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Analysis of the torsional storage modulus of human hair and its relation to hair morphology and cosmetic processing

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Synopsis

Through measurements of three different hair samples (virgin and treated) by the torsional pendulum method (22°C, 22% RH) a systematic decrease of the torsional storage modulus G' with increasing fiber diameter, i.e., polar moment of inertia, is observed. G' is therefore not a material constant for hair. This change of G' implies a systematic component of data variance, which significantly contributes to the limitations of the torsional method for cosmetic claim support. Fitting the data on the basis of a core/shell model for cortex and cuticle enables to separate this systematic component of variance and to greatly enhance the discriminative power of the test. The fitting procedure also provides values for the torsional storage moduli of the morphological components, confirming that the cuticle modulus is substantially higher than that of the cortex. The results give consistent insight into the changes imparted to the morphological components by the cosmetic treatments.

INTRODUCTION

Mechanical tests on human hair are essential tools to determine their properties as guides for product research and development (1). In extension, all morphological components contribute to the parameter values according to their fractions in the cross-sectional area, while a very strong bias towards the outside layers will occur for bending and torsion. Therefore, a strong influence by the surface layer of a hair, i.e., by the cuticle, can be expected for the latter tests. These tests should thus be especially suitable to detect changes on or near the hair surface through cosmetic processes and products, which may pass undetected in tensile testing.

Applying analytical technology described by Persaud and Kamath (2), the torsional properties of European human hair at conditions of low humidity were investigated to

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determine, namely, the precision of the measurements. The potential of the method to detect the effects of cosmetic processes and products was assessed for perm-waving combined with bleaching as a harsh cosmetic process to impart hair damage, as well as for the further application of a shampoo.

Specifically, in investigations of the torsional storage modulus G', a substantial variability of this parameter was observed for human hair, e.g., by Harper and Kamath (3), which impacts negatively on the ability of the method to discriminate between different samples and treatments. A substantial part of this variability can be traced to the fact that the storage modulus for hair is in fact not a material property, but shows a significant dependence on fiber geometry, e.g., a decrease with fiber diameter (2). This phenomenon has been observed over the whole range of humidities (2) and introduces a systematic error, which in turn creates obvious problems with the analysis of the data (4).

This systematic error of G', deriving from the violation of the assumption of fiber geometry invariance, is determined for a set of data for virgin as well as cosmetically treated hair and analyzed on the basis of the cortex/cuticle structure of hair. This leads to a largely improved discriminative power of hair torsional measurements for the influences of cosmetic processes and products, as well as to values for the torsional storage moduli of the two morphological components.

MATERIALS AND METHODS

PRINCIPLES AND APPLICATION OF THE TORSIONAL PENDULUM METHOD

All experiments on hair fibers were conducted on a single fiber torsion pendulum apparatus (TRI/Princeton, NJ) as described by Persaud and Kamath (2). For the measurements, so-called brass crimps with an internal silicone polymer tube (Dia-Stron, Andover, United Kingdom) were attached to both ends of a 5-cm-long hair fiber, leaving an effective testing length of 3 cm. One crimp of a fiber was introduced into the upper clamp of the instrument, while to the other, a cylindrical torsional weight (weight: 5 g) was attached. The geometry of the cylinder and thus its moment of inertia were chosen such as to provide a frequency of the torsional oscillation of about 0.1 Hz for the chosen hair material. For the test, the fiber was twisted through 360° and released. The machine monitored the torsional oscillation movement of the cylinder and determined frequency and amplitudes. The instrument was enclosed in a chamber, which provided controlled environmental conditions (22°C, 22% RH). This low humidity was chosen, since it was expected to provide the best discrimination between cosmetic treatments (3). Twisting angle and the torsional weight impart only low shear and tensile strains, well within the linear viscoelastic region of a hair fiber (5,6). Pre-conditioning and storage conditions were chosen such as to avoid effects of physical ageing (7,8).

