



Unpublished Report

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Project 2.2 Whiter lightfast wools

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| R4.2.3.2 | Report on assessment of the need for a photostability test for fleece wools | 01/10/09 |

Summary

In initial studies the CSIRO photostability test gave a strong correlation between initial wool yellowness (Y-Z) and the change in yellowness $\Delta(Y-Z)$ after exposure to UVB for 4 hours for fleece wool samples held in disposable spectrophotometer cells. Scaling up the test to measure photostability of larger batches of ~600 IN flock samples from the 2007 drop demonstrated shortcomings in the irradiation hardware, leading to significantly more scatter and outliers than were previously observed. This has necessitated the design and construction of a dedicated photostability test rig for fleece wool samples, currently nearing completion at CSIRO.

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28 September 2009

Introduction

A photostability test method for small samples of fleece wool from the IN flocks was recently developed by CSIRO using disposable spectrophotometer cells [1]. Cells were packed with scoured wool to constant density and its colour (X, Y, Z tristimuli) was measured before and after irradiation with UVB for 4 hours. The change in yellowness $\Delta(Y-Z)$ was measured and plotted against initial yellowness Y-Z.

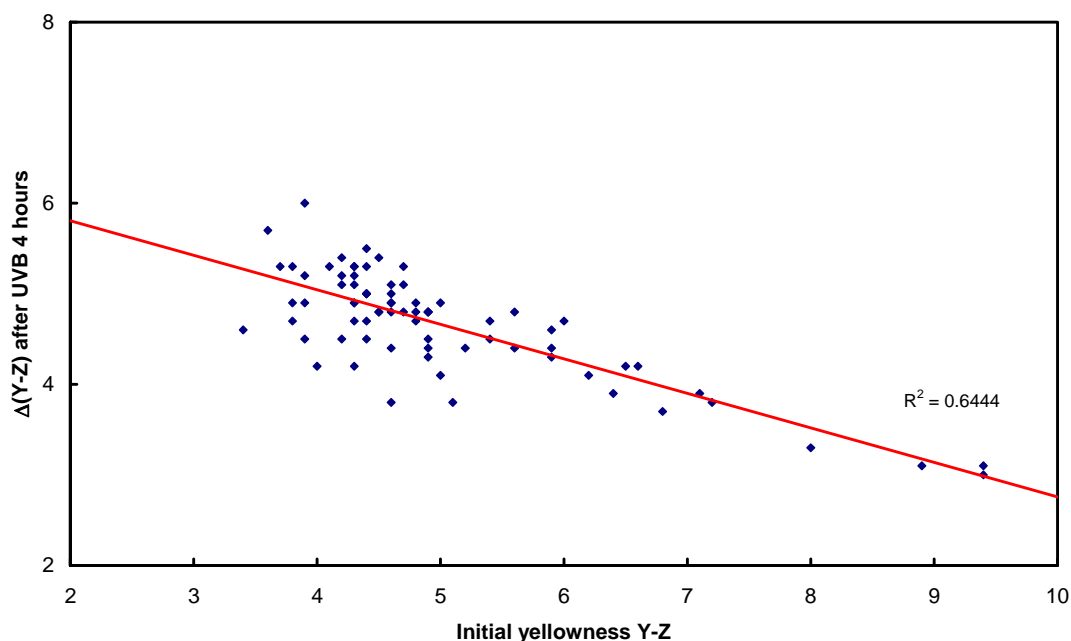


Figure 1 Plot of change in yellowness $\Delta(Y-Z)$ against initial yellowness (Y-Z) for selected IN flock samples irradiated with UVB for 4 hours.

The plot in Figure 1 shows a strong correlation ($R^2=0.64$) between the initial yellowness and the change in colour after exposure to UVB. This result is in agreement with previous work by Lennox and King [2], who showed that whiter wools yellow more rapidly when exposed to UVB and that the ultimate yellowness reaches a limiting value after long exposure times (220 h).

