Colour effect on fibre diameter measurement with OFDA 2000

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Colour effect on fibre diameter measurement with OFDA 2000

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In this study, wool samples from Merino, Corriedale and Lincoln sheep with a wide range of fibre diameters have been dyed and measured for fibre diameter using OFDA 2000 in both dry and conditioned states. Variation in fibre diameter is observed on fibres with different colours in both dry and aqueous conditions. This could be due to an optical effect or more likely due to physical changes in the dyed fibres owing to the presence of dyes. Apart from their physical bulk, dyes may also affect the owing water content of fibres and hence have an influence on the swelling of fibres when they are measured under ambient regain and wet conditions.

Keywords: Colour effect, Fibre diameter, Fibre colour, OFDA 2000, Wool fibre

OFDA 2000 equipment has the advantages of being portable and simple to operate. It can provide rapid (25s), accurate and precise diameter measurements with several thousand fibres being measured with relative ease[1]. OFDA 2000 is particularly well adopted in the wool industry for raw and scoured wool measurement. It has been shown that with suitable sample preparation techniques the equipment can be used at all stages of the wool-processing pipeline, from greasy fleece to yarn and fabric[2]. The use of OFDA is also not limited to wool and can be used more generally for other textile fibres. Recently, a new technique using OFDA 2000 has been developed to enable fibre diameters to be measured in an aqueous environment[3]. With the new technique, significant changes in wool fibre diameter were measured as a function of *p*H and temperature. This newly developed technique has opened up a new application for the use of OFDA equipment.

In the early stages of wool processing, where OFDA 2000 has been widely used for measuring fibre diameter, testing samples have been mainly raw fleece and scoured wool with a natural white colour. Diameters of wool fibre are known to be sensitive to factors such as relative humidity and temperature[4]. In industrial finishing processes, wool is frequently treated in hot water or in solutions with different *p*Hs. Fundamental knowledge of fibre swelling in these situations obtained with OFDA equipment can contribute to understand the outcomes of these processes. Up to now, little attention has been paid on the effect of fibre colour on the fibre diameter measurement results but it has been noted that results appeared to vary when the colour of the sample was changed. It is evident that further investigations are required to understand this effect.

In this study, wool samples from Merino, Corriedale and Lincoln sheep with a wide range of fibre diameters have been dyed and measured for diameter with OFDA 2000 in both the dry and conditioned states in air, and in aqueous solutions at a range of *p*H values. The outcome of this study is expected to add insights into the understanding of the mechanism of OFDA instrument and hence improve the scope for using this technology.

Experimental

Sample Preparation

As has been described in the previous study[3], a special cell for the measurement of fibres was constructed from two sheets of glass of 2 mm thick (184 mm × 187 mm) separated by a 1.5 mm gasket of heat-resistant silicone rubber.

Fibre samples were placed into the cell between the two glass plates. Tweezers were used to tease the fibres out to the required density for measurement in the cell. As the OFDA 2000 uses an image analysis system to identify and measure individual fibres in each of the samples, it is essential that each sample is sufficiently open or teased to allow a large number of individual fibres to be measured. In practice a well prepared open sample will have a far greater number of fibres measured and counted than a denser sample.

Measurement Methodology

The cell was mounted on the cell carrier of the OFDA 2000 instrument (Fig. 1) and held in place with adhesive tape. The scanning behaviour of the

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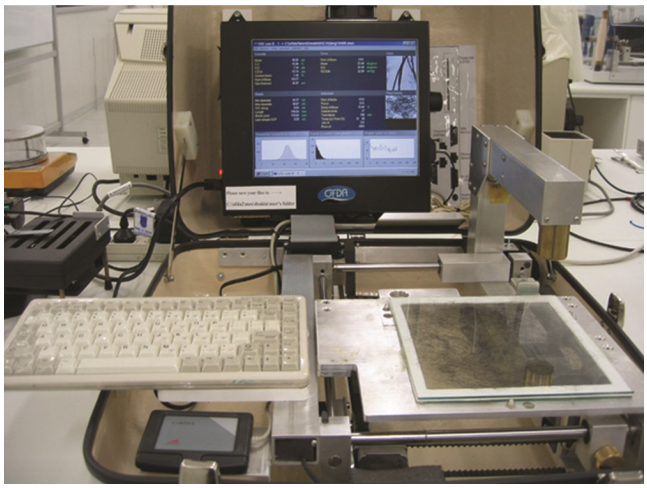

Fig. 1—OFDA instrument with liquid cell in measurement position (source: Textile Laboratory, Deakin University, Australia)

instrument was controlled by software and the parameters of the instrument were modified to ensure that fibres were measured throughout the whole area of the cell and that the focus was adjusted to capture the fibre images in a plane midway between the two glass plates.

