Liquid Coatings on Fibres & Fabrics   Application Note 22

Summary

- Up to 250 times faster than wet chemistry methods

- No hazardous solvents required; no hazardous waste produced

- Easiest, most reliable technique available – suitable for unskilled personnel

- Reliable measurements down to low concentrations

- Ideal for high sample throughput

Introduction

The textile industry is one of the largest industries in global commerce. With many hundreds of years of technological development, today's industry provides an almost unlimited variety of products, many of which are carefully optimised for specific applications. Even high-volume commodity products use a sophisticated understanding of the properties and behaviour of fibres to provide the performance and functionality that is required.

The Role of Liquid Coatings in the Textile Industry

One aspect of the technology routinely utilised is the use of liquid coatings to control and modify the behaviour of fibres. These liquid coatings are applied at a variety of stages in the manufacturing process, sometimes as early as fibre extrusion in synthetic fibre manufacture, but sometimes as late as application to woven or non-woven fabrics.   Liquid coatings are known by a variety of names, such as:

- Spin Finish (SF)
- Finish on Fibre (FoF)
- Oil Pick-Up (OPU)
- Carding Lubricant
- Carding Oil
- Staple Fibre Finish (SFF)
- Texturising Oil
- Coning Oil
- Finish on Yarn (FoY)
- Lubricant on Thread (LoT)
- Yarn Lubricant (LY)
- Needle Oil
- Agents d'Avivage
- Avivagen

The name used depends partly on application, and partly on geography.   To encompass all these variants, the term liquid coating will be used here.

Whatever name is preferred, these liquid coatings are carefully formulated products, often combining many components. Liquid coatings may be oil- or aqueous-based, and may include many substances, such as oils, soaps, surfactants, waxes, anti-statics, and others.

They may be true liquids, emulsions, or suspensions.   They are designed to perform a number of functions, particularly control of inter-fibre friction, static charge, inter-fibre cohesion, heat generation and heat dissipation.   They may also be used to control appearance, feel, and texture.

Application of the correct amount of liquid coating is critical for both performance and economic reasons.   Too much coating can lead to insufficient inter-fibre cohesion, build-up on downstream machinery, uncontrolled fibre behaviour, problems with downstream processes such as dyeing, and, of course, too much coating is an unnecessary increase in cost in a very cost-sensitive industry.

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Liquid Coatings on Fibres & Fabrics

Conversely, too little coating leads to problems from excessive friction, likely to cause yarn breakage, excessive static charge build-up, causing erratic fibre behaviour, and uncontrolled performance in downstream processes.

For these reasons, constant attention to ensuring the application of the correct amount of liquid coating is essential to efficient and profitable fibre, yarn, and fabric processing.   This constant attention means that in a busy manufacturing facility, many tens or hundreds of samples should be measured each day.

Measurement Approaches

The amount of liquid coating on fibres, yarns, and fabrics has been measured for many years.   The traditional method has been to take a set length or weight of sample, and to carry out a chemical extraction using an organic solvent, either at room temperature or an elevated temperature. This is assumed to remove all of the liquid coating from the fibres, transferring it into the solvent. Subsequently, the quantity of liquid coating is determined after evaporation of the solvent or indirectly from the loss in mass of the fibres due to the extraction.   Solvent extraction has a number of features that make it unattractive in an industrial environment:

- It is time consuming
- It requires skilled operators
- It uses flammable and hazardous solvents

- It needs to be conducted in a fume cupboard / fume hood
- The consistency of results depends upon the skill of the operator
- Environmentally acceptable disposal of solvents is costly

The use of Nuclear Magnetic Resonance (NMR) for the measurement of liquid coatings on fibres, yarns and fabrics addresses all of these issues:

- No solvent extraction is required
- Measurement can be carried out by process operators with minimal training
- Results are consistent and operator-independent
- The instrument needs only a benchtop in a reasonably clean and stable-temperature environment; no fume cupboard is required
- Measurement is fast, allowing hundreds of samples to be measured each day

In addition, NMR has other advantages:

- NMR is a bulk measurement technique; it measures the whole sample equally, and is not affected by air voids
- NMR is generally insensitive to colour
- NMR measurement is non-destructive, so measurements can be easily repeated
- NMR is stable over long periods, and has minimal recalibration requirements

The advantages of using NMR for measuring liquid coating on fibres, yarns, and fabrics are widely accepted in the textile industry, and NMR is the method of choice in hundreds or thousands of textile manufacturing plants all over the world.

