# THE EFFECT OF DIAMETER VARIABILITY AND MEDULLATION OF WOOL ON FIBRE FINENESS DISTRIBUTION ANALYSER (FFDA) MEASUREMENTS<sup>1</sup>

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ABSTRACT - The effect of medullation and variability of fibre diameter on FFDA measurements were investigated. For a FFDA fitted with the expanding (new) cell, medullated wools and more variable wools gave finer results than those obtained with the projection microscope. Equations correcting FFDA results for the degree of medullation are presented. It is suggested that calibration using a coarser top than those currently in use would improve measurements on coarse wools.

Index terms: wool fibre diameter variability, calibration, coarse tops, medullation assessment methods.

## EFEITOS DA VARIABILIDADE DE DIÂMETRO E MEDULAÇÃO DA LÃ SOBRE AS MENSURAÇÕES DO "FIBRE FINENESS DISTRIBUTION ANALYSER" (FFDA)

RESUMO - No presente trabalho foram investigados os efeitos da medulação e da variabilidade de diâmetro da lã sobre as medidas de diâmetro no FFDA. Foram desenvolvidas equações para corrigir os resultados oriundos do FFDA, de acordo com o grau de medulação das amostras. Os resultados do trabalho indicaram que na calibração do FFDA deveria ser incluído um "top" de lã com maior diâmetro médio do que os usados correntemente. Isto melhoraria os resultados para as lãs mais grossas.

Termos para indexação: variabilidade de diâmetro da lã, calibração da lã, métodos de avaliação da medulação, avaliação de tops com fibra medulada.

## INTRODUCTION

Lynch & Michie (1976) designed the prototype of the Fibre Fineness Distribution Analyser (FFDA), which is an instrument for the rapid measurement of the mean and distribution of fibre diameter of wool. The principle of measurement is based on the reduction of light intensity when fibre snippets are carried through a laser beam, immersed in a fluid (iso-propanol and water). The FFDA has been shown to be a satisfactory method for the estimation of fibre diameter on non-medullated wools, when factors set out by Irvine & Lunney (1979), Lunney & Irvine (1979 and 1982) and Thompson & Teasdale (1985) are regulated. However, some studies (Lunney & Irvine 1979, Thompson & Teasdale 1984) have reported low accuracy of the FFDA diameter estimates in coarse wools.

Studies on the reliability of FFDA results for medullated wools were made by Van Luijk (1984), whose results suggested the occurrence of bias in the measurement. However, it appears that no investigation has been made to quantify the magnitude of the effects.

All of the results cited above were obtained from instruments equipped with the original parallel sided cell. The FFDA instrument used

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in this study was fitted with an expanding (new) cell (Marler & Irvine 1985) developed at the CSIRO - Division of Textile Physics.

The aims of this study were principally:

- To evaluate the effects of medullation and standard deviation on mean fibre diameter as estimated by the FFDA.

- To develop correction equations taking account of medullation (as assessed by two methods) and standard deviation to improve fibre diameter estimates. This assumes that the effects mentioned above influence FFDA results.

## MATERIALS AND METHODS

Two hundred fourty-two wools, encompassing diameters between 22.0 and 38.0 micrometres ( $\mu$ m) were studied. Wools were grouped into 1.0  $\mu$ m intervals which included non-medullated wools and wools selected to have varying degrees of medullation. The maximum degree of medullation in each micrometre class is shown in Table 1.

### Measurements

### Fibre diameter

**Projection Microscope (PM)** - Diameter measurements for PM were carried out at the School of Fibre Science and Technology, Department of Wool Science, The University of New South Wales, Sydney, Australia. The PM measurements followed the standard method laid down in the IWTO-8-61 (IWTO 1961). Due to the large number of samples, measurements were shared by three operators. Measurements on Interwoolabs standard tops were made in order to harmonize operators when measuring the experimental samples. Snippet length did not exceed 0.6 mm, varying from 0.4 to 0.6 mm depending on the sample mean fibre diameter, as recommended in IWTO-8-61 and later, by Browne & Hindson (1982).

All PM mean fibre diameters were obtained at least at 0.4  $\mu$ m accuracy at the 95% confidence level. The number of fibres measured on each sample, therefore, depended on its variability. At first, a minimum of 400 fibres were measured, from which the variance (s<sup>2</sup>) was calculated and its value

TABLE 1.	Distribution of medullated and non-
	medullated wools within 1.0 µm in-
	tervals of diameter as measured by
	the Projection Microscope (PM).

