AWTA LIMITED

AUSTRALIAN WOOL INNOVATION

PROJECT – EC651

Milestone TMS01

The Definitions of Medullation Threshold Values used by Different Testing Methods to Define an Objectionable Medullated Fibre in Merino Wool.

A literature review prepared

by

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Conclusion

There is no objective definition of a contaminant medullated wool fibre that can be substantiated by published trials. The American Society of Testing Materials (ASTM) has published a definition in ASTM-D 2968-89 that uses the ratio of medulla to fibre diameter to define contaminant medullation. But we have been unable to locate references to a trial or trials that substantiate this value.

Hunter et al. (1996) reported a trial in which experienced mill staff visually detected objectionable medullated fibres of samples in air. The medulla to fibre diameter ratios of the detected fibres ranged from 0% to 90%, indicating that, either the detection method was unreliable or that the medulla to fibre diameter ratio is an inappropriate parameter to define objectionable medullation. However, when testing mohair in benzyl alcohol, Smuts and Hunter (1983) appeared to support the ASTM definition, although the ratio they found was 0.55 compared to 0.60 for ASTM-D 2968-89.

OFDA technology has a definition of objectionable medullated fibres based on fibre opacity and diameter, but this definition is not applicable to bulk presale core testing.

Other techniques for medullation measurement such as projection microscope (pm) and the WRONZ Medullameter use the ASTM definition to directly (pm) or indirectly (Medullameter) to specify a threshold level of medullation for contamination.

Future Plan

Based on this review, the current plan for Project EC651 is to proceed with the processing to dyed fabric of a small (<10kg) quantity of wool that has been contaminated with medullated fibres incorporating a wide variety of medulla to fibre diameter ratios and fibre diameters. Contaminant fibres identified in the fabric will be extracted and their fibre diameter, shape (flat or cylindrical), medulla diameter to fibre diameter ratios determined.

In this manner threshold level of medullation should be determined from the trial.

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1. Introduction

Medullated fibres are characterised by having a central canal (Medulla) containing cell residues and air pockets, running in either a continuous or fragmented form along their length (Smuts 1987). Certain medullated fibres (Objectionable medullated fibres) in wool can be a cause of problems in some end products because they are noticeably different when viewed by the naked eye against a background of surrounding fibres. Complaints related to the presence of objectionable fibres are about their chalky white appearance and apparent inability to dye to the same shade as a normal solid fibre, and also to a lesser extent, their effect on handle, stiffness and prickliness.

Medullated or kemp fibres can occur on the leg and face of merino sheep, or in response to nutritional changes due to environmental factors (Sommerville 2004). These short fibres are generally not included in, and are removed from, fleece wool during shearing and clip preparation so that the Australian Merino industry has a hard-won reputation for having wool free of objectionable medullated fibre. Recently, another potential source of medullated fibre contamination has arisen, i.e., from the transfer of medullated fibres from exotic fibre-shedding breeds of sheep to Merinos. Claims for compensation based on medullated fibre contamination of fabric, have resulted in industry concern that the reputation of the Australian Merino clip may be affected by the presence of medullated fibres (Morgan 2003).

A certain percentage of medullated fibres of mohair are removed during mechanical processing, particularly during carding and combing. Nevertheless, generally a small number of objectionable medullated fibres may persist into top and play an important role in determining its quality and price in the market place (Hunter, 1993 cited Turpie 1995). For example, the presence of even small amounts of objectionable medullated fibres in a high quality mohair may have a pronounced adverse effect on its value. Higher grades of mohair are largely free from objectionable medullated and medullated fibres, the objectionable medullated fibre content being well below 1% in well-bred South African Mohair (Hunter, 1987).

The distinction between the objectionable and non-objectionable medullated fibres has been the subject of much research and debate, but to date has not been satisfactorily resolved (Turpie et al. 1995). A round trial conducted specifically to compare the subjective assessments of objectionable medullated fibres by laboratories from various countries, showed that assessments differed widely. The trial results confirm the errors involved in visual observation given the subjective nature of visually classifying a fibre as objectionable or not (Turpie et al.1995). However, most of the work has been done on mohair, as medullation has been shown to be a problem for this fibre, whereas medullation generally is not considered as a contaminant in Merino wool.

The purpose of this review is to summarise detailed information on:

- The definition of medullated and Kemp fibres;
- The test methods involved in medullated fibre measurement and their definitions for different types of medullated fibres;
- The loss of medullated fibres in processing; and,
- Dyeing behaviour of medullated fibres and any influence dyeing has on defining the threshold value of the objectionable medullated fibres.

2. Definition of medullated fibres

2.1 Introduction

Medullated fibres have been defined as fibres that are generally chalky white in appearance, even after dyeing, and which may be referred to as objectionable fibres (Hunter, 1987). Sometimes these objectionable fibres are referred to as kemp fibres (ASTM definition). The term kemp fibres is also associated with very thick, brittle, chalky white fibres, which have been shed by the animal rather than been shorn. Kemp fibres are also objectionable fibres, but their probability of loss during processing is very high (Hatcher 1999). Therefore it is important to define each type of visually objectionable fibre both to differentiate the different types from each other, and to determine their influence on the final product.

2.2 Medullated fibres

A medulla can be defined as the central portion of some animal fibres consisting of a series of cavities formed by the medullary cells. In some fibres, e.g. wool kemps, the medulla forms the greatest portion of the fibre and is surrounded by a comparatively thin layer of cortex (Appleyard, 1978). These medulla cells are formed from the germinal layer around the tip of the papilla. As they pass up the follicle and keratinise, the cell contents condense around the nucleus forming a corona from which trabeculae radiate, resulting in a network of hollow, air-filled, polyhedral-shaped interstices (Auber 1949, cited Ross 1955). As shown in Figure 1, these interstices of intercellular origin and the trabeculae around them are commonly known as medulla cells. The vigour theory (Dry 1941, cited Ross 1950) of fibre growth and medullation explains that a fibre is large and medullated because a very vigorous follicle produces more fibre material than it is able to keratinise, resulting in a medullated central core.

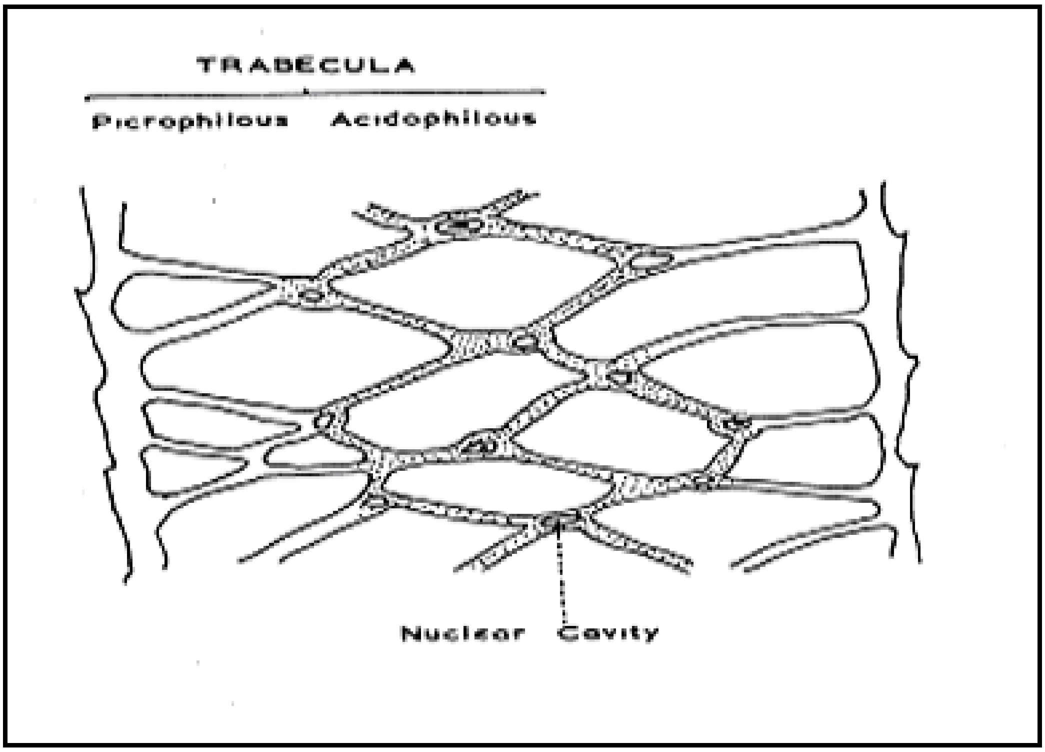

Figure1. Inside structure of medulla (Auber 1949, cited Ross 1955)

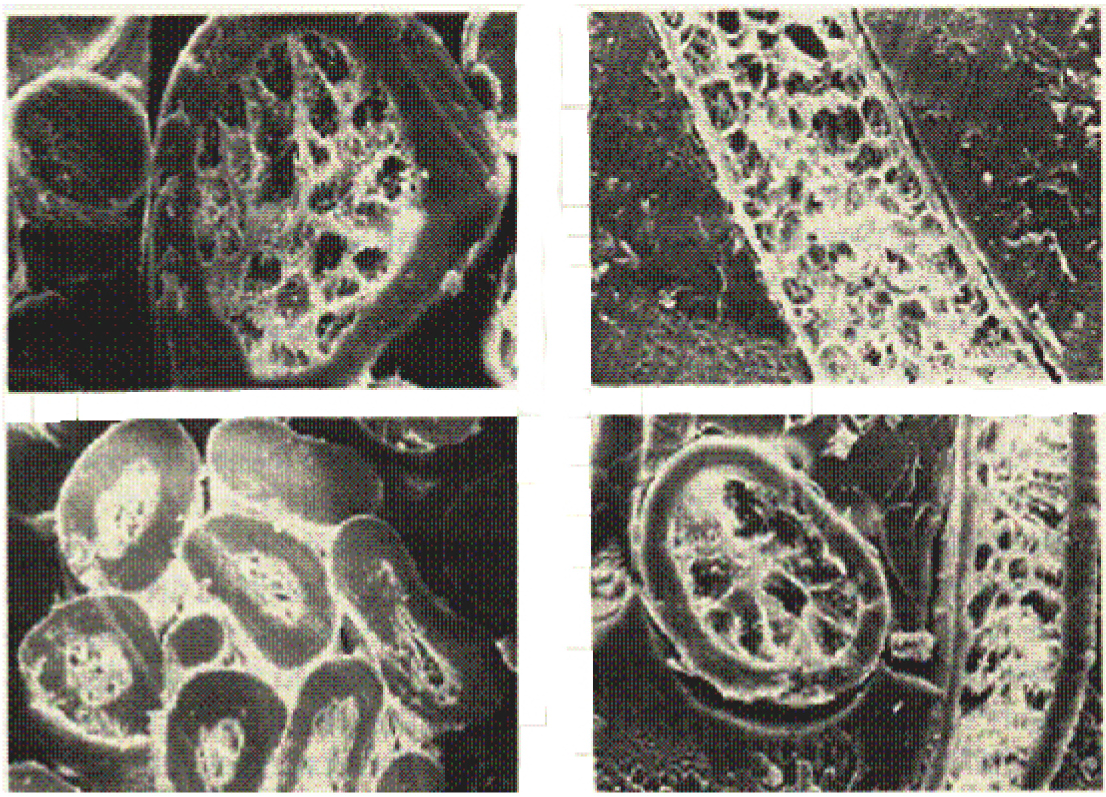

Figure 2. Some cross and longitudinal sections of medullated Mohair illustrating the cellular structure of the medulla (Hunter 1987)

2.3 Classification of Medulla

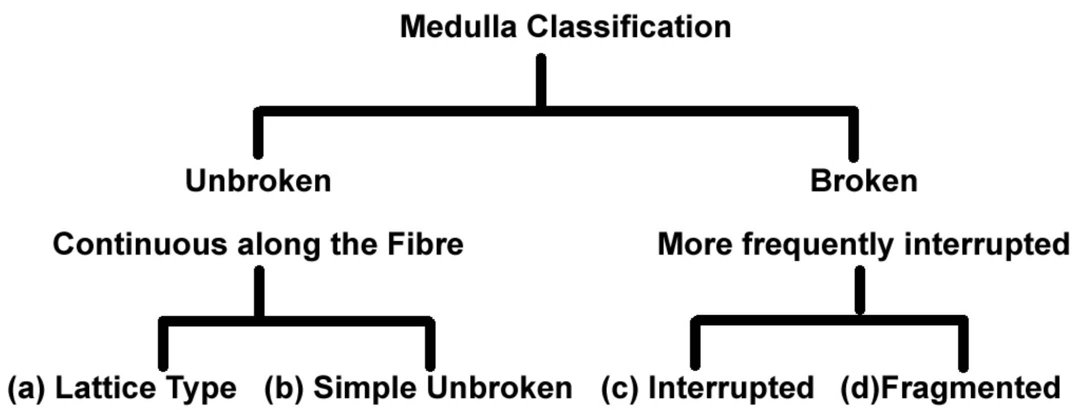

Figure 3. The classification of types of medulla (Wildman, 1954)

The four major classifications of medulla are:

a. Lattice type

This type of medulla is very wide in proportion to the total width of the fibre and consists of a network of keratin 'struts' which outline polyhedral shaped spaces, each of which is continuous with its neighbours. A mixture of gases occupies the spaces within the fibre. When viewed through transmitted light in the microscope lattice medulla appear dark.

