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Determination of the Contaminant Threshold level for Medullated Fibres in Merino Wool Fabrics

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SUMMARY

1. Knitted and woven fabrics were produced from Merino tops carefully contaminated with medullated fibres of a wide range of diameters and med ratios. The fabrics were dyed to different shades selected to highlight medullation contamination, and subsequently inspected at a commercial mill by quality control officers. Contaminated fibres were extracted then microscopically examined and analysed at AWTA Ltd, Research and Development Division. The results showed that:
 - A Med Ratio of 0.8 in a fibre of 50µm diameter is the appropriate threshold for contamination in fabrics made from merino wool.
 - The following factors affected the rate or grade of contamination on fabrics:
 - Med Ratio;
 - Fibre Diameter;
 - Length of the fibre;
 - Number of contaminant fibres; and
 - Fabric colour.
 - An increase in Med Ratio, diameter or length of the medullated fibre, or the numbers of marginal contaminant fibres resulted in an increase in the incidence of faults detected in a fabric. An increase in fibre diameter, for Med Ratios 0.9 or higher, generally resulted in a “bright” fault being detected.
 - The much higher incidence of faults visible in the dyed compared to undyed fabrics indicated that strong colours (eg. burgundy and teal) show more faults than lighter shades (eg. ecru shades).

1. BACKGROUND

The Australian Merino industry and the textile industry, which uses its wool, are dedicated to supplying high quality fabrics. However a small number of claims for compensation based on medullated fibre contamination of fabrics, has raised industry concern that the reputation of the Australian Merino clip may be affected by the presence of medullated fibres (Morgan 2003). Medullated fibres are defined as fibres that are generally chalky white in appearance, even after dyeing, and which are considered objectionable (Hunter, 1987). However, there is no objective definition of a contaminant medullated wool fibre that has been verified by published trials. Currently a Med Ratio value (Medulla Diameter / Fibre Diameter) of 0.6, from the American Society of Testing Materials (ASTM) Standard 2968-75, is used in commercial presale testing for medullated fibres (AWTA testing method). However, based on published results (Balasingam and Mahar, 2005) this may lead to an overestimation of medullated fibre contamination experience by commercial processors. There is consequently a need to generate more information about the level of medullation that causes faults in dyed fabrics.

This report describes a project funded by Australian Wool Innovation (AWI) aimed at determining the threshold at which a medullated fibre becomes an objectionable contaminant. Knitted and woven fabrics were produced from Merino tops carefully contaminated with medullated fibres (approximately 7000 fibres/ 10Kg) of a wide range of diameters and med ratios to determine the lowest medullation threshold selected as a contaminant by a commercial mill. The fabrics were dyed to different shades, selected to highlight medullation contamination, and subsequently inspected by experienced fabric inspectors at the mill. Contaminated fibres were extracted from the fabrics then microscopically examined and analysed at AWTA Ltd, Research and Development Division.

2. PROJECT AIM

To determine the commercial threshold level at which a medullated fibre is considered objectionable in a Merino wool fabric (knitted or woven).

3. METHOD AND MATERIAL

3.1 Trial design

The trial design followed eight sequential steps:-

- i. Selection and supply of a top heavily contaminated with fibres containing a wide range of medullation;
- ii. Blending the contaminated top with a larger mass of Merino top;
- iii. Detection of medullation in the blended top;
- iv. Conversion of top into yarn and Manufacture of both knitted and woven fabrics;
- v. Dyeing of fabric to selected shades;
- vi. Inspection of fabric;
- vii. Extraction of medullated fibres and quantification of degree of medullation (Med Ratio), fibre diameter, and the thickness of the fibre wall ((fibre – medulla) diameter);
- viii. Analysis of the measurement results.

3.1.1 Selection and supply of a top heavily contaminated with fibres containing a wide range of medullation

The number of contaminant fibres required was calculated based on the following assumptions:

- A contaminant rate of 10 fibres/m² was required in the finished fabric;
- An average fibre mass was 30µg;
- There were no processing losses

The four (4) sources of medullated fibres are listed in Table 1, along with their range in fibre diameter and Med Ratio.

