleum ether for 12 hrs. and finally dried in air. Each sample was opened by hand to remove completely the
extraneous matter remaining after scouring and extraction.

b) **Hydrolysis**

Approximately 100 mg. each of sample was taken in a glass tube of 1/2" O.D. To this 8 ml of 6 N HCl was added, air was expelled by heating and the tube was then sealed. The sealed tubes were kept in oven at 110°C for 24 hrs. to complete the hydrolysis. After that the hydrolysate was filtered, washed with distilled water and made it 10 ml. each. From these hydrolysates 3 ml. each was taken out and excess acid was evaporated under vacuum at 35°C to make a dried residue.

c) **Analysis**

The residue obtained above was diluted to 50 ml. with pH 2.0 buffer. Out of this 50 µl was injected into the auto-amino acid analyser (Beckman Instrument Inc., Palo Alto CA 94304) for quantitative amino acid analysis of these samples. The chromatogram was obtained with the help of integrator attached with the instrument. The calculations were carried out against the reference of the standard mixture of amino acids containing 2.5 µmol / ml. each except cystine with 1.25 µmol/ml. The standard was also diluted with buffer
in 1:10 ratio before injecting the same 50 µl into the analyser.

3.2.2 Morphology and Anatomy

As one of the important criteria of fibre identification, surface studies of these animal fibres with respect to scale pattern, scale margins and scale length were performed with optical as well as scanning electron microscope (SEM). Besides, the internal structure of mohair, Angora rabbit hair and wool was also examined under transmission electron microscope (TEM) and the results obtained in these studies, regarding their respective cortex formation, have been correlated with different physical and mechanical characteristics of these fibres.

3.2.2.1 Optical electron microscopy

Optical microscopy is one of the technique being applied to explore the scale arrangements in different types of wool and speciality hairs. Although several methods are in practice but the most common method of taking pictures by polaroid technique on an optical microscope was utilized. The fibres were first washed with water and then with petroleum ether to remove dust, grease and oily matter from the raw fibre surface. No mounting fluid was used for preparing slides. Micrographs were taken at 200-500
3.2.2.2

Scanning electron microscopy

The procedure used to prepare the fibre samples for examination under SEM were chosen to give minimal chemical disruption of the geometrical scale pattern of the fibres. The fibre samples were first rinsed in cold water followed by petroleum ether and then dried to remove body grease and oils from the raw fibre. The fibres were then mounted on the grid, coated by gold-platinum alloy and scanned on S-4-10 Cambridge stereoscan with the magnification range 800 to 2400.

3.2.2.3

Transmission electron microscopy

The specimens were first washed with cold water to remove dust and dirt and then rinsed in trichloroethane to remove body grease and oils from the raw fibre. The fibres were then stained in a 5% aqueous silver nitrate solution for 18 hrs in the dark. This staining procedure gave good contrast for examining the microfibrils, without the need for the potentially damaging reduction step used with osmium staining\textsuperscript{283}. The samples were then quickly rinsed in distilled water to remove extraneous silver ions before the action of light could convert them to an insoluble form. Then the samples were dried in an
oven at 105°C for 45 min. before being returned to room temperature in sealed vials and infiltrated with 2:1 acetone/epon resin followed by 1:1 acetone/epon, 1:2 acetone/epon for 6-12 hrs. in each case. Finally, the fibres were infiltrated in 100% epon resin for 3 hrs then they were embedded in fresh 100% epon resin at 60°C for 24 hrs. This dehydration technique was favoured as minimal damage is known to occur to fibres dried in this fashion, whereas, dehydration in ethanol is known to extract material from the cortex. Sections were cut on an LKB Ultratome M-78000 and then stained with 1% aq. uranyl acetate for 1/2 hr. and lead citrate for 10 min. These sections were examined under a Philips EM 410 LS microscope.