The lattice type of medulla is present in many coarse, persistently – growing fibres from sheep (e.g., of some mountain breeds), in coarse kemp fibres, and in coarse outer coat fibres from several mammals, such as reindeer, red deer (Wildman, 1954).

b. Simple unbroken

A simple continuous central canal, thin or thick, but not as thick usually as the lattice type: it appears dark when viewed through transmitted light. This type occurs in a wide variety of animal fibres (Wildman, 1954).

c. Interrupted medulla

Relatively narrow and completely interrupted or bridged at irregular intervals by cortical cells. This type of medulla occurs in many fibres such as those of medium quality, example in Romney Marsh wool (Wildman, 1954).

d. Fragmental Medulla

In this category, the medulla is fleeting, occurring only irregularly as fragments in the centre of the fibre (Wildman, 1954).

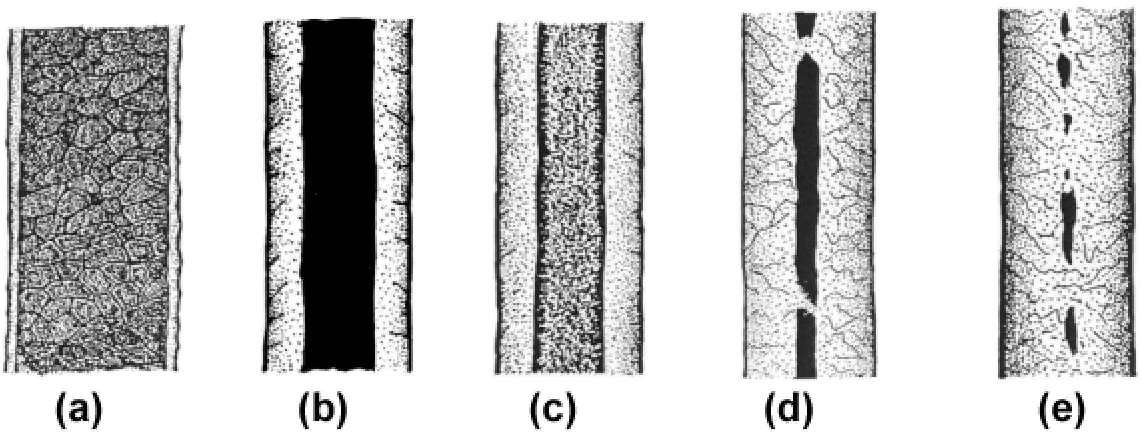

Figure 4. Diagrammatic representation of different types of medulla.

a) Unbroken (wide) lattice. (Usual appearance when not in-filled by mounting medium)

b) Simple unbroken. Medium width. (Usual appearance)

c) Simple unbroken. Medium width. (Appearance when in-filled by mounting medium)

d) Interrupted.

e) Fragmented.

Fundamentally wool fibres can be classified as:

1. non medullated fibres;
2. Medullated fibres; or,
3. Kemp fibres

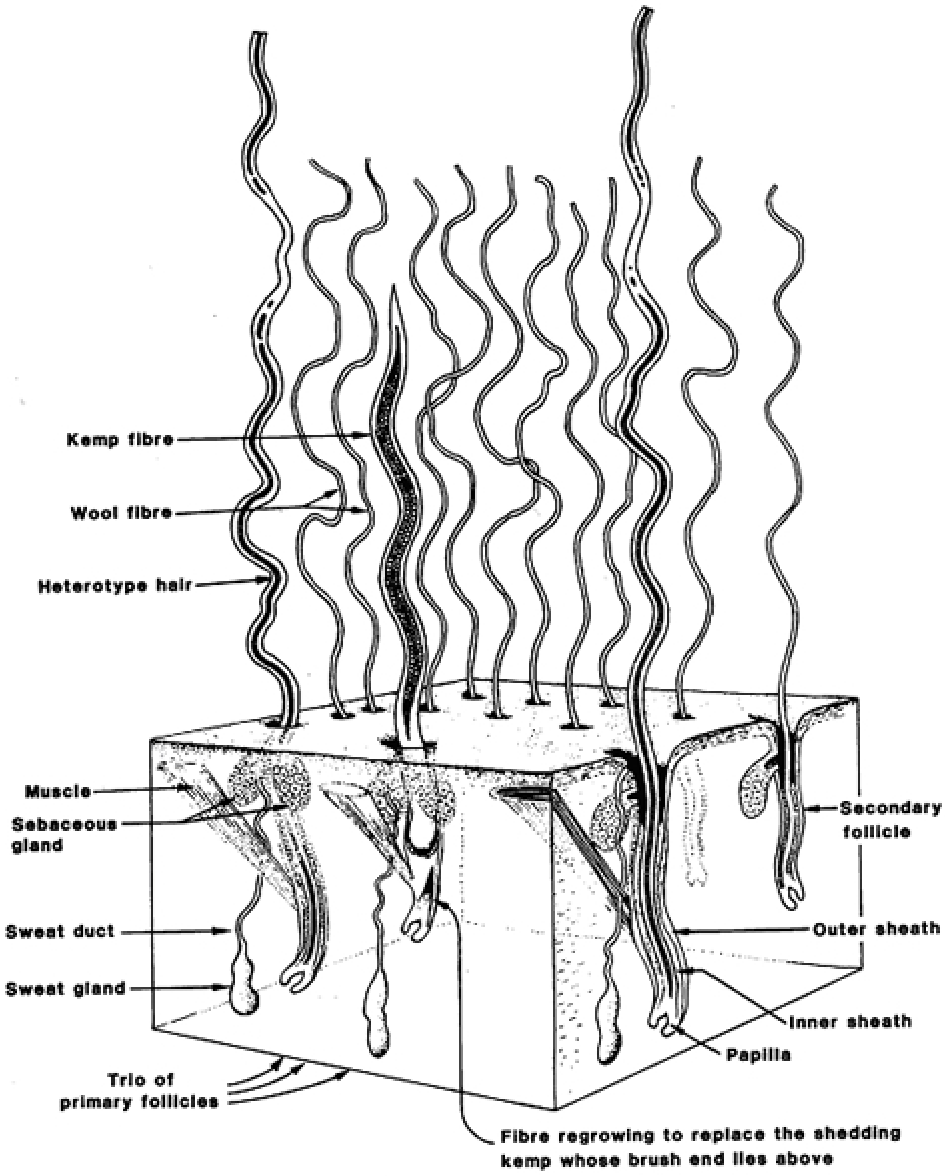

Figure 5. Stereo diagram of an idealized wool follicle group to show the three types of fibre grown and the two types of follicles in the skin (Ryder and Stephenson 1968).

2.4 Classification of visually objectionable fibres.

Medullated fibres are generally chalky white in appearance, even after dyeing, and may be classified as:

- Kemp fibres;
- Highly medullated fibres; or
- Flat fibres.

2.4.1 Characteristic of kemp fibres

Figures 6 and 7 illustrate the Root and Tip of a shed kemp fibre (Wildman, 1954).

Figure 6. Root

Figure 7. Tip

A kemp is quite dissimilar to a “normal” wool fibre. Kemps are not grown continuously from the one follicle. After a period of growth kemp fibres are shed and another kemp fibre may grow from the follicle at a later stage. This growth pattern contrasts with normal Merino fibres, which generally grow continuously. A kemp fibre sheds from the follicle and is scattered throughout the fibres within a staple, often laying transverse to the direction of growth of the “normal” fibres in a staple.

Kemp fibres are flat white or chalky white in colour, tapering towards the tip and, at times, show a bulbous root end (Figure 6). Crimp is not apparent though an open wave or undulation usually appears. Kemp fibres are usually coarser than “normal” wool fibres with which they associate and, almost invariably, are medullated. It is generally considered that kemp fibres do not possess the high degree of elasticity common to “normal” wool fibres. However, this may well be related to the irregularity of fibre diameter and medullation rather than to any chemical differences, which are not conspicuous. The fibre is comparatively “brittle” and lacks wool’s resilience.

The main feature of a kemp fibre is the wide latticed medulla occupying most of the width of the fibre, so that the cortex forms only a narrow ring around the outside.

Kemps are undesirable in good wool not only because of their coarseness, but because the wide medulla makes kemp fibres dye a paler shade than a “normal” wool fibre. This effect is enhanced by the reflection of light from the surface of the medulla, which makes undyed kemps appear chalky white. They are often added to some wool to give “effects” in fancy cloth, and they are an essential characteristic of certain kinds of tweed.

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2.4.2 Normal medullated fibres

Not all normal medullated fibres are problem fibres. These fibres vary in their degree of medullation as well as in their fibre diameter. Some of these medullated fibres stand out as being optically different and therefore objectionable.

2.4.3 Comparison of medullated and kemp fibres

Medullated fibres are intermediate between "normal" non-medullated wool and kemp fibres. Medullated fibres are longer than kemp fibres. Medullated fibres shed less frequently than kemp fibres. Kemp fibres that have moulted into the fleece have a quite different root end known as a brush. Kemp fibres have a pointed tip because they grow to a moderate length before being shed. Fibres from a normal clipped fleece would be expected to have two cut ends, whereas the skin end may have a fairly clean cut, the other end which was the cut previous year and is now at the tip of the fibre, is usually worn and frayed.

2.4.4 Flat fibres

Fibres, which look like a flat ribbon and are generally chalky white in appearance even after dyeing may be classified as flat fibres.

Highly medullated fibres or kemp fibres where the medulla diameter is almost the same as the outside diameter have very thin walls that will collapse to a flat ribbon (Baxter et al 1991). Flat fibres appear paler than the normal fibres in the dyed fabrics (Mahar, Pers.com). The optical characteristics of flat fibres are completely different from ordinary kemp or other highly medullated fibres. Hunter (1987) also found certain thin walled medullated fibres have a flattened (Bean shaped or ribbon like) appearance but he explains this could have happened during processing or in handling of fibres.

