Presentation of the first results of the Round Trial on fineness of cashmere and mohair

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1. Introduction

The introduction of a fast, but accurate and standardised, method for measuring the fineness of ultrafine wools and speciality fibres (especially cashmere) has always been a vital interest of breeders, processors and fibre experts.

In the context of the EU cashmere project (AIR3-CT94-0822), the OFDA (Optical Fibre Diameter Analyser) has been proposed by the metrology group at DWI (German Wool Research Institute in Aachen, Germany) as a suitable method for measuring quality and yield of cashmere in raw fleeces. INRA (Institut Nationale de la Recherche Agronomique) has also proposed the OFDA method for the measurement of mohair. In the mean time, the OFDA method has become a standard test method (IWTO-47-95) for measuring mean fibre diameter (MFD) of sheep wool fibre samples. The application of this IWTO standard is not extended to cashmere and mohair yet, because there is still a lack of reproductible results from inter-laboratory round trials. The aim is to demonstrate that the OFDA technique should be adopted as a European and World standard for cashmere and mohair.

There is also a lack of fine fibre samples as standards when using the OFDA as a tool for measuring small samples for MFD and this required to be rectified before the round trial commenced. Therefore, the round trial will also investigate if the results of the fineness measurement could be influenced by the introduction of a standard (13-18 microns range) in the calibration.

Besides the OFDA method, the cross section method (CS) using an image analysis system, the scanning electron microscopy (SEM), the Laserscan method (LM) and especially the already standardised microprojection (PM) method are also used. A comparison between the values gained by the different methods was also made.

The outcome of the round trial should be that the OFDA method exhibits a high precision such as it can be then recommended as the European and World standard method for cashmere down and mohair.

2. Participants

Twenty seven laboratories (research institutes and industrial companies) were invited to take part in the round trial. Four laboratories did not submit either their written agreement or measurements results to DWI, so that there are finally a total number of 23 participants: 21 laboratories from eight European countries, and also one from Australia and one from New Zealand. The participating laboratories were as follows:

Denmark

- Danish Institute of Animal Sciences, Research Center Foulum, Tjele

France

- INRA, Cr Toulouse, Station d'Amélioration génétique des Animaux, Castanet-Tolosan

- Institut Textile de France Sud ITF, Aussillon Mazamet

Germany

- Deutsches Wollforschungsinstitut (German Wool Research Institute) DWI, Aachen
- Faserinstitut Bremen e.V., Bremen
- Bremer Woll-Kämmerei BWK , Bremen
- Zwickauer Kammgarn GmbH, Silberstraße
- Schoeller Eitorf AG, Eitorf
- Kammgarnspinnerei Stöhr GmbH, Mönchengladbach

Great Britain

- Macaulay Land Use Research Institute MLURI, Aberdeen
- British Textile Technology Group BTTG, Leeds

<u>Italy</u>

- Stazione Sperimentale per la Seta, Milano
- Lanificio Luigi Colombo s.p.a., Borgosesia
- Lanificio Ing. Loro Piana & C. s.p.a., Quarona
- Istituto di Recerche e Sperimentazione Laniera "O. Rivetti", Biella

Portugal

- Centro Tecnológico das Indústrias Têxtil
 - et do Vestuário de Portugal Citeve/Sede, Vila Nova de Famalicão
- Centro Tecnológico das Indústrias Têxtil et do Vestuário de Portugal Citeve/Delegacão, Covilhã
- Kammgarnspinnerei Stöhr Portugal, Famalicão

<u>Spain</u>

- Textile Physics Research Laboratory - Intexter, Terassa

Australia

- Victorian Institute of Animal Science, Fibre Quality Department, Victoria

New Zealand

- SGS Wool Testing Services, Wellington

Switzerland

- J. F. Müller + CO AG, Therwil
- Zellweger Uster Textile Technology, Uster

In what follows, each participant is referred to by a number, in order to ensure anonymity. The numbers have been arbitrarily assigned by DWI and do not correspond to the order of the above list of participants.

3. Methods

Besides the OFDA method, four further methods have been applied for measuring the MFD:

- PM (Projection Microscope)
- LS (Laserscan)
- CS (Cross section method)
- SEM (Scanning Electron Microscope).

The participating laboratories were asked to apply the whole range of measurement systems available to them. In practice, the laboratories have applied the following methods:

OFDA method only:
OFDA and PM method:
OFDA and LS method:
OFDA and CS method:
PM method only:
PM and SEM method:
CS method only:
13 participants (Lab 1-3, Lab 5-10, Lab 12-14, Lab 16)
2 participants (Lab Lab 11, Lab 15)
1 participant (Lab 14)
1 participant (Lab 4)
4 participants (Lab 17, Lab 19-21)
1 participant (Lab 18)
2 participants (Lab 22-23).