Table 1. Sources of medullated fibres

Source	Diameter(μm)	Med Ratio
IH Top 7	50-100	0.20-0.71
Tukidale	62-173	0.33->0.9
Damara Kemp	62-200	>0.9
Highly contaminated Top	50-87	0.4->0.9

Approximately 900 fibres were individually inserted into 12 m of Interwoollabs calibration Top 7(Series 14), as well as 100g of highly contaminated top as shown in Figure 1. The top was then sent to CSIRO for conversion into dyed fabric.



Figure 1 Adding medullated fibre

3.1.2 Blending the contaminated top with a larger mass of Merino top

A critical aspect of this trial was the successful acceptance of the introduced medullated fibres by the gilling and blending machinery, i.e. the introduced fibres being able to blend intimately with the other fibres in the top during the blending sequence. CSIRO was able to devise a blending scheme which achieved this result. A 12m length of top contaminated by AWTA was blended with approximately 10 Kg of approximately 20.7 μm top at CSIRO, Textile and Fibre Technology according to the scheme summarised in Table 2.

Table 2. Blending scheme for inserting the contaminated top

Stage	Machine	No. of Ends	Draft	Ratch (mm)	Speed (m/min)
1	Ingolstadt	5	4.73	30	50
2	Ingolstadt	3	4.73	30	50
3	Ingolstadt	4	4.73	30	50
4	Ingolstadt	6	4.73	30	50
5	Ingolstadt	6	6.58	35	60
6	Ingolstadt	4	6.58	35	60

Stage 1: one end of short fibre length sliver plus one end of long fibre top seeded with medullated fibre (top supplied by AWTA split into two ends of equal length) and 3 ends of uncontaminated top supplied by CSIRO (20.7 μm , 24.3% CVD, 69.7 mm-H, 57.1% CVH);

Stage 2: one end of sliver from stage 1 plus two ends of uncontaminated top;

Stage 3: two ends from stage 2 (output sliver split into two equal lengths) plus two ends of uncontaminated top;

Stage 4: five ends from stage 3 (output sliver split into five equal lengths) plus one end of uncontaminated top; and,

Stages 5 and 6: two blending stages of output from stage 4; i.e. no further addition of uncontaminated top

3.1.3. Detection of medullation in the blended top

Samples of the blended top were examined using the AWTA Dark and Medullated Fibre detection system to count the number of medullated fibres detected. This procedure was to confirm that the top contained a sufficient number and variety of fibres to continue the trial. Results of this examination are shown in Table 4. The standard presale core test was adapted to test top samples for this trial.

3.1.4 Conversion of top into yarn and Manufacture of both knitted and woven fabrics

The blended top was drawn into a roving, spun into a yarn and either woven or knitted into a fabric according to the following scheme:-

DRAWING

Table 3. Details of sliver drawing

Stage	Machine	Ends	Loading (kTex)	Draft	Front Ratch (mm)	Speed (m/min)
1	GN6	6	114	7.6	40	60
2	GN6	6	88.6	7.6	40	80
3	GV-11	3	57	6.8	-	95

ROVING

Machine: Sant'Andrea RF 4A Vertical Rubbing Rover

Draft: 12.4

Ratch: 140 mm

Spiral: No. 12

Speed: 100 m/min

Roving count: 560

SPINNING

Machine: Zinser 421 E1
Yarn count: 1/36 Nm (28 Tex)
Yarn type: Solospun
Draft: 17.9
Twist: 85 α_m (511 tpm); Z-twist
Spindle speed: 9,000 rpm
Traveller no.: 25

KNITTING

Machine: FAK sample knitting machine
Gauge: 23 needles per inch
Diameter: 10 inches
Cover factor: 1.37

WEAVING

Machine: Somet Thema, flexible rapier

Warp:

- 2/40 Nm; yarn supplied by CSIRO and therefore not contaminated with dark or medullated fibre
- 20.5 ends per cm

Weft:

- 1/36 Nm Solospun yarn contaminated with dark and medullated fibre as spun for knitting
- 27 picks per cm

3.1.5 Dyeing of fabric to selected shades

Knitted and woven fabrics were dyed to two different shades selected to highlight medullation contamination, at CSIRO, Textile and Fibre Technology according to following scheme.