AWTA had previously developed a photostability test for fleece wools using a carded web and this was applied to the 2007 IN samples. However, in contrast to yellowness, analysis of the photostability data showed no genetic variation and a heritability close to zero [3]. Also yellowness was not correlated to photostability, in contrast to expectations based on the results of Lennox and King [2]. It was therefore decided to compare the AWTA and CSIRO photostability data from the Hamilton IN flock, and results are shown in Figure 2.

Clearly the CSIRO test, showing a strong correlation between initial colour and photostability appears to be the most reliable and is in agreement with Lennox and King. One possible shortcoming of the AWTA web method is that fibres in a web are free to move and any movement in the surface fibres after the initial colour measurement and during exposure will affect the results. The final position of some exposed fibres may be beneath the measured surface and some unexposed fibres may be measured, which will

affect the precision and repeatability of measurements. Another problem may be variation in the density of the web, since density is known to affect wool colour measurements.

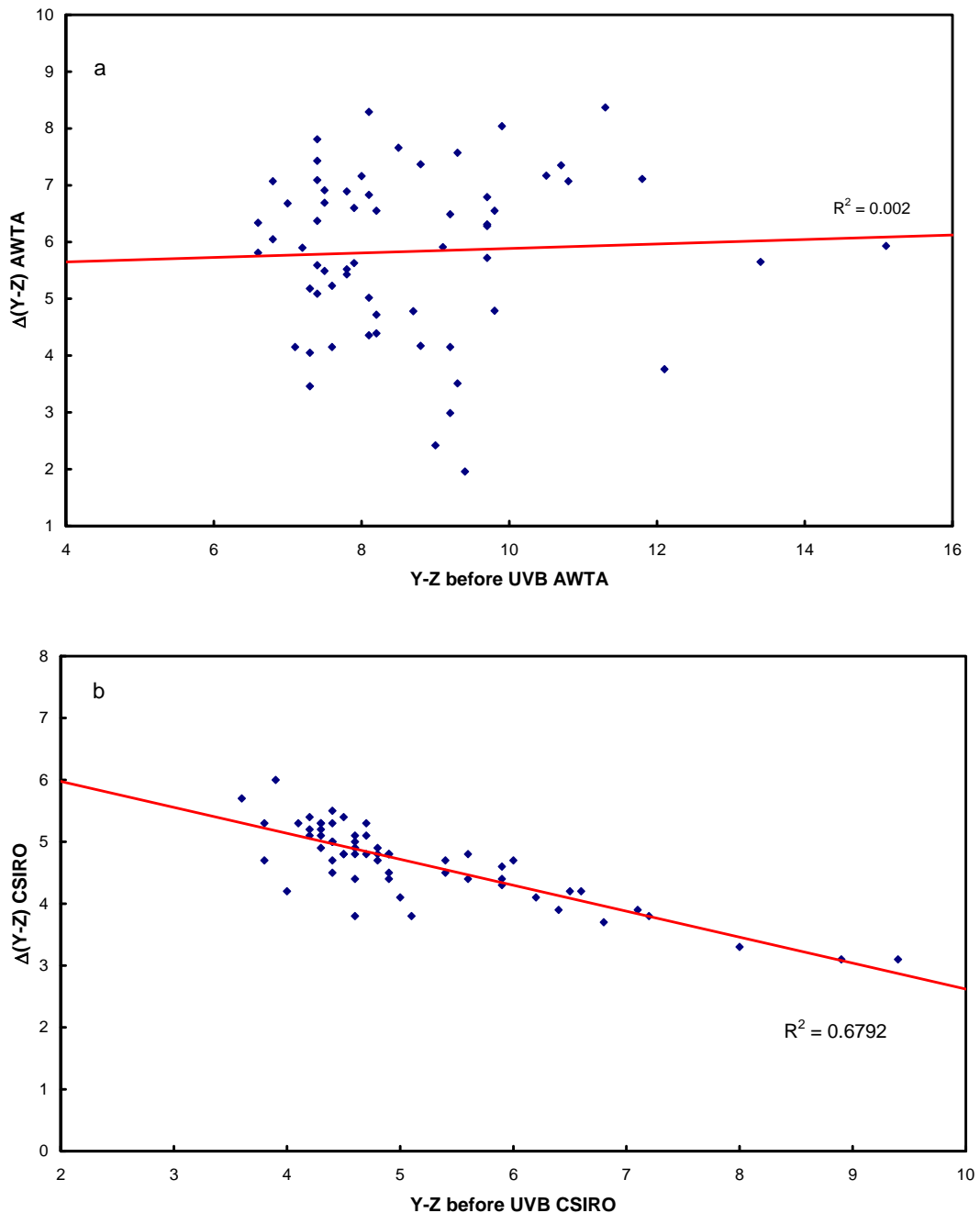


Figure 2 Plots of change in yellowness $\Delta(Y-Z)$ against initial yellowness (Y-Z) for Hamilton IN flock 2007 drop using (a) AWTA photostability test and (b) CSIRO method.

The samples used in the initial CSIRO report were chosen from the available IN samples to represent a wide range of initial yellowness (Y-Z) values and included Hamilton 2007 drop samples. However it can be seen from Figures 1 and 2b that for a particular initial value of Y-Z, a small range of $\Delta(Y-Z)$ values exists, showing that fleece wools having the same initial Y-Z may photoyellow at different rates. This range may be due to both

genetic and/or environmental factors. For example, trace metals are known to increase the rate of protein oxidation by free radicals by catalysis. It has been shown that wool produces hydroxyl radicals [4] and hydrogen peroxide [5] when irradiated with UV and visible wavelengths under wet conditions.

