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INSTRUMENTATION FOR RAPID DIRECT MEASUREMENT OF COTTON FIBRE FINENESS AND MATURITY

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Abstract

A major problem associated with cotton fiber quality is related to the control and management of cotton fiber maturity and fineness from breeding through spinning. At the center of the problem is the absence of rapid and accurate measures for cotton fiber maturity and fineness. The problem exists despite the very significant impact that fiber maturity and fineness can have in the spinning mill and on the quality of fabric. Confounding control of fiber maturity and fineness is the widely accepted and used Micronaire test method, an airflow technique that measures a combination of fiber maturity (fiber wall thickening) and fiber fineness (weight per unit length). A consequence of using the Micronaire is that cotton can be classified inappropriately. For example, fine mature cotton can have the same Micronaire value as coarse immature cotton. Thus, there is a need for a new measurement technique to separate these. Recognition of fiber quality is of particular importance to the Australian cotton industry where varieties of fine, mature cotton have been wrongfully discounted because low Micronaire values were taken as indicating immature cotton. Introduced in this paper are two prototype instruments, developed by CSIRO Textile and Fibre Technology in conjunction with the Australian Cotton Industry, that measure cotton fiber maturity and fineness directly and rapidly.

Background

Whereas the lateral expansion (intrinsic fineness) and elongation (length) of the single cell that becomes the cotton fiber are largely controlled by the genetics of a cotton plant, the laying down of cellulose in the cell wall is affected by environmental factors. Early termination is the most common cause of immature fiber although in most situations not all bolls on a plant are affected. When the cotton plant loses its leaves, from water stress, premature defoliation or cold weather, fiber development is stopped within two to three days (Walhood 1960). Moreover, while an individual boll may be just starting the fiber elongation phase of its development other bolls on the plant will have fibers well into the wall thickening phase. Hence, at any one time there will be a range of bolls on a plant at different stages of maturity.

As a result of environmental influences there is a large degree of variation in fiber maturity between cottons. This variation is also seen in the intrinsic fineness (perimeter) of cotton even though in the application of the Micronaire test it is assumed this is constant for samples of similar genetic background. Together, Figures 1 and 2 illustrate the folly of this assumption. Figure 1 shows Micronaire values between 3.8 and 4.9 for 35 Australian Upland (*Gossypium hirsutum*) cottons, and therefore of similar genetic background, harvested in the same year. The Micronaire values are plotted against direct measurements of maturity, described in terms of the degree of fiber wall thickening. The degree of thickening is defined as the ratio of the cross-section area of the fiber wall to the area of a circle of the same perimeter as the cross-section and is widely regarded as the most satisfactory expression of maturity (Lord and Heap 1988). Although a positive trend between Micronaire and maturity is seen the 'best fit' equation describes less than 22 percent of the variation between these parameters for this set of samples. Restricting the set of cotton samples to those in the 'premium' Micronaire range, i.e., between 3.8 and 4.2, reduces the relationship further. Figure 2 shows Micronaire values for the same set of cotton samples plotted against direct measurements of their intrinsic fiber fineness. Because perimeter is largely determined by the plant's genetic make-up it stands well as an intrinsic measure of fiber fineness. Figure 2 shows no relationship between Micronaire and intrinsic fineness. Even allowing for the relative imprecision of direct measurements (400 sections were measured per sample) the difficulty in separating the effects of maturity and fineness by the Micronaire method is apparent from these figures.

The Micronaire method was developed over 50 years ago and was originally thought to give values that indicated gravimetric fineness (mass per unit length). The principle of operation is that the resistance to airflow through a randomized compressed plug of fibers of known mass is measured. From the established theoretical relationship between the measured airflow resistance and the surface area per unit volume of the fiber a value for fiber fineness could be calculated for solid fibers with regular cross sectional shape. However, cotton fibers have a residual protoplasm called the lumen and are therefore hollow. Cotton fibers also have irregular cross sectional shapes, which combined with the presence of the lumen complicate the interpretation and usefulness of the Micronaire value. Analysis by Lord (1956) revealed that the Micronaire value (X) actually measured a composite of fineness (H) and maturity (M); as expressed by Equation (1) below:

$$MH = 3.86*X^2 + 18.16X + 13 \qquad (1)$$

The limitations of the Micronaire are readily apparent from this equation, namely that as the Micronaire value is related to the product of fiber fineness and maturity, its interpretation is ambiguous, i.e., a coarse immature sample and a finer more mature sample can both have the same Micronaire value. Furthermore, where average Micronaire values of two cottons are the same and the corresponding maturity and fineness co-ordinates are the same the situation can arise where one cotton sample has a greater proportion of immature fibers than the other. The cotton with the greater proportion of immature fibers will have different processing properties and thus yarn and fabric produced from them will be of different quality. For example, shiny or dye resist neps are associated with very immature fibers within a sample that are not measured by the Micronaire or other methods that give average values only.