**Mechanical and Thermal Characteristics**

**Mechanical properties**

In view of the significance of mechanical properties of keratin fibres and the scarcity of systematic work on Indian mohair and Angora rabbit hair, the study of tensile characteristics was undertaken for individual fibres under both dry and wet states. Tensile properties were determined on Instron Model No.1112. Fibres were taken randomly from a lot of hairs. Preconditioning was performed at a relative humidity of 65 ± 2% or 100% at a temperature of 21 ±
1°C. Gage length of 2 cm., cross-head speed and chart speed of 1.0 cm/min and 10.0 cm/min. respectively were opted. About 50 specimens were recorded for each type of hairs. Average stress-strain curves were plotted from these 50 observations for each type of sample. The mechanical properties which have been considered for the characterisation of these keratinous fibres are breaking stress and initial modulus where the difference in cross-sectional areas was compensated by dividing each measured force dependent property by the cross-sectional areas of each fibre and reducing the property to force per unit cross sectional area in terms of dynes/cm². Other principal mechanical properties investigated are stress at 2, 15 and 20% extensions, breaking strain, yield point, secant modulus, tangent modulus, work of rupture, reduction in work upto 30% extension and work factor.

**Thermal Degradations**

As compared to wool, not much emphasis has been laid on thermal degradation studies of speciality hairs. The aim of these studies is to establish the differential identification of the two peaks as microfibril peak and matrix peak in DSC curves of mohair and rabbit hairs and their comparison with wool. Be-
sides, TGA curves of these speciality fibres were plotted to find out the percent weight loss with the temperature and were compared to endotherms of wool. Further, DSC peak temperatures and TGA temperatures of maximum rate of weight loss, under nitrogen atmosphere at a heating rate of 10°C/min., were also compared.

**Thermal gravimetric analysis**

The fibre sample was washed, dried and then cut into small pieces and sieved to a size of 50 mesh. The fluffy material obtained after grinding was compressed into a pallet form in compression molding machine at a pressure of 10 metric tons, out of which 5 ± 1 mg. sample was used. Thermal degradation was carried out under N₂ as a purging gas at flow rate 300 ml/min. using a Stanton Redcroft TG-770 thermogravimetric analyser. Heating rate of 10°C/min. was used to heat the sample from room temperature to 725°C. Primary thermograms were obtained by plotting the percent residual weight against temperature.

**Differential scanning calorimetry**

The fibre sample was washed, dried and then passed through a grinder to cut into small pieces. The fluffy material obtained after grinding was first sieved to a size of 30 mesh and then compressed into
a pallet at a pressure of 10 metric tons. Differential scanning calorimetry was performed with 6.0-7.0 mg. of fibre sample and N₂ as a purging gas. Du Pont 910 DSC analyser was used with the heating rate of 10°C/min from room temperature to 450°C. Degradation studies were also carried out after annealing fibres at 180°, 190° and 200°C for 2 to 15 minutes. The areas under the peaks were measured by the use of planimeter.

**WET PROCESSING**

Considering the lack of research work on wet processing, specifically of mohair, it was planned to optimize scouring and bleaching parameters of mohair with minimum damage to fibre surface and its lustre. Different factors of concentration of sodium carbonate, soap, salt and temperature etc. of mohair scouring with minimum damage to fibre were standardized. Different types of Non-ionic and Anionic detergents, procured from Ahura Chemical Products Private Ltd., were used to study their scouring efficiency. Use of emulsion of water with liquid paraffin oil and castor oil along with the non-ionic surfactants¹⁶⁰ was also examined. Two techniques, the optical microscopy (by determining swelling of fibre under 0.1N NaOH solution)¹⁶² and the scanning electron microscopy, have been utilized to investigate the topological changes
taking place during conventional commercial scouring by aqueous system as well as optimized emulsion scouring method. This study also compares the extent of removal of grease from these fibres during variety of applied scouring procedures. Besides, the bleaching parameters of ringlet mohair were standardised under both alkaline and acidic conditions to obtain whiter mohair fibres. Not only the bleaching agent but also the varying bleaching parameters such as time, temperature and nature of stabilizer have been applied to develop a process to produce desired degree of whiteness with least damage to fibre surface.