It was therefore decided to repeat photostability measurements from the 2007 drop for those flocks where no tip shearing was conducted, ie. Kirby, Turretfield and Hamilton, a total of 600 samples, using the CSIRO method. The genetic analysis for photostability would then be repeated to assess the heritability of photostability, and determine whether variance in photostability demanded an ongoing test for the IN flock samples in future years.

Experimental

Approximately 0.512 g (+/- 0.005 g) wool samples were evenly compressed into 3.2 cm³ volume (packing density 160kg/m³) in a polymethyl methacrylate (PMMA) disposable cuvette (Brand) and secured with a 10 mm Perspex cube with a 1 mm hole drilled through the centre, as described previously [1]. The packing density was equivalent to the IWTO-56-03 requirement. Dry wool was at ambient temperature and humidity prior to packing.

The photostability test was carried out using a small light box containing a Philips TL20W/12RS (UVB) tube (Figure 3). Sample cuvettes were held in contact with the tube using loops of stretch fabric (Figure 4). Whilst this arrangement was found to be satisfactory for carrying out small numbers of photostability tests for research purposes, it was found to be not well suited for the irradiation of the large numbers of samples required for a standard photostability test method, as discussed below.



Figure 3 UVB lightbox housing Philips TL20W/12RS (UVB) tube. Five black elastic fabric sleeves for holding sample cuvettes in contact with the tube are shown.



Figure 4 Sample cuvettes mounted against the UVB tube.

The colour of wool samples was measured before and after irradiation by placing the cuvettes over the small area view (SAV) aperture (0.8cm x 1.0cm) in the inspection port of a Gretag Macbeth Color-Eye 7000A spectrophotometer. The spectrophotometer was configured with a D65 light source and a 10° collection angle with the spectral component included (SCI). The tristimulus values X , Y and Z were measured by averaging 2 reads and used to calculate the wool yellowness (Y-Z) and change in yellowness $\Delta(Y-Z)$.

Samples from the Kirby and Turretfield flocks were irradiated in large batches for 4 h using this method, and results are reported below.

Results and discussion

Plots of $\Delta(Y-Z)$ against initial Y-Z for Turretfield and Kirby samples from the 2007 drop are shown in Figures 5 and 6. There is a very large difference in R^2 between these plots and the original studies shown in Figures 1 and 2b, showing that there is far more scatter in these Turretfield and Kirby results.