From the spinners' perspective, both fiber maturity and fineness are key parameters with sometimes opposing effects on mill productivity and yarn and fabric quality. For example, yarn is specified in terms of its weight per unit length and fiber fineness determines the number of fibers in a given yarn cross section. The use of finer fibers increases the number of fibers in the cross section of a given yarn, which improves spinning efficiency and yarn evenness. Equally cotton fiber maturity is an extremely important property to spinners and fabric manufacturers because it determines how well fibers will process both from a chemical and a physical perspective. Immature fiber, that is fibers with little or no fiber wall thickening, are associated with the formation of small entanglements called neps, irregularities in processed fiber assemblies including finished yarns, non-uniform dyeing of fabrics and decreased processing efficiency.

While knowledge of cotton fiber maturity and fineness has always been important with regard to managing and avoiding the above problems, there is an increasing need for faster and more accurate measurements. The reasons for this are:

- Faster automated processing machinery which to be profitable needs to operate efficiently.
- Increasing demand for higher quality yarns and fabrics.
- Smaller mill blending lines means that bales of immature cotton cannot be blended out.
- Increasing competition from synthetic fibers whose properties are better specified and more consistent.

Discussion

Direct and Indirect Test Methods

There are a number of methods for measuring fiber maturity and fineness though no one method is able to do so both accurately and with the speed requirement for classing purposes. The methods currently used range from direct methods of measurement, e.g., measuring fiber wall thickness and perimeter from magnified cross-sections, to indirect methods that indicate or predict maturity or fineness relative to some other fiber parameter.

While being theoretically more accurate, direct methods have historically been limited by the considerable experimental skill required for sample preparation and the experimental error arising from the limited numbers of fibers that can be practically measured. Furthermore, these tests often require the operator to make subjective assessments on the form of the fiber, another potential source of error. Despite these limitations, direct or reference methods have generally been used primarily to calibrate faster indirect methods.

Indirect methods are favored by cotton marketers and processors because they provide fast results. However, effects of other fiber features tend to bias the results. The Micronaire test method is an example of an indirect method because it measures the specific surface area of the fiber. Fiber maturity and fineness are functions of specific surface area and either can be calculated from the Micronaire result after the other is known.

Other indirect methods include the 'Shirley' Fineness and Maturity Tester (FMT), Near Infrared Reflectance spectroscopy (NIR), the Uster Advanced Fiber Information System (AFIS) maturity and fineness module and the Uster 'Spectrum' High Volume Instrument (HVI). Of these the most successful has been the 'Shirley' FMT, which is calibrated using the British Standard tests for fiber maturity by the swelling in concentrated sodium hydroxide method and fiber fineness (linear density) by the cut and weigh method. The FMT represented an attempt at resolving the ambiguity of the Micronaire value. The FMT measures the resistance to airflow through a porous plug at two different specimen compressions. The two compression ratios give rise to two estimates of specific surface, the differences between which vary with fiber maturity (Hertel and Craven 1951). Table 1 lists the indirect test methods mentioned here and the reference methods with which they were originally calibrated, or as in the case of NIR calibration methods that have been attempted.

It is not the purpose of this paper to comment on the specific shortcomings of each indirect test method for fiber maturity and fineness. However, it is pertinent to comment that test biases and the practicalities of each method aside, the largest obstacle in the development and application of a successful indirect test method has been the absence of rapid, accurate and precise reference measurements of fiber maturity and fineness. In all cases the absence of a direct and rapid reference method has restricted their potential as methods for acceptance testing.

The Commonwealth Scientific Industrial Research Organization (CSIRO) Division of Textile and Fibre Technology in conjunction with The Australian Cotton Industry is developing two new image analysis based technologies that automate and make more objective direct standards for measuring fiber maturity (SiroMat) and fineness (Cottonscan). In the case of SiroMat the standard method for maturity by polarized light microscopy is automated and in the case of Cottonscan the standard method for fineness by linear density by the cut and weigh method is automated. Making these methods automatic means that the objectivity, precision and speed of otherwise accurate test methods is improved.