NMR Methods

The NMR instrument can distinguish between the hydrogen signal from solids (such as fibre, yarn, textile or nonwoven fabric) and liquids (such as an oil-based coating). The same instrument can (with a different probe) also measure fluorine based liquid coatings.

Two variants of the NMR technique are available:

- The weighing method
- The non-weighing method

The choice of which is more appropriate depends upon a number of factors, including the level of liquid coating applied, the accuracy of results required, and the sample throughput needed.

All samples can be measured by the weighing method. In this method, the liquid signal from the NMR is ratioed against the total mass of solid (fibre) and liquid (coating) obtained by weighing the sample. The weighing method is the most accurate method, and is also most suitable where low levels of liquid coating are applied (for example <0.3 %).

The non-weighing method also uses the liquid signal from the NMR measurement, but in this case the liquid NMR measurement is ratioed against the total NMR signal (from both solid and liquid). This avoids the need to weigh the sample, and, whilst not quite as accurate as the weighing method, it is faster (no weighing is required), and sufficiently accurate for most applications. The Oxford Instruments **MQC+** instrument can be used with either the weighing or non-weighing measurement.

Fibre Types

An enormous and ever-expanding range of fibre types are available. The main types can be broadly categorised into three classes, and several subclasses;

- Natural Fibres
    - Cotton
    - Wool
    - Linen
    - Silk
- Reconstituted Fibres
    - Rayon (Viscose)
    - Cellulose Acetate
    - Cellulose Triacetate
- Synthetic Fibres
    - Polyester
        - PolyEthylene Terephthalate (PET)
        - PolyButylene Terephthalate (PBT)
    - Polyamide (Nylon, Tactel, Cordura)
    - Polypropylene (PP)
    - PolyAcryloNitrile (PAN, Acrylic, Dralon, Orlon, Acrilan)
    - Elastane (Spandex, Lycra, Polyurethane-Polyurea Copolymer)
    - PolyVinyl Alcohol (PVA, PVOH)
    - Aramid (Kevlar, Twaron, Nomex)
    - Carbon

A similarly broad range of liquid coatings has been developed, each tailored for use with particular fibres, and often specifically developed to optimise the fibre properties for a particular use or application.

NMR measurements are commonly used for synthetic and reconstituted fibres, such as polyesters, polyamides, polypropylene, elastanes, rayons, cellulose acetates, and aramids.

Making Measurements by NMR

Making a measurement of liquid coating level by NMR is a very simple process with 5 to 7 steps, depending on the method chosen.

1   A sample (usually a fixed length of fibre or yarn, or a defined area of textile) is taken from the production line.

2   The sample is pushed into a sample tube, which may be made of either glass or FEP (a polymer that does not contain hydrogen).

3   If the weighing method is being used, the sample is weighed in a tared tube.

4   The sample is then compressed to the optimum height using a PTFE stopper.

5   For accurate measurements, the tube containing the sample is equilibrated at room temperature or in a conditioning block at an elevated temperature (as appropriate).

6   The sample is then inserted into the instrument. Measurement time is usually 1 minute per sample, however may be longer for very low concentrations (< 0.3%).

7   The coating level is then automatically displayed on the screen of the instrument.

Nylon fibre calibration

$R^2 = 0.9995$

Signal Ratio vs % Spin Finish
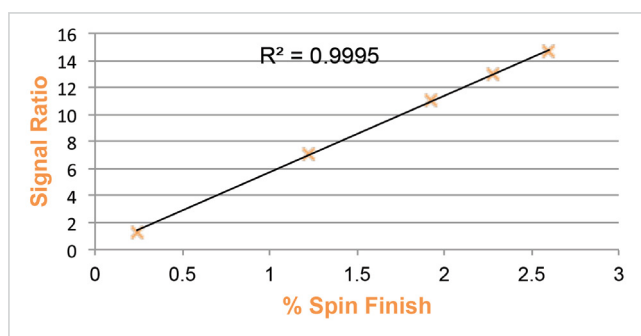
Polyvinyl alcohol (PVA) fibre calibration