Measurements were carried out with the whole plate scanned a number of times, and a minimum of eight thousand fibres were measured in each scan. The mean diameter of the final sample was then calculated, together with the standard deviation of each of the mean values.

Wool Fibre Samples

Wool fibre samples from Merino, Corriedale and Lincoln sheep were obtained as locks from single animals. The fibre samples were first cleaned by washing twice with Hydrapol TN450 (1g/L) and sodium carbonate (0.5 g/l) at 60°C for 10 min and afterwards rinsed well with warm water, dried and conditioned at 20±2°C and 65±2% relative humidity.

Drying, Conditioning and Wetting of Fibre Samples

For dried samples, wool fibres were placed in an oven at 105°C for 60 min on an open cell. The cells were then closed, removed from the oven and the fibre diameters were measured as soon as possible.

For conditioned samples, wool fibres were placed in a conditioned room at 20+2°C and 65+2% relative humidity for 24 h before measurement of the diameters.

For wet measurements, solution was first put in the cell and then the fibre samples were immersed in the solution and then teased out. The top glass sheet was put in place. During this procedure, any bubble introduced inside the cell was carefully removed. Fibres were soaked in the solutions contained in the cell for at least two hours before measurement. For wool sample in particular, previous study [5] has shown that at room temperature swelling of wool fibres takes around 20 min to reach equilibrium. Two hours duration was assumed to have been more than sufficient for fibres to reach their maximum diameters.

Table 1—Dyeing recipes for Merino top

Sample code	Description	Recipe
A	Blank dyeing (Lanaset method)	1% acetic acid 1.5% sodium acetate 1% Albegal SET
B	Red dyeing (Lanaset method)	5% Lanaset Red 2B 1% acetic acid 1.5% sodium acetate 1% Albegal SET
C	Black dyeing (Lanasol method)	5% Lanasol Black CE 1 g/L Miralan Q 1% acetic acid 1.5% sodium acetate 2% Albegal B
D	Acid Orange dyeing	1% C. I. Acid Orange 10 15% sodium sulphate 4% sulphuric acid
E	Blank acid dyeing	15% sodium sulphate 4% sulphuric acid

Buffer Solutions for Measuring *p*H-induced Fibre Swelling

Four different *p*H values were chosen for the experiments. Recipes for the buffer solutions were as follows: *p*H 2.1 buffer solution was obtained by mixing 0.06M sodium di-hydrogen phosphate and 0.06M phosphoric acid; *p*H 4.8 buffer solution was obtained by mixing 0.4M acetic acid and 0.4M sodium acetate; *p*H 7.2 buffer solution was obtained by mixing 0.1M sodium di-hydrogen phosphate and 0.1M di-sodium hydrogen phosphate; and *p*H 10 buffer solution was obtained by mixing 0.1M di-sodium carbonate and 0.1M sodium hydrogen carbonate. The ionic strength of each buffer was kept 1M.

Black Dyeing for Merino, Corriedale and Lincoln Top (Coarse Fibre Sample)

Wool sample size (2 g) was dyed using 5% Lanaset Black R, 2% acetic acid, and 1% Albegal SET. The temperature was raised from ambient to 100°C at 1°C per minute and held for 60 min.

Dyeing of Merino Top

Wool sample of the size 2 g was dyed using the recipes as shown in Table 1. The temperature was

raised from ambient to 100°C at 1°C per minute and held for 60 min.

The Lanaset dyes (Huntsman) are described, in general terms, by the manufacturer as mixtures of 1: 2 metal complex, acid and reactive dyes [6]. It has been claimed that Lanaset Red 2B contains Colour Index Acid Red 361, which is an acid dye and the chromophoric anion has a molecular mass of 519 (Fig. 2a)[6]. Lanasol Black CE (Huntsman) is a reactive dye[7] for wool, with two bromo acrylamido groups, two sulphonic acid groups and the chromophoric anion has a molecular mass of 904 (Fig. 2b)[8]. In this work, because there was no requirement for the dyes to be fast to machine washing, the Lanasol dyeings were washed off with water only and not after-treated with alkali, as is normal in commercial practice.