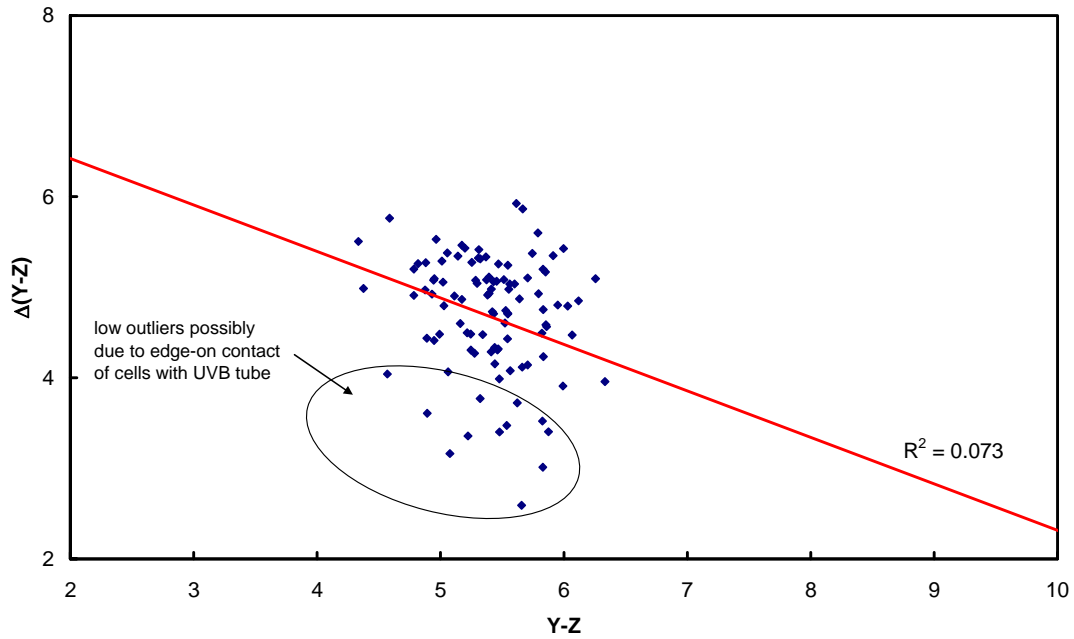


Figure 5 Plots of change in yellowness $\Delta(Y-Z)$ against initial yellowness (Y-Z) for Turretfield IN flock 2007 drop using CSIRO photostability method.

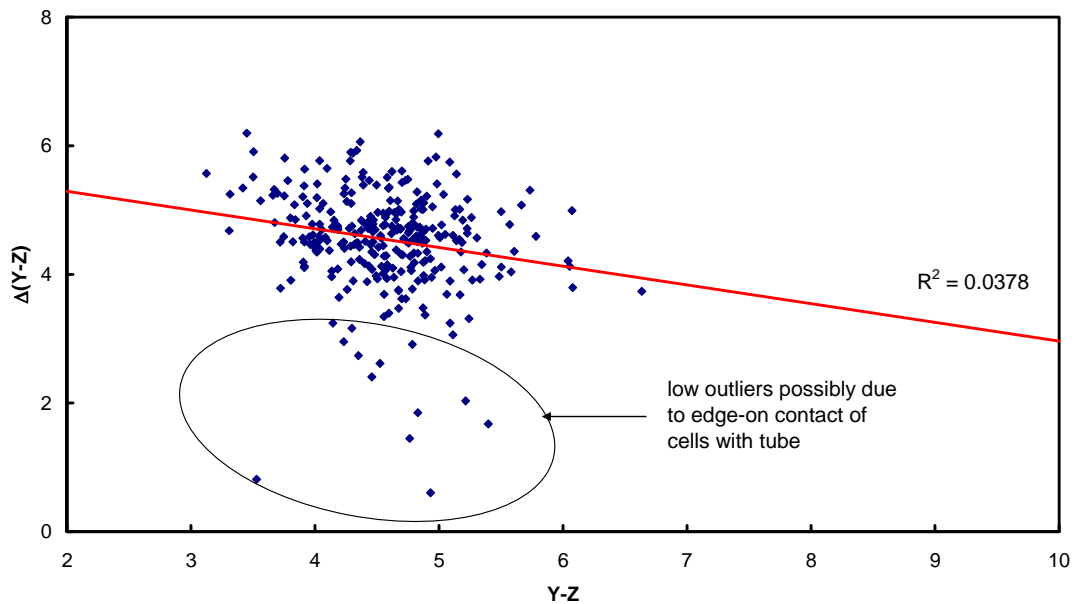


Figure 6 Plots of change in yellowness $\Delta(Y-Z)$ against initial yellowness (Y-Z) for Kirby IN flock 2007 drop using CSIRO photostability method.