$R^2 = 0.9948$

Signal/Mass vs % Spin Finish
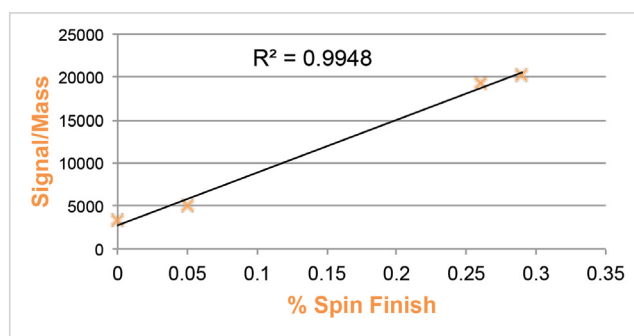
Calibration

Measurement of liquid coating by NMR is a secondary technique, therefore it is necessary to create a calibration, using real samples for which the liquid coating level has been measured using a reference technique.   In theory, only two standards are required to produce a linear calibration; however this is reliant on the accuracy of the reference technique.   To be prudent, it is recommended that 3 – 6 reference samples are used, with liquid coating concentrations evenly spread over the range of interest.

NMR is a comparative technique therefore its accuracy is only as good as that of the reference technique used to create the calibration.

Once the initial calibration has been set up, calibration maintenance can be accomplished using our Calibration Maintenance Standards (CMSs).   The CMSs can be periodically used to correct for any slight drifts in the instrument, without needing to measure a new set of reference samples. They are stable over very long periods, and can be used to maintain the highest performance with minimum effort.

Performance

Concentrations as low as 0.05% liquid coating have been measured by NMR using the weighing method; there is no upper limit to the concentration that can be measured. Samples must be dried prior to testing if moisture is in excess of 10%, but this is rarely the case with synthetic fibres. Ultimately the method and performance is dependent on the fibre and liquid coating being used, therefore, if not already measured by NMR, it is recommended that samples are tested by our applications specialists.

Conclusion

NMR is an attractive, accurate, fast, reliable and easy-to-use technique for the measurement of liquid coating levels in the textile industry. It is widely accepted, and makes a meaningful contribution to the most efficient operation of a textile plant.

Complete Packages

Oxford Instruments offers two packages especially tailored to the measurement of liquid coatings on textile fibres.

The Standard package is:

- Oxford Instruments **MQC+**23 NMR Analyser
  - 0.55 Tesla (23 MHz) high homogeneity magnet
  - Probe for 18 mm diameter sample tubes (7 ml sample volume)
  - Integrated system controller (no external PC required)
  - Integrated flat-screen display
- **MultiQuant** software including **RI Calibration**, **RI Analysis**, and the **EasyCal** 'Spin Finish' application, including both weighing and non-weighing method capability
- 18 mm glass tubes or 18 mm FEP tubes
- PTFE tube stoppers (to fix the sample height)
- Stopper insertion/removal tool
- Instrument tuning sample
- User manual
- Method sheet

For larger samples, we recommend the Advanced package which consists of:

- Oxford Instruments **MQC+**23 NMR Analyser

  - 0.55 Tesla (23 MHz) high homogeneity magnet

  - Probe for 26 mm diameter sample tubes (14 ml sample volume)

  - Integrated system controller (no external PC required)

  - Integrated flat-screen display

- **MultiQuant** software including **RI Calibration**, **RI Analysis**, and the **EasyCal** 'Spin Finish' application, including both weighing and non-weighing method capability

- 26 mm glass tubes or 26 mm FEP tubes

- PTFE tube stoppers (to fix the sample height)

- Stopper insertion/removal tool

- Instrument tuning sample

- User manual

- Method sheet

Optional items are:

- A dry block heater with holes for sample conditioning at 40°C (18 mm or 26 mm)

- A precision balance (only required for weighing method)

- A set of four Calibration Maintenance Standards for Spin Finish

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