COMPARATIVE FIBER EVALUATION OF THE MESDAN AQUALAB MICROWAVE MOISTURE MEASUREMENT INSTRUMENT

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Abstract

Moisture is a key cotton fiber parameter, as it can impact the fiber quality and the processing of cotton fiber. The Mesdan Aqualab is a microwave-based fiber moisture measurement instrument for samples with moderate sample size. A program was implemented to determine the capabilities of the Aqualab for rapid, precise and accurate cotton fiber moisture measurements. Calibrations and operational protocols for the Aqualab were established, using the oven drying method by both the Scirocco and SRRC laboratory ovens as initial references. In preliminary evaluations, very good moisture content agreement was observed between the Aqualab and different oven methods over a wide moisture range. Weight impacts were slight, and excellent analytical was observed.

Introduction

The moisture content (MC) of cotton fiber is often an important quality and processing parameter. Recent concerns and interest in fiber MC are often due primarily to 1) its impact on fiber physical properties, especially for instrumental measurements at non-standard environmental conditions, 2) its impact on textile processing, 3) previous incidents of "high moisture" cotton bales in the marketplace, and 4) its impact on cotton storage and overseas. (Anthony, 1982; Fortenberry, 1965; Lawson, 1976; Lord, 1961; McQuigg and Decker, 1961) The primary globally recognized standard method for measuring the MC of cotton fiber is the oven method, in which the gravimetric measurement of fiber weight loss is determined when a weighed fiber sample is heated at a specified temperature and time. (Montalvo and Von Hoven, 2008) Previous evaluations have established the capabilities, both technical and logistical, of several laboratory MC/water content measurements (compared to the oven method as reference). (Rodgers et. al, 2010) The best overall comparative results were obtained with most small "oven" analyzers and Near Infrared (NIR) analyzers.

Microwave analyzers offer the potential to yield rapid and accurate measurements of MC. They potentially would be faster (much lower analysis time) than the small oven analyzers and less expensive and less complicated to calibrate than the NIR analyzers. The Aqualab instrument (Mesdan S.p.A., Italy) is a microwave-based moisture measurement instrument that measures MC, moisture regain (MR), and commercial weight. For fiber measurements, sample weights are normally of ≥100 grams, and the instrument is calibrated with the Scirocco oven/conditioning system (Mesdan S.p.A., Italy).

A program was implemented 1) to determine the capabilities of the Aqualab for rapid, precise and accurate cotton fiber moisture measurements, 2) to determine the impacts of key variables, and 3) to determine the ability to use different reference methods. All evaluations were performed at the USDA-Agricultural Research Service (ARS)-Southern Regional Research Center (SRRC). The Aqualab MC results were compared to the calibration method(s) used.

Experimental

Mesdan recommended calibration procedures for the Aqualab consist of 3 calibrations (10 measurements per calibration) on 3 distinct cotton samples, representing clean-mixed-"trashy" (high trash content) cottons (C1, C2, C3). The 10 measurements for each calibration were prepared at 5 temperature-relative humidity (RH) conditions (standard 21.0^{O}C/65% RH and low-to-high conditions from 15^{O}C/45% RH to 27^{O}C/80% RH). The Aqualab sample weight was a minimum 125 grams (sufficient fiber sample to fill the Aqualab sample chamber). Reference moisture values were obtained with the Scirocco oven (minimum of 150 gram sample). In addition, a combination calibration (Combo) was prepared by combining the samples from C1-C3 and adding 5 measurements at varying temperature/RH from three (3) 2013 crop year samples. To determine the Aqualab ability to be calibrated by different reference methods, SRRC oven (7.5 gram sample) MC values for the above samples were used to develop SRRC oven calibrations (C1S, C2S, C3S, ComboS).

For the preliminary comparative MC evaluations, a set of 10 diverse cottons (5 domestic and 5 international varieties) were used to evaluate each individual and the combined Aqualab calibrations. The SRRC oven moisture values for each sample served as the reference values. Both Scirocco and SRRC oven calibrations were evaluated. For these samples, all measurements were made at standard conditions (21.0^{O}C/65% RH). In addition, the Combo calibrations were evaluated with 8 additional samples composed of 4 cottons conditioned at 15^{O}C/45% RH and 27^{O}C/80% RH.

For the sample weight and precision evaluations, samples from the cottons used to develop calibrations C1, C2, and C3 were prepared at 75, 100, 125, and 150 grams (±0.1 gram for each sample). Six measurements were made for each sample using the designated cotton-specific calibration for those samples (C1, C2, or C3) and for the Combo calibration.

The primary MC comparison parameters were Standard Deviation of Differences, SDD (the standard deviation of the differences in moisture results between the designated reference method and the compared method for each sample; a residual analysis) and two outlier ranges (percentage of samples with differences between the designated reference and compared method of >±0.30% moisture (tight limit) and >±0.50% moisture (commonly used limit)). Low SDD and low number of outliers indicate improved method agreement for MC. Analytical and analysis precision and the impact of sample weight was determined from the within standard deviation (SDw) for the 6 measurements made on each cotton sample at each sample weight.

