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Proposal for calibrating laboratory preparation systems and instruments to measure curvature on raw wool

By

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SUMMARY

This report examines the possibility of using measurements on 2mm guillotined IH top snippets to calibrate the preparation and measurement system used for raw wool fibre curvature measurement.

It is concluded that on the basis of this preliminary work, it should be feasible to overcome one of the major constraints in progress on a test method for fibre curvature.

It is recommended that the fibre curvature working group be re-convened to re-examine the working group draft test method and to organise a round trial to test the proposed calibration concept.

INTRODUCTION

The measurement of fibre curvature has attracted increasing interest since it first became widely available using the OFDA (Brims, 1993) and subsequently the Laserscan (Dabbs et al, 1994). Use of the measurements was the subject of the Commercial Technology Forum held at the IWTO conference in Barcelona in 2002.

There have been many investigations into the use of curvature for measuring crimp and related characteristics of wool. Lobb et al (1997) reported on comparisons between OFDA and Laserscan measurements of curvature and highlighted some of the preparation factors that influence the measurement. Fish et al (1999, 2000) reported extensively on various aspects of preparation effects, and highlighted the fact that virtually all aspects of sample preparation may have an influence on mean curvature measurements. These papers also mentioned one problem that has delayed development of a draft test method for curvature - the lack of calibration standards or procedures.

Baxter (2002a) showed that the OFDA 100 and 2000 models are inherently relatively accurate when tested using graticules, and concluded that *"Variability between instrument calibrations, together with measurement variability, indicates that the overall accuracy and precision of a single measurement on a graticule is of the order of ± 1 to 7 º/mm, depending on the level and degree of variability of curvature."*

In examining the performance of the OFDA2000 instrument operating in 100 mode, Baxter (2002b) noted that the precision of the two models of the OFDA instrument used in a controlled trial in two laboratories was ± 4º/mm on a range of tops, but ± 14 º/mm on scoured wool samples. The large discrepancy in the 95% CL estimates was considered to be due to minicoring differences between the two laboratories. This may have been exacerbated by the fact that because of the trial design, only two laboratories were involved, (which was nevertheless adequate for the purpose of the project). The concerns raised by previous authors about sample preparation effects are immediately apparent in this comparison of precision estimates for the two sample types.

In another paper to be presented to this conference, Baxter and Johnston (2002) indicate that the precision of measurements of curvature using a large number of OFDA2000 instruments is in the order of ± 7 °/mm on full-length top sliver and ± 9 °/mm on greasy wool staples.

It is apparent from this abbreviated history that measurements of mean fibre curvature on tops and greasy staples appear to be a good deal more stable than on minicored snippets prepared from scoured raw wool, and that the reproducibility of measurements on tops using the OFDA instruments is almost as good as may be obtained on graticules of known curvature values.

PROPOSED CALIBRATION TECHNIQUE

It has already been shown that OFDA instruments are inherently accurate in measuring curvature of graticules, without any supplementary calibration. However, it was noted in Baxter 2002a that if necessary, the accuracy of the measurements from individual instruments could be improved slightly if they were themselves calibrated from graticules. This leads to the question as to how accurately industry requires mean fibre curvature measurement to be undertaken. It has been suggested that the levels already referred to for (uncalibrated) measurements on graticules (up to ± 7 °/mm) may be perfectly adequate. A debate needs to be held to answer this question, but in the meanwhile it is sufficient to note that, if necessary, some improvement may be possible.

Experience suggests that the precision of measurement of mean fibre curvature on scoured raw wool is not adequate without standardising the preparation systems. Other authors have noted that many variables may play a contributory part in unacceptably extending the reproducibility on this form of wool. These may include such items as the scouring methods, length of time for conditioning, minicore compression levels and tip sharpness, and type of spreader in the case of the OFDA instruments. Because of the difficulties of standardising all these potential sources of variability, progress on the working group draft for curvature measurement virtually stalled after the initial draft was presented to IWTO (Ranford 2000).

