Australian Government
Cotton Research and Development Corporation

FINAL REPORT 2008

If you are participating in the presentations this year, please provide a written report and a copy of your final report presentation by 31 October.
If not, please provide a written report by 30 September.

Part 1 - Summary Details

Please use your TAB key to complete Parts 1 & 2.

CRDC Project Number: **CTFT17**

Project Title: International inter-laboratory trials to develop standard reference cottons for fibre maturity and fineness.

Project Commencement Date: 01/07/06 **Project Completion Date:** 30/06/08

CRDC Program: 6. Value Chain

Part 2 – Contact Details

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Part 3 – Final Report Guide (due 31 October 2008)

Background

The commonly used Micronaire value for cotton is related to both fibre fineness and maturity. CRDC and the CCC CRC have been funding work at CSIRO Textile and Fibre Technology to develop new technologies to measure fibre fineness and maturity seperately i.e. to overcome the deficiency in the micronaire measurement. One difficulty is that there are no internationally recognised standard cotton samples that can be used for calibrating new instrumentation. Researchers at the USDA and Texas Tech University have been tackling this problem by coordinating the development of a standardised set of cotton samples specifically for this purpose. It is envisaged that this set of well characterised cottons will last for in excess of 10 years as the primary calibration for all other 'new' techniques for measuring cotton fineness and maturity. CSIRO Textile and Fibre Technology has been participating in this trial as a third independent test laboratory.

This project represents a continuation of the previous work undertaken under CRDC funded project CTFT7C.

At a technical level, this project was designed to enable CSIRO to participate in this important scientific initiative to develop international industry wide standard reference cottons for fibre maturity and fineness. As well as being of technical importance, continuation of this project has also had a strong strategic component in (a) being an important vehicle for relationship building with key international cotton organisations and R&D agencies in the area of fibre quality measurement and (b) CSIRO gaining access to the reference cottons so that they could be utilised in the two related fibre quality instrument development projects funded by the Australian cotton industry (through the CRDC and CCC CRC).

Objectives and Extent Achieved.

The following table from the original project application lists the objectives as originally planned.

Obj No.	Objective	Milestone	Performance Indicator	YR 1	YR 2
1	Validate the CSIRO approach for measuring fibre cross-sectional area vs the US approach.	Undertake a comparative trial of the two approaches using shared substrates.	Good correlation between the two approaches analysing shared images of fibre cross-sections.	x	
2	Collect CSIRO data on a small range of cottons	A small number of cottons will be tested using the CSIRO system to obtain average fibre fineness and maturity values for each sample.	Report summarising the data for the cottons tested.	x	
3	Comparison of CSIRO data and US data	For a small number of cotton samples, comparison of CSIRO vs US results for average fibre fineness and maturity will be undertaken	Analysis complete highlighting level of agreement between laboratories.	x	
4	Go/no go decision point regarding continuing the project into the second year.	Based on the outcomes of Objective 3, recommend whether the project needs to continue into the second year.	Recommendation of go/no go decision justified.	x	
5	Resolution of any discrepancies between laboratories	Undertake further analysis, measurements and dialogue to obtain consensus on assigned values	Consensus obtained.		x

Progress Against Objectives:

Objective 1:

As reported in the May 2007 progress report Objective 1 represented the major technical and scientific challenge for the project. As the project progressed it became apparent that this objective was considerably more challenging than first thought and would require more effort to complete than originally planned. Given that this was central to the whole project the

whole focus of the project during Year 1 was to tackle this objective i.e. this objective became the priority task. This was satisfactorily achieved and proved to be a major scientific breakthrough/achievement. Progress in the other lower priority objectives planned for Year 1 was limited.

Objective 2:

A subset of 11 cottons covering the range of fibre maturity and fineness values from the 104 US reference cottons were chosen and all the images for this subset of cottons were collected in Year 1. It was decided not to waste resources analysing these images until the challenges associated with Objective 1 were resolved.

Objective 3:

This objective was not tackled.

Objective 4:

This objective was completed, despite Objective 3 not being tackled. Based on the important and exciting progress with Objective 1, it was recommended that the project proceed to Year 2.

Objective 5:

This objective, which became the focus of the project during the second year of the project, was successfully completed.