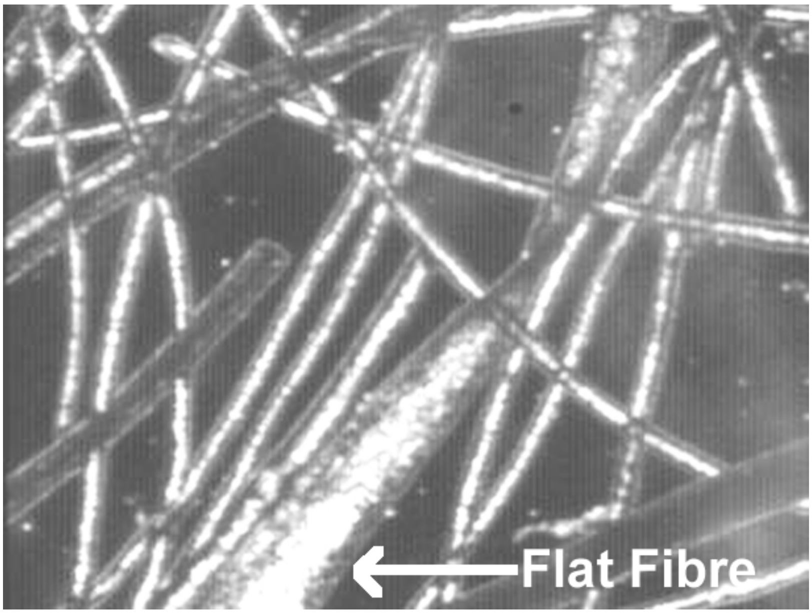


Figure 8. Flat fibre (www.OFDA.com, 2004)

It is important to quantify the loss of these different types of medullated fibres in worsted processing to estimate the actual risk of medullated fibre contamination in worsted fabrics.

Work done by AWTA Ltd (Table 3) shows that most contaminating fibres from a contaminated fabric are highly medullated and have a flat ribbon-like appearance. Further studies are needed to confirm this and AWI is currently surveying the wool processing industry to recover fabric samples contaminated with medullated fibres (Project EC596).

3. Medullated Fibre Contamination in Australian Merino Wool

As previously noted, medullation is unusual in the Merino, only occurring rarely in very coarse fibres. Specific subtypes of Merino sheep, including the Australian superfine, Dhone and the South African Meat Merino (SAMM), have low pigmented and medullated fibre risk (Hansford 2003). But some studies have found fibre shedding in Merinos in response to changes in nutrition (Doney, 1966; Fraser and Short 1960, Lang1964; Ryder 1967, cited Hatcher 2002).

Wool producers in some areas in Australia have introduced “exotic” sheep (e.g. Damara) to diversify their commercial enterprise to include meat production. In order to quickly build up sheep numbers, rams of these exotic sheep are being crossed with merino ewes (Fleet et al 2002). Cross breeding and running crossbred lambs with Merino ewes may facilitate the increase in the incidence of objectionable dark and/or medullated fibres in Merino wool. The increasing incidence of kemp fibre in Australian wool in recent years is shown in Table 1.

Table 1. Percentage of auction sold wool appraised as containing kemp fibre (Source: Fleet et al, 2002)

Region	1990/2000	2000/2001	2001/2002
Western	0.02	0.17	0.42
Northern	0.01	0.02	0.07
Southern	0.02	0.02	0.03
Total	0.02	0.05	0.12

The common exotic breeds with medullated fibres are Dorper and Damara.

Dorper sheep have a short shedding wool fleece containing “Floating kemp” and was introduced from South Africa during the mid 1990’s (Fleet 2003). The Damara is a fleece shedding sheep breed also imported from South Africa. Damara fleece has a high content of coarse fibre with pronounced medullation (Fleet 2003). Most of the pigmented fibres from Damara sheep are very dark and /or highly medullated and have high fibre diameter averaging, 68µm (Fleet, 2001).

Contamination by medullated fibre can occur in different ways: medullated fibres moved from the non wool growing areas of the Merino sheep, such as face, legs and belly into wool growing areas; or, direct contact between sheep, for example when mating exotic ram breeds to merino ewes, and merino ewes raising cross bred lambs between birth and weaning (Hatcher2002). Also contact with common areas, e.g. shearing yards where fibres from exotics can shed onto races, fences, gates, on the ground etc, and can be subsequently picked up by Merino sheep.

The Federation of Australian Wool Organisations (FAWO) initiated a program to assist in the protection of Australia’s reputation as a supplier of premium Merino white wool by managing the risk of dark and medullated fibre contamination. The aim of this program is to provide buyers and processors with a reliable and quantified measure of the risk in dark and medullated fibre in sale lots. This is achieved by assigning a dark and medullated risks rating from 1 (best) to 6 (worst) to each sale lot. Ratings 1 and 2 are suitable for white or pastel shade end products. The risk rating is derived from the information provided by the growers, such as if their stock have been in contact with exotics, if crutched, if crutched within 3 months of shearing, age, sex, wool description as shown in figure 9.

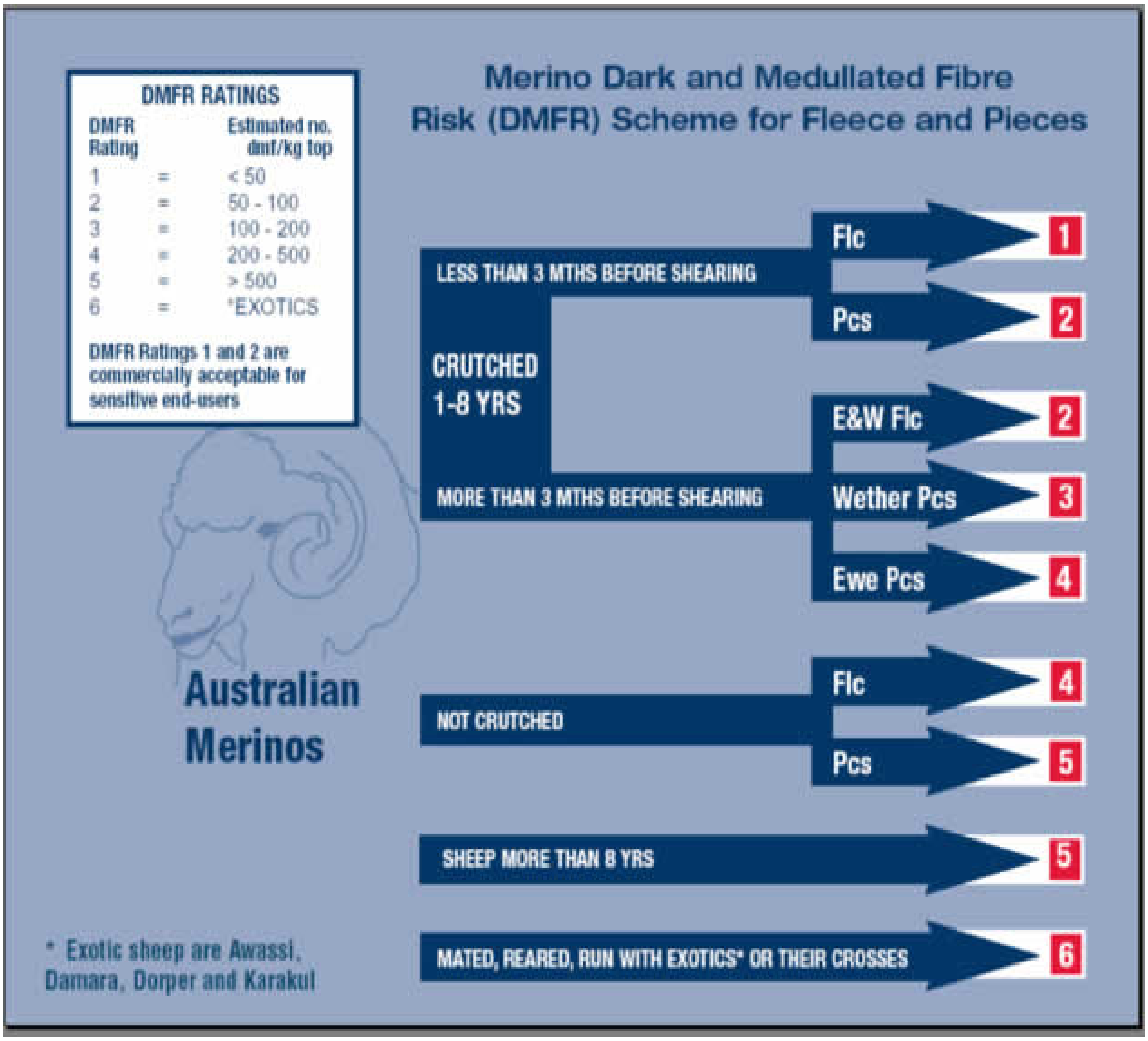


Figure 9. Merino Dark and Medullated Fibre Risk Scheme for Fleece and Pieces

The DMF Risk scheme only covers Merino Fleece and Pieces wool from farm lots. All other wool types are not included and hence a risk rating cannot be provided to the markets. Recent findings and concern about wool contamination arising in merino crossbreeding with exotic breeds have shown the importance of a presale test to provide buyers with confidence about these risks in several Merino cross breeding situations.

In cases where the DMF risk scheme is not applicable Medullated and Dark fibre testing provides an alternative method for indicating risk of contamination.

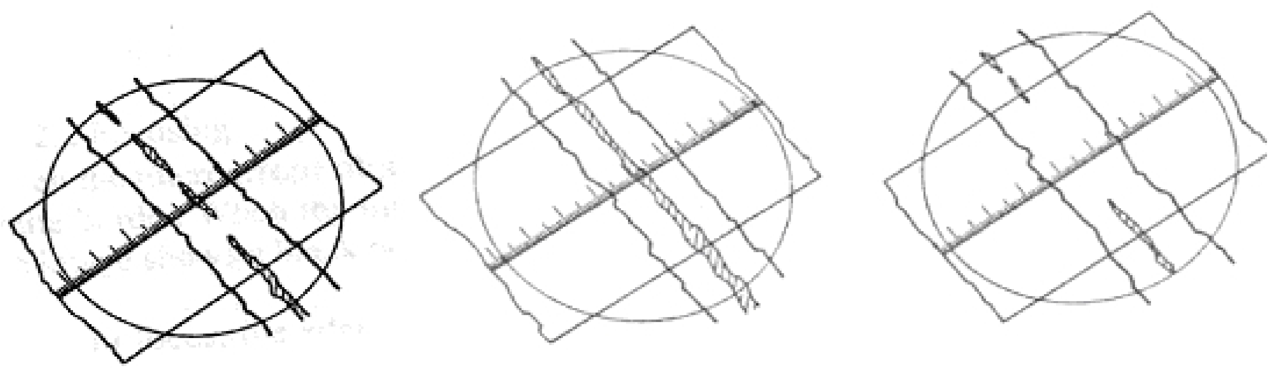
4. Test Methods for Measurement of Medullation.

Because of the low concentration of medullated fibres in Australian merino wool a significant amount of sample needs to be measured to provide a reliable measurement. Most of the methods except WRONZ Medullameter and Near Infrared Reflectance analysis, involved in the measurements are time consuming and labour intensive. Therefore it is impracticable to measure large amounts of sample in a commercial test. Also it is important to measure or differentiate the objectionable medullated fibres from other medullated fibres. Not all the available methods differentiate the different type of medullated fibres.

It is appropriate to examine each method, its associated particular technology, and its definitions of the objectionable medullated fibres, in order to assess the test methods for application to presale testing of Australian wool sale lots.

4.1 Microscopic counting method

Microscopic counting is a direct method of medullation measurement. It is possible to measure different types of medullation and record the medulla diameter distribution by using this method. This is the internationally accepted medullation reference method for wool and is also accepted for certification purposes (IWTO-8-97, 1989). The problem with this method is that it is extremely slow and operator intensive. The diagrams in Figure 10 show the fibre images, and rules for counting, of the different types of fibre medulla using projection microscope (IWTO-8-97, 1989).