Table 1 shows a general overview of all participants and methods of MFD measurement.

Table 1: List of	participants	and of MFD	measurement methods.
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Participant			Method		
Lab No.	OFDA	PM	CS	LS	SEM
1	Х				
2	Х				
3	Х				
4	Х		Х		
5	Х				
6	Х				
7	Х				
8	Х				
9	Х				
10	Х				
11	Х	Х			
12	Х				
13	Х				
14	Х			Х	
15	Х	Х			
16	Х				
17		Х			
18		Х			Х
19		Х			
20		Х			
21		Х			
22			Х		
23			Х		

4. Materials

4.1 <u>Cashmere</u>: dehaired, washed, commercial samples. According to the cashmere supplier, the MFDs of the samples measured by using a projection microscope (IWTO-8), were as follows:

No 1	Chinese white	14.49 µm
No 2	Chinese brown	14.60 µm
No 3	Chinese white	14.94 µm

No 4	Afghan	15.89 µm
No 5	Iranian fawn	17.36 µm

4.2 <u>Mohair</u>: being used as old mohair top standards from the IMA (International Mohair Association), supplied by DWI:

No 6	27.3 μm
No 7	29.1 µm
No 8	34.5 μm
No 9	38.0 µm

5. Results

5.1 Cashmere

Descriptive statistics (arithmetic mean and 95% confidence limits) were calculated separately for each laboratory and each sample.

The results are shown in Figures 1 to 5 and represent the range of data around the mean fibre diameter for each sample and each laboratory. The errors bars show the confidence limit (C.L.). The Tables corresponding to the Figures are shown in Appendix 1.

Figure 1: Mean fibre diameters and confidence limits for sample 1 (cashmere) for each laboratory.



Figure 2. Mean fibre diameters and confidence limits for sample 2 (cashmere) for each laboratory.





Figure 3. Mean fibre diameters and confidence limits for sample 3 (cashmere) for each laboratory.

Figure 4. Mean fibre diameters and confidence limits for sample 4 (cashmere) for each laboratory.



Figure 5. Mean fibre diameters and confidence limits for sample 5 (cashmere) for each laboratory.



In general, there is less variation in the results within the laboratories using OFDA method than the other methods. The results obtained with the other methods were more variable.

The confidence limits are very compact for the OFDA method.

5.2 Mohair

As for cashmere, the arithmetic mean and the confidence limit were calculated separately for each laboratory and each sample.

The results are shown in Figures 6 to 9 and represent the range of data around the fibre diameter for each sample and each laboratory. The error bars show the confidence limits. The Tables results are in Appendix 2.

Figure 6. Mean fibre diameters and confidence limits of sample 6 (mohair) for each laboratory.



Figure 7. Mean fibre diameters and confidence limits of sample 7 (mohair) for each laboratory.



Figure 8. Mean fibre diameters and confidence limits of sample 8 (mohair) for each laboratory.



Figure 9. Mean fibre diameters and confidence limits of sample 9 (mohair) for each laboratory.



As for cashmere, the same regularity and confidence limit trends were observed for the results of the mohair samples.

6. Preliminary statistical analyses

The approach described by Sommerville (1997) has been taken. This method attempts to demonstrate that there are differences between the methods used to determinate the Mean Fibre Diameter. This approach was used on both cashmere and mohair results. However, the outcomes of the statistical analyses to date have to be taken with caution, for they only represent a first stage in the analyses. For example, the difference between laboratories using the same method has not been studied at this stage.

6.1. Cashmere

Several series of comparison have been undertaken. They were as follow:

- Optical Fibre Diameter Analyser compared to the Cross Section method.
- Optical Fibre Diameter Analyser & the Cross Section methods compared to the Projection Microscope method.
- Optical Fibre Diameter Analyser, the Projection Microscope & the Cross Section methods compared to the IWTO-8 standard (The IWTO-8 standard uses the Projection Microscope

method to measure the mean fibre diameter. The values of the mean fibre diameters of the samples 1 to 5 were issued according to this IWTO-8 standard).

The OFDA method produced results which were significantly different from those obtained using the PM or CS methods. The difference between the CS and the PM methods was not significantly different from each other. When comparing to the IWTO values, the OFDA & PM methods gave results which were significantly different from the IWTO-8 values, while the CS one did not.

