COMPARATIVE EVALUATIONS OF LABORATORY FIBER MOISTURE MEASUREMENT METHODS

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Abstract

In the global marketplace, the accurate and precise measurement of cotton fiber moisture is becoming more important. Moisture is a key quality and processing parameter for cotton fiber, and several moisture measurement techniques and commercial instruments are readily available, but how well do they agree with each other? A program was implemented to compare and determine the capabilities of various laboratory moisture instrumentation and measurement methods. Several moisture instruments were evaluated and validated with a common set of domestic and international cotton samples. The moisture techniques were divided into 4 broad categories—thermal, chemical, spectroscopy, and "electric." The moisture results were compared with those obtained by a thermal/oven drying method. Results to date indicate that most fiber moisture techniques agree to within ±0.5% moisture with the oven method for greater than 90% of the samples analyzed, but only a few agree to within ±0.3% moisture with the oven method for greater than 80% of the samples analyzed. A comparative matrix has been developed.

Introduction

Cotton fiber moisture is an important quality and processing parameter (Lord, 1961). The primary influences of moisture on the quality of cotton fibers and products are its impact on 1) fiber physical properties, 2) fiber to final fabric processing, and 3) different moistures and fiber-moisture responses for different cotton varieties. (Anthony, 1982; Lawson, 1976; McQuigg and Decker, 1961) Interest in fiber moisture has significantly intensified over the last 5 years, due in no small measure to:

- Concerns on the impact of "non-standard" environmental conditions on instrumental measurements of fiber quality, especially in developing countries where standard environmental conditions may be difficult to establish and maintain for high volume testing instruments. Standard environmental conditions are 65% Relative Humidity (RH) and 70^{O}F (Fortenberry, 1965).
- Reports of occurrences of "high moisture" bales in the marketplace (>7.5% bale moisture).
- Impact of high moisture on cotton storage and overseas shipments.

In the U.S. and internationally, the primary means for measuring the moisture content(s) of fibers—both natural and synthetic—is the use of an oven to dry the sample, and the weight change is calculated as the moisture content. (Montalvo and Von Hoven, 2008) However, there are several different technologies for measuring moisture, most of which use the gravimetric/oven method as the reference method. These various moisture measurement methods have been divided into four main groups: weight loss/gravimetric, chemical, spectroscopy, and "electric." Weight loss/gravimetric methods remove the sample's moisture (often by heating the sample at a specified temperature), then the dried sample is weighted to calculate the moisture content. Examples of weight loss/gravimetric methods include oven heating, IR/halogen lamps, and microwave. Chemical methods primarily involve a colorimetric measurement of moisture that has been extracted from the fiber. An example of the chemical methods is the Karl Fischer Reagent colorimetric titration measurement. Spectroscopy methods involve measurement of absorptions of energy by the OH group of the water molecule or the free, unbound water molecule itself, and the quantity of moisture is determined by the intensity of the absorption. Examples of spectroscopy methods include Infrared (IR), Near Infrared (NIR), microwave, and Nuclear Magnetic Resonance (NMR). "Electric" methods are those that measure changes in the fiber's electric charge/capacitance/conductance/resistance with changing moisture content in the fiber. A comparison of the four main groups of fiber moisture measurements is given in Table I.

Table I. Comparison of specific properties of fiber moisture measurement methods.

PARAMETER	**WT. LOSS**	**CHEMICAL**	**NIR**	**ELECTRIC**
SPEED	**SLOW**	**SLOW**	**VERY FAST**	**FAST**
MOISTURE LOCATION	**WHOLE SAMPLE**	**WHOLE SAMPLE**	**SURFACE**	**WHOLE SAMPLE**
MAINTENANCE	**MINIMAL**	**MODERATE**	**SLIGHT**	**MINIMAL**
"PORTABILITY"	**VERY POOR**	**VERY POOR**	**FAIR→EXCELLENT**	**VERY GOOD**
OTHER IMPACTS	****High Turn-Around Time**	****Moderate Turn-Around Time**	****Large Number of Samples **Chemometric Calibrations **Moderate Cost**	****Surface Contaminants **Metal Ions/Other Conductive Species **No Calibration Adjustment**

A program was implemented 1) to compare the method agreement between various Agricultural Research Service (ARS) thermal weight loss moisture measurement methods and 2)to compare and determine the capabilities of various laboratory moisture instrumentation and moisture measurement methods for cotton fiber. A consistent set of diverse domestic and international cottons will be used for the comparative evaluations. Initial emphasis will be on spectroscopy, weight loss/gravimetric, and "electric" moisture measurement instruments. The best overall ARS oven gravimetric oven method will be the reference method for the comparative evaluations. The program was a joint project between Cotton Incorporated and the Cotton Structure & Quality (CSQ) research unit with the Southern Regional Research Center (SRRC) of the Agricultural Research Service (ARS).

