

In the December/January edition, the interactions between cotton cellulose and moisture, and how these affected fibre properties and drying rates, were described. In this instalment the authors review the methods used to measure moisture of seed-cotton and lint, particularly during ginning.

Measuring moisture in cotton

By Stuart Gordon, Marinus van der Sluijs, Andrew Krajewski and Susan Horne

Measurement of moisture is critical particularly at harvest and through the gin, because of the influence these processes have on fibre quality. Dry cotton can be harvested cleanly and efficiently but may suffer undue damage in the gin. On the other hand harvesting and ginning wet cotton leads to significant issues in processing and quality.

A number of methods are used to measure moisture in seed-cotton, lint and fuzzy seed. Each has its advantages and shortcomings. Understanding these is important in order to be able to properly assess moisture.

Commercial regains

There is no commercial regain value for raw cotton for most international trade. A value of 8.5 per cent is specified in Rule 15 of Egyptian sales contracts and in Rule 105 of the Liverpool sales contract for Egyptian and Syrian cotton. Although such moisture regain values are intended for determining the commercial weight of fibre when it is bought or sold, obtaining a moisture content value of 8.5 per cent in any raw cotton is difficult to achieve under regular ambient conditions.

A value of 8.5 per cent is easier to achieve in processed cotton like yarns and fabrics that have been scoured, bleached (mercerised) and/or dyed. These processes remove the fibre's wax layer, which acts as a barrier to liquid water. In the case of bleached, mercerised and dyed cotton, the structure of the cotton cellulose

is also changed, making it more accessible to water molecules.

A maximum moisture regain of 11 per cent is placed on fuzzy cotton seed sales contracts although moisture is rarely measured and no price adjustments are applied on the basis of moisture regain. It is notable that other oilseeds, such as canola, linseed, and so on gain a two per cent deduction in price for each one per cent above the allowed moisture level when seed is destined for immediate processing, or a 1.5 per cent deduction for each one per cent above the allowed level, plus a drying charge, when seed is received for storage.

Modules of seed-cotton with moisture contents in excess of 12 per cent, as measured by hand-held resistance type moisture meters, prompt immediate action from growers and/or ginners because of the potential damage that can be created by the action of microbes on the cotton cellulose.

Approaches in measuring moisture

The methods for measuring moisture in cotton lint can be classified into six groups based on the technique and on the type of cotton material being tested, such as compacted or loose seed-cotton, loose lint moved by air in ducting or compressed baled lint.

Moisture measurement methods can be based on:

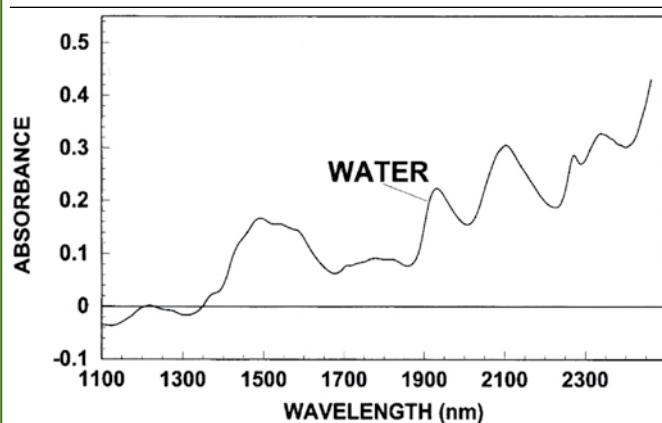
- Thermal drying;
- Chemical;
- Spectroscopy;
- Measurement of electrical or dielectric properties;
- Compression properties of cotton lint; and,
- Modelling of moisture uptake or loss based on temperature and relative humidity isotherms.

Thermal (gravimetric) methods – lint and seed-cotton

Standard methods for measuring lint and seed moisture largely rely on thermal drying of the sample. For these methods, the retention of bound moisture in the sample and liberation of volatile chemical components from fibre and particularly seed often confounds the ability to achieve a static 'dry' weight from the specimen.