SiroMat

Polarized light microscopy is a technique that has been used extensively in textile and industrial fiber identification, particularly for fibers that exhibit birefringence, i.e., fibers that behave like a uni-axial optical crystal. The optical axis in birefringent fibers is usually parallel along the fiber axis with the refractive index being dependent upon the plane of polarization of the incident light. When plane polarized light is transmitted through a birefringent object the light ray is split into two mutually perpendicular vibrating fast and slow rays, which propagate through the object at two different speeds. Upon emerging from the object a phase difference occurs between the fast and slow rays. When recombined into a single ray by passage through a second polarizer (analyzer) the rays interfere with each other, which in turn create different interference colors that highlight crystalline aspects of the specimen.

A standard test for determining the maturity of cotton fibers based on the interference colors produced when cotton fibers are viewed under polarized light microscopy has existed for many years. The method has previously been overlooked because classing the fibers on the basis of color was subjective and having an operator manually count fibers was too slow. For the test the operator had to make an arbitrary assessment of the colors assumed by the fibers and this subjectivity contributed to large discrepancies in the results from different laboratories. The Standard Method (ASTM 1980) in fact warns against using the method for acceptance testing because "laboratory precision can be poor." Furthermore, the test is too slow for routine test applications both in terms of specimen preparation and test time.

In addition it has been thought that the method was biased by fiber fineness (Lord and Heap 1988) or, by implication, the path length of light through the fiber. A recent survey of the interference colors assumed by different cottons by Gordon and Phair (2002) showed that there was no difference in color on the basis of genetic origin or intrinsic fineness. Three different cotton species were included in the survey and thus wide ranges of cross-sectional parameters (cross-sectional wall area and perimeter) were represented. Individual fibers were selected on the basis of the color classification by Grimes (1945), i.e., blue/orange for immature fibers and bright yellow for mature fibers. Each fiber was then photographed as a longitudinal section using a digital color camera before being sectioned to obtain cross-section wall area, perimeter and degree of thickening values. Interference colors were compared on the basis of their hue in a Hue Saturation and Intensity (HIS) digital color model. The comparison showed that the interference colors transmitted by a fiber related directly to a prescribed range of values for degree of fiber wall thickening, and were not co-dependent upon fiber perimeter or cross-sectional area as previously thought. Figure 3 shows the degree of thickening and cross-section wall area results for individual immature and mature fibers, as defined by the polarized light microscopy test, from three species of cotton; *Gossypium hirsutum*, *G. barbadense* and *G. arboretum*. The interference colors of immature and mature fibers alike did not differ between species despite gross differences in cross-section dimensions, i.e., cross-section area and perimeter.

Color digital cameras, color image analysis software and higher powered computers have made automation of the polarized microscopy test viable and allow test times of less than two minutes per sample to be achieved. Moreover, the sample does not require conditioning before testing. Thus, the SiroMat method determines fiber maturity based on the colors fibers assume when viewed under a polarized light microscope set up according to the ASTM standard. Cotton fibers are automatically scanned and analyzed meaning that selection of fibers or fiber sections and interpretation of their color is no longer subject to operator interpretation. As well as measuring average fiber maturity the method is also able to measure the distribution of mature and immature fibers in a sample. Figure 4 shows the CSIRO incarnation of the polarized light microscopy test and Figure 5 shows the relationship between SiroMat results and maturity ratio as measured by the 'Shirley' FMT.

Cottonscan

The Cottonscan instrument evolved from preliminary experiments with the Sirolan-Laserscan, a technology that has become the industry standard for measuring wool fiber diameter. The original principle was to first prepare a known mass of fiber snippets each of fixed length (approximately 2 mm) using the Sirolan-Laserscan guillotine technology and the aligned fiber beard normally prepared for an HVI strength test. The Sirolan-Lasercan was then used in a novel mode of operation to count the number fibers in the sample mass. This gives a direct measurement of the average weight per unit length of the fiber snippets in the sample, i.e., the fineness of the sample (Naylor and Sambell 1999 and Naylor 2001). Further combining this measurement with an independently measured Micronaire value (from a HVI) the average fiber maturity can be calculated using Lord's well established empirical relationship between Micronaire, maturity ratio and fineness (Lord 1956).

