MOISTURE IN COTTON BY OVEN DRYING

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Abstract

Cotton fiber properties are strongly dependent on the moisture content in the fiber matrix. The standard test methods for moisture in lint cotton are almost all based on oven drying. All of the loss in weight by oven drying, due to moisture and other volatiles, is attributable to moisture. The U.S. cotton industry questions the reliability of the oven drying test and has requested that this laboratory develop improved standard test methods. The approach taken in this paper is to review the importance of moisture to the industry, the standard test methods by oven drying, present new quality control measures to ensure the laboratory techniques are in control, and compare oven drying results with preliminary results using the Karl Fischer technique. The practical implication of the review of the oven drying method is that it confirms the need by the industry for improved standard test methods for moisture in cotton. There is a lack of information regarding the accuracy of the oven drying techniques. Two new quality control measures are presented to help assure that the weight data is independent of drift or bias in the analytical balance and environmental changes. As expected, oven drying and Karl Fischer results showed differences in moisture contents.

Introduction

Moisture dramatically impacts cotton fiber properties and its processing. When cotton is to be harvested, how it is to be stored, when it should be ginned, how it is to be compressed into bales, and how it is to be spun are heavily impacted by moisture (Backe, 2002). Because of its importance in so many aspects of the cotton lifecycle, accurate moisture measurements are vital to the industry. The objective of this paper is to present a brief review of the importance of moisture in the industry and the standard test methods for moisture in cotton by oven-drying. Also, moisture contents by oven drying are compared to preliminary results by the Karl Fischer technique along with simple quality control measures.

Moisture's Importance in Cotton Fiber Properties and Processing

There are a few comprehensive review papers that discuss the importance of moisture in all facets of cotton processing. The most recent is "Historical Review on the Effect of Moisture Content and the Addition of Moisture to Seed Cotton before Ginning on Fiber Length" by Byler published in 2006 in the Journal of Cotton Science (Byler, 2006). In Backe's book "Cotton Moisture Harvesting through Textile Processing" a very comprehensive historical and contemporary review of literature is presented. This is a wonderful resource published in 2002 by the Institute of Textile Technology (Backe, 2002). Some of the publications highlighted in these two review papers overlap. Other recently published papers discuss the importance of moisture on all facets of the cotton lifecycle. A few highlights are presented here omitting the details of the original publications.

In Byler's 2006 paper, fiber length is the focus of moisture's impact at the gin. Low moisture was found to be most detrimental to fiber length in the ginning process. Ginning at moistures contents greater than 6% not only improved fiber length, but yarn strength even more dramatically. This paper also contains a table of studies published prior to 1990 showing moisture ranges and its statistical significance on properties like staple length, grade, uniformity, length and yarn strength. Ginning moisture levels were recommended at 7%. At lower rates, short fiber contents increased during ginning; for each percentage of fiber moisture decrease, short fiber content increased by 1%, which in turn, caused a 2.6% decrease in yarn break factor. In a study from the late 1940s to the early 1950s, five major findings were highlighted concerning drying temperatures. At higher temperatures, color and leaf grade improve whereas fiber length, strength, yarn strength decrease in value. The ideal moisture content for ginning is between 6.5% and 8% and if below 4%, it becomes detrimental to spinning quality and fiber quality. Fiber length was reduced when ginning was conducted below 3.1% moisture compared to moisture values of twice that. Single fiber strength decreases with decreasing moisture content from 15% to 3%. Thus, a decrease in drying temperature reduces color and leaf grade while improving fiber length, strength and yarn strength. Spinning quality is better when ginning moisture is held to between 6.5% and 8% (Byler, 2006).

Moisture alters many physical properties of cotton, particularly during ginning. Cotton fiber moisture content is often in flux due to changes in relative humidity. High moisture levels are detrimental to ginning as are too dry temperatures, which reduce length despite an improvement in grade. One study investigated cotton picked at different times of day under different relative humidities. At higher humidities, composite grades were lower while mean fiber length and upper half mean length were higher.