Thermal (gravimetric) methods involve heating a pre-weighed fibre or seed-cotton sample to dryness for a prescribed period and then weighing the dried sample. The regain or moisture regain is then expressed as the ratio of mass of absorbed water to oven-dry mass of fibre (dry-basis). Moisture content is the ratio of mass of absorbed water to the total fibre mass (wet-basis). The equations for calculation of these expressions are listed below:

FIGURE 1: Typical near infrared absorbance spectrum for cotton lint showing main moisture band at 1930 nm

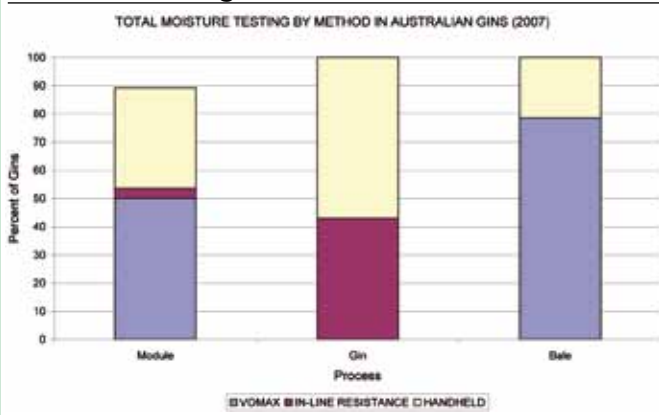


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FIGURE 2: Electrical methods used by Australian gins to measure moisture at the module, in the gin and at the bale



Equation 1: Moisture Regain (%) = [(M-D)/D] x 100

Equation 2: Moisture Content (%) = [(M-D)/M] x 100

Where: M = mass of (wet) specimen as received; and,
D = mass of oven-dried (dry) specimen

Both values are usually quoted as percentages and both quantities are often used inter-changeably leading to confusion about the quantities expressed. Measurement of moisture in this way is described in standard methods for determining the moisture content or regain of cotton fibre. Best results are obtained in physical laboratories where drying ovens and weighing scales are routinely calibrated and where the tared weight of drying vessels can be accurately determined.

There are no specific Australian standards for measuring moisture in cotton so the American Society for Testing and Materials (ASTM) standard is used. The standard designated ASTM 2495-07 describes the determination of the amount of moisture in cotton by oven-drying and is applicable to raw cotton, cotton stock in process and cotton waste.

In the standard lint specimens must be at least 5 grams and weighed to within ±0.01 grams. Specimens are dried in an oven at 105°C ±2°C for at least one hour or until the change in mass after a cooling or stabilisation period between successive weighing at intervals of at least 15 mins, is less than 0.1 per cent of the specimen mass. The difference between the original mass and the oven-dry mass is calculated in per cent, either as moisture content or moisture regain (see Equations 1 and 2). The standard covers the drying and weighing of specimens under a set of standard conditions. Alternative procedures for weighing the dried specimens are given – one procedure uses an oven balance (an oven with a balance in situ) while the other uses a desiccator to cool the specimen before weighing after each drying period.

Fuzzy seed

The Rules for Testing Seeds from the Association of Official Seed Analysts (AOSA) do not include standards for seed moisture determination. The International Seed Testing Association (ISTA) makes provisions for seed moisture testing of several crops and includes cotton in the category for which an oven drying temperature of 103 ±1°C for 17 ±1 hours is recommended. An ISO standard (ISO665:2000) exists for determining the moisture and volatile matter content in oilseeds, which is applicable to ginned cotton seeds, but not seed-cotton.