Scouring

Scouring studies were performed on ringlet type of mohair. In each case, the fleece after conditioning under standard conditions (65 ± 2% RH, 20± 1°C temp) was divided into samples of 5 gm. each. In order to obtain uniform and comparable samples a rotational sampling procedure was adopted in which successive handfuls of samples were taken from different areas of fleece in rotation. After preliminary opening, samples were scoured in 3 or 4 bowl system, with varying concentrations of soap and soda in each bowl, for 5 minutes in each bowl with 1:80 material to
liquor ratio. After scouring, the samples were squeezed by hand pressing. Scoured samples were air dried and vegetable matter was removed by careful hand picking. Samples were then vacuum dried overnight at 60-70°C to constant weight and allowed to condition at 20°C and 65% RH. Residual grease was ascertained by the alcohol extraction and the damage to the fibres was estimated by swelling in 0.1 N NaOH under microscope. Further, the damage to the fibres was also scanned through scanning electron microscope.

**Bleaching**

Scoured ringlet mohair has been used for these bleaching studies. In each experiment 1:50 material to liquor ratio was applied. Both - alkaline and acidic bleaching conditions, using hydrogen peroxide as an oxidant, were investigated where the concentrations of hydrogen peroxide was monitored by colorimetric titrations. Alkaline bleaching bath consisted of hydrogen peroxide from 0.3 - 1.8% w/w, trisodium phosphate (as stabilizer for controlling dissociation of hydrogen peroxide) at 4.4 gm/lit. concentration, sodium carbonate to adjust pH 8-10 and lissapol-D paste (1gm/lit) non-ionic detergent as wetting agent. Acidic bleaching bath consisted of hydrogen peroxide 0.3 - 1.2% w/w, trisodium phosphate, lissapol-D-paste
and sulphuric acid or formic acid to adjust pH3.

The evaluation of whiteness of the bleached samples was carried but by using Spectronic 20 spectrophotometer by recording reflectance at 420 nm, 450 nm, 470 nm, 500 nm, 550 nm, 600 nm and 650 nm wavelength. The whiteness index was evaluated using the Jacquemart's formula.

\[ WI = \sqrt{(CF^2 + (100-AS)^2)} \]

Where CF is the coloring factor and the difference between the reflectance at 650 nm and 450 nm.

AS is the average of reflectance noted at 420, 450, 520, 550, 600 and 650 nm.

The reflectance at 470 nm gives a direct measure of whiteness.

\[ R = \text{reflectance at 470 nm}. \]

The higher the value of WI lower is whiteness while greater the value of R better is whiteness.

**YARN PROCESSING**

Considering the lack of research work on processing of mohair and Angora rabbit hairs as such and their blends with different varieties of wool, it was planned to study the processing parameters of mohair/wool and rabbit hair/wool blends with varying ratio of components. In all, four types of adult
mohair (ringlet type, pure Angora heavy type, rough kempy and pashmina type) and a kid mohair were blended with different varieties of wool (chokla, ramb. X chokla and merino), based on their fibre diameter and length, with 40 to 80% proportion of mohair. These blends along with pure mohair and wool were processed into yarn on woollen system. Similarly, it is highly required to plan a detailed study for optimizing the blend ratio of rabbit hair and wool with respect to smooth spinning and better yarn quality. Thus, the Angora rabbit hairs were blended with merino wool in different proportions from 20 to 70% and semi-worsted tops were prepared which were spun on rollers adjustable charkha. Different observations were made and difficulties faced during processing of these yarns were analysed and concluded comprehensively.