Experimental

Three ARS locations—CSQ at SRRC; the Cotton Quality Research Station (CQRS) in Clemson, SC; the Ginning Research Laboratory in Stoneville, MS—use the oven gravimetric method to determine the moisture content of the fiber. However, the oven method used at each laboratory and location are very different from each other (Table II). For the comparison of ARS oven moisture methods evaluations, three domestic cottons were used and moisture measured by each method by both static and forced air ovens. It should be noted that the oven and weigh mechanism used for the Ginning Research Laboratory method was modified for the standard ovens at SRRC, as the Ginning Research Laboratory uses a custom-made oven system, which is not available at SRRC that permits the weighing of the dried sample in the oven (hot).

For the comparative moisture instrumentation and methods evaluations, a set of 20 diverse cottons (15 domestic and 5 international varieties) were used to evaluate each instrument/method. The reference method was the ARS moisture measurement method that yielded the best overall results and properties (technical capabilities, total sample used, analysis time, etc.). The moisture results from each instrument/ method were compared to the reference method moisture results. To date, 10 instrument systems/methods have been evaluated and will be discussed—Arizona Instrument Computrac Max 2000, Brabender MT-C Automated Tester, Mettler HR 83 Moisture Tester, CEM Smart Microwave Moisture Analyzer, Y412B Chinese Moisture Meter (2), Strandberg M-400 Portable Moisture Meter (surface probe, yarn probe), Bruker MPA NIR Analyzer, and Brimrose Luminar 5030 NIR Analyzer. Manufacturer recommended procedures were followed.

Table II. Comparison of ARS gravimetric/oven moisture measurement methods.

ITEM	SRRC	CQRS	GINNING LAB
DRY TEMP (^{O}C)	105	105	105
WEIGHT (g.)	10 g	1.5 g	20 g
DRY TIME	Overnight	24 hrs	2 hrs
RUNS/SAMPLE	3	5	4
CONTAINER	Mason Jar	Weighing Bottle	Perforated Cylinder
OVEN TYPE	Forced Air or Static	Forced Air	Forced Air
WEIGH MECHANISM	Cool, Then Balance	Cool, Then Balance	Hot, Sample in Oven/ External Balance

Results and Discussion

Comparison of ARS Moisture Measurement Methods

Three ARS locations—CSQ at SRRC; the Cotton Quality Research Station (CQRS) in Clemson, SC; the Ginning Research Laboratory in Stoneville, MS—use the oven gravimetric method to determine the moisture content of the fiber. However, the oven method used at each laboratory and location are very different from each other (Table II). A program was implemented to compare the method agreement between various ARS oven gravimetric moisture measurement methods.

The moisture results for the three moisture methods were in overall very good agreement, with all methods agreeing within ± 0.3% moisture for all samples (Figure 1, forced air oven method). In general, the CQRS method yielded slightly lower moisture results compared to the Ginning Lab and SRRC methods, but it also used the least amount of fiber and yielded no outliers. Although the moisture results for the static and forced air oven methods were very similar, the best overall moisture agreement was observed with the forced air oven method for all cottons. Based on these results, the CQRS method with a forced air oven was used as the oven reference method for the comparative analyses of moisture measurement instruments/methods.