6.2. Mohair

Several series of comparison have been undertaken. They were as follow:

- Optical Fibre Diameter Analyser & Projection Microscope methods compared to the Cross Section method.
- Optical Fibre Diameter Analyser & the Cross Section methods compared to the Projection Microscope method.
- Optical Fibre Diameter Analyser, Projection Microscope, Laserscan, Cross Section and Scanning Electron Microscope methods compared to the IMA standard (values given to the participants at the start).

The OFDA and PM methods gave results which were significantly different from the CS method. However, the results of the OFDA and PM methods did not show any significative differences between each other.

When comparing to the IMA standard values, the results of the OFDA, PM and CS methods were significantly different from it. The LS and SEM methods did not show results significantly different from the IMA standards.

7. Conclusions

The preliminary results show general agreement between laboratories using the OFDA technique to measure the fibre diameter of the cashmere and mohair. There was greater variability within and between the other methods. However, the mean values differed between methods. Further statistical analyses will explore within-laboratories variability and help to elucidate why the different methods differed from one another before final conclusions can be drawn from the Round Trial.

8. References

Sommerville P.J. (1997). Measurement of the Fineness of Superfine Wool: A comparison of Airflow, Laserscan and OFDA. International Wool Textile Organisation, Boston Meeting.

Appendix 1: Tables I to V: Results of the cashmere samples.

method used	OFDA															
laboratories	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
mean (um)	15.4	15.4	14.9	15.2	14.8	14.8	15.2	15	15.1	15.2	15	14.8	15	15.1	16.2	14.9
C.L. (um)	0.06	0.11	0.07	0.06	0.10	0.06	0.12	0.06	0.10	0.10	0.06	0.07	0.08	0.12	0.14	0.11

Table I: Basic Statistics for sample 1 (cashmere)

method used	PM	PM	PM	PM	PM	РM	РM	CS	CS	CS	LS	SEM
laboratories	17	18	19	20	21	11	15	22	23	4	14	18
mean (um)	15.4	16.5	15.1	15.7	15	14.8	16.3	14.7	14.8	14	16.1	14.7
C.L. (um)	0.33	0.22	0.44	0.27	0.25	0.26	0.31	0.25	0.28	0.37	0.39	0.27

Table II: Basic Statistics for sample 2 (cashmere)

method used	OFDA															
laboratories	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
mean (um)	15.2	14.9	14.7	15	14.7	14.8	15.5	14.7	14.9	14.8	15	14.5	14.6	14.6	14.7	14.6
C.L. (um)	0.07	0.11	0.08	0.06	0.11	0.07	0.15	0.07	0.11	0.11	0.07	0.08	0.10	0.11	0.08	0.11

method used	PM	CS	CS	CS	LS	SEM						
laboratories	17	18	19	20	21	11	15	22	23	4	14	18
mean (um)	15.5	15.7	14.8	15.3	15	14.3	14.9	14.2	15	13.5	15.3	14.8
C.L. (um)	0.37	0.22	0.40	0.29	0.24	0.28	0.27	0.27	0.31	0.36	0.32	0.39

Table III: Basic Statistics for sample 3 (cashmere)

method used	OFDA															
laboratories	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
mean (um)	15.3	15.3	15	15.2	15	14.7	15	14.8	15	15	15	14.8	15.1	14.9	14.8	15
C.L. (um)	0.07	0.11	0.08	0.07	0.11	0.07	0.11	0.07	0.09	0.10	0.07	0.08	0.08	0.10	0.08	0.11

method used	PM	PM	PM	PM	PM	PM	РM	CS	CS	CS	LS	SEM
laboratories	17	18	19	20	21	11	15	22	23	4	14	18
mean (um)	15.7	16	14.7	15.2	14.9	14.6	15.3	14.7	14.7	13.8	15.6	14.3
C.L. (um)	0.36	0.29	0.34	0.26	0.23	0.29	0.28	0.20	0.30	0.36	0.31	0.34

Table IV: Basic Statistics for sample 4 (cashmere)

method used	OFDA															
laboratories	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
mean (um)	16.4	16.3	16.1	16.6	16.1	16.1	16.5	16.3	16.2	16.3	16.3	15.8	16	16.2	16.1	15.9
C.L. (um)	0.07	0.11	0.08	0.06	0.11	0.07	0.12	0.07	0.09	0.11	0.07	0.08	0.07	0.11	0.08	0.11

method used	PM	CS	CS	CS	LS	SEM						
laboratories	17	18	19	20	21	11	15	22	23	4	14	18
mean (um)	16.4	18	15	18.4	16.1	15.6	16.4	15.7	13.9	17	16.9	14.9
C.L. (um)	0.35	0.25	0.37	0.32	0.24	0.31	0.27	0.33	0.27	0.38	0.32	0.36