The current Cottonscan instrument shown in Figure 6, is a computer controlled implementation of the direct gravimetric determination of fiber fineness i.e. the total fiber length and weight of a prepared sample of fiber snippets is determined to yield the average weight per unit length. To this extent the invention is similar to that described by Naylor and Sambell (1999) and Naylor (2001). However in the current prototype instrument, rather than counting fiber snippets one by one as done previously in the Sirolan-Laserscan, a large number of fiber snippets are photographed in one image. Modern computer image analysis techniques are then used to determine the total length of the fiber snippets in the image. This significantly increases the potential speed of operation of the instrument. For example the current prototype operates at about 90 seconds per measurement and it is hoped that further optimization will allow a measurement time of 30 seconds.

Figures 7 and 8 illustrate typical results from the Cottonscan. In these graphs the results obtained from the Cottonscan for the average fineness and maturity values for a set of 6 well blended cotton samples are in good agreement with results obtained from FMT measurements.

The primary differences between the SiroMat and the Cottonscan are that SiroMat measures maturity of individual fibers directly and so can give information about the population and distribution of maturity values in a sample whereas Cottonscan measures average fiber fineness and then infers an average maturity value from an existing empirical relationship. In terms of operational capabilities the SiroMat approach is slower than Cottonscan and is focused on being a tool in quality assurance laboratories rather than the HVI market. On the other hand, the Cottonscan measurement time is within HVI analysis times and as such effort is focused on incorporating this technology within HVI lines.

Conclusion

CSIRO and the Australian Cotton Industry are currently developing two new instrument technologies that automate and make more objective direct standards for measuring fiber maturity (SiroMat) and fineness (Cottonscan). In the case of SiroMat the standard method for maturity by polarized light microscopy is automated and in the case of Cottonscan the standard method for fineness by linear density by the cut and weigh method is automated. Making these methods automatic means that the objectivity, precision and speed of otherwise accurate test methods is improved. Reported here are preliminary data from these methods that demonstrates their potential value to the cotton industry world wide.

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References

A.S.T.M. Designation D1442-80, Standard Test Method for Maturity of Cotton Fibers, Sodium Hydroxide Swelling and Polarized Light Procedures, (1980).

Gordon, S. G. and Phair, N. L., Unpublished data, CSIRO Textile and Fibre Technology, (2002).

Grimes, M. A., Polarized Light – Preferred for Maturity Tests, *Textile World*, **95:** 196-201 (1945).

Hertel, K. L. and Craven, C. J., Cotton Fineness and Immaturity as Measured by the Arealometer, *Textile Res. J.*, 21: 765-774 (1951).

Lord, E., Air Through Plugs of Textile Fibres, Part II, The Micronaire Test for Cotton, *J. Textile Inst.*, 47: T17-T47 (1956).

Lord, E. and Heap, S. A., The Origin and Assessment of Cotton Fibre Maturity, International Institute of for Cotton, 40 pp. (1988).

Naylor, G.R.S. and Sambell J. Measuring Cotton Fineness Independently of Maturity Using the Sirolan-Laserscan. Beltwide Cotton Conference, 1, 679 (1999).

Naylor, G.R.S. Cotton Maturity and Fineness Measurement using the Sirolan-Laserscan. . Beltwide Cotton Conferences (2001).

Walhood, V. T., Growth in Immature Bolls After defoliation, *Proc.* Beltwide Cotton Physiology and Defoliation Conf., 14 – 20 (1960).

Table 1. Indirect Methods of Measuring Fiber Maturity and Fineness.

Test Method	Calibration	Industry Use
'Shirley' FMT	British Standard test methods: • Fiber maturity by swelling in caustic soda method. • Fiber fineness by cut and weigh method.	Quality control labs in spinning mills and some classing houses
NIR	• Cross-section measurements of maturity and fineness • Micronaire • Causticaire • FMT maturity and fineness	Research only
Uster AFIS Fineness and Maturity Module	• Cross-section measurements of maturity and fineness • Micronaire	Research only
Uster 'Spectrum'	• Micronaire • HVI Strength • HVI Extension	Quality control labs in spinning mills and classing houses