One primary parameter determined by the test is the torsional storage modulus G':

$$G' = \frac{Jl\omega^2}{I} \qquad (1)$$

where J is the moment of inertia of the pendulum, l the length of the fiber, I the polar moment of inertia of the fiber, and ω the frequency of oscillation, with:

$$\omega = \frac{2\pi}{T} \tag{2}$$

where T is the time taken for one oscillation. Equation 1 applies if the damping of the torsional oscillation, i.e., the dissipation of torsional oscillation as frictional energy, is low. Though hair is inherently a viscoelastic material, it is consistently below its glass transition temperature for low to medium relative humidities (9), showing in consequence long relaxation times and thus little damping (7,10,11).

The specific cross-sectional shape of a human hair, though irregular along its length, is generally assumed to be elliptical, so that the polar moment of inertia is given by:

$$I = \left(\frac{\pi}{4}\right) \cdot (a^3 b + a b^3) \tag{3}$$

where a is the long and b the short semi-axis of the ellipse, respectively.

Arithmetic means for G' were determined from individual values for T, as determined along the oscillation curve and according to Equations 1 and 2. Five repeat measurements were conducted for each fiber, averaged and taken as the G′-value for that fiber. Values for fibers tested for a sample were further summarized by their arithmetic mean, variance, standard error (S.E.), and the limiting value for the 95% confidence range (1.96 S.E.) (4). Data sets were compared using the Fisher least significant difference (LSD) test (12). This is essentially a multiple t-test and as such very non-conservative (4). It therefore appears generally well suited for testing in the context of cosmetic products and processes. Also separate t-tests for specific pairs of samples were conducted.

CHARACTERIZATION OF HAIR GEOMETRY

All tests and treatments were conducted on dark brown, commercial, Caucasian hair (International Hair Importers & Products Inc., Glendale, NY). The fibers were washed with 3% sodium lauryl sulfate (BASF, Duesseldorf, Germany), rinsed thoroughly with warm water, and allowed to air-dry under ambient conditions.

To determine the cross-sectional shape of a fiber, the assumption of general ellipticity was made. For each fiber, prepared for torsional testing, the smallest and largest diameters were determined at five equidistant positions of a 3-cm-long fiber through a 360° rotation by means of a Laser Scan Micrometer (LSM-500, Mitutoyo, Kanagawa, Japan), as implemented by Dia-Stron Ltd., United Kingdom. At each point along the fiber, the smallest and largest diameter was determined. The arithmetic means of the measurement data were used to calculate the overall short and the long axis of the elliptical fiber cross section. Diameter measurements were conducted under conditions of constant, but ambient laboratory climate (approximately 22°C, 55% RH). The diameter data thus obtained were taken as basis for the determination of the moment of inertia of a fiber according to Equation 3. No corrections were applied for the limited changes of cross-sectional shape at the conditions of torsional measurement (22 ± 2°C, 22 ± 2% RH).

HAIR TREATMENTS

Hair tresses were taken from the collective of virgin hair, to be referred to as *V* in what follows, and submitted to a permanent waving treatment with 7% thioglycolic acid at pH 9.5, adjusted with ammonia. The solution was applied in excess to the tresses for 30 min.

This was followed by extensive rinsing and reoxidation with 2.2% hydrogen peroxide solution, adjusted to pH 4.0 with citric acid for 30 min. Finally, the tresses were again extensively rinsed and then dried with a hair drier at ambient temperature. To further sensitize the hair, it was subsequently subjected to a strong bleaching treatment with an 8% hydrogen peroxide/persulfate combination at pH 9.4 for 30 min. Subsequently, the hairs were extensively rinsed and dried with a hair drier at ambient temperature. In what follows, hair from tresses, which have undergone this treatment sequence will be referred to as *WB* (permanently waved and bleached).