Methods

Under the project USDA, Texas Tech University and CSIRO have been collaborating in a three way inter-laboratory trial to develop reference cottons for cotton maturity and fineness. Experimentally this is very detailed and tedious work, carefully preparing cross-sections of cotton fibres and then using optical microscopy and image analysis to directly measure the fibre perimeter and cross-sectional area in order to calculate fibre fineness and maturity. For each sample a large number of individual measurements are necessary to obtain good

representative data for the particular sample. To date Texas Tech University have measured over 100 cottons.

The limited but important CSIRO contribution is to independently (a) validate the ability of an independent laboratory to implement the experimental procedure developed in the US and (b) confirm the results i.e. the assigned fibre maturity and fineness values for a small number of the reference samples. This has utilised CSIRO's skills in fibre manipulation, sample preparation for microscopy, optical microscopy and digital image analysis.

CSIRO have taken the approach of developing its own image analysis software to both save purchase costs and also to act as a truly independent validation/test of the US procedures. In retrospect this decision has proved to be critical to the major advance that CSIRO has been able to make in this project. For each fibre cross-section, the two key measurements are the length of the outside perimeter of the cross-section and secondly the cell wall area in the cross-section. From these two parameters the fibre maturity and fineness are calculated. In the previous related project (CTFT7C) CSIRO were able to confirm the fibre perimeter measurement on individual cotton fibres but were not able to duplicate the cross-sectional cell wall area values obtained in the US. Understanding and resolving the apparent discrepancy in the cell wall area values between the CSIRO and US data has been the primary technical focus of this project.

The major technical breakthrough in the project has related to identifying a major inherent limitation in the resolution of the optical microscopy experimental techniques developed and utilised by researchers at the USDA and Texas Tech University. A new technique using SEM was developed to overcome this limitation. This is described below.

Results

The source of the discrepancy between CSIRO and US cell wall area measurements.

Figure 1 shows schematically a 'typical' fibre cross section. The relevant cotton fibre cross sectional area A_C is the total area inside the outside perimeter of the fibre A_T less the area of the lumen A_L. ie

$$A_C = A_T - A_L$$

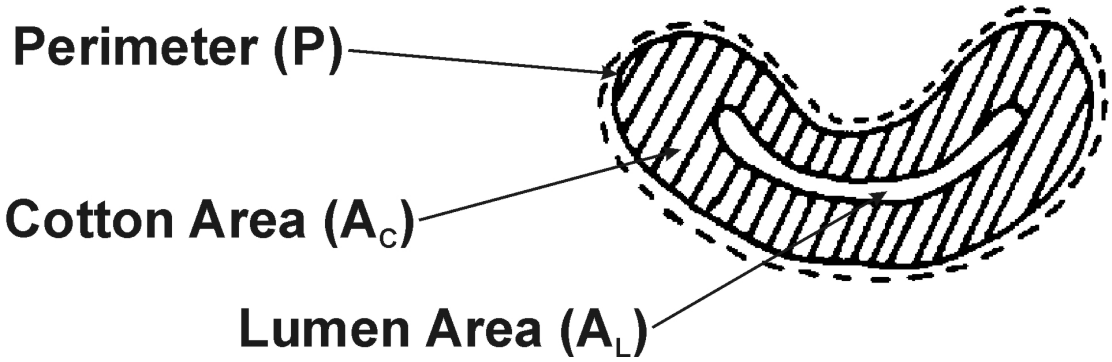

Figure 1. Schematic diagram of a cotton fibre cross section. (The total area A_T is defined to be the sum of the cotton area A_C and the lumen area A_L)

A major technical challenge is that the outside boundary of the fibre and also the lumen boundary are often not well defined as illustrated in Figure 2.