Diameter interval	Number of measur	PM maximum medullation	
(µm) 	Non-medul.	Medul.	(%)
22	6	7	10.3
23	6	9	13.3
24	5	4	7.8
25	4	9	13.9
26	5	10	18.3
27	4	9	9.5
28	6	12	22,6
29	10	4	8,8
30	6	10	11.5
31	12	13	17,7
32	8	8	17.0
33	4	8	14.4
34	7	11	16.7
35	7	14	19.7
36	6	3	15,9
37	2	6	22.7
38	3	4	9.5

replaced in the equation below. This, therefore, indicated whether or not additional measurements on the sample should be made to reach the desired significance level.

 $\mathbf{n} = (\mathbf{t} \, \mathbf{s/e})^2$ 

where

- n = number of fibres to be measured
- t = 1.960 (Student's value at 95% confidence level and infinite degrees of freedom)
- e = 0.4 (allowance in micrometre for error about the mean)

This procedure is in accord with ASTM-D-2130 (1972) and led to the measurement of a large number of fibres mainly in coarse wools in which the variance was higher.

#### Fibre Fineness Distribution Analyser (FFDA)

Fibre diameter measurements by the FFDA instrument were carried out at the CSIRO-Division of Textile Physics. These measurements were obtained for all 242 wools measured by Projection Microscope.

The test specimen (approximately 0.5 g of fibre snippets) was obtained employing the hand mini-coring technique (Buckenham et al. 1979), using a 2 mm diameter cutting tip. The minicored material was washed with petroleum ether, air dried and conditioned at  $20^{\circ}$ C and 65% RH for at least three hours, although conditioning of specimens may not be necessary, since their regain does not affect the results (Thompson & Teasdale 1985).

As previously mentioned, the instrument used contained the new cell, which, due to changes in fibre orientation in the flow, allows a single FFDA calibration (with tops TI46 and T147 from CSIRO, Division of Textile Physics) to cover a wide range of diameter measurement. In addition, according to Marler & Irvine (1985), the change in fibre orientation has contributed to "increase (ing) the measurement acceptance rate from 25% to 50% and remove (ing) the bias in the measurement of coarse wools". The FFDA was calibrated using software supplied so that fibres from 3  $\mu$ m to 71  $\mu$ m were detected (J.W. Marler, pers. comm.). The total count rate was kept below 50 ct/sec. and a limit of 2000 accepted counts was used for calculations.

Proving runs using standard reference tops (viz. T146 and T147), were made prior to the first measurement, and subsequently after each group of fifteen samples. The FFDA diameters were obtained at 0.2 to 0.3  $\mu$ m measurement precision, at the 95% confidence limit.

An inspection of the PM measurements of the experimental samples revealed that no fibre was outside the range of  $3-71 \,\mu m$ , indicating that the calibration used was suitable for these samples.

### **Degree of medullation**

Two different methods of medullation assessment were used on individual samples:

### **Projection Microscope (PMM)**

The percentage of medullated fibres in all of the 242 wools was estimated at the same time as diameter measurement (IWTO-1964). In the present study, however, narrow and continuous medulla were not distinguished at the place of measurement.

#### Medullameter (MM)

The percentage area of medullation in 86 of these

samples was assessed photo-electrically by the SAWTRI medullameter, at the South African Wool Textile Research Institute (SAWTRI), South Africa.

The principle of measurement, instrument calibration, sample preparation and testing procedures are set out in Lappage & Bedford (1983) as the SAWTRI Medullameter in based upon WRONZ design (Smuts et al. 1983). According to the former authors the Medullameter readings "provide detailed knowledge of medulla distribution as well as an overall index which can be expressed as area of medulla as a ratio to area of fibre". The values observed were found to be closely related to percentage area of medullation as measured on a Projection Microscope.

### Data analysis

The data were subjected to regression analyses, which were carried out using SPSS program (Nie et al. 1975). Two approaches were followed to investigate the proposed aims.

- To determine the extent to which FFDA measurements (Y) are dependent on PM diameter  $(X_1)$ , SD of diameter  $(X_2)$  and medullation (PMM or MM)  $(X_3)$ , two sets of multiple linear regressions were fitted, which are described in the general model:

$$Y_{i} = B_{0} + B_{1}X_{1i} + B_{2}X_{2i} + B_{3}X_{3i} + E_{i}$$
(1)

where:  $E_i$  = residual error of observations (u = 0 and  $\sigma^2$ )

- Assuming other factors were found to be important, and an attempt was made to supplement FFDA measurements to predict the "true" diameter (PM value) more accurately by considering the reduced model:

$$Y_{i} = B_{0} + B_{1}X_{1i} + B_{2}X_{2i} + E_{i}$$
(2)

where:

 $\begin{array}{l} Y_i = \text{observation on the } i^{th} \ \text{PM diameter} \\ B_o = \text{intercept} \\ B_i = \text{partial regression coefficients} \\ X_{1i} = \text{effect of the } i^{th} \ \text{FFDA diameter} \\ X_{2i} = \text{effect of the } i^{th} \ \text{PMM or MM} \\ E_i = \text{model residual variance} \end{array}$ 

The SD of the FFDA fibre diameter was included in model (2), but its results are not presented because this variable was only used to produce an adjusted regression equation (multiple partial regression).

