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Fundamental Principles of Fibre Fineness Measurement

Part 5

Gravimetry

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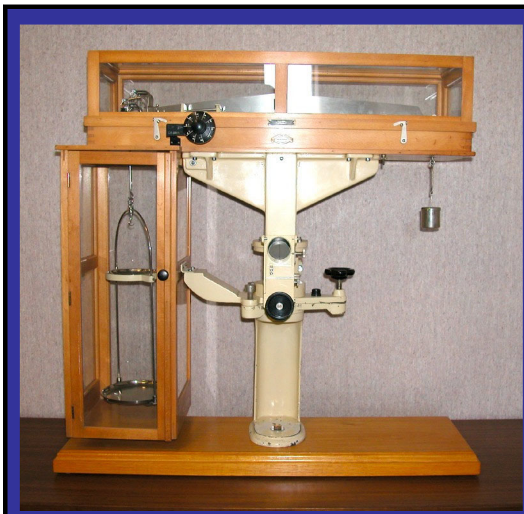
GRAVIMETRY

Principle

IWTO initially adopted its unit of fibre fineness as the weight in milligrams of 10 metres of wool fibres at a regain of 18.5% (Von Bergen, 1932). The method relied on weighing a definite number of fibres cut to a certain length and expressing the mean fineness in terms of the weight of a standard length at a standard regain. Subsequent applications of the gravimetric method used the relationship between mass, volume and density to define the fibre fineness in terms of its cross-sectional area.



Gravimetry, or mass (weight) measurement, is probably the oldest analytical technique known to man.

$$Density = \frac{Mass}{Volume} = \frac{Mass}{Length\ x\ Area}$$

Therefore $$Area = \frac{Mass}{Density\ x\ Length}$$

By assuming a circular cross-section, and a uniform density, the fineness can be expressed in terms of the mean diameter of a circle of equivalent cross-sectional area.

$$D_g^2 = \frac{4}{\pi} \cdot \frac{m}{\rho l}$$

Where D_g = mean diameter of the equivalent circle;

m = mass of the fibre sample;

l = total length of the fibres in the sample; and

ρ = mean density of the sample.

The fibre fineness can then be defined as the *root mean square diameter,* i.e.

$$D_g = \sqrt{\frac{4m}{\pi\rho\, l}}$$

For the gravimetric method, although the measurement is based on a sample consisting of a discrete number of fibres, each fibre is effectively represented at all points along its length. In other words, if we imagine all the fibres in the sample to be laid end on end the method effectively measures the average cross-section over the whole length, and then calculates the average diameter on the assumption all the fibres are circular. The measurement is therefore an estimate of the mean for the bulk.

In contrast, as we have already seen, the Projection Microscope profile method, measures individual fibre snippets at a single point randomly located along the length of the snippet. Providing that the sample is length biased, and each snippet is measured only once, the profile measurement also estimates the mean thickness of the fibres, D_p, in the bulk. Thus if we assume circularity, and d is the diameter at any point along a fibre,

$$D_g = \sqrt{\sum d^2 / n}$$

and $$D_p = \frac{\sum d}{n}$$

Now $$\sum d^2 = n\left(\frac{\sum d}{n}\right)^2 + n\sigma^2$$

where $\sigma =$ the standard deviation of d.

From these equations it follows that:

$$D_g = \sqrt{\left(D_p^2 + \sigma^2\right)}$$

or the root mean square diameter

$$D_g = D_p\sqrt{1 + c^2}$$

where $c =$ fractional coefficient of variation of d.

In Palmers notation (see For Technophiles - January 2002) this is defined as $\sqrt{\left(l, d^2\right)}$.

Development

The gravimetric method has never been advanced to a standard test method. Nevertheless it was widely used in the period 1930 – 1950.

Von Bergen (1932), reported the results of comparisons of gravimetric measurements on wool tops, compared with measurements based on fibre cross-sections and fibre widths determined by optical examination through a microscope. A selection of the data he reported is summarised in Table 1.

Table 1: Comparison of Gravimetric Measurement of fineness of top with two Microscope Methods

Quality Number	Gravimetric Method		Width Method
80's	19.6		19.5
70's	20.4	20.7	20.8
64's	22.3		21.9
60's	24.3	24.4	23.5
58's	25.7		24.8
56's	28.1	27.7	26.9
50's	31.1		30.4
48's	32.9	33.8	33.0

Von Bergen remarked, "....*there was an astonishing conformity of results*".

Palmer (1948, 1951) reported the results of the 1948 inter-laboratory diameter and length experiment involving 15 international laboratories and using 6 tops. This followed an earlier experiment on a smaller scale conducted in 1947, which was designed to test the reproducibility between laboratories of three different methods, one involving optical measurements by microscope and the remaining methods being two different gravimetric methods.

One of these methods, developed by WIRA (Wool Industry Research Association) obtained a sample of fibres by a cut squaring procedure. The length of each fibre was measured by stretching between two pairs of forceps. The measured fibres were collected, cleaned, conditioned and weighed. The weight of the fibres was multiplied by a constant and divided by the total length of all the fibres measured. The square root of this gave the length proportioned root mean square diameter.