Excessive moisture dramatically hinders separation of cotton from seed resulting in wads of cotton clogging up machinery, while low moisture causes static electricity, which also stops machinery. Fiber properties were also impacted by moisture, with the exception of the 2.5% span length. At low moisture levels, below 3.5%, the uniformity ratio suffered, whereas at mid moisture levels, 5% to 7%, and high moisture levels, 10% to 15%, the uniformity ratio was better. However, trash was more abundant and harder to remove at the higher moisture levels. Thus, an optimal moisture must exist, and was found to be 7%. With this optimal moisture, large neps were also found to be reduced (Jade, et al., 2001).

In one study, moisture was used during storage to reduce the stickiness of the cotton. Cottons containing 15, 30 and 40% moisture were studied for 5, 11, and 15 days at 10^{o}C. At the lower moisture level, cotton stickiness was reduced while cotton quality was maintained. At higher moisture levels, yellowness increased while strength decreased. When ammonia or urea was added to the 30% moisture level to reduce any microbial growth, the quality is maintained while the stickiness is reduced (Chun and Brushwood, 1998).

In Backe's book "Cotton Moisture Harvesting Through Textile Processing", there are several chapters quoting directly from published literature on moisture's affect on the various stages of cotton processing from harvesting, storage, ginning, pressing, baling and processing. Much of the book focuses on published recommendations of moisture contents at various stages of processing. Some of the properties affected by moisture are included in 1.

Table 1. Properties Affected by Moisture

Property	Main Effect	Additional Effects
Length	Variety	Temperature, water stress, breakage at gin
Uniformity	Ginning & cleaning at high heat	Field weathering, low strength
Micronaire	Weathering & management	Low mic: cool night temperatures during boll maturity; high mic: high temperatures during boll maturity
Strength	Variety	Excess heating at gin
Reflectance	Fungal growth; moisture during weathering	
Yellowness	Prolonged exposure to moisture in module; high gin temperatures	Premature defoliation, early frost

Cotton is extremely sensitive to changes in relative humidity. In one study, dried cotton contained 5.25% water content (dry basis at 85^{o}F and 55% relative humidity) whereas at the same temperature but at 95% relative humidity, the moisture content was 12%. Cotton can loose one to two grades after ginning if it was picked at 6% to 8%. It was recommended by the National Cotton Council and the Cotton North Carolina Extension Services that cottons should be harvested with moistures below 8% to 10% in the early 1960s. Just as high moistures can lower grades because the cotton is incapable of cleaning and ginning well, thus being trashy, low moistures result in poor quality cotton since strength decreases with a decrease in moisture and static electricity prevents proper ginning. It is recommended that seed cotton be stored for any length of time with moisture below 12% (Backe, 2002).

When ginning, moisture is critical in optimizing the stages of the gin. Too high moisture limits the seed cotton's ability to separate into locks. Unlike the locks, clumps of fibers can reduce the efficiency of the ginning process and even bring it to a halt. Trash removal is most efficient with moistures between 6% and 8%, optimally at 7%, which has been recommended from the 1950s (Backe, 2002).

When compressed in the bale, cotton moisture can change as the relative humidity changes. How densely the bale is packed, the temperature, the wrap type and how much of the bale is exposed to air dictate the bale moisture. Based on actual ginner recommendations, 8% moisture content is the ideal content for baled cotton. This also is the recommended moisture content at the bale press (Backe, 2002).

At higher relative humidity, cotton becomes more elastic, making drawing easier while producing stronger yarns. For cotton spinning, temperatures range from 80°F to 83°F, humidities are kept from 44% to 58% relative humidity, and moisture regains are kept from 5.9% to 7.7%. These ranges indicate the need for future research to recommend the optimal moistures for all stages of yarn manufacturing (Backe, 2002). On October 10, 2003 the National Council recommended that moisture levels of cotton bales at the gin not exceed a targeted level of approximately 7.5 percent.