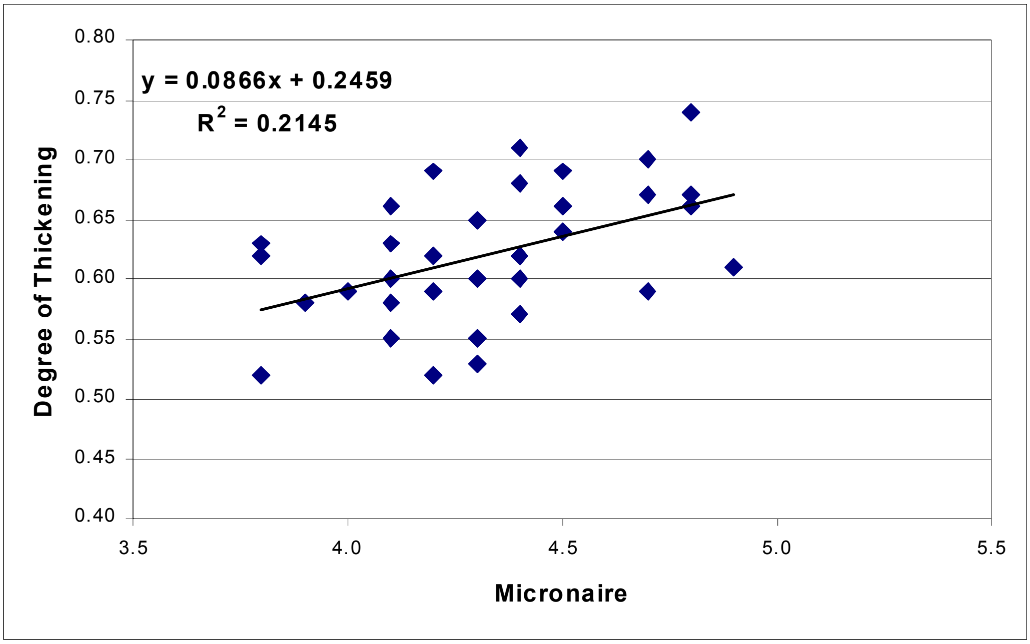

Figure 1. The relationship between Micronaire and degree of thickening results for 35 Australian Upland cottons harvested in the same year.

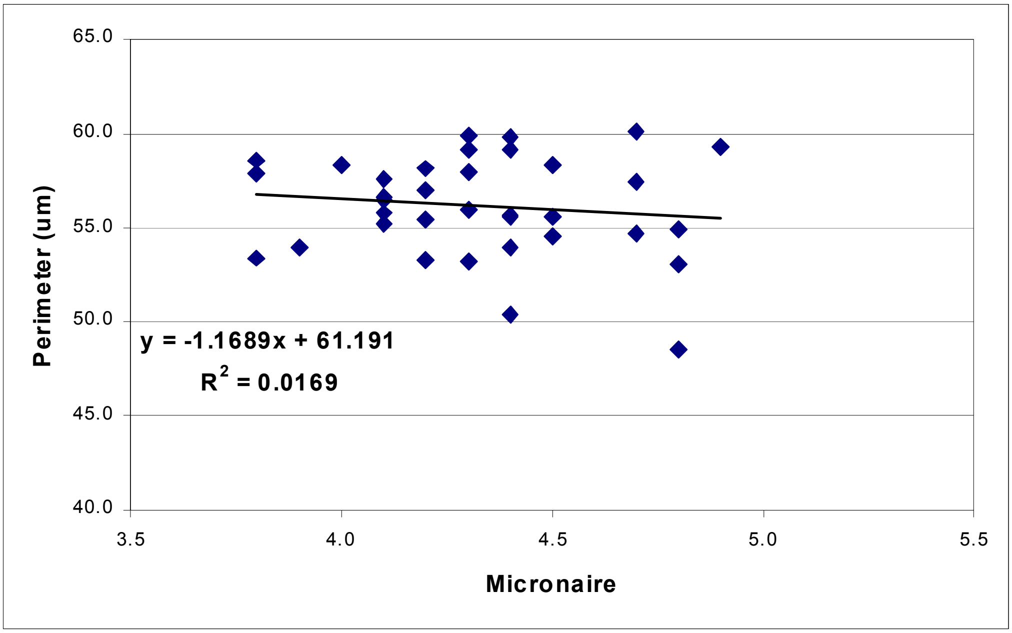

Figure 2. The relationship between Micronaire and perimeter results for 35 Australian Upland cottons harvested in the same year.