**Processing of Mohair**

**Preliminary processes**

Preprocessing treatments such as burr picking, scouring and carbonising are highly required, specifically in case of wool, for making these fibres suitable for subsequent blending, carding and spinning processes. Burr picking was carried out manually. Prior to carbonisation, scouring is a must to remove dust, dirt, grease and oil to make vegetable surface freely available for the reaction of acid. Scouring was
performed according to standard method IS 1349-1964 in which three bowl system was applied containing 3 gm. of anhyd. sodium carbonate and 1 gm. of lissapol-D per litre of water at 52 ± 5°C. Carbonisation was performed following traditional steps of sulphuric acid treatment (4%) at a temperature of 30-36°C with 5 minutes immersion time in the presence of 0.2% sodium lauryl sulphate, drying at 70-80°C to remove excess water, baking at 130°C for 15 minutes, crushing of the charred materials and then neutralizing the fibres first by rinsing in cold water and subsequent washing in weak soda ash and finally with water. As the vegetable matter contamination is low in mohair fibres, mechanical means are preferred for their removal instead of chemical carbonising procedure where both fibre lustre and strength are highly susceptible towards speciality chemicals generally used in the process. Besides, a specialised scouring technique is required mainly due to the oxidised nature of wax associated with mohair fibres which generally poses an intricate problem in its removal upto the required level of 0.2% residual grease by a conventional scouring procedure applied for wool. Thus, raw mohair fleeces were scoured by an emulsion method using 4% of paraffin oil by weight of a fibre grease at 62°C temperature in presence of non-ionic
detergent, lauryl alcohol. 7 moles ethylene oxide.

**Blending or Mixing**

Mohair and wool were weighed to produce 2 Kg. of clean fibre per processing batch. Blends with the ratio of mohair/wool of 80:20, 60:40 and 40:60 were prepared and mixed by passing twice through a blower. Batch blending was applied in which the component with smaller percentage was mixed in two different instalments. Each ingredient was taken piled on the running table which drop it into suction trap in a duct coupled to a fan which blows the materials to a dust removing cyclone.

**Oiling of the stock**

Lubrication is usually carried out to minimise breakage of fibre in carding as well as to reduce fly waste and static electric charges. The mineral oil emulsions also serve to increase the cohesion of the fibres in a loose sliver which facilitate drafting, condensing and spinning. As the mohair could only be scoured to approximately 1.4% of residual grease, 2-4% of lubricant was applied by sprayer in mohair/wool blends, whereas only 2.0% lubrication was required in 100% mohair processing due to sufficient amount of residual grease which itself acts as lubricant. Contrary to mohair, processing of wools as such
required higher percentage of lubricant i.e. 4% due to their easily scourable greasy matter to an optimum level of 0.2% by the simple conventional scouring procedure.

**Woollen carding**

After the blends have been thoroughly mixed and oiled for a complete amalgamation of different ingredients, they were subjected to a two card system. Carding constitutes the last operation in which wool fibres open properly. Carding is considered an important process because a good and even woollen yarn can only be spun out from an evenly carded wool. Rovings, a ribbon of carded wool in which the fibres are rubbed together into a round continuous strand with no twist, were prepared from the carded stock obtained from finisher card.

**Woollen yarn spinning**

Woollen yarn spinning is only a single spinning operation following carding which permits no further cleaning or mixing as compared to worsted yarn spinning where combing and gilling provides further cleaning and mixing operations. Here the main functions are to thin or draft and impart twist. In fact, the thinning is only upto the order of 50%. Thus, the carded rovings should be free from impuri-
ties, homogeneous in fibre composition, and as uniform as possible in weight per unit length. As the ring spinning frame is being rapidly accepted by the woollen trade because it suits all grades of wool and mixture of wool with other fibres for knitting yarns as well as high grade tweed yarns, the rovings of different wool/mohair blends were spun into yarns on a woollen ring spinning frame.

**Processing of Angora Rabbit Hairs**

**Preliminary Processes**

In case of Angora rabbit hairs, it was not required to undergo preprocessing treatments such as burr picking, scouring, carbonising and dusting due to lack of waxy and vegetable matter, whereas merino wool was subjected to these treatments according to traditional standard methods.