Standard Tests

Currently, there are several standard methods for measuring cotton fiber moisture (Table 2). There are two ASTM standards and a published standard that tailors the ASTM method for the ginning industry, all involving oven drying. Several laboratories have also modified the standards to meet their specific time and criteria for testing as needed. There is a global standard that also involves oven drying that many countries have adopted.

One of these standards is ASTM 2495, which calls for the oven drying of cotton fibers. This method requires a fan-forced ventilation oven at 105°C +/- 2°C (220°F +/- 4°F) equipped with a balance. If the oven does not have a balance, weighing bottles and desiccators are needed. With fibers with at least 50% cotton a sample of 10 grams is required, whereas, if the sample is less than 50% cotton then a 5-gram sample is required. Sampling is discussed at the gin cleaners or condensers, mill openers and cleaners, and conveyers; sample quickly and place specimen in containers immediately. As for number of specimens required, three are needed for the oven dry method and two for the desiccator method. Preconditioning is not required since the purpose is to measure moisture at various stages of cotton processing. The original specimens are to be weighed. If very dry, the sample must be weighed in the container first then the weight of the container can be subtracted. If the sample is not very dry, containing more than 2% moisture, the sample can be weighed directly. The sample is then dried. If the sample is less than 50% cotton, then more than five hours of drying is needed. With a cotton fiber content of greater than 50%, the fibers are to be dried for at least one hour, with the test ceasing when the change in weighing is less than 0.1% of the original mass in subsequent fifteen-minute interval weighings. When the desiccator procedure is followed, this can be an extremely lengthy test since the specimens must cool to room temperature before weighing. The precision statement was last updated in 1969 for both within and between laboratories. As for accuracy, the method is considered a referee method with an admitted small bias due to ambient air that is used in the procedure (ASTM 2495, 2006).

Withdrawn in 1998, test method ASTM 2654 measured the moisture in textiles in addition to fibers. This method did require preconditioning of the samples. Four procedures are described. The first procedure takes ambient air that is heated to 105°C (220°F) in an oven. The sample is kept in the oven until the loss in mass levels off. It is intended to give an idea of moisture content, not an accurate measure. It was designed as a tool to reject materials that contain too much moisture. The second procedure takes standard textile testing conditioned air heated to 105°C (220°F). This test was to give the trade a means to accept a shipment. The third procedure takes specimen at moisture equilibrium, usually textile testing conditions, and oven dries them from air at a specific temperature and relative humidity. The fourth procedure takes a sample dried at a low temperature in a vacuum to arrive at the actual moisture regain. Each procedure is discussed in detail as to sample selection, preparation and testing, with many similarities to the ASTM 2495 method still in use. No precision or accuracy statement is given (ASTM 2654, 1997).

In order to tailor the ASTM methods for ginning research, Shepherd penned the "Standard Procedures for Foreign Matter and Moisture and Analytical Tests Used in Cotton Ginning Research" in 1972. This method has been cited in recent literature on moisture testing. This method calls for oven drying 50 grams of seed cotton at 105°C to 110 °C in a fan-forced ventilation oven with an internal balance. The method continues with a five-hour drying procedure followed by weighing and calculating moisture content. A two-hour procedure is also given with a formula to give the equivalent moisture content of the five-hour procedure. The precision was found using ten tests per lot giving a standard deviation of 0.5% and a 6.4% coefficient of variation. The method does state that there is no accuracy statement "because the true value of moisture content in seed cotton cannot be established by an acceptable reference method" (Shepherd, 1972). For a 95% probability, the number of tests that are required are tabled as five specimens needed for a 0.5% specific difference (Shepherd, 1972).

A method for oven drying ginned lint is also presented. As with seed cotton, a fan forced ventilation oven, with a balance, at 105°C to 110°C is needed. The sample size for this procedure is 20 grams. The ginned lint must be dried at least on hour using the same moisture content equation presented in the seed cotton procedure. The precision, using 10 tests per lot is 0.3% standard deviation and 5.9% coefficient of variation. The same statement of accuracy is presented. The number of tests needed at the 95% probability level is 5 for a specific difference of 0.3% (Shepherd, 1972).