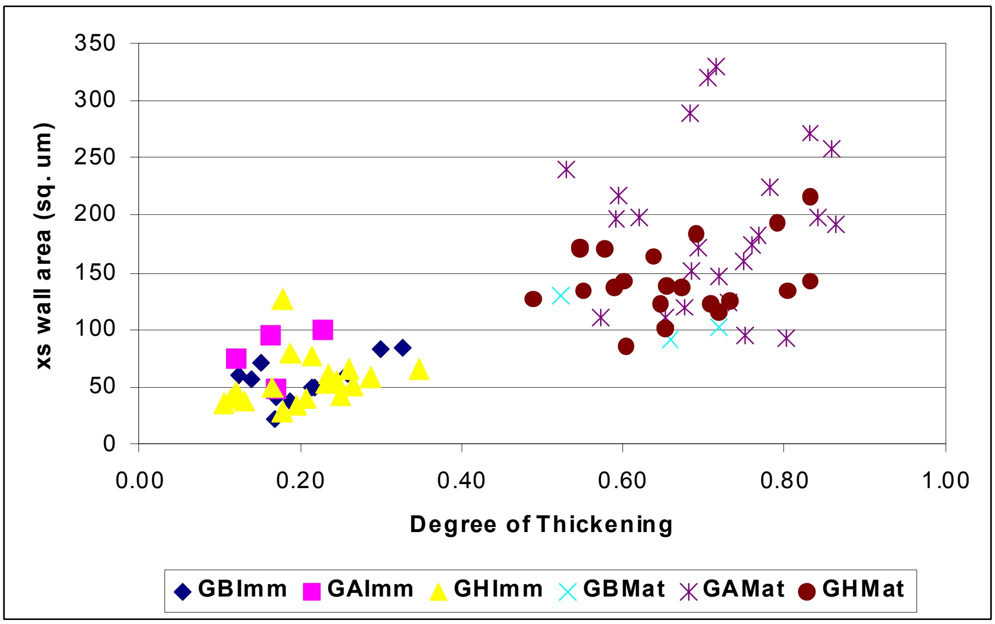

Figure 3. Immature and mature fibers segregated on the basis of the polarized light microscopy test plotted to show the relationship between interference colors, degree of thickening and cross-section wall area.