Other versions include those described by British and German

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Cotton Trader - Matthew Bradd

Ecom Commodities Pty Ltd
Suite 1, Level 19,
Tower A, Zenith Centre
821 - 843 Pacific Highway
Chatswood NSW 2067
P 02 9419 8300

Agents

Steve Dalton

AgVantage Commodities Pty Ltd
Namoj, Gwydir,
Mungindi and Boomi
P 02 6792 2962

Polly Gibbons

Front Gate
Darling Downs, Goondiwindi,
St George and Dirranbandi
P 0418 385 656

Don Cooper

Cooper Consulting
Central Queensland
and Dawson - Callide
P 0428 794 698

Peter Horton

Gilgandra Marketing
Co-operative Ltd
Macquarie Valley
P 02 6847 1116

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Institute Standards. In the ISO standard the moisture content of a test specimen is determined, either on the material as received (pure seed and impurities) or, if required, on the pure seed alone, by drying at 103°C ±2°C in an oven at atmospheric pressure, until a practically constant mass is reached as per the Standards for cotton fibre moisture determination.

Specimens should be prepared according to ISO664:2000, which describes extraction of oils and volatiles in methanol from the specimen before grinding, and then tested by drying according to ISO665:2000.

Chemical methods

Chemical analysis for moisture content involves a colorimetric or volumetric titration measurement of moisture that has been extracted from the fibre and/or seed. The most widely used chemical method is the Karl Fischer titration measurement of moisture content, which was originally based on a reagent containing pyridine (C₅H₅N), iodine and sulphur dioxide according to the Bunsen equation without the excess of water. Equation 3 shows the original formulation of the reaction.



The most important advantage of the Karl Fischer method over conventional thermal drying methods of moisture determination is its specificity for water.

Modern, automated Karl Fischer apparatus allows the moisture content of the captured specimen to be maintained and measured in sealed test bottles. But these automated versions are expensive and not trivial in their methodology.

Spectroscopic methods

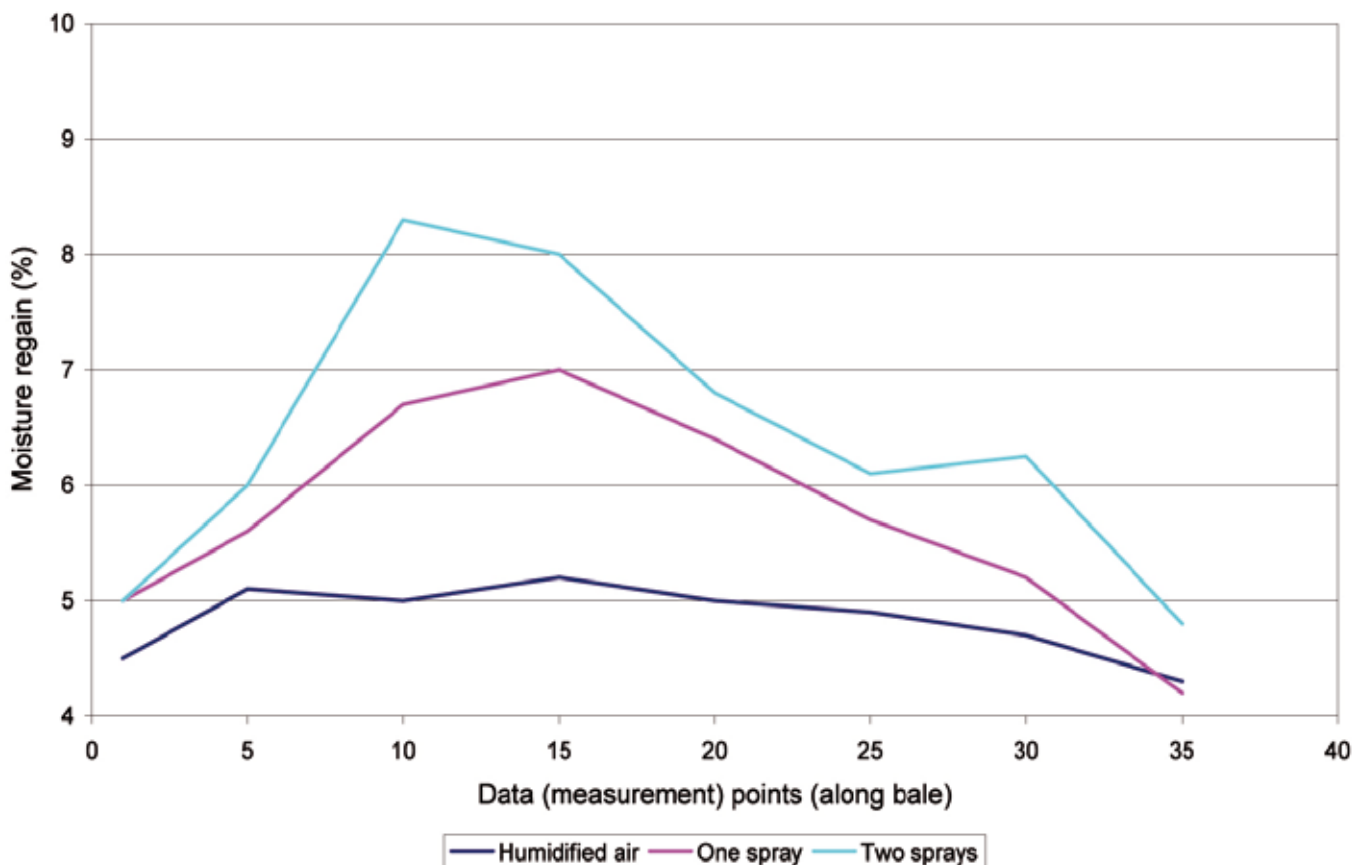
Spectroscopic methods for moisture determination involve the use of a spectrometer to measure the amount of electromagnetic energy of particular wavelengths absorbed (or reflected) by water molecules in different bonding states within the sample. The quantity of moisture is measured by the intensity of the moisture absorption (or reflectance) spectra.