To further reflect the general context of hair cosmetic treatments as well as to target more subtle changes of hair, a commercial shampoo was applied, which claims to be able to repair the hair surface. The shampoo was applied on WB-type hair fibers already fitted with crimps and thus ready for torsional testing. The hairs were wetted and then covered with the shampoo for 30 min. Subsequently, the shampoo was rinsed off for 1 min and then the hair dried with an air drier for 15 min at ambient temperature. The long contact time was chosen to maximize the potential effect of the shampoo as well as to reflect the effects of repeated applications. In what follows hairs, which have undergone all treatments will be referred to as *WBS* (permanently waved, bleached, and shampoo treated).

THE CORE/SHELL MODEL FOR HAIR

To analyze the dependence of G' on the moment of inertia, as documented for all samples in Figure 1, a core/shell model for hair is applied. For an individual human hair, the cortex is surrounded by a layer of cuticle cells, which are arranged in a tile-like fashion with each cuticle cell being in contact with the cortex as well as being visible on the fiber surface (13). The resulting tilt angle away from the fiber surface is 2–3° (14,15). In the cross section of a hair fiber, this arrangement presents itself as a sequence of concentric layers of cuticle cells around the cortex. The thickness of each layer is that of an individual cuticle cell and for human hair generally determined as approximately 0.5 μm. The total number of cuticle layers for undamaged hair near the scalp is expected to be 6–10, leading to an initial cross-sectional thickness of the cuticle layer of 3–5 μm (13).

For the hair fiber collective used for these investigations, the overall cuticle thickness was found microscopically in cross sections to be largely independent of diameter and about 3 μm, equivalent to six layers. In view of the relatively short length of a sample fiber (3 cm effective length) and its probable origin with respect to the scalp (approximately 10 cm for

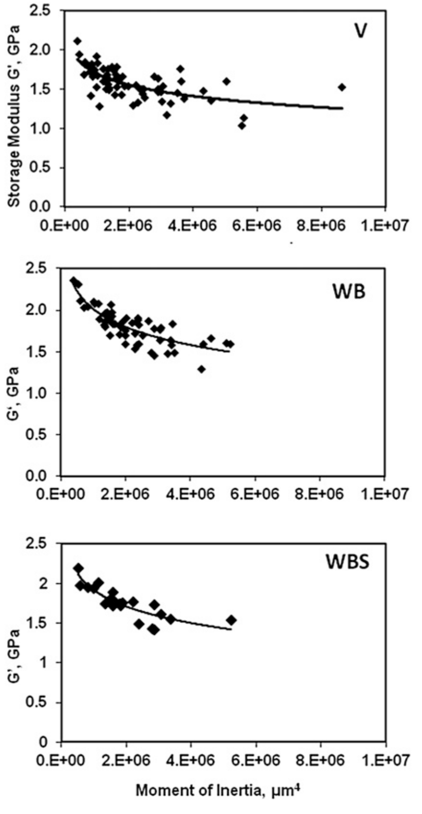

Figure 1. G′-values for individual fibers from samples of virgin (V), perm-waved and bleached (WB), plus shampoo-treated (WBS) hair at 22°C and 22% RH. The values are plotted against moments of inertia (Equation 3). The solid lines through the data are fits according to Equation 4 with the parameter values given in Table I.

commercial hair), this value is assumed to be constant along the fiber, in good agreement with observations of cuticle-wear patterns by Garcia *et al*. (16).

Implementing the ring/core-structure of cuticle and cortex, Equation 1 yields:

$$G' = \frac{(G'_{co} I_{co} + G'_{cu} I_{cu})}{I} \quad (4)$$

with the total moment of inertia of a fiber given by:

$$I = I_{co} + I_{cu} \quad (5)$$

where subscripts *co* and *cu* relate to cortex and cuticle, respectively. Treating the cuticle, based on the argument made above and for the current hair sample, as a hollow shaft with a constant wall thickness of 3 µm, I_{cu} is calculated by a suitably modified version of Equation 3 (17). Given the principal objectives of the investigation, no correction was applied at this stage to correct for potential changes of cuticle thickness through cosmetic processing.