Narrow medulla   Continuous Medulla   No medulla

Figure 10. Fibre images of different types of medulla during projection microscope measurement (IWTO-8-97, 1989).

4.1.1 Standard test definitions for medullated fibres in wool and other animal fibres by micro projection (ASTM Standard D2968-89, 1989).

- Medullated fibre – an animal fibre that in its original state includes a medulla
- Med Fibre – a medullated animal fibre in which the diameter of the medulla is less than 60% of the diameter of the fibre.
- Kemp fibre (Objectionable Fibre) - a medullated animal fibre in which the diameter of the medulla is 60%, or more, of the diameter of the fibre.

ASTM standard definitions have been commonly used in different methods of measuring medullation

4.2 WRONZ Medullameter

The WRONZ Medullameter provides a rapid method for measuring medullation in wool. The principle of measurement is that the medulla is exposed by rendering the surrounding fibre invisible. This is achieved by immersing the wool in a mixture of Benzol Alcohol and oil of aniseed, a mixture whose refractive index is equal or very close to that of wool (Lee 1999). When wool is immersed in a medium whose refractive index is equal to that of wool there is no reflection of light at the medium/wool interface. The medulla is substantially an air filled void, with efficient light reflectance at the keratin/medulla interface. The amount of light reflected from a sample of wool fibres is related to the total or overall degree of medullation present in the sample. In the Medullameter, the sample is illuminated by a diffuse light source and the amount of light refracted and transmitted is measured by a photoelectric detector. A single millivolt output, indicating the level of light detected, is produced. This value is transformed mathematically to produce a measurement of the degree of medullation (Figure 11, Lappage & Bedford, 1983).

The degree of overall medullation is expressed as an index, which can be calibrated against a measurement made by projection microscope. As such it is an indirect method of measuring the level of medullation in a sample. The measurement of medullation represents the total medullation present in the specimen of wool. This instrument does not distinguish between a large number of finely medullated fibres and a small number of heavily medullated fibres and also requires frequent calibration (Lappage & Bedford, 1983).

But it has been shown that the Medullameter reading is mainly a measure of fibres with med ratio >0.6, other medullated fibres with a med ratio <0.6 apparently making only a small contribution to the total Medullameter reading (Hunter et. al 1983).

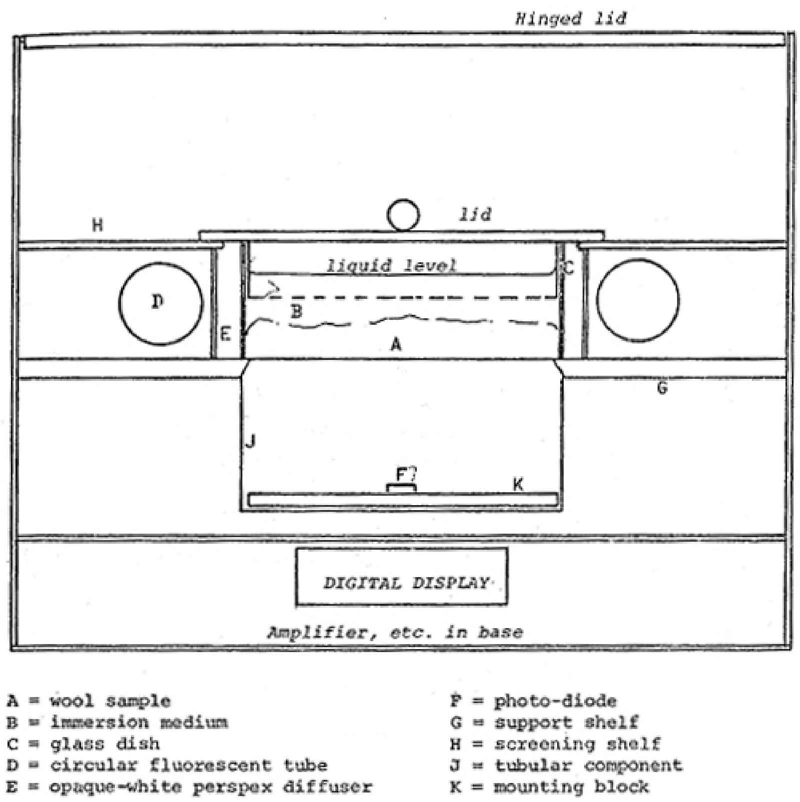


Figure 11. Diagram of WRONZ Medullameter (Lappage and Bedford 1983)

4.3 Near Infrared Reflectance Spectroscopy (NIRS)

The NIRS is an empirical method, which uses statistical inference to calibrate the reflections of selected near infra-red wavelengths across a spectrum using samples with known levels of medullation in the sample. The matching of wavelengths to particular levels of medullation was "made purely on statistical performance"(Ranford & Hammersely 1991 cited Lee 1999). Calibrated NIRS instruments can then be used to measure the level of medullation of wool within the calibration range (Lee 1999). Research work has been carried out in several laboratories on using NIR analysis for predicting wool base, fibre diameter, colour and chemical residuals, but application of these measurements has been restricted to quality control purposes in a small number of commercial scours (Ellery et al.2000 cited Baxter, 2002), rather than being used as a basis for trading.

4.4 Measurement of medullation using OFDA

The OFDA 100 (Brims and Peterson 1994) measures the "opacity" of individual fibres and this is related to the medullation. Fibre Opacity is defined as the ability of a fibre to transmit light perpendicular to fibre length. Fibre opacity is calculated by summing the light transmitted by the fibre in dark field mode, normalised by dividing by the fibre diameter. (For the purposes of providing a base for calibration, a fibre is 0% opaque when the amount of light transmitted by the fibre is the same as that transmitted by a glass fibre of the same diameter.) This normalised number is converted to an Opacity % by calculating its ratio to a calibrated sum for a glass fibre. 80% Opacity is generally regarded as representative of the normal medullated fibre population. Medullated fibres that have collapsed, i.e., flat fibres, may have lower opacity values (Brims and Peterson 1994).

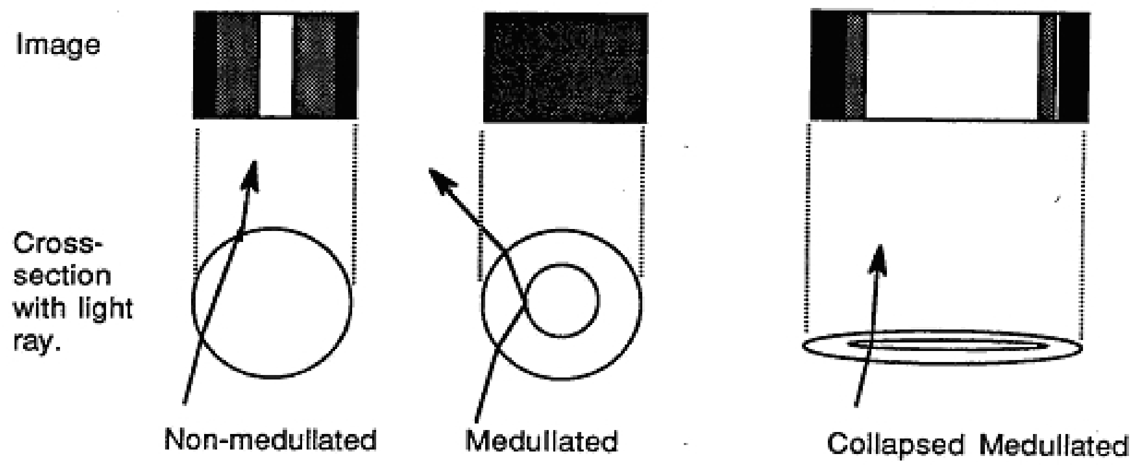


Figure 12. Fibre image under dark field illumination (Brims and Peterson 1994).

Figure 12 shows the three cases of fibre cross-section under dark field illumination. And it explains that the greater the ratio of the medulla diameter to the outside diameter, the greater the opacity due to the internal reflection from the medulla. The reflection from the medulla is what produces the "chalky ", non-lustrous, appearance. A fibre where the medulla diameter is almost the same as the fibre diameter will have a very thin wall and will collapse to a flat ribbon. The flat surfaces allow the light through, but the width of the central light band is greater than that of a non–medullated fibre (Brims and Peterson 1994). This allows the OFDA software to divide fibres into 3 classes depending on their dark field image, as follows:

Medullated: opacity > medullation threshold (80%used here);

Non-medullated: (opacity<medullation threshold) And (light width<40µm); or,

Flat: (opacity<medullation threshold) and (light width>40µm).

Figures 13 and 14 illustrate the opacity histograms for a cashmere and a mohair sample (Brims and Peterson 1994).

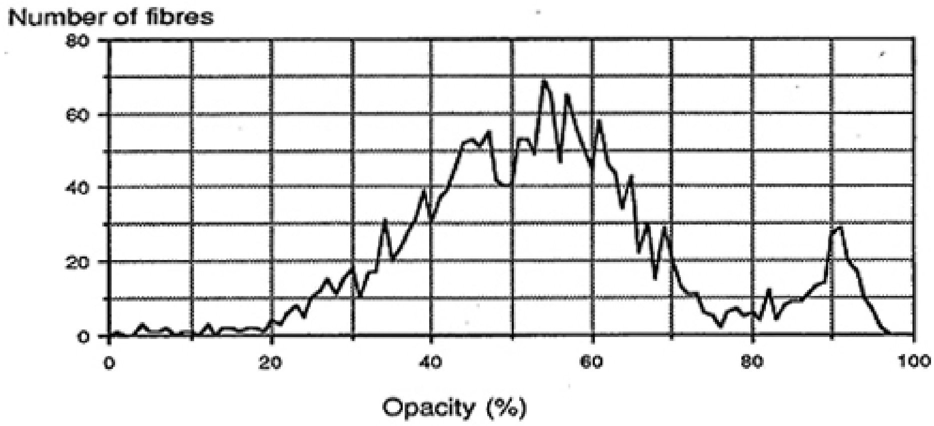


Figure 13 Opacity Histogram for a highly medullated Mohair sample

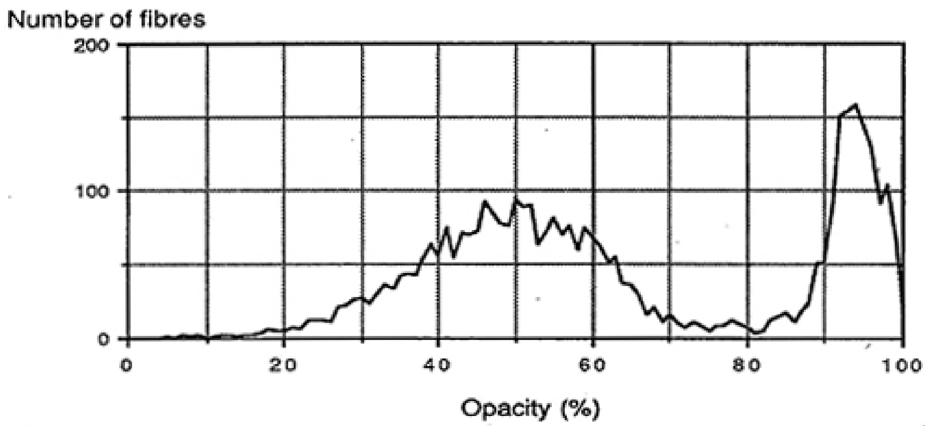


Figure 14 Opacity Histogram for a 10% Yield Cashmere sample.