The most used region of the electro-magnetic spectrum for this application is the near infrared (NI) region, for example, between 700–2500 nm. In particular the 1490 nm and 1930 nm wavelengths are known to be strong absorption regions for the OH group in water, and are often referred to as moisture bands.

For cotton, spectral differences with changing moisture are observed at 1490 nm, although a significant and distinct absorption due to cotton cellulose itself is also observed at this wavelength. At 1930 nm, the strong OH absorption is due to moisture in the fibre and not cotton cellulose itself. The OH absorption at 2170 nm also correlates strongly with the moisture content of the sample. Figure 1 shows a typical NI absorption spectrum of cotton. Other parts of the infrared region with longer wavelengths (above 2500 nm) also give information about moisture, although these are less sensitive in measuring the specific absorption energies of cotton cellulose and water molecule bonds.

Near infrared (NI) spectroscopy is used routinely to measure moisture and other chemical and physical attributes in a wide range of agricultural commodities, such as in the wheat grain industry where NI spectroscopy is used to assess moisture, protein and a range of other quality attributes. But its application to cotton is limited by the ability of NI radiation to penetrate the sample

FIGURE 3: Bale moisture profile in three bales subject to different moisture restoration treatments as measured by Uster Technologies FBM system



more than about one mm depth. As well, to ensure precision and accuracy, NI specimens need to be compressed to a constant density during analysis. Further, the difficulty in applying delicate (and expensive) spectroscopic instruments into rugged cotton harvesting, ginning and warehousing environments also limits their application.

Electrical methods

Electrical methods for measuring moisture in lint or seed-cotton specimens are based on measuring changes in electrical charge due to the moisture content of cotton and to some extent the presence of mineral salts in water and on the cotton. Electrical charge is typically measured in terms of resistance or permittivity (measured in relation to micro and radio-wave transmission).

Conductivity is low (resistance is high) in very dry cotton but is higher in cotton with some moisture. As such, static electricity is not generally a big problem in cotton except when it is very dry. Resistance plates and probes are used widely to measure moisture in seed-cotton or lint samples pre, in situ and post-gin. Microwave and radio-wave transmission sensors, which respond to the much larger dielectric of water, are used in denser seed-cotton and lint samples; pre and post ginning as modules or bales.