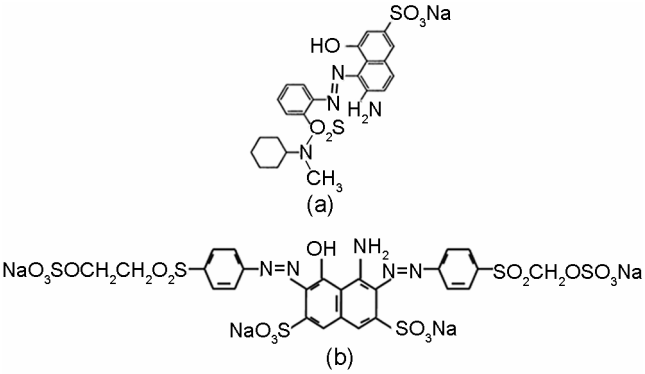

Fig. 2—Structures of (a) C I Acid Red 361, and (b) Lanasol Black CE[6-8]

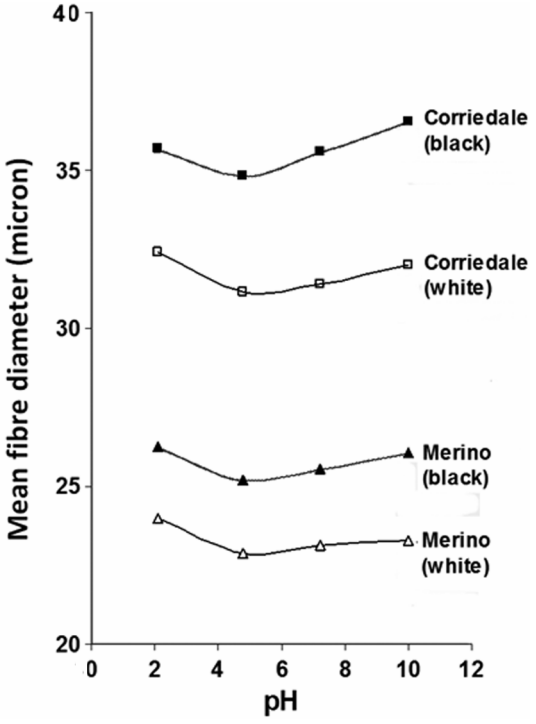

Fig. 3—Mean diameters of undyed and black dyed wool fibres in aqueous solutions at various *p*H

Albegal SET is an amphoteric levelling agent which has affinity for both dye and fibre and Miralan Q improves the quality of the wool at all stages of processing. These are proprietary auxiliaries recommended by Huntsman for wool dyeing[6].

Results and Discussion

Diameter Measurement

Table 2 shows the mean diameters of undyed and black dyed Merino, Corriedale and Lincoln fibres. The greater diameter values are observed on the dark fibres in all cases, in both dried and conditioned states.

Similar observations are made on the fibre samples measured in water. In Fig. 3, the measured mean diameters of white and black wool fibres in water are given at various *p*H. At all *p*H, both Merino and Corriedale fibres give higher diameter values when dyed black.

Table 2 shows that the coarse Lincoln samples (both dyed and undyed) could be measured in air, only the black dyed Lincoln fibres could be measured in water. This suggests that the large diameter fibres (especially when measured in aqueous condition) require to be made opaque by dyeing them in dark shade before the image analysis system is able to reliably detect the edges of the fibres.

Measurements on Merino and Corriedale fibres also show a variation in wool swelling as a function of *p*H. Fibre diameter is found to be minimum at the wool isoelectric point of *p*H 4.8 and the fibre diameter increase at both lower and higher *p*Hs. The changes in fibre swelling with *p*H are attributed to the variations in ionic attraction and repulsion between the amphoteric wool protein chains. These results are consistent with those obtained from previous studies[3, 9].

Table 2—Mean diameters of undyed (white) and black dyed wool fibres in air, dry state and conditioned state

Wool	Mean diameter, µm (SD)			
	Dry fibre		Conditioned fibre	
	Undyed	Black dyed	Undyed	Black dyed
Merino	19.28 (0.05)	19.78 (0.02)	20.28 (0.02)	20.79 (0.01)
Corriedale	26.52 (0.03)	28.33 (0.04)	27.99 (0.06)	29.41 (0.05)
Lincoln	41.19 (0.01)	42.94 (0.05)	43.57 (0.07)	45.91 (0.04)

Colour Effect on Fibre Diameter Measurement in Aqueous Solution

The effect of fibre colour on fibre diameter is shown in Fig. 4, which shows the mean diameters of Merino fibres with various dyed colours (Table 1), as a function of *p*H. It is observed that the highest diameter values are measured with the darkest fibre sample C (black), followed by less dark coloured fibre samples B (red) and then D (orange), within experiment error. The lower fibre diameters are shown in the light coloured (white) samples A and E respectively. The fibre diameters of these two controls treated by different dyeing methods show no significant differences.