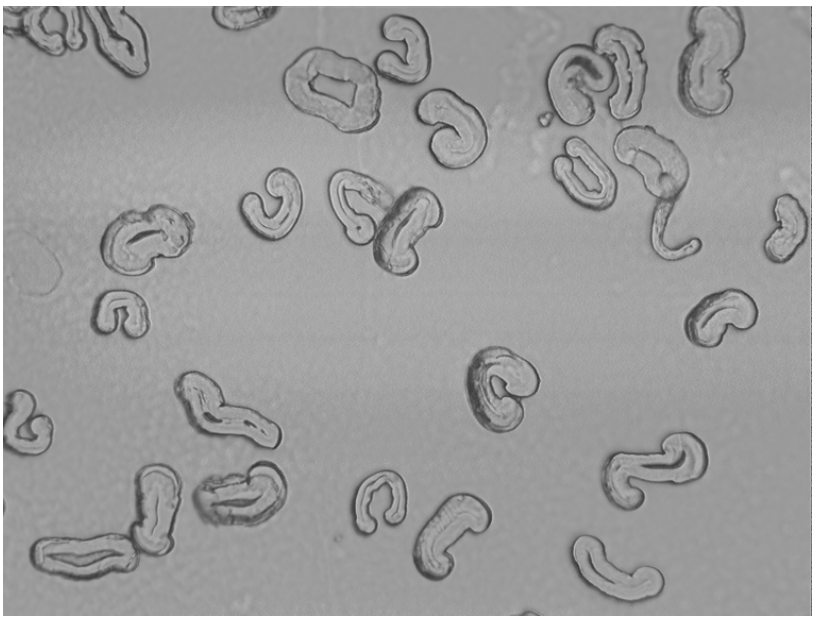

Figure 2. A typical image from the optical microscope.

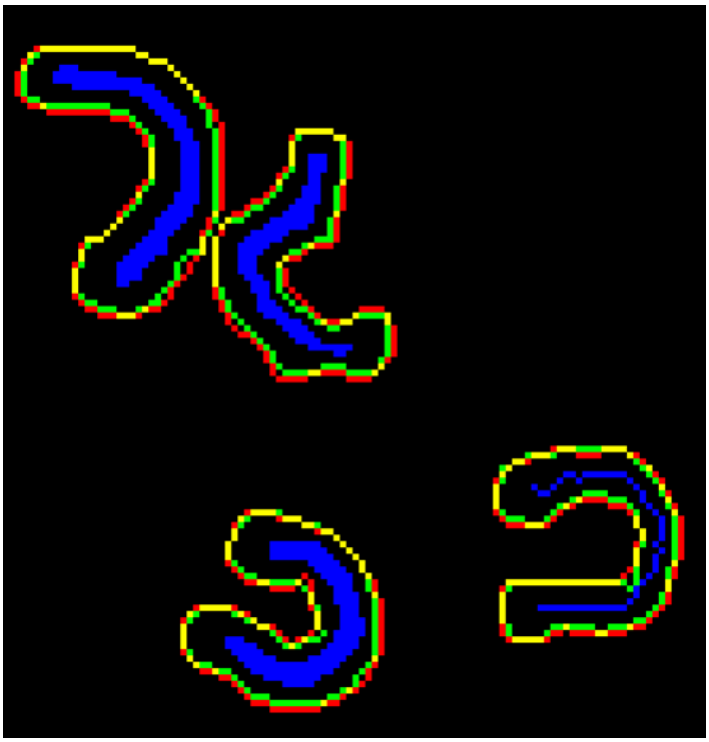

Figure 3. Illustration of the differences between the CSIRO and US software's determination of the outside boundary of a cotton fibre cross section. (CSIRO in green, US in red, and co-incident in yellow.

Figure 3 illustrates the outcome of a comprehensive study comparing the CSIRO and US results. In this figure, the outside edge of the fibre as identified by the US software is depicted in red and the corresponding CSIRO border is in green. Yellow is used to indicate where the outputs from the two systems are coincident. It can be seen that the outside edge of the fibre determined by the CSIRO software often lies one pixel inside the border determined by the US algorithm. A study of many cross-sections has confirmed this effect. Figure 4 shows the quantitative comparison of the CSIRO and US values for the total area A_T. The points in red illustrate the original discrepancy with the CSIRO areas being some 10-15% smaller than the corresponding US values. The points in blue illustrate the effect of removing one layer of pixels from the US results. It can be seen that this has a considerable effect on the relationship in Figure 4, with the CSIRO data now being slightly larger than the 'modified' US values. (This might be expected, given the observation as illustrated in Figure 3, that the CSIRO boundary is generally but not always inside the US boundary). The key finding is that moving the boundary by only one pixel causes a systematic shift in the measured area of 10-15%.