Results and Discussion

Comparison of Scirocco and SRRC Oven Methods

The Aqualab is calibrated for MC and MR with the high mass Scirocco oven (minimum of 150 grams), but the SRRC and other oven methods oven uses a much lower fiber mass (20 grams or less per sample; 7.5 grams total for the SRRC oven method). In order for the Aqualab to be calibrated to other reference methods, good method agreement should be obtained between the Scirocco oven MC results and the MC results from other reference methods. The Scirocco MC results were in very good agreement with the SRRC oven MC results, with good linearity and high R^2 (Figure 1). In general, the Scirocco MC results were normally 0.5-1.0% moisture higher than the SRRC oven MC results.

Preliminary MC Fiber Comparisons

Mesdan recommended calibration procedures for the Aqualab consist of 3 calibrations on 3 distinct cotton samples, representing clean-mixed-trashy (high trash content) cottons (e.g., C1, C2, C3). The operator would then choose the calibration to use based on the relative perceived “cleanliness” of the fiber sample. However, this method is subjective and may lead to MC differences between different operators, especially if the 3 calibrations have different MC values due to their calibration. Thus, the feasibility and capabilities of a “combination” calibration (e.g., Combo), composed of the samples in the original 3 calibrations plus data from 3 additional cottons, was examined.

In preliminary evaluations, the Aqualab measurement was very fast, with a fiber measurement time of <5 seconds per readings and 2 readings per sample; total analysis time, including data entry and sample loading, was < 1 minute per sample. Very good MC agreement was observed between the Aqualab and SRRC oven methods for the Scirocco-developed cotton specific calibrations C1, C2, and C3 as well as Combo combination equation, with low residuals and a low number of outliers (Table I). The Aqualab MC results were approximately 0.5-1.0% higher than the SRRC oven MC values, which is in very good agreement with the observed differences between the previous Scirocco oven MC results and SRRC oven MC results. For the 10 samples run at standard conditions, a minimum of 90% agreed within ±0.5% MC for all calibrations. The Combo calibration results were among the best overall, and this calibration was examined for both high and low MC obtained from high and low temperature/RH conditioned samples (n=18 total). Excellent residuals and outlier results were observed, verifying the Combo calibration’s capability to accurately monitor large changes in MC from ~4.0-10.0% MC.

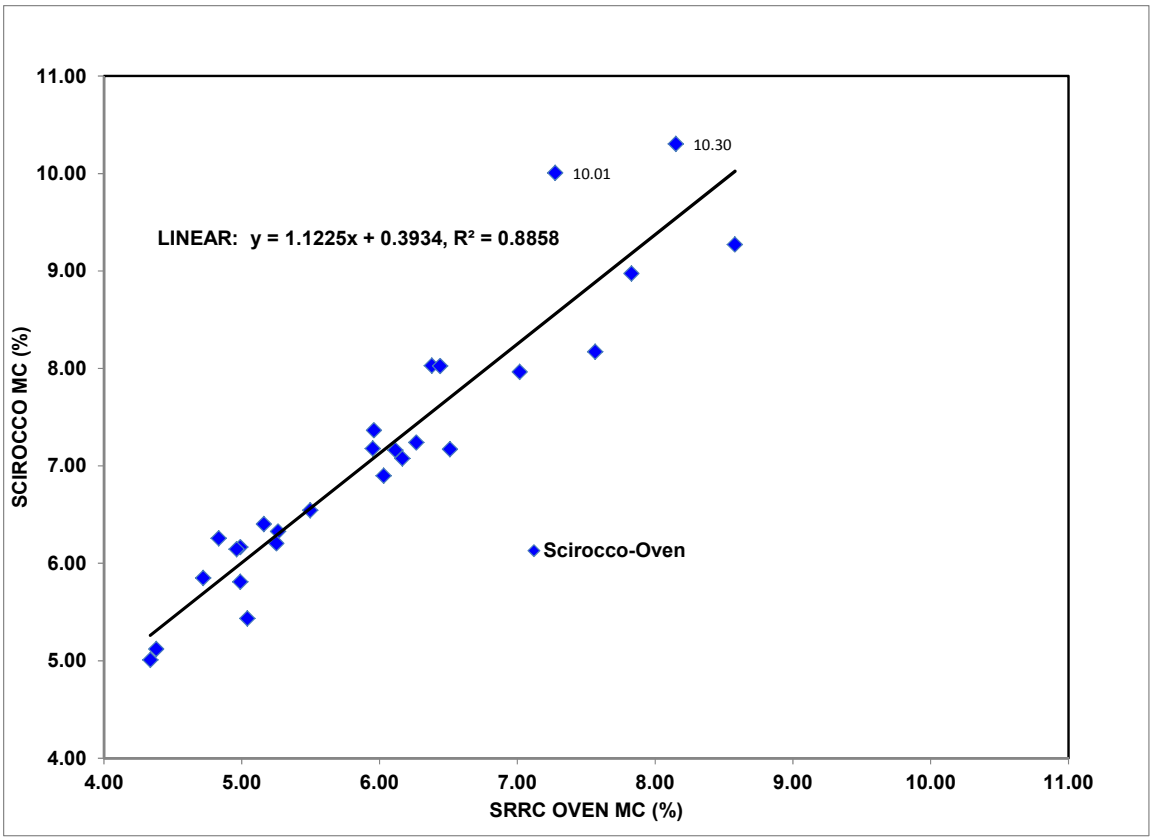

Figure 1. Gravimetric oven comparison, Scirocco oven vs. SRRC oven.