**Blending or Mixing**

Rabbit hair and merino wool were weighed to produce 5 Kg. of clean fibre per processing batch. To ensure an even opening and blending, the Angora rabbit hairs/merino wool blends in the ratio of 20:80, 30:70, 40:60, 50:50 and 70:30 were passed twice through opening machine. Similar to mohair/wool blends, batch blending was applied in which the component with smaller percentage was mixed in two
different instalments. Each ingredient was taken piled on the running table which drop it into suction trap in a duct coupled to a fan which blows the materials to a dust removing cyclone.

3.4.2.3

**Oiling of the stock**

6% of lubricant oil was applied by sprayer to check breakage of fibres in carding as well as to reduce fly waste and static electric charge.

3.4.2.4

**Woollen Carding**

Woollen card with two card system was applied to open these fibre blends properly in the form of top.

3.4.2.5

**Gilling**

Since the woollen card is more concerned with blending, parallelism of the fibres is not upto the mark as is required in worsted carding. To obtain the yarn with uniformity, great fineness, with a consequent higher cost, gilling of tops is usually performed through gill boxes. Thus, the gilling operation was carried out initially with Gill box made indigenously by Supertex Mech. Engg., Ludhiana and then followed by German Gill box to straighten and parallelise the fibres.

3.4.2.6

**Combing**

Combing gives the yarn to its smooth, clean and
uniform diameter and appearance as against the rough, hairy, bulky and irregular diameter and appearance of woollen yarn. The choice of the comb depends largely on the type of wool to be combed and also their length, fibre diameter and amount of noil permissible are taken into consideration. The French worsted combing or the rectilinear combing is known for making good slivers of fine and short wools. Besides, French comb not only combs very short wools but handle longer wools quite satisfactorily, hence it was planned to go for French combing, particularly for the blends of Angora rabbit hairs and merino wool where both the components of blends are fine and rabbit hair is comparatively shorter than merino wool. French comb of 14 inch wide working width was used.

**French finishing gilling**

The slivers from the combs are still quite uneven and non-uniform. Evenness of the sliver is so essential to the success of future drawing operation and to the final yarn itself. Here, the blending was also accomplished by drafting and doubling slivers along the lines of previous gilling operations. It was performed by two gillings on pindrafting machines.
3.4.2.8 **Drawing**

The actual reduction in sliver weight takes place during drawing. It is necessary to employ several successive drafting operations between the top and the rovings. French or continental drawing system was employed where only false twist was applied and mechanical drafting was controlled by Porcupine rolls.

3.4.2.9 **Yarn spinning**

Ring spinning frame was applied consisting of drafting, insertion of twist and winding steps.

3.5 **YARN CHARACTERISATION**

In view of the importance of the correlation between fibre properties and ultimate yarn characteristics and the lack of information on this aspect, it was decided to embark on a study aimed at establishing the effects of the various inherent fibre properties on their yarn quality. The processed yarns from different blends of mohair/wool and Angora rabbit hair/wool were evaluated for their yarn number, skein strength, twist per meter, breaking tenacity, extension of single yarn, yarn evenness, yarn abrasion etc. and a correlation was established between yarn properties and the basic fibre properties from which they are processed.
Yarn Number or Count

Yarns were conditioned at 65% RH and 21°C temperature prior to reeling to ensure a correct length of skein. Lea were prepared on a reel having 1 meter girth, with fifty revolutions which produces a skein of 50 meters. The skein gage was used to check the reeling tension. Skeins were then scoured in boiled water with 0.5 gm. soap (lissapol-D)/lit, oven dried at 105°C to remove finishing materials. In all twenty skeins were prepared, scoured, dried and weighed in each case to calculate the yarn number of the moisture free yarn in direct system.

\[ T^1 = \frac{W}{50} \times 1000 \]

Where

\[ T^1 = \text{yarn number in direct system} \]
\[ W = \text{average mass of dried skein} \]

Adjusted yarn number was then calculated considering the moisture regain factor

\[ T = T^1 \times \frac{(100 + C)}{100} \]