A. Knitted samples:

Navy

Prescour @ 60 degrees C with 1 g/l Texal HZ

1% Albigal SET
10% Glauber salt
pH 4.5 with Acetic acid

2.6% Lanaset Navy

98 Degrees C for 60 mins.

Burgundy

0.4% Lanaset Yellow 2R
1.6% Lanaset Red 2B
0.6% Lanaset Navy R

B. Woven samples:

Teal

Prescour @ 60 Degrees C with 1g/l Texal HZ

1% Albigal A
10% Glauber salt
pH 4.5 with Acetic acid

0.133% Nylosan Yellow E2RL
0.03% Nylosan Red EBNL
0.88% Nylosan Blue EBGL

98 Degrees C for 60 mins.

Burgundy

1% Albigal SET
10% Glauber salt
3% Formic acid 90%

0.4% Lanaset Yellow 2R
1.6% Lanaset Red 2B
0.6% Lanaset Navy R

3.1.6 Inspection of fabric

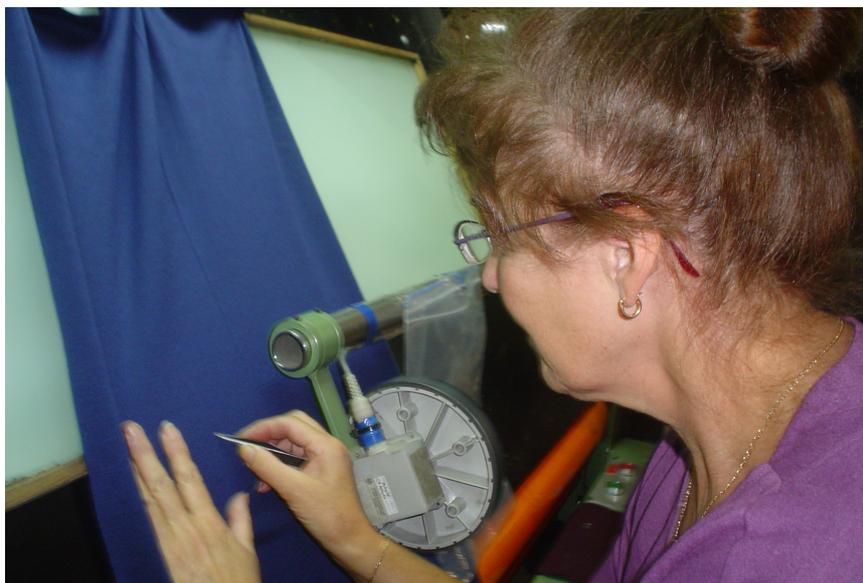


Figure 2 Medullated fibre extractions by fabric quality control inspector at Macquarie Textiles.

The fabrics were initially examined at AWTA Ltd to confirm the presence of medullated fibre contamination and then sent to Macquarie Textiles Groups (MTG) for commercial inspection. Two (2) inspectors were instructed to ignore fabric and yarn structural faults and to concentrate on medullated fibre faults.

The fabrics were inspected from a distance of approximated 1.5m from the fabric surface. The inspectors were asked to identify medullated fibre faults in the fabrics, and also to differentiate very bright faults from faults of marginal brightness.

Upon completion of inspections the fabrics were returned to AWTA and all fibres detected by the MTG inspectors were removed for microscopic examination and measurement.

3.1.7. Microscopic measurement

Twelve clearly objectionable (bright) and 12 marginally objectionable (marginal brightness) fibres were extracted from the knitted fabrics, and 10 clearly objectionable and 14 marginally objectionable fibres were extracted from woven fabrics. These fibres were mounted in water and examined under the microscope (magnification - 20 times). Approximate diameters and med ratios were measured using a graduated eyepiece in the microscope. The graduations were calibrated from a Projectina graticule. The smallest step in the eyepiece was equivalent to 2.48 μ m.

3.1.8 Analysis of the measurement results.

The microscope measurements were analysed based on the grade of faults (bright or marginal) in the knitted and woven fabrics separately.

4. RESULTS FOR THE BLENDED TOPS AND UNDYED FABRIC.

4.1 Medullation detection of blended tops.

Samples of the blended top were examined using the AWTA Dark and Medullated fibre detection system to count the number of medullated fibres. Results from two (2) samples of top are shown in Table 4. The levels of medullated fibre contamination found in these samples are indicative of a highly contaminated top.