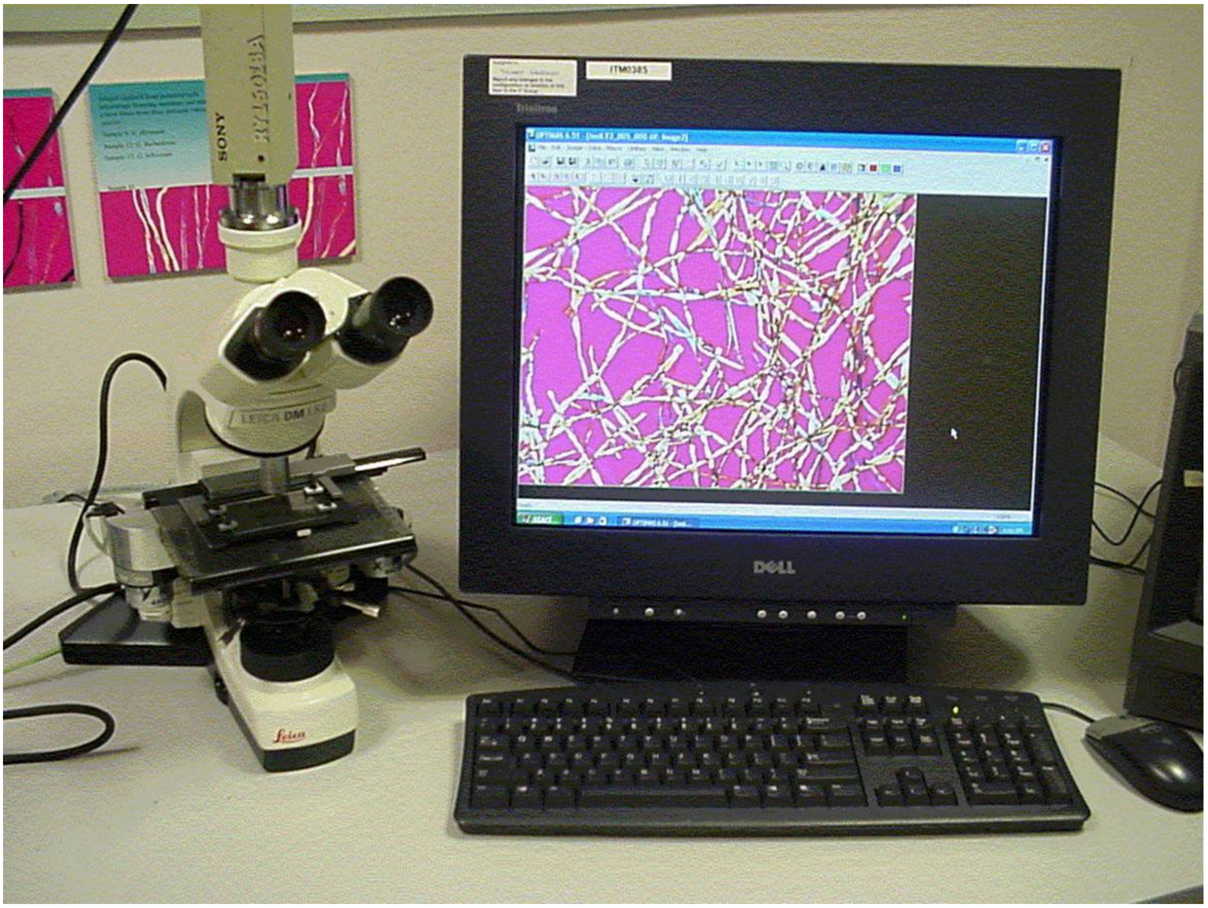

Figure 4. The SiroMat instrument.

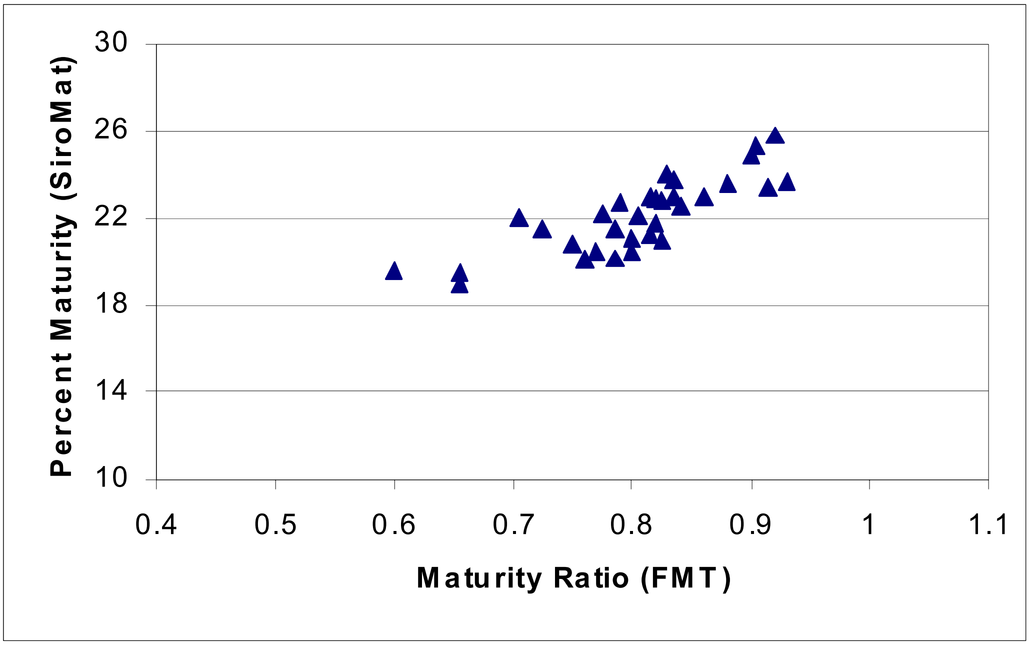

Figure 5. SiroMat 'percent' maturity results (1 x 2 mg replicate) versus maturity ratio as measured by the 'Shirley' FMT (average of 2 x 4.00 g replicates).

Figure 6. The Cottonscan instrument.