Where

\[ T = \text{adjusted yarn number in direct system} \]
\[ T^1 = \text{yarn number of moisture free yarn in direct system} \]
\[ C = \text{commercial regain in percent.} \]
Skein Breaking Strength

The circumference of the skeins used to determine the
lea strength is not so critical but as the skein
breaking tenacity has to be calculated, hence the
method of reeling preparation was performed with the
control to reeling tension as in case of yarn number.
Total, twenty five number of skeins were prepared on
1 meter reel, with 50 turns in each case. Then
skeins were conditioned under the standard atmosphere
for textile testing (65% RH and 21°C temperature) for
about 4 hrs. Cross-head speed of instron was adjusted
at 300 mm/min. under standard testing conditions of
65% RH and 21°C temperature. To calculate the break­
ing tenacity and count strength product, the broken
skeins were weighed and the average yarn number was
evaluated. Average breaking load was calculated from
the observed values. From the skeins breaking load,
skein breaking tenacity was calculated by applying
the equation.

Skein breaking tenacity, gm f/tex

\[ \frac{L}{(2 \times 50 \times T)} \]

Where

\[ L = \text{average breaking load, gm f;} \]
\[ T = \text{average yarn number, tex} \]
Twist per meter

The samples were conditioned under 65% RH and the testing temperature was maintained at 21°C. The movable clamp of the tester was adjusted to maintain the gage length 50 mm. During testing the tension on the movable clamp was fixed at 0.25 gm f/tex. Twist was removed completely by turning the rotatable clamp until the yarn element are parallel which are determined by passing a needle between the untwisted fibres from one clamp to another. Direction of the twist was also recorded directly from the twist tester. About 100 readings were recorded in each sample. The average amount of twist was calculated as turns per meter. Twist factor was also calculated by the equation.

\[
\text{Twist factor} = \frac{t}{\sqrt{T}}
\]

Where

- \( t \) = twist in turns/cm;
- \( T \) = yarn number in tex

Tensile properties of single yarn

Each sample of yarn was first preconditioned at 25% RH at 50°C. After preconditioning they were brought to moisture equilibrium of 65% RH and 21°C temperature in about 4 hrs. The instron was operated at a X-head speed 300 mm/min. with the gage length of 25 cm. Testing was also performed at 65% RH and 21°C
temperature. Breaking load and breaking elongation were recorded directly from the instrument for fifty specimens. Average breaking load, gmf; average breaking tenacity, gm f / tex; average percentage of breaking extension along with their coefficient of variation were calculated.

3.5.5  
**Yarn Evenness**
From each of the yarn packages a skein was reeled with sufficient length for testing. As the strand to be tested should have a uniform moisture content along its length, first the strand was conditioned at 65% RH at 21°C for 4 hrs and also the testing was carried out under similar atmospheric conditions. Yarn unevenness of nine specimens in each case was evaluated on Uster model B.I. As the unevenness values are to be compared Lb and Lw were kept constant in each case. Travelling speed on strand was maintained at 100 Yd/min and each specimen was run for about 10 min. Unevenness was recorded directly from the instrument in U% besides, thick and thin places and nep s/100 m. Average mean of unevenness was calculated for each sample.

3.5.6  
**Flex Abrasion**
Samples were prepared by mounting 20 fibres, parallel to each other and side by side in each case, for
measuring the relative resistance to abrasion. The length of test specimens was maintained at 200 mm. Five specimens were tested for each sample of yarn. The specimens were preconditioned by bringing them to moisture equilibrium in the standard atmosphere for preconditioning, then bringing them to moisture equilibrium for testing in the standard atmosphere for textile testing i.e. 65 ± 2% humidity and 21 ± 1°C temperature. For wool, mohair and their blended yarns the head weight/tension weight ratio was kept as 0.5/2.0 lbs, whereas in the case of Angora rabbit hair/merino yarns, it was maintained as 0.5/1.0 lbs. Each specimen was abraded against the blade until ruptured on Universal wear tester.