Figure 2 shows the methods of moisture determination used by Australian ginners. The results come from the recent gin best management practice (BMP) audits carried out by CSIRO. The Figure shows that most gins use an electrical method of moisture determination at all three 'stages' of ginning. Microwave systems were used to determine moisture in modules in 50 per cent of gins, and nearly 80 per cent of gins used a microwave system to measure bale moisture.

Hand-held resistance instruments were most used (55 per cent of gins) to determine moisture levels during ginning. The frequency with which hand-held resistance sensors are used is not known as records are not usually kept. The remainder of gins employed fixed in-line resistance instruments to test moisture during ginning.

A range of opinions is held about how accurate the results are from each of these sensors. The predominant view of resistance-based sensors is that they give satisfactory indications of wet or dry cotton, but their accuracy in the significant five per cent to eight per cent moisture range is not good enough for reliable metering of moisture or gas (for drying).

Microwave sensors employed for module and bale moisture measurements are believed to be more accurate, although as discussed later there are now commercial pressures to manage bale moisture and in particular the distribution of moisture in bales more closely.

CSIRO have recently tested a large capacitance-based device with the ability to measure the moisture of lint and/or seed-cotton instantaneously and continuously as it is transported through gin ducting.

Resistance-based methods

Resistance-based moisture sensors do not work well when water droplets are sprayed directly on the surface of the fibre. The combination of absorbed and surface moisture can cause errors in resistance-based moisture sensors, as the conductive effect of surface moisture and contaminants increases the measurement (of current). Nor do these sensors work well when moisture content is very low.

Furthermore, the specific resistance of lint at low moisture contents becomes very large, with a converse reduction in current.



A consequence is that resistance-based sensors need specially designed circuitry in order to manage the very wide range of currents produced between test electrodes subject to a normal range of moisture contents, for example, four per cent to 10 per cent. It is also the case that distortion and noise in measurements are amplified when the specimen is not applied correctly or consistently between or around electrodes.

When any type of resistance-based portable moisture meter is used, the accuracy and precision can be improved by:

- Following the manufacturer's recommendations;
- Using uniform, homogeneous samples of about the same weight;
- Wearing gloves to prevent moisture transfer from hand to sample;
- Placing each sample in the measuring cup immediately to minimise moisture change;
- Compressing each sample uniformly with the same amount of pressure applied each time;
- Checking instrument calibration; and,
- Replacing the battery or connecting mains power when needed.

Microwave methods

While techniques involving the transmission of microwaves (electromagnetic radiation with wavelengths between 10^{-3} and 0.3 m) through materials to determine moisture have been known for 50 years it was not until the late 1990s that a number of technical and cost difficulties associated with their application were overcome.

One issue particular to cotton bale moisture determination has been the alignment of the microwave transmitter so that the microwave energy from the transmitter to the receiver could pass fully through a bale in the alignment direction of the compressed layers that form a bale.

The boundaries of these layers have different densities and as such can cause the microwave energy to be internally reflected or scattered, affecting the accuracy of the predicted result. The VOMAX microwave system, which is built in Australia and widely used here and the US, claims to overcome this problem by directing and controlling the transmission and reception of microwave energy orthogonally through layers of the bale, rather than being restricted to the direction between the boundaries of one layer. Another aspect of the VOMAX microwave system is the range of

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frequencies transmitted; the range transmitted is noted to fit well with the geometry of the transmitter and receiver.

Existing microwave systems have a disadvantage in that they average moisture over large sensing areas and as such miss localised high moisture areas – for example, areas within the bale that might be in excess of 7.5 per cent. The US Department of Agriculture (USDA) has recently examined a method of providing a moisture profile of the bale using a new tomographic microwave imaging technique, and earlier in 2005 Uster Technologies introduced a bale moisture measurement system called the 'Final Bale Moisture' (FBM) system to measure the moisture profile of a bale.