For testing fibers, yarns and fabrics, AATCC 20A describes an oven method that does not require a fan-forced ventilation oven and an oven balance. Samples of at least 1 gram need not be conditioned prior to being heated in a 105° C to 110° C oven. The samples are removed from the oven, capped, cooled and then weighed after 15 minutes and, subsequently, in 30 minute intervals until the sample weight is constant. No accuracy or precision results are reported for this method (AATCC 20A, 2002).

A global standard, ISO 6741 exists mainly to determine a correct invoice weight, after drying and perhaps cleaning, where the textile material is bought and sold by weight. Large samples of at least 50 grams are not preconditioned before being weighed and heated in an oven at 105 ± 2° C (ISO 6741, 1989). Standard atmosphere is used for the air entering the fan-forced ventilation oven. If no oven drying duration is specified for the consignment, the sample is dried and weighed every 5 min until the difference in sample mass between two successive weighing is < 0.05 %.

Table 2. Standard Oven-Drying Moisture Measurement Tests and Their Quoted Accuracy and Precision

Method	**Status**	**Accuracy**	**Precision, Single Operator**
ASTM 2495	Current	None	4 reps, 0.374% critical difference
ASTM 2654	Withdrawn	None	None for procedure 3
Shepherd, seed cotton	Current	None	5 reps, 0.5% specific difference
Shepherd, lint	Current	None	5 reps, 0.3% specific difference
AATCC 20A	Current	None	None
ISO 6741	Current	None	None

Materials and Methods

For the quality control measures, a closed sample weighing bottle that had been cleaned was used to monitor the balance. A 1 gram bleached and scoured cotton sample in an open weighing bottle was used as a quality control measure of the environment around the balance. Both the empty bottle and the bottle with cotton were weighed prior to the weighing of the empty sample bottles, the filled sample bottles, and the heated sample bottles.

For the unknown cottons, six cottons were obtained from the Agricultural Marketing Service (AMS) in Memphis. Empty sample bottles were weighed and then filled with 1 gram samples and reweighed. Cottons were heated in an oven to 105-110°C for 24 hours. The samples were then cooled in a desiccator for one hour and then weighed. All weights were made in a standard conditioned laboratory.

Results and Discussion

To accentuate any nuances in the environment around the balance, an empty and closed weighing bottle and a 1 gram cotton sample in an open weighing bottle is kept in near proximity to the balance. These quality control samples are weighed prior to all other weight measurements to ensure the balance and environmental conditions are in control. When examining the quality control data, the weights of the empty bottle, Figure 1, vary only slightly (~ 1mg) indicating the balance is acceptable for use. The balance was not re-tared during the collection of the data in Figures 1 and 2. Additionally, several problems had been resolved before charting the data displayed in the figures. Contamination of the balance pan had resulted in a positive bias or drift in Figure 1. This was traced to residue on the wire shelves in the oven. Oven drying of cotton samples had corroded the shelves. Both oven shelves and balance pan were cleaned.

The open weighing bottle containing the 1 gram cotton sample, which had been bleached and scoured, shows a dramatic decline in weight (negative bias, ~ 30 mg) as the lab went out of condition, from 65% relative humidity to approximately 40% relative humidity (Figure 2). The weight of the cotton decreased over 2.5% with the change in relative humidity in the lab. This simple quality control measure indicates just how dramatically conditions can affect sample weight, and consequently, measured moisture content. This simple technique provides a direct way to determine if environmental conditions affect the weighings in the oven drying technique.