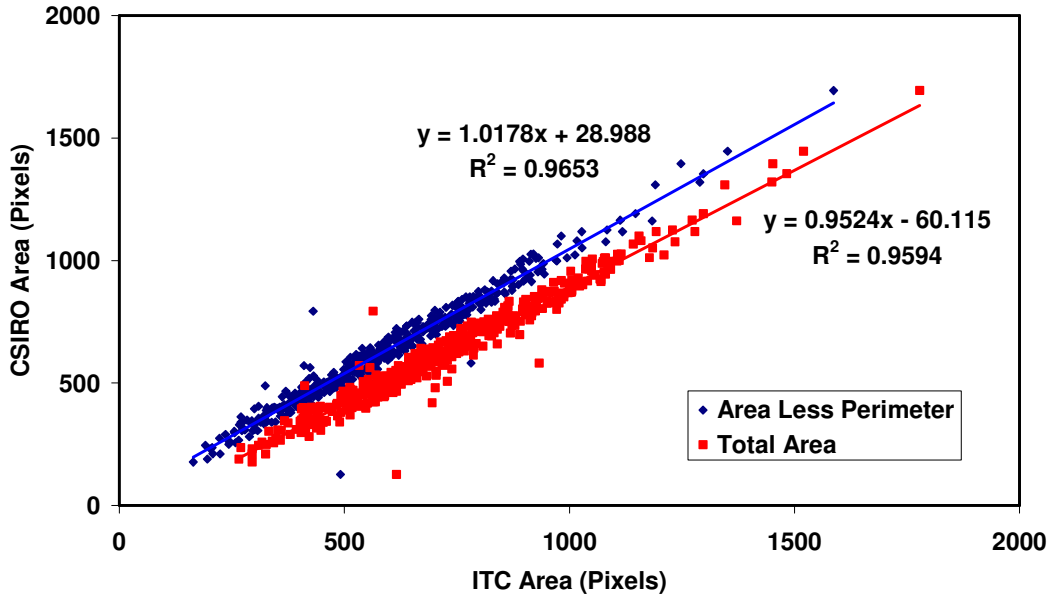

Figure 4. Comparison of CSIRO and Texas Tech University (ITC) estimates of the total area A_T. (In Figure 1, $A_T = A_C + A_L$)

Some differences in determining the area of the lumen A_L have also been identified which may also contribute a smaller but significant systematic difference between the reported CSIRO and US data.

Given the resolution of the original images it is a moot point as to which is the 'correct' answer, however the work has highlighted a potential new significant systematic uncertainty in the results. This is important given that these cottons and their reported values are to be used globally as the central reference point for calibrating other cotton fibre fineness and maturity measurement systems (including Siromat and Cottonscan).

New approaches to correct cell wall area measurements.

Within the limited scope of this project a small experimental program was undertaken to observe the prepared slides of cotton cross-sections under the scanning electron microscope (SEM) rather than under the optical microscope. The SEM is known to have much higher resolution than the optical microscope and hence this approach should avoid the problems noted above with the optical microscopy. Figure 5 illustrates a typical SEM image. The increased resolution compared to the image is apparent. Figure 6 shows the comparison of

cell wall area values obtained from the SEM versus the CSIRO optical microscope values for two different cotton samples. Note the systematic differences between the two values with the SEM value being (around) six percent smaller than the values obtained by CSIRO under the optical microscope. Given that the CSIRO optical microscopy values are already observed to be some 20 to 40% smaller than the equivalent US values this is indeed an important result. Technical difficulties in locating images in the SEM mean it is not feasible to reproduce the imaging of all cross-sections in the SEM. However from this SEM work it is possible to estimate a suitable correction factor to apply to the CSIRO optical microscope data and more importantly to the US data.

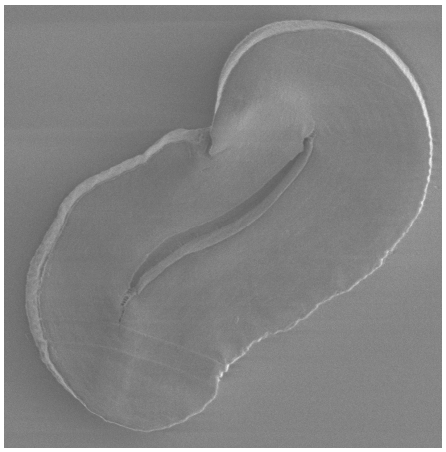

Figure 5. A typical image from the SEM.