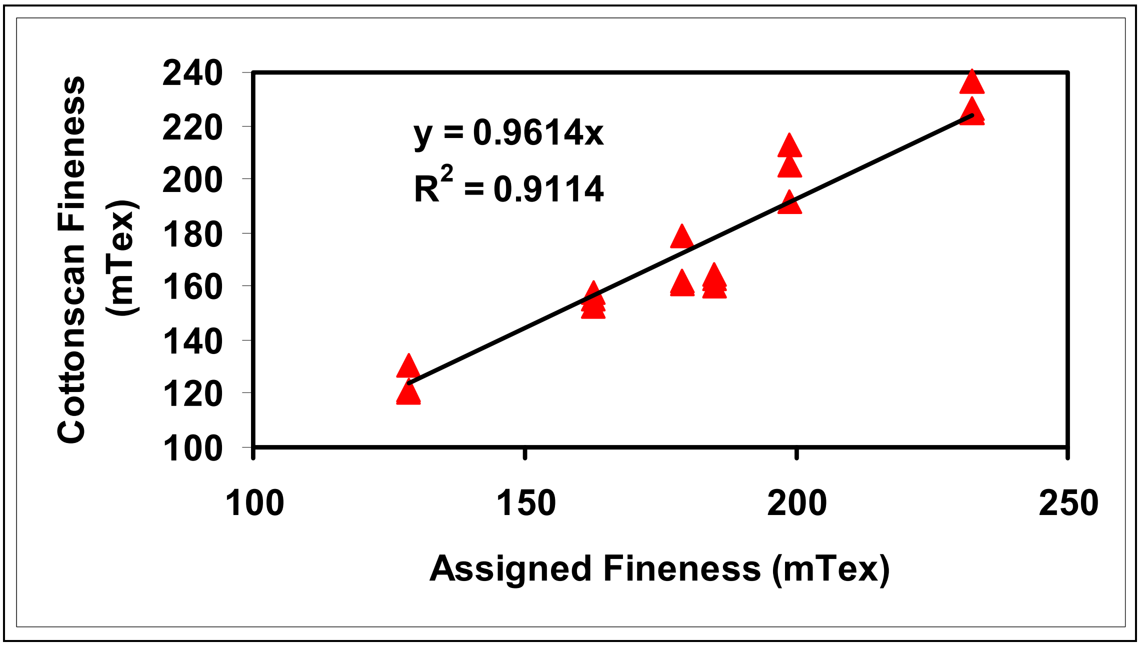

Figure 7. Cottonscan average fineness versus fineness as measured by the 'Shirley' FMT.

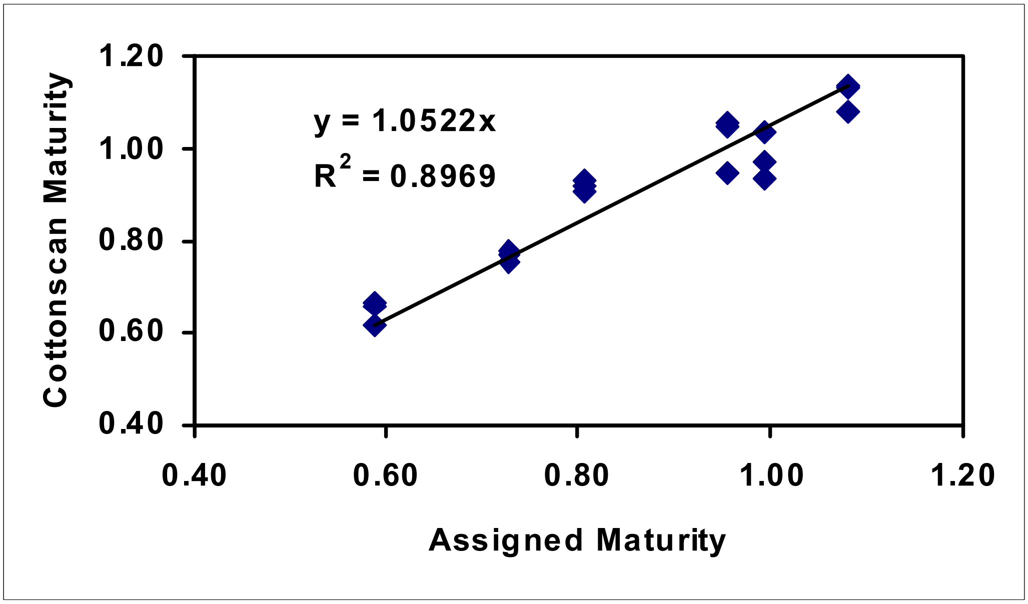

Figure 8. Calculated maturity ratio from Cottonscan and HVI data versus maturity ratio as measured by the 'Shirley' FMT.