Figure 3 shows the trend line of the mean moisture content (one replicate on each of five testing dates, average values plotted) for the AMS cottons versus micronaire. A similar trend was observed with preliminary Karl Fischer results, although the values are somewhat smaller. We are developing this new reference method for moisture in cotton. In brief, the sample is heated in a oven, the moisture released is transported by a dry carrier gas into the reaction cell where it is titrated with iodine. The end point is determined by platinum indicating electrodes. During all measurements of sample weights for the oven method, the analytical balance and temperature and humidity of the testing lab were in control.

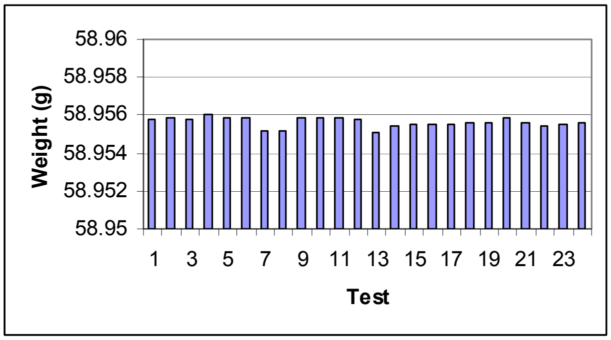

Figure 1 Quality control with closed empty bottle.

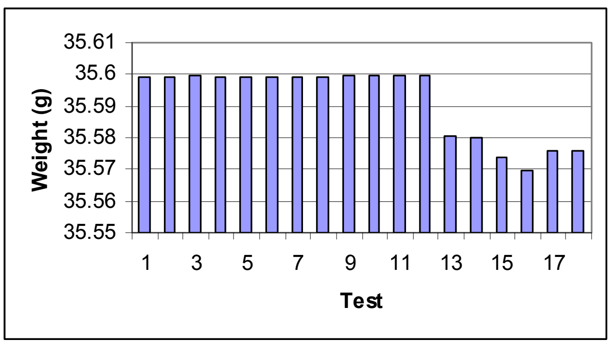

Figure 2. Quality control with bleached and scoured sample in open bottle.

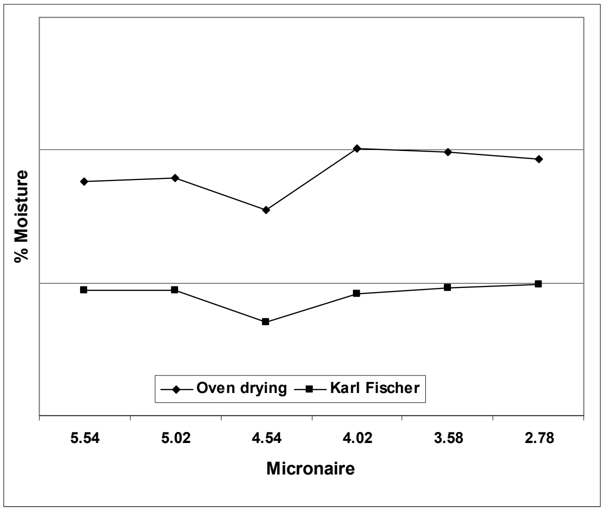

Figure 3. Percent moisture of six cottons.

Conclusions

As can be seen from the reviews of literature from many years of research, cotton fiber moisture is a very important aspect of cotton harvesting and processing. Moisture affects many important properties of cotton such as length, strength, and uniformity. Many recommendations for optimal moisture for various phases of the cotton lifecycle are present, while there is still a need for research to optimize moisture contents for some other aspects of processing. Because cotton fiber moisture is so important, its measurement is vital. With the accuracy of the current standard measurements in question by the cotton industry, and its precision information not updated for over three decades, there is a need for updated measurement technique(s). As Shepherd's 1972 modification of the ASTM 2495 standard states, there is no acceptable reference method because of a lack of demonstrated accuracy. The need for a accurate, precise reference method(s) with reduced bias would be a great help in determining the true moisture content of cotton fiber samples. For these reasons, some fundamental research is needed on cotton fiber moisture and its measurement. Simple quality control checks are presented and recommended.

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