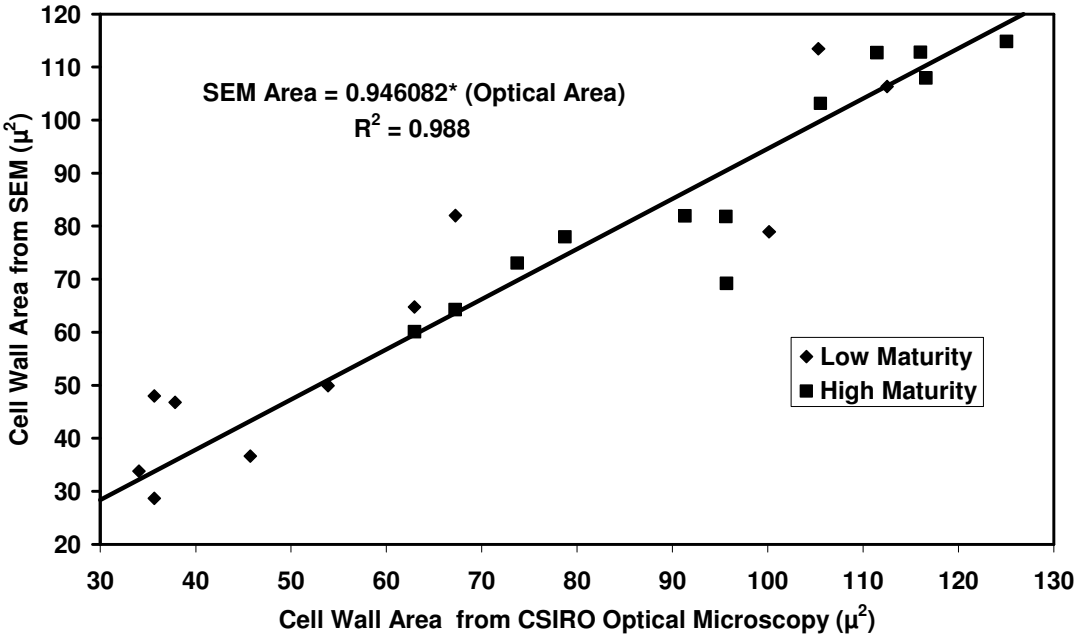

Figure 6. Comparison of the cell wall area measured by the SEM relative to the value obtained by optical microscopy.

Applications to Cottonscan and SiroMat.

As noted above, one of the strategic focuses of the project was to gain access to the reference cottons for use in calibrating the Cottonscan and Siromat. Figure 7 shows the use of these cottons in the Cottonscan project. One would have anticipated that the observed slope on this graph of 1.77 would represent the density of cellulose namely approximately 1.52 g/cc. The current work would suggest that the assigned values from the image analysis work are over estimated and an appropriate correction to the data on the horizontal axis would reduce the observed slope in Figure 7.

These cottons have also been used in a similar way in the Siromat project.