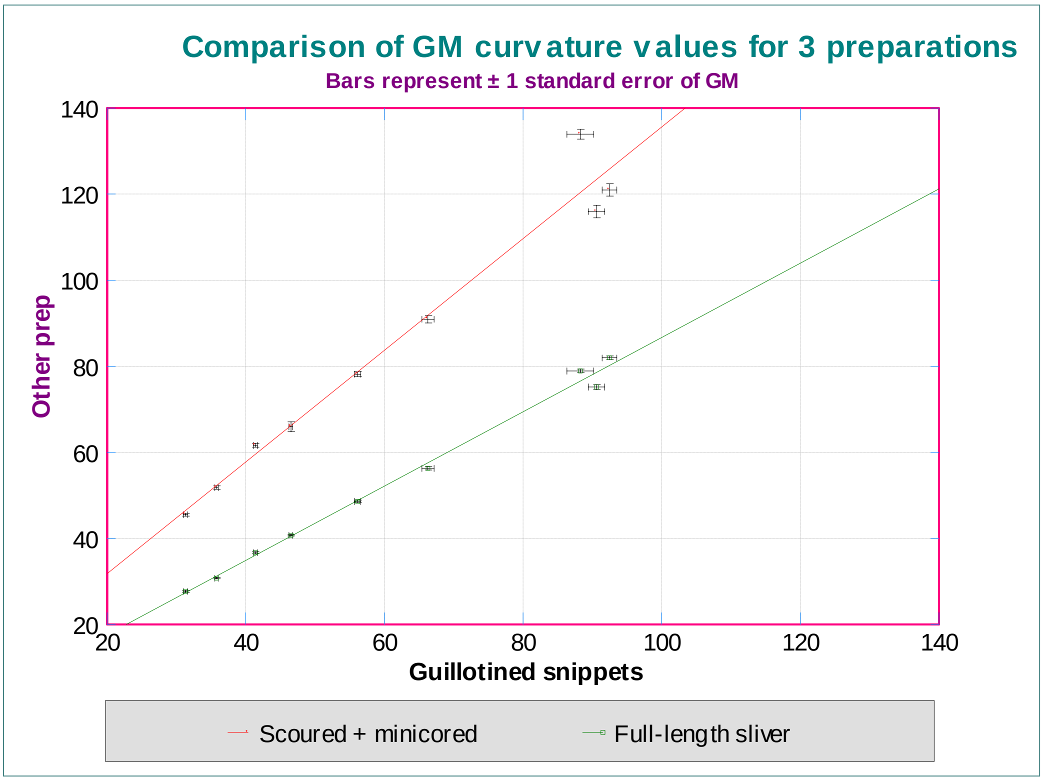
The results of the various round trials noted above suggest that similar levels of precision are possible when measuring both graticules and tops, using either full-length sliver in the case of the OFDA2000, or 2mm snippets obtained with a guillotine in the case of the OFDA100 or OFDA2000 in 100 mode. This immediately suggests the possibility of using tops to "calibrate" the laboratory processing system that is needed to measure the curvature of raw wool. This also seems a logical course, in that most measurements undertaken on raw wool are carried out in order to predict the processing performance or outcomes after the wool has been processed.

OFDA2000 measurements on full-length sliver, whilst being as reproducible as OFDA measurements on 2mm guillotined snippets of top, are, however, also biased with respect to the latter, being on average approximately 87% of the latter for IH tops. It has been suggested that since the sliver presented to the OFDA2000 is in a relaxed state and has not been subject to any form of pre-measurement stress, that the OFDA2000 style of measurement should be adopted as a standard state. However, since more measurements of curvature on tops are undoubtedly undertaken worldwide using guillotined snippets, it seems more sensible at this stage to consider 2mm guillotined snippets as the baseline. Nevertheless, as can be seen in figure 1, it should be feasible to convert between one state and the other.