Typically, a histogram is produced ranging from zero to 100 percent opacity, but containing two distinct populations with two distinct peaks. One population commences at a value approximately 80% opacity and is generally regarded as representative of the normal medullated fibre population (IWTO –57-98). The opacity histograms (Figure 13 and 14) clearly illustrate the existence of two populations of fibres.

Further (Turpie, D.W.F, 1995) studies showed that the number of medullated fibres at and above 94% opacity has also been shown to correlate extremely well with projection microscope results for objectionable medullated fibres from the same laboratory, also using ASTM definitions of medullated fibres. Therefore the threshold recommended for the determination of objectionable medullated fibres is now 94% for both wool and mohair (IWTO –57-98). Brims and Paterson (1994) explained that animal fibres that are collapse are generally much greater than 40µm, and in their work flat fibres less than 40µm ribbon width were not detected.

4.4.1 IWTO–57-98 Standard Definitions for OFDA measurements of medullation

Flat medullated fibres:	Fibres with Opacity <80% that produce a wide light band (>40µm) under dark field illumination and have diameter>60µm
Objectionable medullated fibres:	Fibres with Opacity > 94% and diameter>25µm
Normal medullated fibre:	Fibres with opacity ≤ 80%
Total medullation	Sum of normal medullated and flat medullated fibres usually expressed per 10000 snippets counted or % by number.

4.5 Original AWTA Ltd method for medullated fibre measurement of core samples.

In 2001, research was under taken by AWTA Ltd and the South Australian Research and Development Institute (SARDI) on contamination of Merino wool caused by contact with one breed of exotic sheep, the Damara (Sommerville, 2004, Mahar *et al, 2001*). A key outcome of this research was that contamination from this source is detectable in the core samples routinely used for Yield and Micron testing, thus allowing a test to be developed for woolgrowers. In the early 1980's CSIRO developed an instrument, the Dark Fibre Detector (Figure 15), which is used for detecting the level of Dark (sometimes Medullated) fibre contamination in wool top.

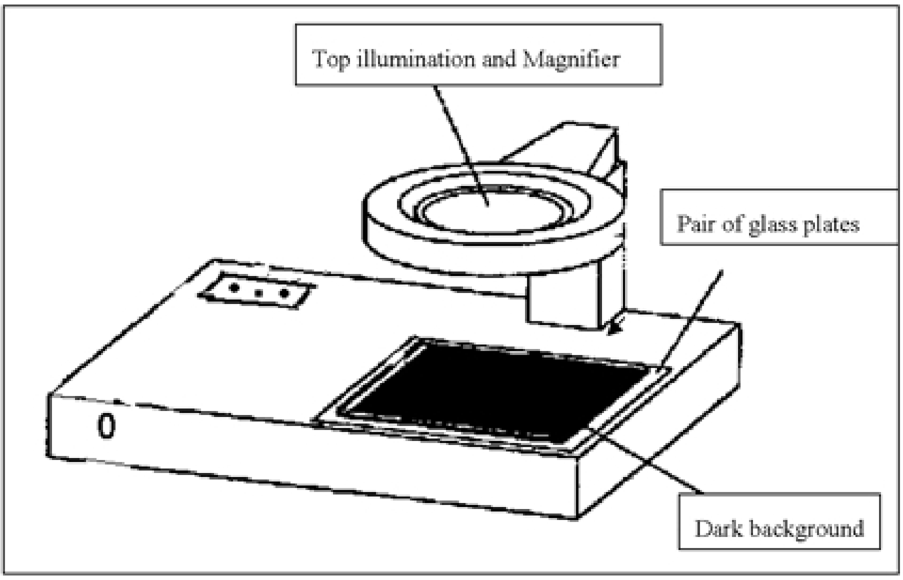


Figure 15. Modified CSIRO Dark Fibre Detector for medullation measurement

A test specimen of approximately 0.2 g is spread evenly on a glass plate to a density of about 10g/m², then covered with a second glass plate without disturbing the fibres in the web. The folded glass slide is placed on a dark background and inspected in the sequent as indicated in Figure 16. Wool is opened mechanically, medullated fibre faults detected and counted. The specimen is examined using 2 x magnification when illuminated by white light. This method is based on visual detection and comparison of the brightness of the detected medullated fibre to that of medullated fibre No. (3) of the AWTA Ltd medullated fibre reference web.

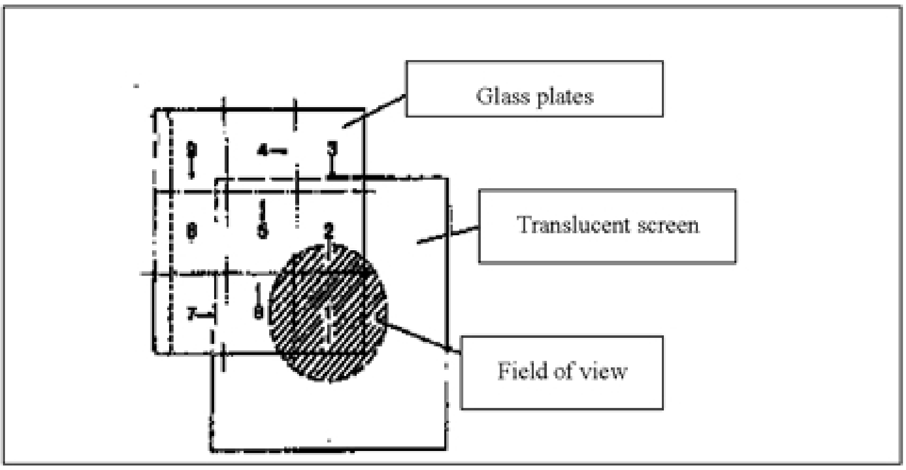


Figure 16. Measurement of glass plates

4.5.1 Definition of the threshold detection in the original AWTA Ltd method

The ASTM D2968-89 definition of an objectionable fibre was used in this trial. This definition is " a medullated animal fibre in which the diameter of the medulla is 60%, or more, of the diameter of the fibre".

4.5.2 Reference web for medullated fibre measurements

The Research and Development Division of AWTA Ltd developed a reference web to quantify the commercial occurrence of medullated fibre contamination (Mahar, Pers.com).

The Medullated fibre reference web consisted of 5 medullated fibres and a thin strip of nylon bale filament. Fibres with brightness equal to or less than fibre 3 were considered as contaminant medullated fibres.

Table 2: Reference web for medullated fibre measurements

Scale	Description
1	Diameter of the medulla is 80-99.5% of the diameter of the fibre
2	Diameter of the medulla is 70-80% of the diameter of the fibre
3	Diameter of the medulla is 60-70% of the diameter of the fibre
4	Diameter of the medulla is 50-60% of the diameter of the fibre
5	Diameter of the medulla is less than 40% of the diameter of the fibre
Pack	White nylon filament from a wool pack

For the AWTA Ltd screening test, the results for dark and medullated fibres are currently reported as three categories:

Low - 0-5 fibres /10g;

Medium - 5-10 fibres/10g and;

High - >10 fibres /10g sample.

4.6 AWTA Ltd Benzyl Alchohol Test Method

From, July 2004, AWTA Ltd introduced a new method for medullation measurement based on research by CSIRO (AWTA et al. 2004), which was funded by Australian Wool Innovation.

4.6.1 Principle of this method

When normal white fibres are immersed in a solvent of the same or very similar refractive index they become transparent and very little reflection occurs. Wool fibres are virtually transparent when immersed in benzyl alcohol because it is a clear colourless liquid and its Refractive Index (R:I) is 1.540 very similar to the wool refractive index of wool 1.553 (Faust 1956). But medullated fibres due to their internal medulla continue to reflect incident light so that against a black background they appear white in benzyl alcohol.

4.6.2 Procedure of this method (AWTA, 2004)

1. Four specimens of scoured and carded wool (each of 5 grams) are prepared from the core sample, which has been submitted to AWTA Ltd for Yield & Micron testing. The carding process is specifically designed to present the sample as a thin, carded web with a relatively uniform thickness.

2. The carded webs are placed in specially designed rectangular transparent plastic bags.

3. An experimentally determined volume of benzyl alcohol is added, and the top of the bag is heat-sealed.

4. The sample is immersed in the solvent by placing the bag on an inclined surface and squeezing the bag between two rollers as the bag moves down this surface. All the air and any excess solvent expelled to the top of the bag. Another heat seal is applied separating the bag into two sections:

    a. The upper section (approximately 25%) contains the expelled air and excess solvent.

    b. The lower section contains the wool fibres totally immersed in the solvent in a thin uniform layer.

5. The lower section of the bag is placed on a modified Dark Fibre Detector. These modifications include an x-y stage (the area of sample to be examined is larger than the area of the glass slides used in the previous system) and a simple device for placing a black background under the sample.

6. The same procedure as used previously is then followed to examine the specimen for dark and medullated fibre contamination.

This method is also based on visual detection; once detected, the brightness of the medullated fibre is compared with the medullated fibre from the AWTA Ltd Medullated Fibre Reference Web (see Figure 17).

4.6.3 Definition of the threshold detection in the AWTA Ltd, benzyl test method

The specification of the Reference fibre for the benzyl alcohol method is: – a medullated animal fibre in which the diameter of the medulla is 60% of the diameter of the fibre and the diameter of the fibre is 50μm.

The results for dark and medullated fibres are currently reported as three categories:

Low - 0-10 fibres /10g,

Medium - 10-20 fibres/10g and

High - >20 fibres per 10g sample.

Figure 17. Modified dark fibre detector for AWTA benzyl test method

4.7 Comparison of existing medullation measurement methods.

The projection microscope method is a direct method of measuring medullation but, is unsuitable for extensive use in both commercial and research due to the cost involved in measuring a large number of samples. The average time taken by two operators to measure 500 fibre sites is approximately two hours and the method relies on a considerable amount of interpretation by operators (Edmunds, pers.com., Ranford, pers.com., cited Lee 1999).

The WRONZ Medullameter and Near Infrared techniques are rapid indirect methods. They count/measure all medullated fibres, not just those fibres which are regarded as contaminants. These methods are still not commercially acceptable for high volume testing. They need microscope measurement results for calibration and they do not differentiate between the different types of medullation.

The OFDA differentiates the different types of contaminating fibres, but is not a direct method of detecting medullation and the sample size for each measurement is very small (10,000 snippets in each measurement). Hansford (2003) in her review noted that the OFDA100 is not commonly used in Australia for the measurement of medullation and that OFDA100 overestimates the medullation in Merino wool.

The AWTA Ltd methods are commercially available but the original method is labour intensive and time consuming and is thus extremely costly. Due to the small size of specimen (0.25g-0.5g) that can be examined at one time, several (20-40) such specimens must be examined to achieve the level of sensitivity required.

Compared to the other methods, the new AWTA Ltd benzol test method is commercially acceptable because it tests a large size of sample (20g) relatively quickly. The medullation of several hundred thousand fibres is seen at a glance and measure only fibres that are considered to be a cause of contamination.

Even though several methods are available, all methods depend on the ASTM definition of the threshold level contamination to calibrate or evaluate their measurements. Currently there is no research, which establishes that the ASTM D2968–89 definition of a threshold level for medullation contamination is very suitable for merino wool.

5 Measurements on medullated fibres

Smuts *et.al*. 1983 visually graded the medullated fibres as kemp (all fibres having a milky or chalky appearance when viewed in air against a black background) or Med fibres (those which appeared obviously “milky” in benzyl alcohol when viewed under incandescent light after all the kemp fibres had been removed) and examined these medullated fibres with a microscope. Their work explained (See figure 18[1]) that kemp and med fibres are generally coarser than the non-medullated fibres in a sample. Most of the fibres containing broken and fragmented medullas have a med ratio below 0.5. The fibres containing “lattice“ type medulla have a med ratio above 0.5. There is no relationship between the unbroken type of medullated fibres and the med ratio of those fibres` and the diameters of the unbroken medullated fibres, which are defined as kemps ranged from 35μm to 200μm.