Table 4. Results of Medullation detection of blended tops

Subsample no	Number of medullated fibres/5g		
	Less than the current threshold level	At a level near the current threshold level	Greater than current threshold level (Bright)
1	126	50	26
2	116	52	36
Ave	121	51	31

This examination indicated there were a reasonable number of medullated fibres covering a range of med ratios in the top, so the decision was made to proceed to the next step in the trial and to produce the contaminated fabrics.

4.2 Examination of undyed fabrics

Two samples of the undyed (ecru) knitted fabric were also examined for Dark and Medullated fibre contamination using a minor adaptation of the AWTA presale DMF test, to count the number of medullated fibres in the sample. The samples were approximately 544cm² and 420cm² in area.

Table 5. Results of the undyed knitted fabrics.

AWT400a	Operator 1	Operator 2	AVE
Very Bright(>60%)	24	20	22
Marginal(~60%)	34	33	34
Less Bright(<60%)	59	43	51
	117	96	107
AWT400b	Operator 1	Operator 2	AVE
Very Bright(>60%)	20	19	19.5
Marginal(~60%)	18	21	20
Less Bright(<60%)	40	45	42.5
	78	85	82

These results indicated that:

- (i) There were high levels of medullated fibre contamination present in the fabric;
- (ii) There was an approximately even distribution of contaminated fibres over the 2 samples tested.
- (iii) There was a range of Med Ratios (i.e, significant numbers of fibres below and above the current threshold) in the knitted fabrics.

5. RESULTS AND DISCUSSION

5.1 Examination of fibres

A total of 46 contaminant fibres were identified as contaminants by MTG officers. A total of 0.5 m² of knitted and 6 m² of woven fabric was examined.

Results of the microscope measurements of diameter and Med Ratio are presented separately according to the fabric structure (woven or knitted), colour and grade of fault (bright or marginal) in Tables 6 to Table 13 for the following fibre and fabric combinations respectively.

- Bright fibres in navy knitted fabric;
- Bright fibres in burgundy knitted fabric;
- Marginally contaminant fibres in navy knitted fabric;
- Marginally contaminant fibres in burgundy knitted fabric;
- Bright fibres in teal woven fabric;
- Bright fibres in burgundy woven fabric;
- Marginally contaminant fibres in teal woven fabric; and,
- Marginally contaminant fibres in burgundy woven fabric.

Table 6. Results of microscope examination of bright fibres extracted from the Navy colour knitted fabric

Fibre No.	Diameter(µm)	Med Ratio	Comments
1	173	>0.9	Damaged flat fibre
2	116-136	>0.9	Flat fibre
3	74-89	0.2-0.9	60% of the fibre, Med Ratio>90%
4	30-99	0.3-0.9	Kemp fibre 75% of the fibre Med Ratio>90%and diameter > 80 µm
5	174	>0.9	Flat fibre

Table 7. Microscope results for bright fibres in the Knitted Burgundy colour fabric

Fibre No.	Diameter(µm)	Med Ratio	Comments
1	50-100	>0.9	Kemp fibre, with flat, damaged portions
2	74	>0.9	Flat fibre
3	25-50	>0.9	Kemp fibre, containing flat damaged portions, > 50% of the fibre 50µm
4	100	>0.9	80% of the fibre contains flat damaged portions
5	124-136	>0.9	Flat fibre
6	100	>0.9	Flat ribbon like fibre
7	74	>0.9	Damaged flat fibre

Table 8. Microscope results for Marginal Fibres in Knitted, Navy colour fabric.

Fibre No.	Diameter(μm)	Med Ratio	Comments
1	50-57	>0.9	Flat fibre
2	74-87	>0.9	Flat fibre
3	55	>0.9	70% of the fibre damaged, flat fibre
4	50	0.8-0.9	75% of the fibre Med Ratio>0.9
5	62-74	>0.9	Flat fibre
6	74	>0.9	Highly medullated fibre

Table 9. Microscope results for Marginal Fibres in Knitted Burgundy colour fabric.