In order to test the hypothesis that measurements undertaken on 2mm guillotined snippets of sliver can be used to calibrate a raw wool processing system, data has been abstracted from calibrations undertaken over a 2 year period on a number of OFDA instruments. The data was restricted to the series 13 IH tops. Calibrations of OFDA instruments in our laboratory are usually performed for both raw wool and for sliver. In the case of raw wool measurements, it is necessary to cut the IH top into short lengths, and then scour, dry, condition and minicore the samples in a manner that directly simulates the measurement of greasy wool (IWTO-47). Whilst not needed for the calibration and validation processes, a number of these calibration records have included curvature measurement.

Figure 1 compares the mean curvature values for the raw wool calibration process in our laboratory against the corresponding curvature values obtained by guillotining IH slivers in the normal manner. One additional ultrafine top that is used in our internal validation procedures is also included. As indicated above, the plot also includes data from OFDA2000 full-length sliver measurements where corresponding guillotined snippet measurements have been undertaken on the same instrument.

Figure 1: Comparisons of mean curvature values for series 13 IH tops



PRELIMINARY VALIDATION

Unfortunately preliminary validation data could only be obtained from a limited database. Since all the scoured preparation in this database had been carried out in one laboratory, albeit at several different times, only a limited assessment could therefore be made of whether this concept would be effective in reducing between-laboratory variance. However, data was also available for two OFDA2000 instruments that had been calibrated with guillotined snippets (in 100 mode) and also with full-length sliver. As can be seen in Figure 1, there is a large difference between the values obtained on full-length sliver and minicored scoured wool, and this was therefore utilised to simulate a significant between-laboratory difference.

For 3 OFDA 100 instruments, the mean curvature results on guillotined snippets in one calibration were regressed against the results from a calibration using the same calibration tops but with scoured and minicored snippets. These equations were then used on a separate set of data (either a validation set or a calibration undertaken on a different date) to predict the guillotined sliver values from measurements on scoured minicored snippets. On the two OFDA2000 instruments, the same process was used, except that in this case, the guillotined snippet values were predicted from calibration regressions undertaken with full-length sliver. This process therefore examined the feasibility of predicting the 2mm guillotined sliver values from two extremes - mincored scoured samples and unprocessed full-length sliver.

Two-way analyses of variance were undertaken to estimate the components of variance for between-instruments and the error variance (or residual measurement variance). From this data, 95% confidence limits were calculated for the following scenarios:

1. Measurements undertaken solely on guillotined IH top snippets
2. Measurements undertaken solely on minicored scoured samples of IH top
3. Measurements undertaken on 3 instruments measuring scoured snippets and 2 measuring full-length sliver (to simulate the worse-case scenario)

4. Measurements undertaken with all 5 instruments "calibrated" to predict the guillotined IH top snippet values.

The results of this analysis are shown in table 1.

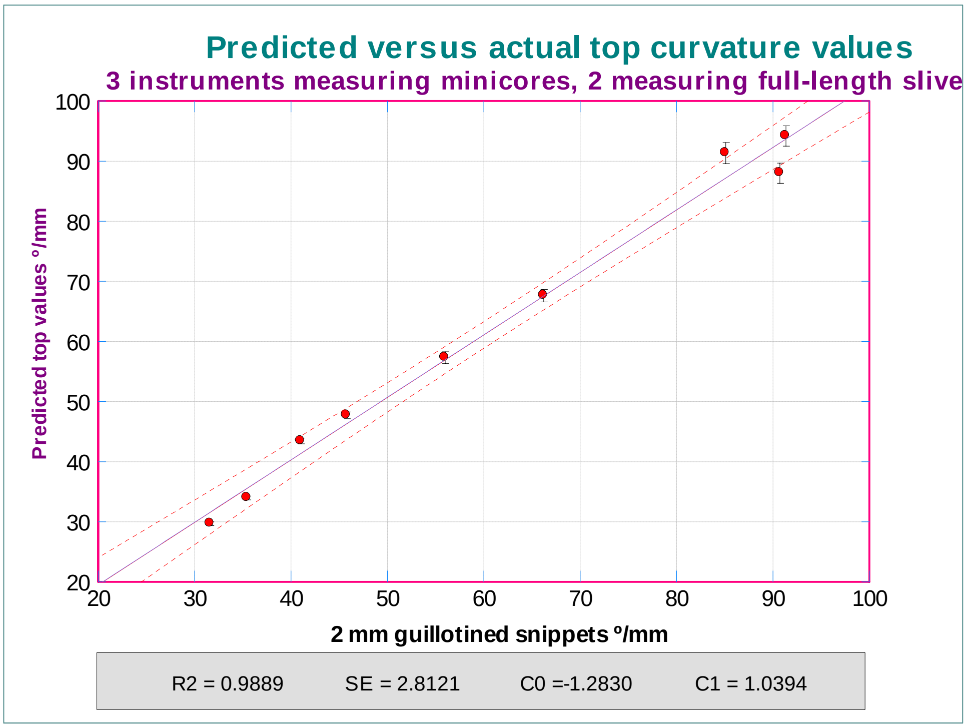
Table 1: Comparison of precision estimates for 4 scenarios