Table V: I	Basic S	Statistics	for sampl	e 5	(cashmere)
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method used	OFDA															
laboratories	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
mean (um)	17.3	17.4	17.3	17.6	17.5	17.1	17.2	17.4	17.3	17.5	17.4	17.1	17.2	17.3	17.3	17.3
C.L. (um)	0.08	0.11	0.09	0.08	0.11	0.07	0.12	0.07	0.13	0.11	0.08	0.08	0.08	0.11	0.08	0.12

method used	PM	CS	CS	CS	LS	SEM						
laboratories	17	18	19	20	21	11	15	22	23	4	14	18
mean (um)	17.6	18.2	15.5	19.1	17.6	17.1	17.7	16.4	18.2	16.8	18	17.1
C.L. (um)	0.38	0.24	0.43	0.34	0.27	0.30	0.33	0.28	0.31	0.36	0.34	0.35

Appendix 2: Tables VI to IX: Results of the mohair samples.

Table VI. Basic Statistics for sample 6 (mohair)

methods	OFDA															
laboratories	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
mean (um)	27.2	27.9	27.1	28.3	28.4	28.3	28.2	27.4	27.6	27.9	27.4	27.6	27.4	27.6	27.3	28
C.L. (um)	0.19	0.25	0.23	0.19	0.24	0.17	0.26	0.16	0.24	0.25	0.19	0.18	0.27	0.25	0.28	0.27

methods	PM	PM	РM	PM	PM	PM	PM	CS	CS	SEM	LS
laboratories	11	15	17	18	19	20	21	22	23	18	14
mean (um)	27.4	27.2	28	28	26.7	33.2	27.7	28.9	27.5	26.6	37.7
C.L. (um)	0.66	0.60	0.77	0.52	1.01	0.76	0.54	0.44	0.63	0.57	0.72

Table VII. Basic Statistics for sample 7 (mohair)

methods	OFDA															
laboratories	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
mean (um)	30.2	30.9	30	30.9	31.1	31.2	31.4	29.7	29.8	31.3	30	30.2	29.8	30.5	30.5	31.7
C.L. (um)	0.34	0.39	0.34	0.35	0.39	0.25	0.40	0.25	0.44	0.40	0.33	0.27	0.38	0.39	0.37	0.40

methods	PM	CS	CS	SEM	LS						
laboratories	11	15	17	18	19	20	21	22	23	18	14
mean (um)	29.1	29.7	30.4	31.1	30.6	32.3	28.7	30.2	30.2	27	30.2
C.L. (um)	0.94	0.82	0.98	0.94	1.74	0.99	0.75	0.76	0.93	0.56	0.97

Table VIII. Basic Statistics for sample 8 (mohair)

methods	OFDA															
laboratories	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
mean (um)	35.5	35.6	34.4	35.5	35	35.8	35.2	35.3	34.6	35.8	35.6	35.2	35.4	35.5	35.6	35.2
C.L. (um)	0.28	0.34	0.35	0.31	0.32	0.22	0.34	0.21	0.64	0.33	0.31	0.23	0.33	0.33	0.32	0.35

methods	PM	CS	CS	SEM	LS						
laboratories	11	15	17	18	19	20	21	22	23	18	14
mean (um)	34.9	34	35.5	35.5	34.4	40	33.7	34	34.9	34.1	35.7
C.L. (um)	0.83	0.82	0.96	0.69	1.26	0.96	0.63	0.80	0.84	0.87	0.89

Table IX. Basic stati	stics for sa	mple 9	(mohair)
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methods	OFDA															
laboratories	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
mean (um)	37.1	39.4	37	38.5	38	39.2	38.9	38	36.8	38.5	38.1	37.9	38.5	37.9	38.2	38.5
C.L. (um)	0.33	0.35	0.36	0.29	0.33	0.24	0.35	0.24	0.42	0.36	0.35	0.25	0.38	0.36	0.39	0.38

methods	PM	CS	CS	SEM	LS						
laboratories	11	15	17	18	19	20	21	22	23	18	14
mean (um)	37	36.9	38.1	37.2	37.5	36.5	37.3	36.5	36.3	35.8	37.8
C.L. (um)	0.87	0.80	1.09	0.62	1.70	0.88	0.62	0.70	0.86	1.02	0.92