Fibre No.	Diameter(μm)	Med Ratio	Comments
1	54	0.8-0.9	Flat fibre
2	52-62	>0.9	Fibre flat damaged portions
3	54	0.86-0.9	Damaged flat fibre
4	74	>0.9	Damaged flat fibre
5	62	>0.9	Highly medullated fibre
6	62-99	>0.9	Kemp fibre, 75% of the fibre flat and damaged

Table10. Results of microscope examination of bright fibres extracted from the Teal colour woven fabric

Fibre No.	Diameter(μm)	Med Ratio	Comments
1	104-178	>0.9	Flat damaged fibre – very bright
2	50-174	>0.9	Kemp fibre
3	198	>0.9	Flat, damaged fibre
4	62-178	>0.9	Flat, damaged fibre with an uneven diameter

Table 11. Microscope results for bright fibres woven Burgundy colour fabric

Fibre No.	Diameter(μm)	Med Ratio	Comments
1	50-148	>0.9	Kemp fibre
2	30-86	>0.9	50% of the fibre >50 μm
3	124	>0.9	Highly medullated fibre
4	74-99	>0.9	Highly medullated and uneven diameter
5	76-99	>0.9	Highly medullated and uneven fibres
6	50-149	>0.9	Kemp fibre

Table 12. Microscope results for Marginal Fibres woven Teal colour fabric

Fibre No.	Diameter(μm)	Med Ratio	Comments
1	101-149	>0.9	Flat fibre , Uneven diameter
2	124	>0.9	Highly medullated
3	50-161	>0.9	Highly medullated fibre, uneven diameter
4	87-163	>0.9	Highly medullated, uneven diameter
5	77-111	>0.9	Flat fibre, uneven diameter
6	74	>0.9	Highly medullated fibre

Table13. Microscope results for Marginal fibres woven Burgundy colour fabric

Fibre No.	Diameter(μm)	Med Ratio	Comments
1	107-148	>0.9	Damaged fibre
2	54-62	>0.9	Flat damaged fibre
3	54-74	>0.9	Highly medullated fibre
4	50	0.5 – 0.85	Uneven medullated fibre found outside the fabric
5	50-74	>0.9	Kemp fibre
6	50-111	>0.9	Uneven diameter
7	89-101	>0.9	Uneven diameter
8	112-124	>0.9	Uneven diameter

5.2 Fibre diameter and Med Ratio of contaminant fibres.

The results show for both fabric colours and constructions and for both 'bright' and 'marginal' contaminant fibres, the minimum fibre diameter of the contaminant fibres was always coarser than the Mean Fibre Diameter of the non-contaminant, or background, fibres from which the fabric was constructed. Also, the diameter of the brightest portion of all contaminant fibres was at least 50 μm .

Not surprisingly, the minimum diameter of a 'bright' contaminant fibre was also 50 μm , though there was only one instance of a 'bright' fibre with such a low diameter. The next lowest diameter for a bright fibre was 74 μm , the average 'bright' fibre diameter was 112 μm , and the range of diameter of the 'bright' fibres was from 50 μm to 178 μm .

The minimum diameter of a 'marginal' contaminant fibre was also 50 μm . The average 'marginal' fibre diameter was 78 μm , and the range of diameter of the 'marginal' fibres was from 50 μm to 163 μm .

The minimum Med Ratio for the 'bright' fibres was 0.90, with most fibres having a Med Ratio > 0.9. The minimum Ratio for the 'marginal' fibres was similar at 0.85, with most fibres again having a Ratio >0.9.

It was determined that there were 1558 fibres/m² of both marginal brightness (Med Ratio~0.6) and less than marginal brightness (Med Ratio<0.6) in the undyed fabric based on examination of the (undyed) knitted fabric in Benzyl Alcohol. These fibres were also present in the dyed fabric, though they were not detected as contaminants.

The Med Ratios of medullated fibres extracted from dyed fabrics were greater than the current threshold value used in the AWTA Ltd presale core test, i.e., a Med Ratio of 0.6 at a fibre diameter of 50 μm . Only dyed fibres with a Med Ratios of 0.8 or greater were considered as contaminants, even though the fabric colours were specifically chosen to highlight medullated fibres.

5.4 Fibre description and comment

As expected and reported in a recent review of medullated fibre contamination (Balasingam, 2005), the extracted contaminant fibres were mainly kemps, flat fibres, highly medullated fibres, or combinations of these as shown in the Figures 3,4,and 5.