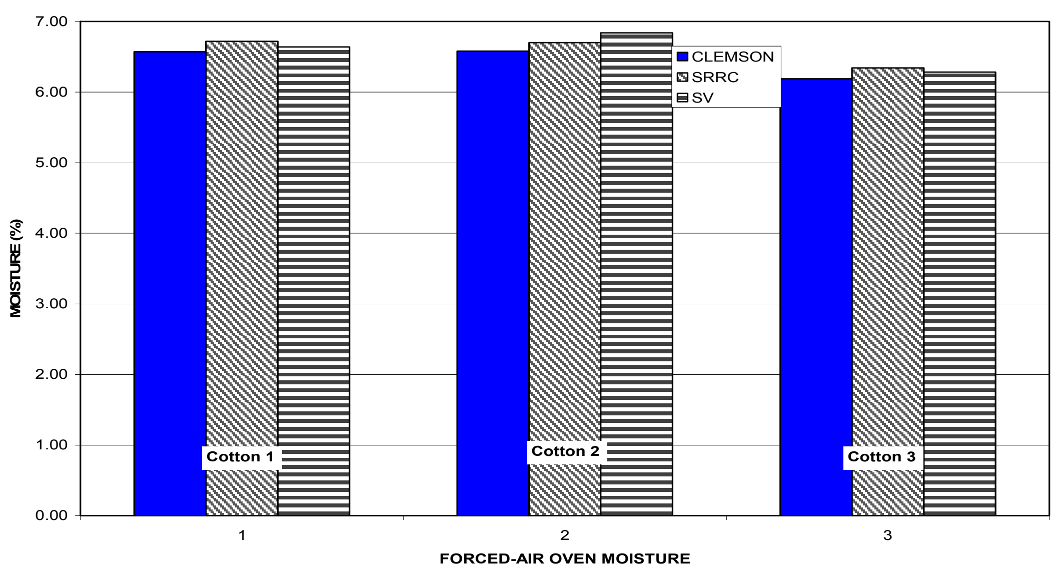

Figure 1. ARS moisture measurement comparisons, forced-air oven.

Comparison of Various Moisture Measurement Instruments/Methods

A program was implemented to compare and determine the capabilities of various laboratory moisture instrumentation and moisture measurement methods for cotton fiber. A consistent set of diverse of 20 domestic and international cottons were used for the comparative evaluations. Initial emphasis was on commercially available weight loss/gravimetric, NIR spectroscopy, and "electric" instruments. Ten (10) instrument systems/methods have been evaluated— the Arizona Instrument Computrac Max 2000, Brabender MT-C Automated Tester, Mettler HR 83 Moisture Tester, CEM Smart Microwave Moisture Analyzer, Y412B Chinese Moisture Meter (2), Strandberg M-400 Portable Moisture Meter (surface probe, yarn probe), Bruker MPA NIR Analyzer, and Brimrose Luminar 5030 NIR Analyzer.

The technical results for the evaluated instrument systems are shown in Table III. The 10 instrument systems included 4 weight loss (WL), 4 "electric," and 2 NIR spectroscopy systems. A primary evaluation end-state criteria was the method agreement between the evaluated system and the reference method, expressed as the percent (%) of samples whose moisture results for the evaluated system and reference method agreed within ±0.3% moisture (target agreement) and within ±0.5% moisture (satisfactory agreement), indicating few major outliers. The best technical results in terms of residuals and outliers were observed for the Bruker and Brimrose NIR systems and the AZI Max 2000 and Mettler HR83 weight loss systems. The NIR systems were the best overall technical systems—and the most expensive. The Max 2000 and HR83 weight loss systems were the units that were most cost effective plus accurate, but a definite slight slope skew was observed for both systems that may impact moisture results at moderately high and moderately low moistures, as shown in Figure 2. Most production environments use the "electric" systems, which performed at acceptable levels for the within ±0.5% moisture criteria, but they were overall the most variable systems and exhibited the most outliers.

Table III. Technical results, comparative moisture instrument systems evaluation (WL = Weight Loss method)

INSTRUMENT	**TYPE**	**% Within ± 0.3%**	**% Within ± 0.5%**
AZI MAX 2000	**WL/Thermal**	**95%**	**100%**
METTLER HR83	**WL/Lamp**	**95%**	**100%**
BRABENDER MT-C	**WL/Thermal**	**75%**	**90%**
CEM Smart	**WL/Microwave**	**65%**	**85%**
STRANDBERG M-400, SP	***Dielectric***	***70%***	***95%***
STRANDBERG M-400, YP	**Dielectric**	**60%**	**70%**
CHINESE (1)	**Conductance**	**65%**	**95%**
CHINESE (2)	**Conductance**	**85%**	**95%**
BRUKER MPA	**FT-NIR**	**100%**	**100%**
BRIMROSE 5030	**Portable NIR**	**95%**	**100%**