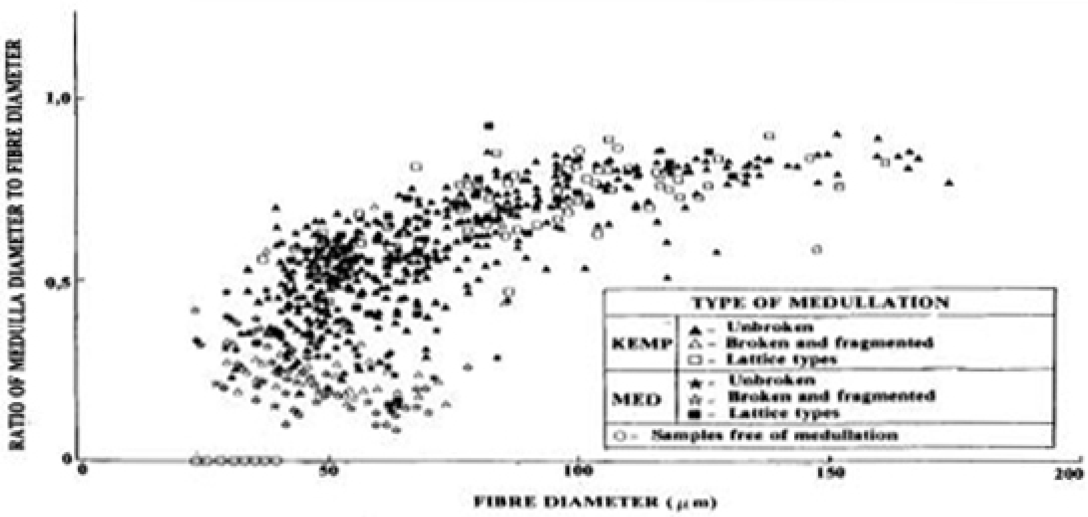


Figure 18. The relationship between the medulla diameter to fibre diameter ratio and fibre diameter for all medullated fibres.

This work has also determined that there is a relationship between medulla diameter and fibre diameter for kemp fibres as shown in Figure 19. Generally fibre diameter increases with medulla diameter for kemp fibres. Kemp fibres, have a coarse medulla and coarser than average fibre diameter within a wool sample. For fibres with a broken medulla, the medulla diameter is less than 25μm.

Fibre distribution curves shown in Figure 20 illustrate that there is considerable overlap between the medulla to diameter ratio distribution curves for kemp and med fibres. The overlapping distribution curves for kemp and med fibres explain one of the difficulties of objectively defining medullated fibres as visually objectionable or not. The Mean Fibre Diameter and mean medulla diameter of the kemp fibres were in the range of approximately 45μm - 55μm and 25μm-35μm respectively.

[1] Figures 18, 19, and 20 represent the best images obtain from the available texts

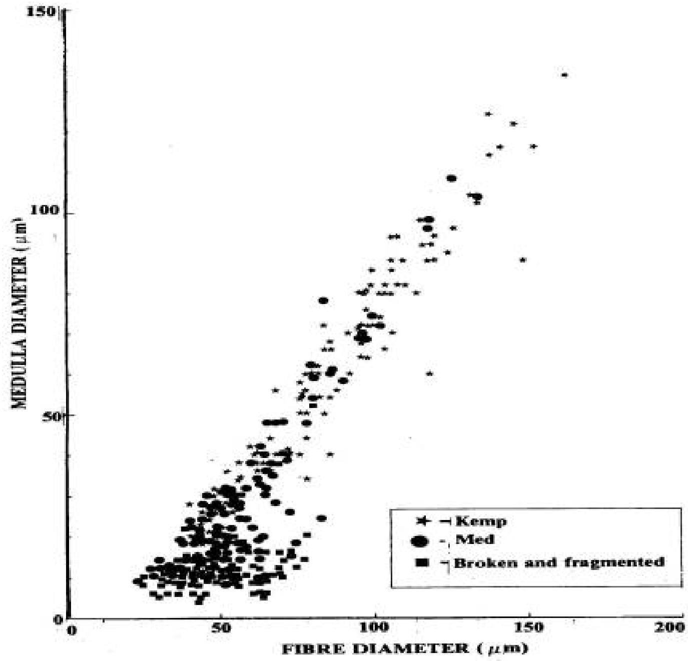
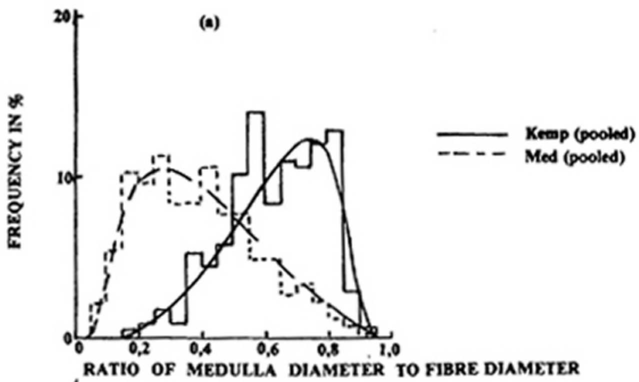
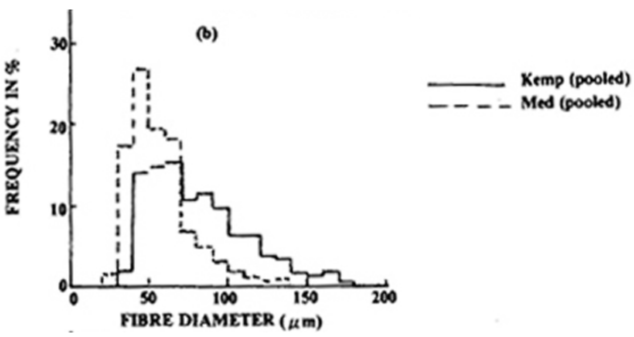


Figure 19. Relationship between medulla diameter and fibre diameter for medullated fibres. (Source – Smuts and Hunter 1983)





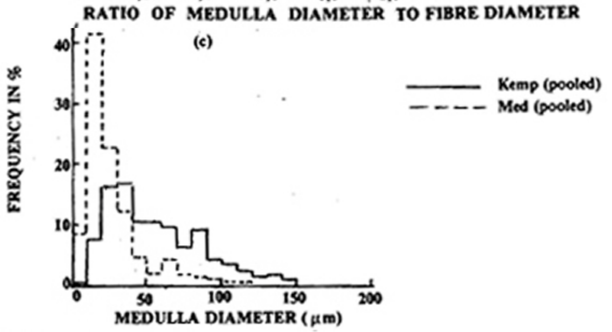


Figure 20: Some distribution curves for various properties of medullated fibres (Source – Smuts and Hunter 1983)

Hunter *et. al.* (1996) investigated wool in order to define objectionable fibres (adjudged visually objectionable against the background of the parent fibres by combing mill staff experienced in such work). The diameter distributions shown in Figure 21[2] for normal and objectionable wool fibres demonstrate that the minimum

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[2] Figures 21,22, 23 and 24 represent the best images obtain from the available texts

diameter of the objectionable fibres was 35 µm, and the Mean Fibre Diameter of the kemp fibre population was approximately 75µm - 85µm.

The med ratio distribution curve in Figure 22 shows that a significant proportion of fibres (2.7%), which have zero medulla ratios, still have a chalky appearance in air. The assessment of these fibres as objectionable may be due to their coarse diameter and this result indicates the uncertainty about the definition of objectionable fibres using visual observation in air.

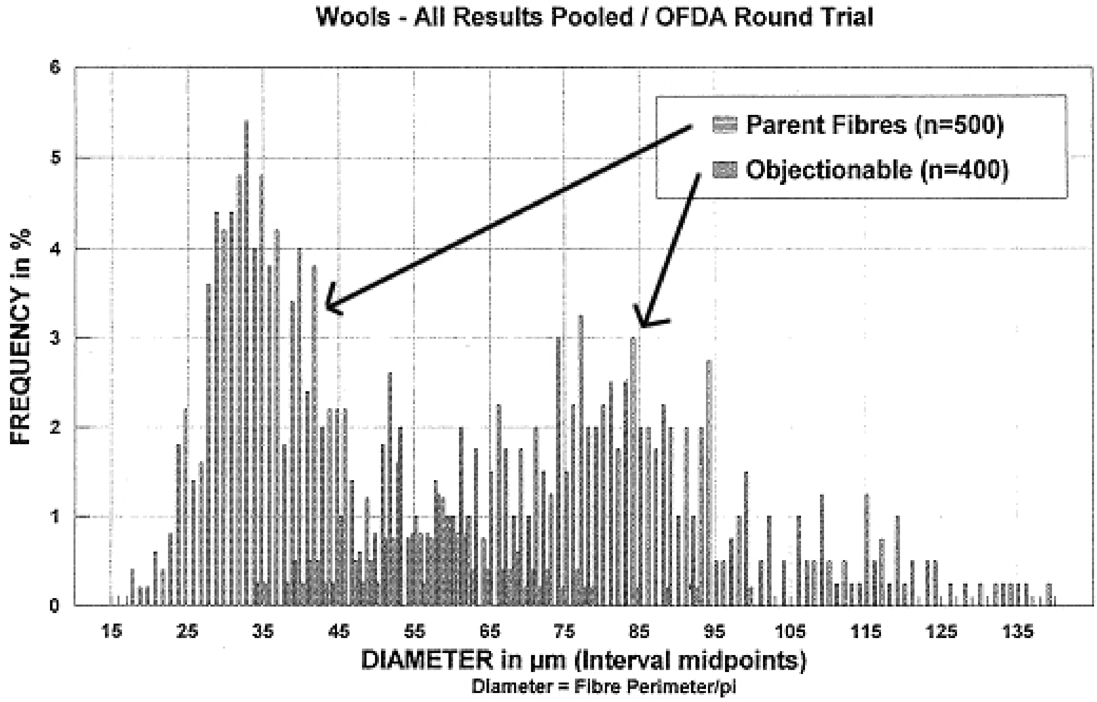

Figure 21. Diameter distribution of wool fibres (Source – Hunter *et.al*. 1996).

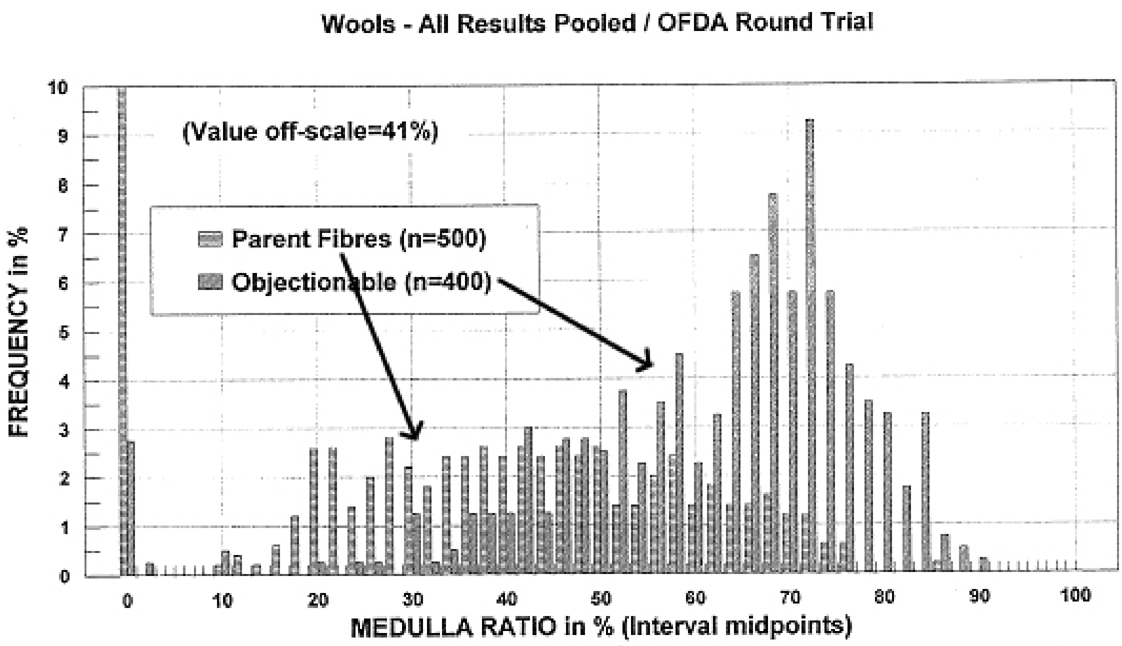

Figure 22. Medulla ratio distributions of wool fibres (Source – Hunter *et.al*. 1996).