The FBM relies on measuring the transmittance of ultra low frequency radiowaves (of 1 kHz) through a bale. The value of both techniques is their potential to provide ginners with feedback on the amount and distribution of moisture being applied at the battery condenser and lint slide.

By way of example, Figure 3 shows the moisture profile of bales treated with humidified air and one and two spray nozzles as measured by the FBM system. While the average moisture content of the bale treated with two sprays is less than 7.5 per cent, the FBM measured a number of points in the bale in excess of eight per cent. It is well accepted now that wet spots in excess of 7.5 per cent are likely to cause fibre quality degradation.

Information about moisture variation in the bale has not yet been deemed critical by Australian ginners and merchants, although its value to high throughput gins using atomiser spray systems to replenish moisture in dry bales is obvious. The company that manufactures the VOMAX instrument has indicated that measurement of bale moisture variation is also possible using their microwave transmission system.

Capacitive methods

Until recently the use of capacitors to measure moisture on the scale required in gins had been difficult to achieve due to the relatively small signal from the lint or seed-cotton and the large amount of electrical interference created in the gin environment.

New electronic circuitry with the ability to resolve small but significant capacitance signals has enabled CSIRO to develop a new sensing device to accurately measure the moisture of both seed-cotton or lint moved through gin ducts. The device combines large area capacitance plates with light detectors to measure the mass



and moisture of material travelling quickly under pneumatic pressure or gravity through gin ducts.

The advantage of the device is that it can accurately measure low or high density cotton or seed-cotton webs transported quickly through ducting, and that the measurement is based on all material in the duct at any particular time.

Compression methods

Measurement of the pressure differences required to compress dry and wet cotton bales has also been used to predict the moisture content of cotton in bales. As described previously, moisture affects the compressing ability and resilience of cotton. The USDA has investigated the pressure required to compress cotton into a bale as a method of moisture measurement.

In 2003 they reported a hydraulic-based system that could predict bale moisture in the range of 3.3 to 7.9 per cent. Measurements were based on bale weight, bale volume and the measured pressure once the bale volume had been reached. Issues with this type of measuring system are the expected restrictions in transferring one compression calibration to other presses that have different geometries, compression ratios and work in different micro-climates – with each of these variables having a significant impact upon the original calibration coefficients.

Modelling and predictive methods

Mathematical models that predict seed-cotton moisture content using the air temperature, air mass flow and seed-cotton mass flow to account for heat transfer from the conveying air, heat added to the room and the seed-cotton, and the heat added to the water in the seed cotton have also been developed. A model calibration by Gillum and Armijo involved two conditioning rates, two mix point temperatures, and two moisture contents. The model gave satisfactory estimates of seed-cotton moisture content, but did not perform well at moisture contents of less than six per cent, the level at which most cotton gins operate.

CONCLUSION

For standard measurements, thermal drying at the prescribed temperature, followed by a dry cooling or stabilisation period and accurate weighing remains the only option.

But the retention of bound moisture in the sample and liberation of volatile chemical components from fibre and particularly seed reduces the true accuracy of this method. In response to this, the USDA is currently exploring use of the Karl Fischer titration method as a standard because of its specificity for the bound water in cotton.

Measurement of moisture in-line during ginning is available, although the resistance sensors commonly employed to measure the moisture of seed-cotton or lint as it passes through gin ducting are limited in terms of accuracy and precision. A new CSIRO capacitor-based in-line sensor looks promising in this regard.

The measurement of moisture via microwave transmission through modules and bales stands as a relatively accurate and robust method. Accurate, in-line methods for measuring the moisture of material during harvesting and particularly ginning will become increasingly important as energy costs and fibre quality premiums rise.

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¹CSIRO Materials Science and Engineering, Henry Street, Belmont VIC 3216.