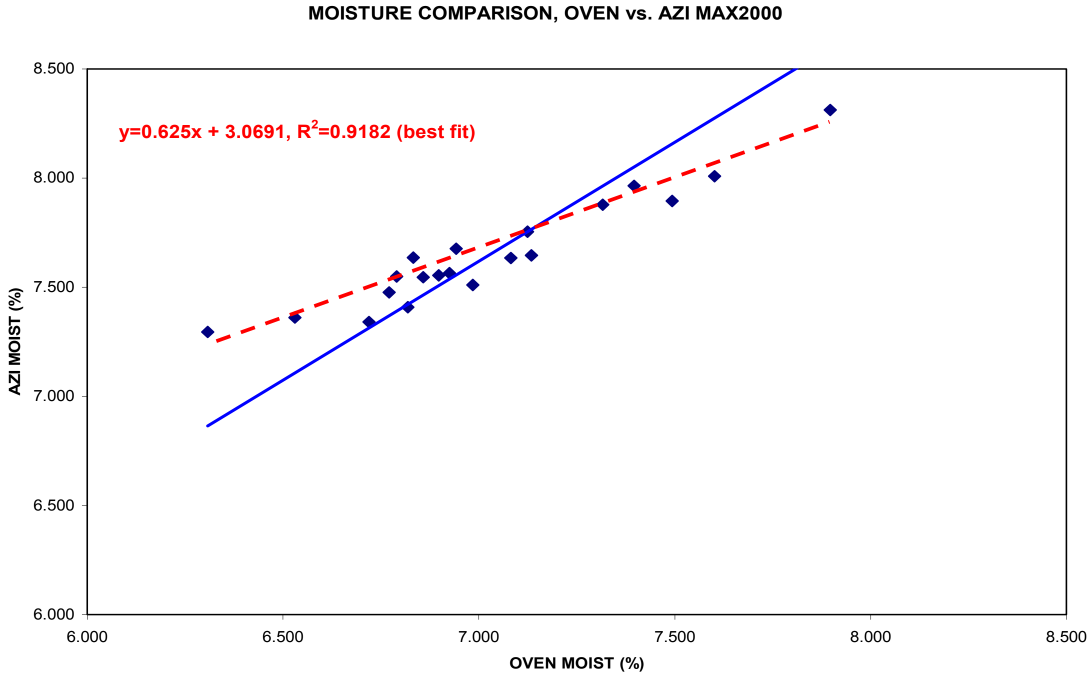

Figure 2. AZI Max 2000 vs. oven reference method moisture results, slope skew.

A comparative matrix was developed to fully compare all evaluated units, with an overview section and technical section. Parameters in the overview section include system/instrument type, sampling, speed, number of samples per run, moisture measurement location, maintenance, portability, non-technical impacts, and cost. Parameters in the technical section include R^2, residuals, % observations within ±0.3% method agreement, % observations within ±0.5% method agreement, and technical impacts.

Summary of Results

A program was implemented 1) to compare the method agreement between three Agricultural Research Service (ARS) thermal weight loss moisture measurement methods and 2) to compare and determine the capabilities of various laboratory moisture instrumentation and moisture measurement methods for cotton fiber. For the 3 ARS gravimetric/oven moisture measurement systems, very good agreement was observed between the 3 laboratory methods. The CQRS method was selected as the reference moisture measurement method for the comparative laboratory moisture instrumentation/methods evaluations. Ten (10) moisture instrument systems/methods were evaluated and validated with a common set of 20 domestic and international cotton samples. The comparative moisture results indicate that most fiber moisture techniques agree to within ±0.5% moisture with the oven method for greater than 90% of the samples analyzed, but only a few agree to within ±0.3% moisture with the oven method for greater than 80% of the samples analyzed. The best overall technical results were achieved with the NIR systems (most expensive). The most cost effective accurate systems were the AZI Max 2000 and Mettler HR83 weight loss systems, but a slight slope skew was observed for their results. A comparative matrix has been developed.

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