A modification of this method involved measuring the length of each fibre under constant tension, by hanging each fibre with a constant mass attached to the free end. The purpose of this was to determine whether the different amount of stretch applied to each fibre by different observers using the forceps technique, was an appreciable source of error.

The alternative method, developed by Maillard and Roehrich, involved sorting the fibres into length groups. Cutting known lengths of fibres from each group and determining their weight enabled the root mean square diameter of each length group to be calculated. The root mean square diameter of the whole material was determined by calculating the weighted mean of the results for the separate length groups.

Palmer concluded that the *modified* WIRA gravimetric method improved the precision of the measurement within and between laboratories, and that the variable amount of stretch applied to the fibres by different operators using the forceps method was an appreciable source of error. He concluded that the Maillard-Roehrich method gave more variability within laboratories, and suggested the major sources of this variation arose from stretching of the fibres when the constant length sections were prepared from each class interval, and from the preparation, conditioning and weighing of the fibres. The trial confirmed that the WIRA gravimetric method and the modified WIRA gravimetric method gave good agreement with the Projection Microscope, for the root mean square fibre diameter, with the Maillard-Roehrich method giving results approximately 0.5 - 1.0 microns higher (Table 2).

Palmer noted that not all laboratories involved in the trial were within statistical control. The error limits quoted in Table 2 are for all laboratories and are therefore slightly higher than for those laboratories that were in control. This particular experiment marked a significant milestone for IWTO in that it was the beginning of the formal development of IWTO standard methods for estimating the fineness of wool fibre.

TABLE 2: Comparison of mean diameter determined by the four methods used for the 1948 Inter-laboratory Diameter Experiment (Palmer, 1948, 1951)

Top Identification	Projection Microscope	WIRA Gravimetric Method	Modified WIRA Gravimetric Method	Maillard-Roehrich Gravimetric Method
CG	19.52	19.69	19.62	20.46
BL	20.52	20.56	20.59	21.08
FJ	21.14	21.56	21.62	21.92
AD	21.46	21.69	21.82	22.20
HI	22.12	22.44	22.56	22.60
EK	24.41	24.56	24.60	25.04
Error (all labs)	0.63	0.60	0.63	0.84

Andrews and Irvine (1969) proposed a method for measuring the gravimetric diameter by using small snippets instead of full-length fibres. The novelty of the method was that the fibres were cut into snippets short enough to be easily specified. Since the number of length measurements required for a test then became too large to be practicable, an estimate is obtained of the total length of snippets in the weighed sample. Firstly the total number of snippets, *N*, was counted using a Coulter Counter. Secondly, the individual lengths in another, much smaller, sample of the snippets were measured with a Projection Microscope and averaged. The product of *N* and the average snippet length is an estimate of the total length of fibre in the sample. In such a numerical sample of snippets, cut from the original sliver or assembly, the total length of snippets in each small interval of diameter must be proportional to the total length of fibres in the same diameter interval in the original assembly. Each fibre is therefore represented in proportion to its length, as is the case with the intact fibre gravimetric method. The precision for the method was reported to be better than 0.2 μm.

Technical Issues

Gravimetric methods do not provide an estimate of the standard deviation (or coefficient of variation) of the estimated fibre diameter.

Their basic limitation rests with the measuring the length of the individual fibres. This limits the precision of the method because of the uncertainty surrounding the amount of stretching that occurs during this measurement. Furthermore, owing to the necessarily few fibres that can be measured in a reasonable time, the sampling error further limits the precision. This is the same limitation that applies to the Projection Microscope. Although Andrews and Irvine (1969) did demonstrate that the method is capable of improvement, little further progress has been made, and for wool the method remains relatively underdeveloped. However, gravimetric measurement is widely used for estimating the fineness of synthetic textile fibres, and in such cases is often the only practical method given the enormous divergence from circularity of many synthetic fibres.

The method does rely on the presumption that the density of wool fibres is relatively constant. This is clearly not the case with medullated fibres, and this limits its general applicability. There is evidence that the fibre density of individual farm lots can vary by small but significant amounts from the generally accepted value of 1.310 g/cm^3 (Van Wyk and Nel, 1940, Connell & Andrews, 1974). This means that for very precise work it may be necessary to measure the density of the sample in order to reduce small differences in estimates arising from density differences alone.

However, if the density of the sample is also measured, then the gravimetric method is one of only two methods that approach the status of primary measurement systems. Also, the gravimetric method provides a totally unambiguous definition of fineness, in that the reported diameter is independent of the shape of the fibre cross-section.

Commercial Issues

The absence of a standard test method is the major commercial limitation of the gravimetric method. Also, the cost of measurements based on gravimetric methods severely inhibit its commercial usefulness, in the same way as the costs of the Projection Microscope measurement have limited the commercial application of the Projection Microscope standard method. Furthermore gravimetric methods do not provide distribution data.

However the gravimetric method does have the potential to provide a primary measurement system, linked directly to SI units, for wool fibre fineness measurement, and thereby provide standard reference material that is traceable to the SI standards for weight and length. This does depend upon the availability of a suitable technique for accurately determining the fibre density. The commercial benefit would be a more fundamental basis for calibrating any appropriate secondary test procedure for use in determining the conformity of deliveries to contract specifications.

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