Table I. MC comparisons, Aqualab MC vs. SRRC oven MC (Scirocco oven calibrations).

PARAMETER	SRRC OVEN MC (%)	AQUALAB CALIBRATED MC (%), SCIROCCO CALIBRATED			
		C1	C2	C3	Combo
AVG	6.62	7.09	7.80	7.19	7.34
SD	0.25	0.16	0.32	0.25	0.21
SDD (residuals)	NA	0.27	0.34	0.31	0.28
n	10/18	10	10	10	10/18
% Within ±0.3%	NA	90%	80%	70%	90%/89%
% Within ±0.5%	NA	100%	90%	100%	90%/89%

Very good MC agreement was observed between the Aqualab and SRRC oven methods for the SRRC oven developed cotton specific calibrations C1S, C2S, and C3S as well as ComboS combination equation, with low residuals and a low number of outliers (Table II). The Aqualab MC results were overall in very good agreement with the SRRC oven MC results (normally within 0.5% MC), and the best overall results were obtained with the ComboS combination calibration. For ComboS, the Aqualab MC results for all standard condition samples were within ±0.3% MC with the SRRC oven MC results (no outliers). For the high and low MC obtained from high and low temperature/RH conditioned samples (n=18 total), very good residuals and outlier results were observed (approximately 80% and higher within ±0.3% MC method agreement). These results also verify the combination calibrations' capability to accurately monitor large changes in MC from ~4.0-10.0% MC.

Table II. MC comparisons, Aqualab MC vs. SRRC oven MC (SRRC oven calibrations).

PARAMETER	SRRC OVEN MC (%)	AQUALAB CALIBRATED MC (%), SRRC OVEN CALIBRATED			
		C1S	C2S	C3S	COMBOS
AVG	6.62	6.11	6.73	6.14	6.51
SD	0.25	0.24	0.18	0.21	0.26
SDD (residuals)	NA	0.29	0.23	0.27	0.24
n	10/18	10	10	10	10/18
% Within ±0.3%	NA	80%	90%	90%	100%/78%
% Within ±0.5%	NA	100%	100%	100%	100%/89%

Samples of the 3 cotton used to develop C1, C2, and C3 were prepared at 75-150 grams, in 25 gram increments, and 6 replicates were performed on each sample. The results are given in Table III. At 75 grams, cotton was present in the unit's sampling zone but no compression was on the fiber; at 150 grams, the sample chamber was full and very compressed. At sample weights of 100 grams and higher, the differences in within SD (SDw) for the 6 measurements were small normally for all calibrations, but the best overall results for each cotton were observed for the Combo calibration. Thus, minimal weight impacts were observed for samples weights of 100 grams and higher. It is recommended that 125 gram be used as the minimum sample weight to have on hand for the Aqualab MC measurement. The small SDw and SDwp values demonstrated excellent analytical and analysis precision for the Aqualab MC analyses.

Table III. Aqualab Precision and Sample Weight Impacts.

COTTON	CAL	SDw (by Sample Weight, grams)				
		75	100	125	150	SDwp
C1	C1	0.19	0.06	0.04	0.09	0.11
	Combo	0.08	0.12	0.06	0.05	0.08
C2	C2	0.32	0.29	0.14	0.16	0.24
	Combo	0.19	0.14	0.10	0.06	0.13
C3	C3	0.21	0.04	0.09	0.05	0.12
	Combo	0.15	0.12	0.10	0.12	0.12

Summary of Results

A program was implemented to determine the capabilities of the Aqualab for rapid, precise and accurate cotton fiber moisture measurements. The Aqualab microwave moisture measurement was very fast, easy to perform, and required no sample preparation. Very good MC agreement was observed between the Mesdan Scirocco high mass (150 gram minimum) oven (Aqualab standard reference method) and low mass (less than 10 grams) SRRC laboratory oven, with the Scirocco yielding approximately 0.5-1.0% higher moisture. Calibrations based on the SRRC oven were developed. In preliminary evaluations, very good MC agreement was observed between the Aqualab and SRRC oven. Minimal weight impacts were observed, especially at weights above 100 grams. High analytical and analysis precision was observed at 100 grams and above sample weights.

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Disclaimer

The use of a company or product name is solely for the purpose of providing specific information and does not imply approval or recommendation by the United States Department of Agriculture to the exclusion of others.

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