	1 - guillotined	2 - scoured & minicored	3 - minicored + full-length	4 - predicted guillotined
Variance between instruments (º/mm)²	8.1	23.3	290.4	15.7
Variance within instrument (º/mm)²	5.5	29.5	109.9	6.5
95% CL for 1 measurement	7.2	14.2	39.2	9.2
95% CL for average of 4 measurements	6.0	10.9	34.9	8.2

It can be seen that the precision of the predicted values from 5 instruments using two very diverse preparation systems (scenario 4) was reduced to similar levels to the precision on guillotined tops alone.

The accuracy of the predicted values from the 5 diverse instruments against the measured values can be judged by figure 2. The dotted lines indicate the 95% confidence limits on the regression, and it can be seen without carrying a full IWTO-0 analysis that a 1:1 line would be included within these boundaries, indicating equivalence on this limited data set.

Figure 2: Comparison of predicted guillotined IH top curve values against measured values



VALIDATION USING THE OFDA2000 IN 100 MODE TRIAL DATA

The OFDA2000 in 100 mode trial involved a total of 4 OFDA2000 instruments and 4 OFDA100 instruments in two laboratories - one an IWTO Licensed Laboratory, and the other a Fleece Testing Laboratory (Baxter 2002b). The trial involved the measurement of both scoured wool and sliver, and hence required each instrument to be calibrated for both wool types. The scoured wool samples were all scoured and dried in one laboratory, and hence the overall precision estimates are slightly reduced in absolute terms from what might be expected from a full-scale round trial. The data can, however, be used to examine the effects of the proposed calibration regime.

The calibration MES files for the 8 instruments were obtained, and the curvature data abstracted for the 16 calibrations. This data was then used to "calibrate" each instrument individually for the measurement of minicored scoured wool using the individual mean curvature values from the sliver calibrations (not the global means). For convenience, linear regression was used, with the calibration equations taking the form:

2mm guillotined mean curve = A + B * 2mm minicored scoured mean curve

There was some evidence that the calibration might be improved by the use of a quadratic form.