There are a number of possible explanations for this, but the two explanations deemed most likely are movement of the sample cells during irradiation, leading to large numbers of low outliers, and possible overheating of the UV tube due to the large numbers of samples irradiated in each batch. These effects are due to the much larger batches of samples irradiated in one cycle that are necessary to expedite the throughput of large numbers of photostability measurements from IN flocks.

Although the samples were placed snugly around the light source, inconsistencies in the final $\Delta(Y-Z)$ results were observed. Inconsistencies may have arisen due to the following observations.

It was difficult to ensure that the cuvette remained in the right plane during irradiation. Some cuvettes had moved so that either some or all of the irradiation face was not in direct path of the lamp. The curvature of the lamp and the flat plane of the cuvettes made it difficult to ensure all samples placed in the sleeve (up to six) remained in the desired position during irradiation. The stability of the cuvette position was only improved when a maximum of four cuvettes were placed in the sleeve which allowed the cuvettes to be balanced around the lamp. However, this was not ideal as it only allowed a maximum of 24 samples to be irradiated at one time and increased the total time necessary to measure the photostability of 600 IN flock samples from the 2007 drop.

Another contributing factor was that the sleeves expanded and became looser during the 4 hour irradiation exposure which allowed some cuvettes to move into an undesired position.

It was sometimes observed after irradiation that a few cells had moved their position relative to the UVB tube from a face-on to an edge-on configuration. Due to this change in geometry, the wool in the edge-on cells was irradiated significantly less, resulting in significant numbers of low outliers. Repeating the photostability measurement on several of these low outliers produced a result in the normal range ($\Delta(Y-Z)$ range 4–6).

It is also possible that larger batches of samples held onto the tube with insulating stretch fabric may have increased the operating temperature of the UV tube, which in turn may have affected UV output.

This situation, particularly the generation of false low $\Delta(Y-Z)$ values, led to suspension of photostability testing and the design and construction of a dedicated UVB irradiator capable of treating 56 samples (7 sets of 8 samples arranged at different levels relative to the UVB tube) in one batch (Figure 7). In this device the cells will be positively located facing the tube and unable to shift position during irradiation. Since it will now be unnecessary to use insulating stretch fabric loops hold sample cells in position, the operating temperature of this tube should remain constant and not affect UVB output. The spectrum and spectral irradiance of the tube will be regularly measured using a spectral analyser.

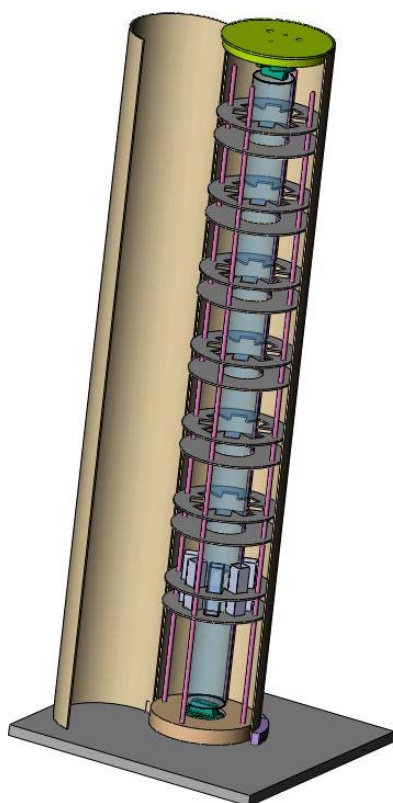


Figure 7 CAD drawing of new photostability test apparatus for fleece wool samples in spectrophotometer cells

The irradiator is currently being assembled at CSIRO (Figure 8) and should be available for operation by October 2009. Due to the uncertainty regarding the data already obtained using the small light box, measurements for Kirby and Turretfield will be repeated using this new device.



Figure 8 New UVB irradiator under construction in CSIRO workshop

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