## **RESULTS AND DISCUSSION**

## Effects of medullation and standard deviation

The results derived from model (1), displayed in Table 2, show that, after adjusting the equations for PM fibre diameter, both medullation and SD, significantly affected the mean fibre diameter as estimated by the FFDA instrument.

In order to graphically show the effects of SD (free of medullation effect) on the FFDA results, the data for 101 non-medullated wools are presented in Fig. 1. The results indicate that bias of measurement with FFDA substantially increased when SD of the samples was beyond the range of SD of the calibrating tops (e.g., T146 with SD=6.24). In view of the correlation of 0.70 between PM diameter and SD found, lower FFDA readings tend to be produced on coarse wools, which agrees with Thompson & Teasdale (1984), who used a FFDA equipped with the parallel sided cell.



FIG. 1. Pairwise comparison of FFDA and PM results according to different values of the mean fibre diameter distribution (SD).

Variable	РММ			ММ			
	Multiple R	B Value	Se (B)	Multiple R	B Value	Se (B)	
РМ	0.973	1.063 **	0.014	0.951	1.010 **	0.027	
MAM	0.975	-0.032 **	0.009	0.955	-0.149 **	0.033	
SD	0.978	-0.290 **	0.050	0.962	-0.379 **	0.085	
Intercept		-0.315	0.302		-0.324	0.781	
Residual							
SD (µm)			0,68			0.71	

 TABLE 2. Multiple regression coefficients; model (1) where the medullation assessment method (MAM; X3) was based on the Projection Microscope (PMM) or the Medullameter (MM).

\*\* (P < 0.01).

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By comparison with the results reported by these authors, the results obtained highlight that the employment of an expanding cell in the FFDA instrument used in this study did not completely remove the bias apparent on diameter estimates on coarse wools (Fig. 2), which is in contrast to the claim by Marler & Irvine (1985).

According to Lunney & Irvine (1982), the PM readings of the calibrating tops influencethe FFDA calibration function even beyond the range of diameter of the tops (10 µm -55  $\mu$ m for the combined T146 and T147), although the confidence on the predicted upper limit is reduced. The results obtained broadly suggest that inaccuracies of measurements were mostly found on samples having fibre readings in this "extrapolation part" (coarse tail) of the calibration function. In agreement with Thompson & Teasdale (1984), it appears that more satisfactory results might be obtained in coarse wools, if a top covering the predicted coarse fibres were included in the FFDA calibration.

There does not seem to be any work evaluating the quantitative effects of medullation on FFDA readings. The results obtained here were consistent in indicating that, irrespective of the medullation assessment method (PMM or MM), the increase in the proportion of medullated fibres



FIG. 2. Pairwise comparison of FFDA and PM mean diameter on non-medullated wools.

substantially decreased the estimates of the FFDA mean fibre diameter. A hypothesis to explain this effect is based on the rejection of measurements because of the production of an atypical pulse when medullated snippets pass through (place the laser beam of measurement). For example, if some light passes through the centre of the fibre (medulla), it might produce a so-called "double humped peak" (atypical pulse or multiple peak, as named by Lunney & Irvine 1979) which, according to the FFDA design, is promptly rejected for diameter computation. In such a situation, there would consequently be an overall decrease in the mean fibre diameter, owing to these fibres being coarser than the unmedullated ones.

In Table 3, results on some highly medullated wools, which also showed the largest FFDA-PM difference, are presented. The mean fibre diameter of both medullated and non-medullated fibres was calculated from the PM measurements. The results indicate that FFDA diameter was based largely on the diameter of non-medullated fibres. This appears to support the previous hypothesis. Nevertheless, the extent of the trends shown in Table 3 is confounded with biases due to fibre diameter variability (as previous seen), principally in coarser wools.

The values lower than normal for specific gravity of medullated fibres (Oliveira 1986) may probably be excluded as a source of variation in the FFDA results. There should not be any "separation" of medullated and unmedullated snippets in the immersion fluid, since due to its low specific gravity (0.8129 g/cc at  $20^{\circ}$ C), all fibres would remain in similar conditions in the moving liquid.

### Prediction of PM fibre diameter

The data obtained in the previous section highlighted the dependence of the mean fibre diameter measured by FFDA on the factors examined. It, therefore, should be possible to obtain better diameter estimates (closer to PM values) when information on degree of medullation and SD are considered.