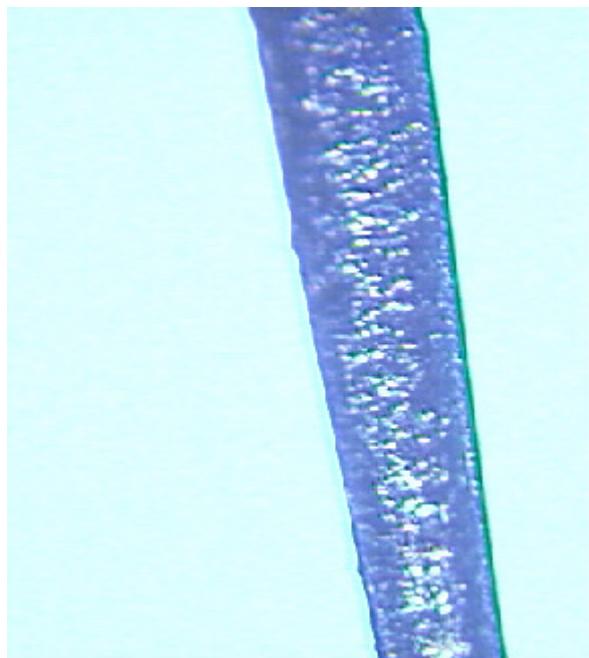


Figure 3 Portion of Kemp fibre (10 x magnification).

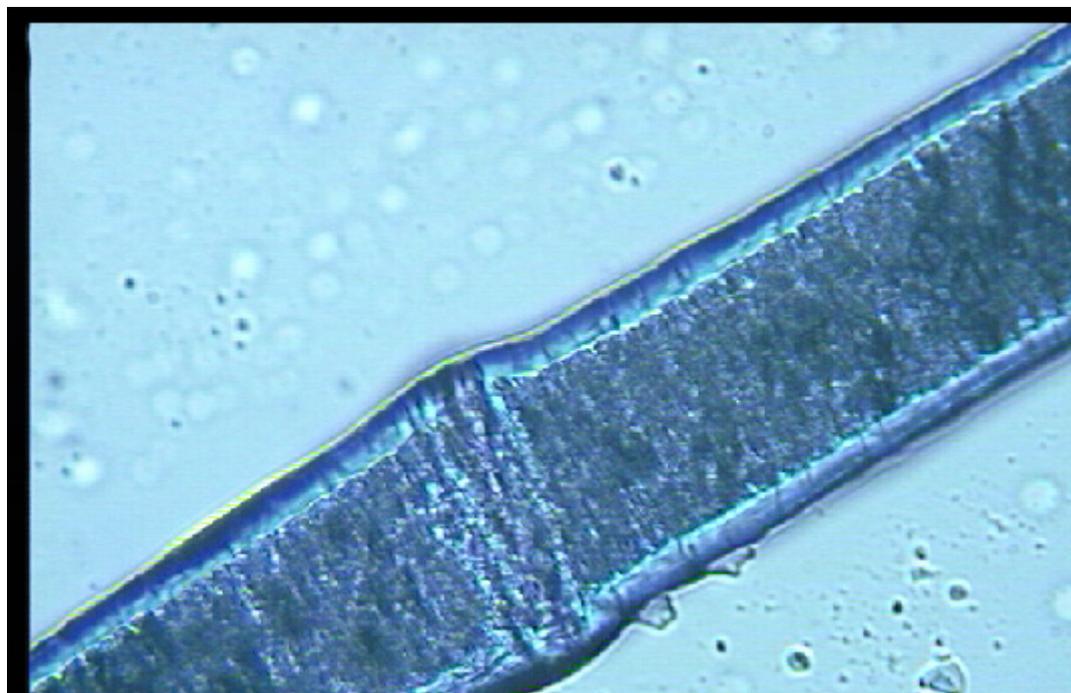


Figure 4 Highly medullated fibre (20 x magnification).



Figure 5. Highly medullated fibre with a normal fibre (20 x magnification)

As can be observed in these figures, the fibres contained a very low proportion of keratin material, the substance which is available to absorb the dye.