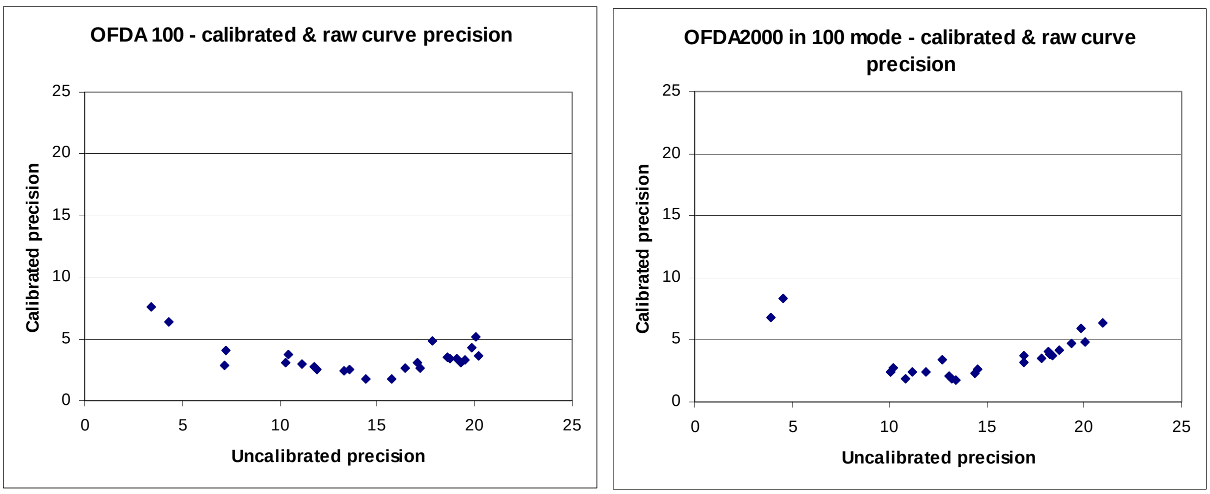
The calibration equations used are shown in table 2, and it can be seen that there is a significant diversity in the regression coefficients.

Table 2: Linear fibre curvature calibration coefficients used for each of the 8 instruments in the OFDA2000 in 100 mode trial

Laboratory	Instrument	Constant A	Coefficient B
A	2000 a	-20.76	0.9107
	2000 b	-19.13	0.9130
	100 a	-23.26	0.9376
	100 b	-23.60	0.9598
B	2000 c	-9.66	0.9027
	2000 d	-6.04	0.8508
	100 c	-6.24	0.8593
	100 d	-6.80	0.8653

These equations were then used to calibrate or convert the individual measured values on each of the 25 scoured wool samples used in the trial to 2 mm guillotined sliver values. The analysis of variance schema used in the original work was then used to calculate the within and between components of variance for the calibrated curvature measurements. The plots in figure 3 illustrate the differences between the uncalibrated 95% confidence limits and calibrated values.

Figure 3: Comparison of calibrated and uncalibrated 95% confidence limits for 2 models of OFDA



It can readily be seen that apart from two samples with already low precision, the calibrated instruments gave very much improved confidence limits. As a further test, and bearing in mind that the original trial detected a very small difference between the mean curvature values from the two models of instrument

(< 1 °/mm), a two-way analysis of variance was undertaken on the entire data set with sample and instrument as the two fixed effects (i.e. ignoring the effect of instrument model).

The 95% confidence limits were then recalculated based on the 8 instruments and the 25 scoured samples, and additionally recalculated for the 27 tops also measured in that trial for comparison. The results are shown in Table 3.

Table 3: Comparison of calibrated and uncalibrated precision estimates for 8 instruments

Basis of measurement	Un-calibrated, scoured wool	Calibrated, scoured wool	Tops (un-calibrated)
95% CL - single measurement	14.5	5.2	5.7
95% CL - average of 4 measurements	13.7	2.8	4.1

The contrast between the calibrated and un-calibrated results is dramatic. Clearly the calibration process has improved the overall precision on scoured wool to close to the values estimated for the commercial tops.

COMMENTS & RECOMMENDATIONS

Using the method proposed, it would appear feasible to predict the mean curvature values for tops from samples processed or handled in other ways, using calibrations based on guillotined slivers. The technique appears capable of providing acceptable accuracy, and precision which is similar or only marginally worse than would be obtained if all the instruments were actually measuring guillotined tops. There seems no reason why this technique would not equally apply to Laserscan instruments.

If greater accuracy and further improved precision were needed, there remains the option to calibrate the OFDA instruments with graticules, or, alternatively, use the Interwoollabs process to generate reference fibre curvature values.

It is recommended that the curvature working group be re-convened in order to establish whether a draft test method can now be progressed, and a trial organised to test the concept on a wider basis.

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