Mean Fibre diameter (µm)		Medullation PMM method	Mean fibre diameter (µm ; SD)				Number of fibres
PM	FFDA	(%)	Med.	Fibres	Non-Med.	Fibres	(PM)
23.7	22.3	13.3	29.3	3.9	22.8	4.1	505
24.2	22.7	7.8	32.4	3.7	23.3	4.8	567
25.3	23.8	8.3	32.8	4.9	24.6	5.2	569
26.3	24.5	11.7	30.4	4.1	25.7	5.2	504
26.4	24.8	18.3	32.7	6.2	25.0	5.5	590
26.8	25.7	8.8	40.0	6.8	25.5	8,1	645
27.5	26.3	7.4	33.7	4.7	26.9	5,4	552
28.8	27.4	22.6	34.0	4.3	27.3	4,9	504
29.3	27.9	8.8	40.0	8.2	28.2	7.6	626
31.1	29.7	11.7	40.9	4.5	29.7	6.6	750
35.3	34.1	12.7	42.3	5.6	34.2	6,8	833
35.6	34.1	11.0	44.8	6.6	34.6	8.5	911
37.6	36.3	22.7	44.5	6.9	35.6	7,8	913

TABLE 3. Pairwise comparison of the PM and FFDA results in some medullated wools and the effect of the distribution of medullated and non-medullated fibres on the mean fibre diameter.

In Table 4, correction equations (obtained from model 2) which are based on two methods of medullation assessment, and previously adjusted for SD effects (FFDA value), are presented. The low percentage of variation not accounted for by the models fitted, can be observed. The errors of prediction were similar and low in both equations, and are comparable with the  $0.5 \,\mu$ m obtained on non-medullated wools by Thompson & Teasdale (1984).

For each medullation assessment method, Fig. 2 and 3 present the amount by which the "true" mean diameter differed from both the actual FFDA values (items a), and those obtained when predicting PM diameter by employing the equations given in Table 4 (items b). Overall, a reasonable improvement in FFDA results can be noticed. The largest difference between uncorrected FFDA and PM (up to 3.0  $\mu$ m) was reduced to about half when FFDA diameter was corrected.

The consist tendency of measurements on the coarser wools to be biased, was not

TABLE 4. Multiple linear partial regression<br/>coefficients; model (2) where the<br/>medullation assessment method<br/>(MAM; X2) was based on the Pro-<br/>jection Microscope (PMM) or the<br/>Medullameter (MM).

	PMN	M	MM		
Variable	B Value	Se (B)	B Value	Se (B)	
FFDA MAM Intercept	0.884 ** 0.030 ** 3.775	0.013 0.080	0,900 ** 0.158 ** 3.184	0.021 0.025	
R (model)	0,981		0.968		
Residual SD (µm)		0.62		0.62	

Regression equations are adjusted for the effect of SD as obtained with the FFDA.

\*\* (P < 0.01).

very pronounced in this study (Fig. 3 and 4; items a), which may have been due to the fact that highly medullated wools were also presented at the finer diameters.

The root mean square relative errors, obtained for the difference between the corrected FFDA diameter and PM values, were 2.0 and 1.8  $\mu$ m, for the PMM and MM methods, respectively. These were much smaller than the 4.7  $\mu$ m reported by Van Luijk (1984), for normal FFDA results on 84 crossbreed medullated wools.

The results indicated that biases of FFDA measurements on coarse and/or medullated wools may be substantially reduced when diameter is corrected in the manner described here.



FIG. 3. Pairwise comparison of mean fibre diameter (m) for samples where medullation was assessed by the PMM method.



FIG. 4. Pairwise comparison of mean fibre diameter (m) for samples where medullation was assessed by the MM method.

## CONCLUSIONS

1. In view of the results obtained and considering the FFDA instrument used, its calibration function, and the procedures of wool sampling and measurement, it can be concluded that the increase in both medullation and standard deviation of wool, significantly decreased mean fibre diameter as measured by the FFDA. Correction equations including different methods of medullation assessment (adjusted for SD effects), which were developed and evaluated, indicated the possibility of obtaining more reliable FFDA results when these are applied.

2. The correction equation was also developed for PMM method, although its use is limited, as it implies medullation assessment by the slow and laborious Projection Microscope. On the other hand, the correction suggested for the MM method could well be routinely employed, since the Medullameter instrument gives faster, more automated, and probably less biased medullation evaluation.

3. It appears that the use of a FFDA instrument equipped with the expanding cell did not completely remove the biases in measurement already observed in coarse wools, when a FFDA with the parallel sided cell was used. The results broadly suggest that, in order to minimize such bias, the inclusion of a coarser top than T146 should be attempted for the FFDA calibration.

4. In summary, it appears that the correction methods of fibre diameter for medullation and standard deviation devised in this study, constitute an important advance in diameter measurement on coarse wools using FFDA.

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