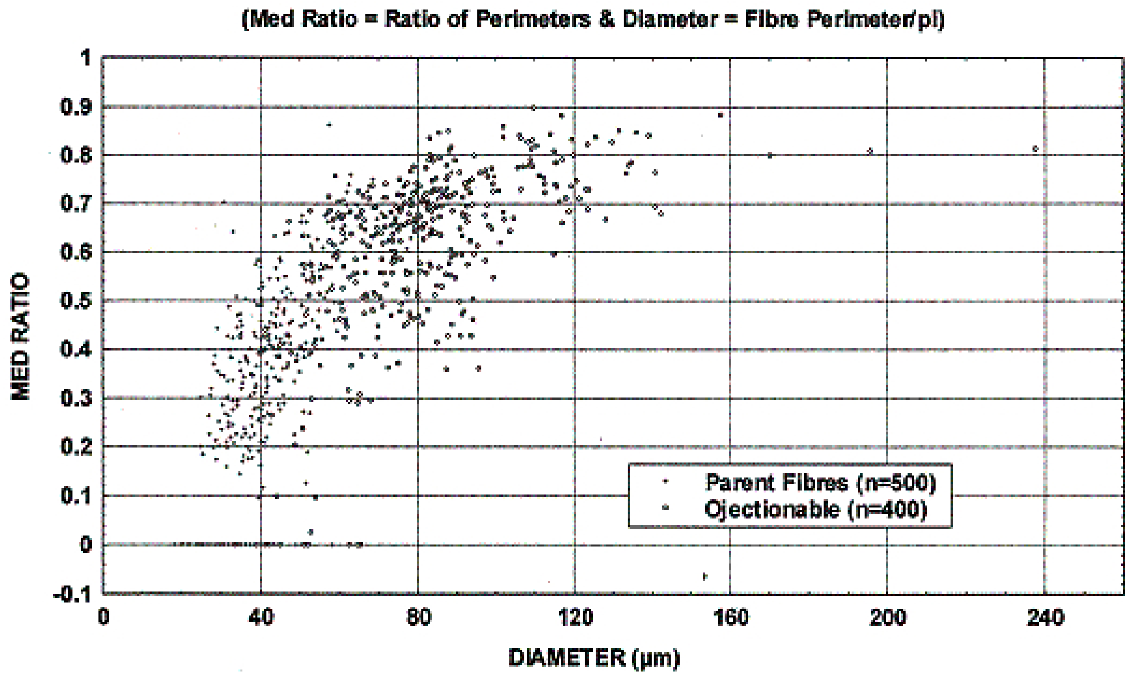

Figure 23. Illustrates the relationship between med ratio and the diameter of the objectionable fibre and parent wool fibres. (Source – Hunter *et.al*. 1996)

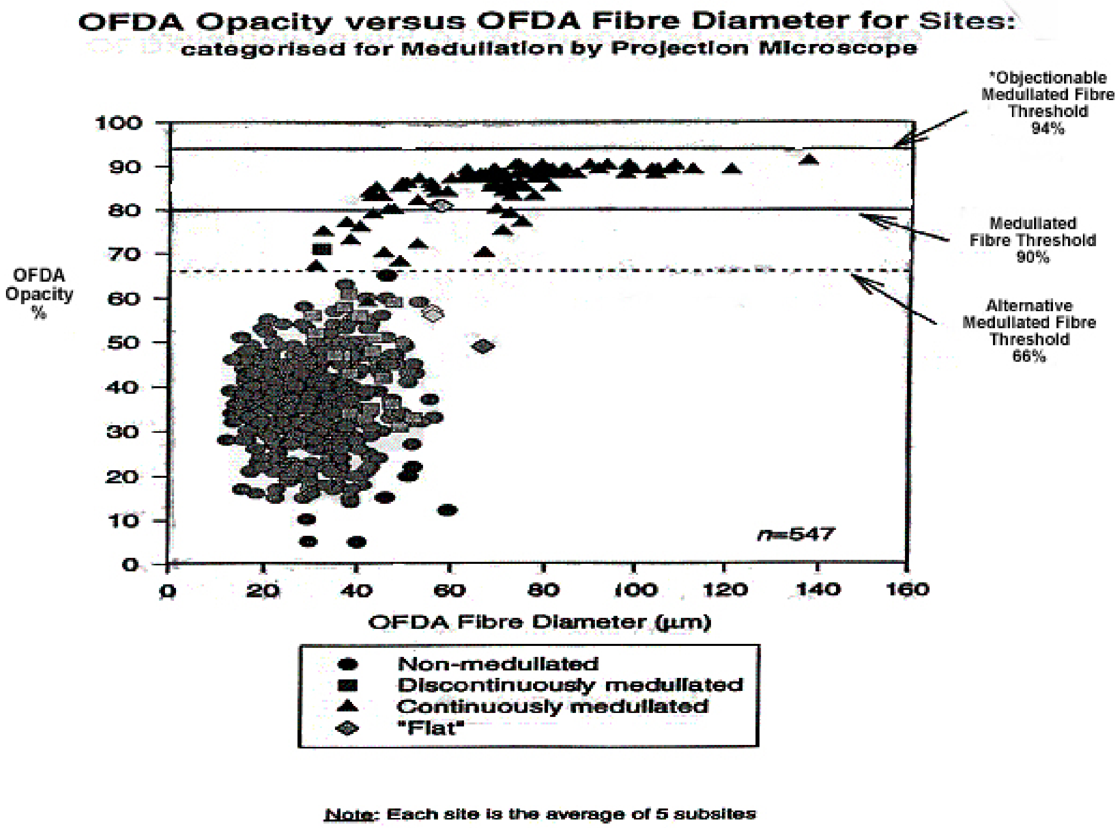

Figure 24. OFDA Opacity versus OFDA fibre diameter. (Source –Lee 1996)

Comparative measurements for medullation were made at the same sites on individual fibres using OFDA and Projection Microscope (Lee 1996). These data were used to investigate several aspects of the relationship between OFDA Opacity and medullation. This work presented in Figure 24 and shows that the minimum diameter of the medullated fibres which have an opacity > 80% is approximately 40 μm and for medullated fibres which have an opacity greater than 94% the minimum diameter is greater than 140μm. But there were no fibres above the 94% threshold identified in this study.

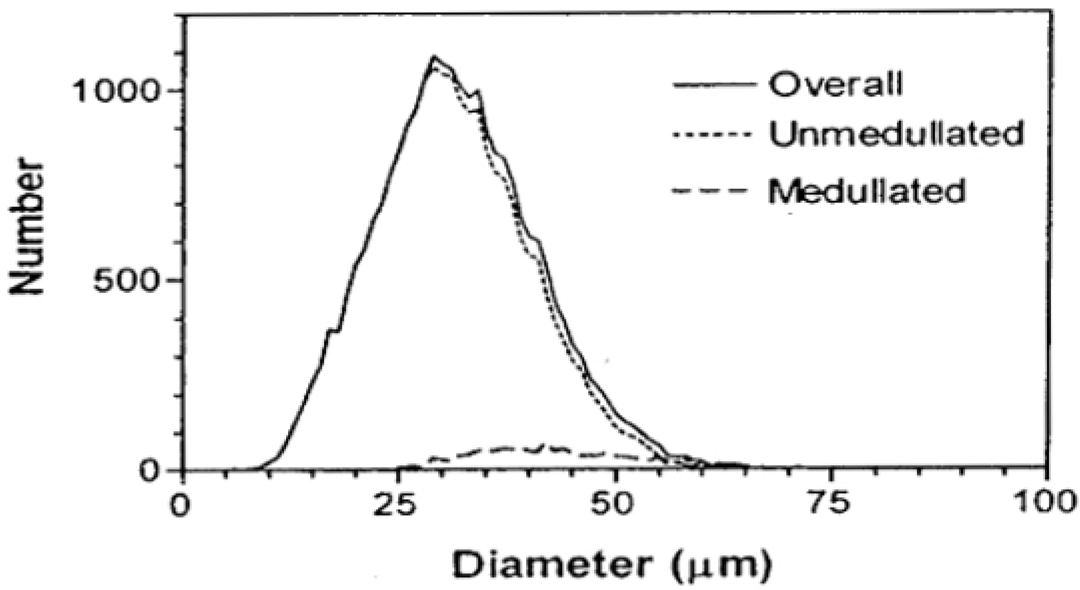

Figure 25: Diameter distribution of medullated and unmedullated fibres for a typical wool. (Source –Glass 2000)

Glass (2000) measured some wool samples of medullated wool fibres by OFDA and reported fibre diameter distribution for medullated and unmedullated fibres as shown in figure 25. The mean and standard deviation of the medullated fibre distribution were 31.7 μm and 9.2 μm, 42.96μm and 9.81 μm respectively. This trial confirms that most medullated fibres are to be found at coarser diameters.

Work was attempted by Hunter (1987) to establish what actually distinguishes objectionable medullated fibres from other medullated fibres on the most common South African mohair. The samples examined individually using projection microscope showed that the medulla diameter to fibre diameter ratio appeared to hold some promise for discriminating between the various groups of medullated fibres over the entire range of mohair diameters, but did not consistently distinguish between the various categories of medullation. The more obvious objectionable medullated fibres, however, generally had ratios above 0.5, while medullated fibres, which do not appear different in air, mostly had ratios below 0.5. A mean ratio in the range 0.5 or 0.55 could therefore be used an approximate criterion for discriminating between medullated fibres, which differ in appearance from other less apparent medullated fibres.

Six contaminant wool fibres from a commercially produced, dark fabric were extracted and examined microscopically and measured using at AWTA Ltd. A summary of the results is shown in Table 3. These results illustrate that very coarse and highly medullated fibres are a major problem in the dark fabrics. However, there is not sufficient data to determine any values for threshold value for medullation contamination.

Table 3. Microscope measurements of some contaminated fibres from a contaminated fabric.

Fibre No	MFD (μm)	Description
1	168.6	Flat 85% medullated
2	173.6	Apparently Flat 96% medullated
3	248	Apparently Flat 80% medullated Medullation varies along length of fibre
4	173.6	Apparently Flat 90% medullated
5	148.8	Flat (Ribbon) 98% medullated
6	173.6	Flat (Ribbon) 98% medullated

Limited information has been obtained from experiments involving the measurement of contaminant medullated fibres on wool. This information shows that medullated fibres are relatively coarser than non-medullated fibres in any natural population. Fibre diameter and medulla diameter to fibre diameter ratio are important characteristics, which offer the possibility of defining a threshold level for contaminant medullated fibres. Most of the research reviewed here supports a value of med ratio of 0.6 and a diameter of 50μm as suitable limits to define contaminant medullated fibres. No work has been found which specifically investigates the threshold level of contaminant medullated fibres on Merino wool. Further study and research are needed to identify a threshold value of contaminant medullated fibres that would be useful for the presale testing for contaminant medullated fibres.