These observations suggest that the presence of dye in the fibres affects the fibre diameter significantly. When the results in Figs 3 and 4 are compared with the mean fibre diameters of undyed fibres (from the same fibre sources) as shown in Table 2, it appears that the measured diameter values on the undyed fibre samples (white fibre samples) are invariably lower than that of the coloured fibre samples.

There are two possible explanations or the apparent variation in mean fibre diameter with colour. Firstly, it is possible that the presence of dyes has actually modified the dry and conditioned diameters as well as the swelling behaviour of the fibres. This could arise because the dyes are relatively large molecules which occupy significant amounts of space within the matrix of fibre and their presence may both increase the diameters of dry and conditioned fibres by limiting

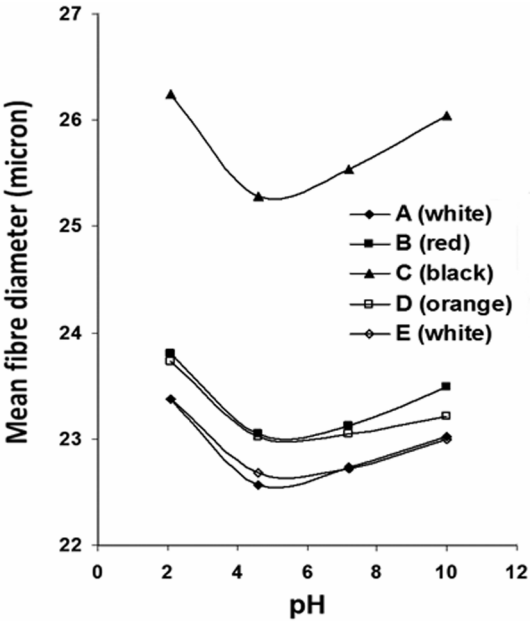

Fig. 4—Variation in dyed Merino fibre diameter as a function of *p*H

the shrinking of the matrix as it loses water when the dyed samples are dried. Reductions in the water content of dyed fibres have already been observed [10]. This is consistent with the dyes occupying hydrophilic absorption sites that presumably are no longer available to contribute to fibre swelling. The fact that the dry and conditioned black fibres give consistently higher mean diameters than the other dyed and undyed fibres lends credence to this hypothesis, because a relatively larger amount of dye (5% o.w.f.) is required to achieve this shade as compared to the lighter shades (1%). Moreover, since the reactive dye is apparently bifunctional, it would have been able to crosslink the matrix protein in the fibre during dyeing while the fibres are in the fully swollen state, and this may have limited contraction of the matrix protein when the fibres are dried.

Secondly, the colour of the fibres might have affected the fibre diameter measurement optical image analysis system of the OFDA 2000 machine, particularly when the fibres are measured in water because the refractive index of water (1.33) is considerably higher than air (1.00) and closer to the refractive index of wool (1.55). The opacity of wool fibres in air illuminated in the OFDA machine relies on the fact that, with a critical angle for light passing from wool into air of about 40°, the illumination geometry (with the fibres illuminated from below with a narrow angle beam) ensures that light that passed into the fibres is unable to escape. However with wool fibres immersed in water, the critical angle is around 59° and in the case with the undyed Lincoln fibres, with their large diameters, it is possible for light to pass through the centres of the fibres. This makes the fibre centres distinctly brighter than the edges of the fibres and completely prevents the fibres from being measured at all. Since the black dyed fibres are relatively opaque, this problem does not arise.

Further work needs to be carried out to investigate and quantify the variations in measured fibre diameters by using dyes of known structure and purity, to measure fibres in media with different refractive indices and to compare the results obtained from the OFDA instrument with those obtained from the more conventional microscopic method of fibre diameter measurement.

It is observed that the fibre diameter measurement with OFDA 2000 could be affected by fibre colour in both dry and aqueous conditions. The darker the colour of fibre samples the greater is the measured diameter.

While these could be due to the artefacts of the optical principle and the measurement process of the apparatus when the fibres are measured in water, it is likely that the observations on dry and conditioned fibres represent real changes in fibre diameter that can be attributed to the physical effects due to the presence of the relatively bulky dye molecules inside the fibres.

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