5.5 The Influence of colour

It would appear that the solid material (keratin) in a medullated fibre absorbs dye stuff to a similar concentration as a nonmedullated fibre dyed in the same bath (Hunter, 1987). The hue and concentration of dyestuff applied to the wool fibres would be expected to influence the degree to which medullated fibres appear different, as darker shades provide more contrast to the lighter appearance of medullated fibres than lighter shades. It was very difficult to detect medullated fibres in the undyed (i.e. light colour) fabric in this trial, yet 'bright fibres were obvious in the dyed fabric.

The different appearance of a contaminant medullated fibre in dyed samples may be caused by:

- An optical effect due to the reduced light path through the fibre wall and consequent reduced opportunity for light to be absorbed by the dye molecules; and,
- Refraction and reflection of light at fibre (keratin) to medulla interface resulting in more light emerging from a medullated fibre than from normal fibre.

According to staff at MTG (ref D. Tester – personal communication), medium to dark colours are the main source of problems with medullated fibre contamination. Very dark shades of black, dark navy and dark brown are less frequently a problem than the bright medium shades, such as the burgundy and teal used in this trial. This conclusion is supported by the observations of experienced fabric technicians.

5.6. The influence of the of the fibre length

The length of the fibre can influence its perception as a fault. A fibre which is marginal for brightness may be perceived as a fault if it is sufficiently long. The fabric examiner from MTG noted that, combinations of fibre length and brightness may make an individual fibre appear unacceptable. The fabric examiners included a 5 cm long fibre with very marginal brightness as a contaminant because of its length, fibres of similar marginal brightness but shorter length were not included as faults.

5.7 The effect of the numbers of medullated fibres in a fabric

The MTG inspectors noted that even a small number of bright fibres is a problem, for fabric quality whereas a small number of marginal fibres is not considered such a problem. But a large number of marginal fibres is considered a problem.

5.8 Comparison of Knitted and woven fabrics

Table 15 Results of comparison of Knitted and woven fabrics

Description of fabric	Area (m ²)	Approx. Weight(g) of contaminated yarn	No of fibres detected/kg
Knitted Navy	0.25	50	220
Knitted Burgundy	0.25	50	260
Woven Teal	3	270	37
Woven Burgundy	3	270	51

Comparison of knitted and woven fabrics shows that the numbers of contaminated fibres detected per mass of yarn were higher in the knitted fabrics than in the woven fabrics. An interesting observation is that the MTG fabric inspectors noticed a number of medullated fibres in the woven fabric structure which were not in the yarn structure. This may indicate that the different actions of weft insertion into a woven fabric compared to knitting may act preferentially to dislodge medullated fibres.

Flat fibres will behave differently in a yarn structure to a “normal” fibre with a circular or elliptical cross sectional shape. The “normal” fibres follow a migrating spiral path through the yarn, which will be difficult for a flat, or ribbon shaped, fibre because of its asymmetric bending and torsional rigidities.

These differences may induce a different fibre path within the yarn for ribbon shaped (highly medullated) fibres, which results in these fibres being preferentially on the yarn surface for part of their length, and particularly for one end of the fibre. These surface fibres may subsequently be broken from the yarn during weaving and become lodged in the woven structure.

5.9 The medulla diameter and thickness of keratin material

The medulla diameter and thickness of keratin material were measured for each of the extracted fibres. There was no evidence that these parameters assisted in distinguishing between the marginal and the bright contaminate fibres.

6. CONCLUSION

- Results obtained on medullated fibres removed from dyed fabric suggested that a Med Ratio of 0.8 in a fibre diameter of 50µm is the threshold for contamination in fabrics made from Merino wool.
- The following factors have been observed to affect the rate or grade of contamination on fabrics
 - Med Ratio;
 - Fibre Diameter;
 - Length of the fibre;
 - Number of contaminant fibres;
 - Fabric colour.
- An increase in Med Ratio, diameter or length of the medullated fibre, or the numbers of marginal contaminant fibres will result in an increase in the incidence of faults detected in a fabric. An increase in fibre diameter, for Med Ratios 0.9, will generally result in an increased probability of a “bright” fault being detected.
- The much higher incidence of faults detected in dyed compared to undyed fabrics indicates that strong colours (eg. Burgundy and teal) show more faults than lighter shades (eg. ecru shades).

7. ACKNOWLEDGEMENTS

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