6. Loss of Medullated fibre in processing

According to Turpie 1985, fettling at regular intervals during carding can reduce overall 'kempiness'. Another source of kemp removal, for cards fitted with flexible card clothing is the burr beater of the card. Turpie also noted that kemp fibres are only loosely attached to the web between the swift and doffer and often drop out at this point. Therefore, attention to machine design, settings and handling can increase the removal of kemp at the carding stage. Earlier work done by Turpie (1971) showed that using an appropriate selection of pin densities and a suitable selection of the settings of the drawing–off rollers can improve kemp removal in the combing process.

Turpie also showed that the shorter coarser medullated fibres went into the noil and the longer finer medullated fibres went into the top in Noble combing process (Table 4). Similar performance would be expected using modern, rectilinear combs. No trial evidence of this pattern of removal of kemp has been found, though anecdotal evidence exists (Gary Robinson, pers.com).

Table 4. Characteristics of kempy crossbred in punch sliver, TOP and NOIL

		Punch Sliver	Top	Noil
Percentage of Medullated fibres		24.0	21.7	34.6
Wool	m.f.l. (cm)	4.5	5.6	1.6
	m.f.d. (μm)	20.8	20.9	19.6
Medullated fibres	m.f.l. (cm)	4.3	6.3	1.8
	m.f.d. (μm)	49.3	40.3	58.9

(Source Turpie 1971) m.f.l – mean fibre length, m.f.d – mean fibre diameter

Subsequent work on wool fibres also showed that fibres such as kemp, which are structurally different from wool, tend to be removed during modern worsted processing. Early–stage processing has completely removed hetero type fibres normal medullated fibres present as contaminants, most likely by mechanical damage during carding (Harrowfield 1987 cited Hatcher 1999). The work done by Hatcher et al.(1999) showed that more than 94% of pigmented kemp fibres from Awassi sheep present in Merino wool were lost during early stage processing with a further 3 % loss occurring through to the finished fabric.

Most of the studies in processing loss are at least a decade or more old, and limited information has been obtained from experiments involving processing losses of medullated fibres from Merino wool (Hatcher, 1999). No studies have specifically investigated the loss of medullated fibres in advanced processing such as high speed carding, and the increased speeds of modern combing and spinning. Further studies and research are needed to identify the loss of these medullated fibres in the processing and the persistence into fabrics. Such a study may also facilitate finding the threshold level of medullation that makes the fibre objectionable. Knowing this threshold would be useful for presale testing for contaminant medullated fibres.

7. Dyeing Behaviour of Medullated Fibres

Studies were undertaken by Smuts and Hunter (1987) to compare the dyeing behaviour of medullated and unmedullated fibres in mohair. Their initial results suggested that the solid material of kemp (i.e. their walls) and normal mohair fibres dyed to approximately the same colour(shade) in many cases as shown in Figure 26. They explained that the different appearance of kemp in a dyed sample appeared to be an optical effect due to the reduced light path through the dye in the fibre wall and refraction and reflection of the light at and within the medulla. Their work also showed that kemp fibres could be more or less apparent (i.e. visually different), depending upon the colour and depth of shade to which the material is dyed. And their later studies indicated that the take-up of premetalised dyes containing chromium may be different for mohair and kemp, possibly due to differences in surface area per unit mass.

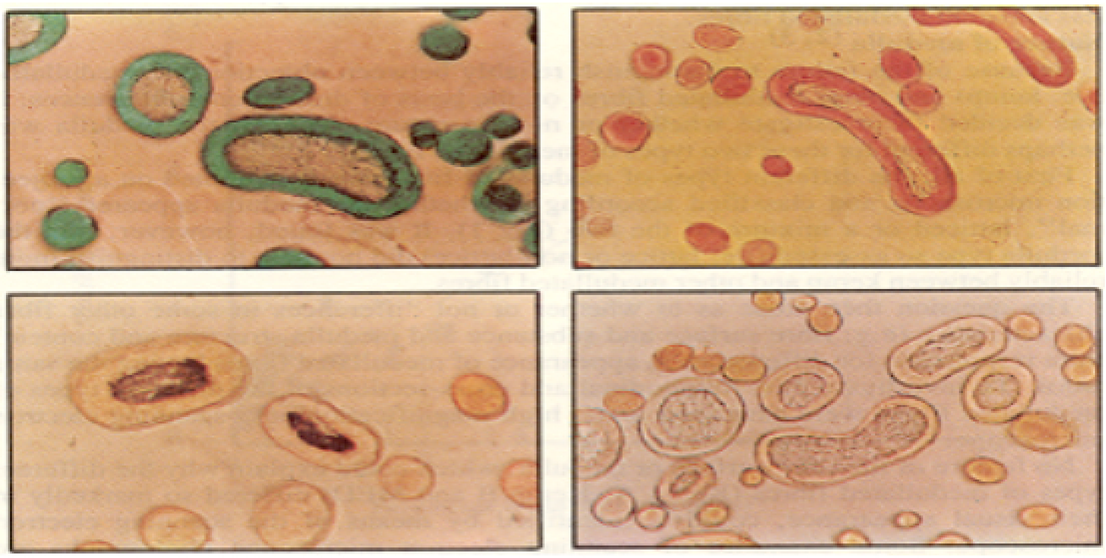

Figure 26. Example of dyed mohair, unmedullated and medullated fibres (Source - Hunter 1987)

Other work on mohair by Smuts and Hunter (1987a) showed that, for the medullated fibres removed from different dyed and undyed samples, the medulla to fibre diameter ratios for the contaminant dyed fibres were slightly higher than those for the undyed fibres. This suggests that the dyeing reduced visual differences between the different types of kemp fibres. In the experiment they graded the medullated fibres as:

- Kemp A (Chalky white fibres which were easily distinguishable in air)
- Kemp B (Chalky white fibres which were less easily distinguishable in air)
- Med fibres (Fibres which were only distinguishable in benzyl alcohol after removal of the chalky white fibre).

The fibre were measured the fibres before and after the dyeing. Indications from this study are that, the threshold medulla to diameter ratio for dyed fibres may be approximately 0.6, as shown in Figure 27.

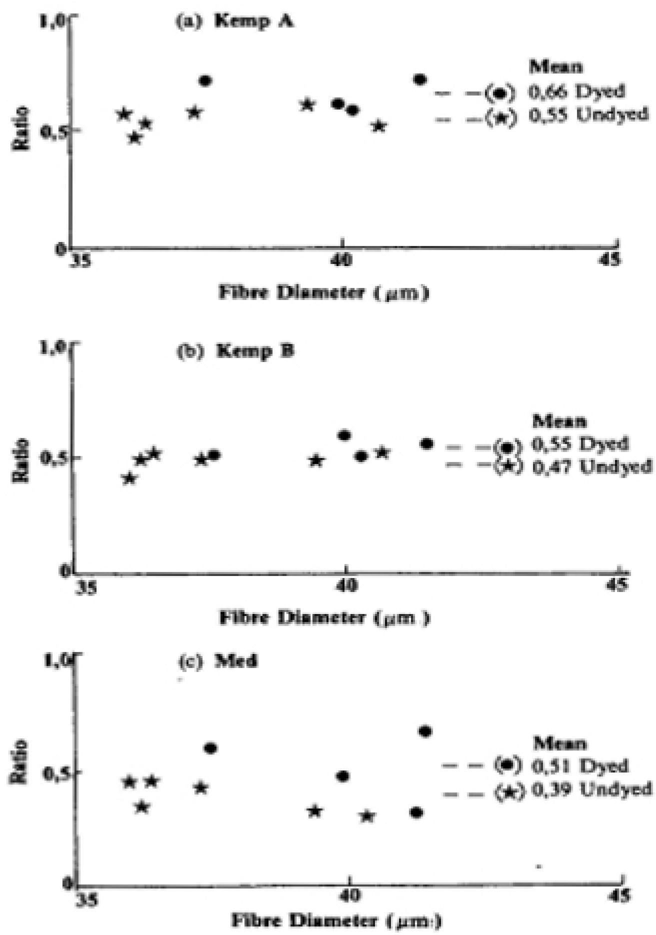

Figure 27. Sample mean ratio of medulla to fibre diameter for dyed and undyed fibres vs. sample mean fibre diameter . (Source – Smuts and Hunter 1987a)

Limited information has been obtained from experiments involving dyeing of medullated fibres. No studies have specifically investigated Merino wool. Further studies and research are needed to identify the effect of different types of medullated fibres in the dyeing process of fabrics. Such a study may facilitate finding the threshold level of medullation makes the fibre objectionable. Knowing this threshold would be useful for presale testing for contaminant medullated fibres.

8. Summary

- Medullated fibres may or may not be referred to as objectionable fibres.

- Objectionable medullated fibres have been defined as fibres that are generally chalky white in appearance, even after dyeing, (Hunter 1987) and may be classified as:

  Kemp fibres;

  Highly medullated fibres; and/or

  Flat fibres.

- Work done on one contaminated fabric sample at AWTA Ltd shows that all contaminant fibres are highly medullated and most have a flat ribbon-like appearance.

- Medullation is unusual in Merino sheep, only occurring rarely in very coarse fibres. These medullated fibres can occur on the face and legs, and can be inadvertently included with the main clip lines at shearing.

- Medullated fibre contamination can also occur by direct contact between sheep, for example, when mating exotic ram breeds to Merino ewes, and when Merino ewes raise cross bred lambs between birth and weaning (Hatcher 2002). Also contact with common areas can be responsible for contamination in Merino sheep, e.g., shearing yards where fibres from exotic sheep can shed onto races, fences, gates, on the ground etc, and can be subsequently picked up by Merino sheep.

- The most common Test Methods for measurement of medullation are:

  1. Microscopic counting;
  2. WRONZ Medullameter;
  3. Near Infrared Reflectance Spectroscopy (NIRS);
  4. Measurement of medullation using OFDA;
  5. Original AWTA Ltd method for medullated fibre measurement of core samples using CSIRO Dark Fibre Detector; and,
  6. AWTA Ltd Benzyl Alcohol Test Method, originally developed by CSIRO.

- Standard test definitions for medullated fibres in wool and other animal fibres by microprojection (ASTM standard D2968-89, 1989) are:.

  - Medullated fibre – an animal fibre that, in its original state, includes a medulla;
  - Med Fibre – a medullated animal fibre in which the diameter of the medulla is less than 60% of the diameter of the fibre; and
  - Kemp fibre (Objectionable Fibre) - a medullated animal fibre in which the diameter of the medulla is 60%, or more, of the diameter of the fibre.

- The ASTM standard definitions have been commonly used in different methods of measuring medullation.

- Compared to the other methods, the new AWTA Ltd benzol test method is commercially acceptable because it tests a large size of sample (20g) relatively quickly. The medullation of several hundred thousand fibres is seen at a glance and the method detects only fibres that are considered to be a cause of contamination.

- Limited information has been obtained from experiments involving the measurement of contaminant medullated fibres in wool. This information shows that medullated fibres are relatively coarser than non-medullated fibres in any natural population. Fibre diameter and the medulla diameter to fibre diameter ratio are important characteristics, which offer the possibility of defining a threshold level for contaminant medullated fibres.

- Most studies of processing loss of medullated fibres are at least a decade or more old, and limited information has been obtained from experiments involving processing losses of medullated fibres from Merino wool (Hatcher, 1999).

- In a trial by Hunter, mohair fibres were measured before and after the dyeing, and results showed that the threshold medulla to diameter ratio for dyed fibres may be approximately 0.6. No information has been obtained from experiments involving dyeing of medullated fibres for Merino wool.

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