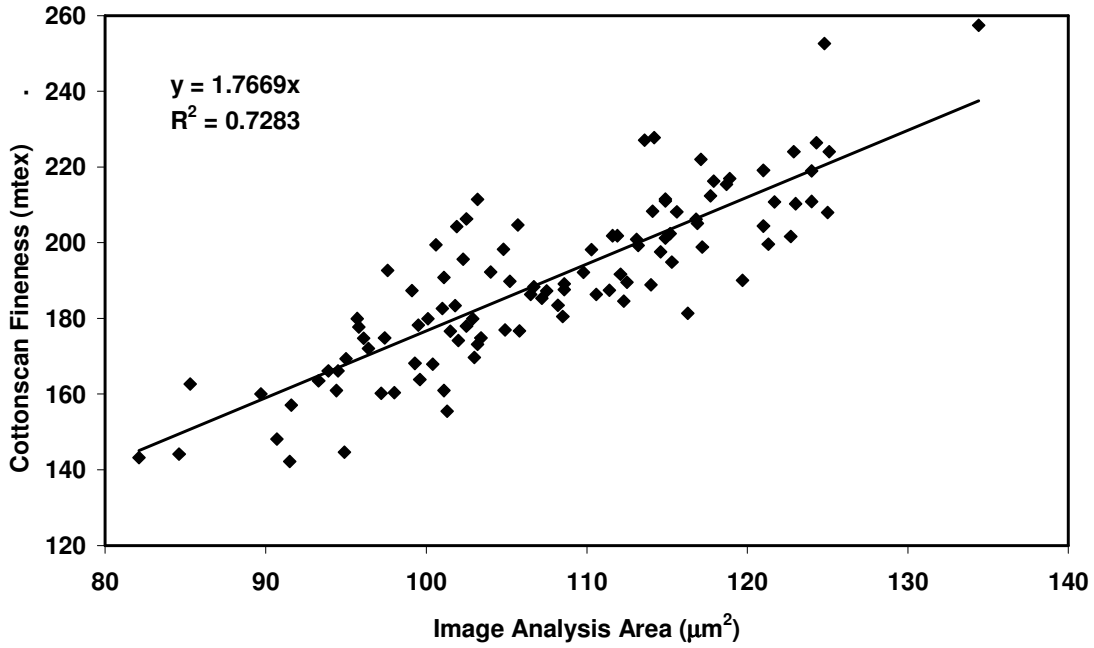

Figure 7. Application of the reference cottons to check the calibration of the Cottonscan instrument.

Outcomes

This small project has been extremely productive and successful. Whilst the project has taken a slightly different path to that originally planned, it has achieved the broad goals of the project and completed most specific objectives. The major technical outcome is that a significant systematic error in the assigned values of fibre maturity and fineness of an

important set of 104 reference cottons has been identified. The project has also facilitated a close working relationship between two key research groups in the US in the area of fibre quality measurement.

Conclusion

This relatively small project has been extremely productive and successful on a number of fronts as follows:

(a) Technical progress has been significant in an important aspect of cotton metrology.
(b) Strategically the project has indeed contributed to building the reputation of the CSIRO group internationally, built links with key international researchers and provided valuable access to the reference cottons for other project initiatives.

Extension Opportunities

It may also be possible to interact further with the US collaborators to formally revise the assigned values of maturity and fineness for the set of reference cottons. Given the nature of the project no direct extension activities to the Australian cotton industry are appropriate.

Given the importance of the work to the international cotton community, it may be beneficial to give a report on the outcomes should a suitable occasion arise i.e. a Beltwide meeting or Bremen 2010, particularly if attendance at such a meeting was already planned for other reasons.

This project has been completed and a major scientific paper has been submitted for publication. Two further scientific papers may be possible from this project but this would require further project resources.

Publications

Higgerson, G.J., Pate, M. and Naylor, G.R.S. 'Determination of Cotton Fiber Maturity and Linear Density (Fineness) by examination of Fiber Cross-sections. Part 1: An Assessment of the Limitations of Optical Microscopy'. Text. Res. J. (submitted in Aug 2008) (A copy of this paper has been previously supplied to CRDC via Dallas Gibb)

Part 4 – Final Report Executive Summary

The commonly used Micronaire value for cotton is related to both fibre fineness and maturity. CRDC and the CCC CRC have been funding work at CSIRO Textile and Fibre Technology to develop new technologies to measure fibre fineness and maturity seperately i.e. to overcome the deficiency in the micronaire measurement. One difficulty is that there are no internationally-recognised standard cotton samples that can be used for calibrating new instrumentation. Researchers at the USDA and Texas Tech University have tackled this problem by coordinating the development of a standardised set of cotton samples specifically for this purpose.

The broad aim of the project is that CSIRO would collaborate with the two US groups and act as as a third independent test laboratory to validate the procedures and results obtained in the US. This is particularly important as it is envisaged that the set of well characterised cottons with their assigned values of fibre maturity and fineness will last for in excess of 10 years as the primary calibration internationally for all other 'new' techniques for measuring cotton fineness and maturity.

The project has been extremely successful. Working in collaboration with the US groups the Australian initiative has identified a significant systematic error in the results obtained by the US groups. The cause of the error has been identified. It relates to the limited resolution of the techniques being employed to image the cotton fibres. Further an alternative approach has been identified to overcome this problem.