Equation 4 was fitted to the data in Figure 1 using the Excel solver function and targeting the minimum of the residual sum of the squared errors for G'. This yields the residual variance s_R^2 as:

$$s_R^2 = \frac{\sum_i (G'_{i,exp} - G'_{i,th})^2}{N-1} \quad (6)$$

$G'_{i,exp}$ are the individual experimental values for the storage modulus, while $G'_{i,th}$ are the related values on the fitted curve for the same moment of inertia.

The overall quality of the fits of the curves is given, in the usual way (4) by the coefficient of determination r^2, which gives the fraction of data variance explained by the curve fit, so that:

$$r^2 = 1 - \frac{s_R^2}{s_T^2} \quad (7)$$

where s_R^2 and s_T^2 are the residual and the total variance, respectively.

RESULTS AND DISCUSSION

BASIC RESULTS

Figure 1 shows the individual results for G' for virgin (V) and treated fibers (WB & WBS), plotted against the moment of inertia of the individual fibers (Equation 3). The lines through the data are theoretical fits on the basis of the core/shell model (Equation 4).

Despite substantial scatter, the data for G' show in all cases a pronounced overall drop with increasing moment of inertia and thus with overall fiber diameter. The data confirm that the torsional storage modulus of hair is not a material constant. This drop with moment of inertia is in agreement with observations by Persaud and Kamath (2) at 50% RH, but in contrast to earlier observation by Wolfram and Albrecht (18), who found such a change only for hair fibers in water but not at 65% RH.

The assumption, backed by our experimental observations, that the thickness of the cuticle is independent of fiber diameter, implies that the cross-sectional fraction of the cuticle decreases with increasing fiber diameter. In view of Figure 1 this, firstly, leads to the qualitative conclusion that G' for the cuticle has to be substantially different from that of the cortex, in agreement with earlier considerations (2,19,20) and in contrast to more recent observations (21,22). In fact, the results, secondly, imply that the modulus of the cuticle has to be larger than that of the cortex, in agreement with and supporting the observations by Parbhu *et al*. (23).

Table I gives the arithmetic means of the storage moduli of the three samples and the related total data variances (s_T^2). Figure 2 graphically summarizes the experimental results for G' in the form of a box-and-whisker plot. In this presentation G' shows a substantial and significant increase (LSD test, $p < 0.001$) with the perm and bleach treatment (WB). From a materials point of view, this leads to the conclusion that the harsh chemical treatment makes the hair fiber overall stiffer, at least at this low humidity. Effects at higher humidities may be quite different and even reverse (3). The decrease of G' with the subsequent shampoo treatment (WBS), which may be attributed to a positive action of the product in terms of "softening," is in this context not significant on the 95% confidence level ($p = 0.29$). The latter observation corroborates the observation that data sets for samples WB and WBS in Figure 1 would strongly overlap, if plotted together. This seems to indicate that the discriminative power of the torsion method may thus be satisfactory for harsh hair treatments, but may not be adequate for more subtle ones, in apparent agreement with previous observations on a comparable set of samples (3). The values for G' as such, as given in Table I, are in good general agreement with previous observations by Bogaty (24) and Harper and Kamath (3) for virgin as well as cosmetically processed hair.

ESTIMATING THE TORSIONAL STORAGE MODULI OF CORTEX AND CUTICLE

The errors signified by the boxes and whiskers in Figure 2 are implicitly assumed to be random. Figure 1 shows for all samples, however, that the errors, e.g., given by the total variances s_T^2 (see Table I), contain in fact a significant systematic component, namely the drop of G' with the moment of inertia. Fitting Equation 4 to the data sets in Figure 1, this drop of G' is used to separate the torsional storage moduli of cortex (G'_{co}) and cuticle (G'_{cu}). These two moduli values are given in Table I, together with the coefficient of determination (r^2) of each fit. The fits account in each case for a major fraction of the overall data variance.

Figure 3 gives a graphical presentation of the data in Table I. It emphasizes that G'_{cu} is much larger than G'_{co}, supporting the view that the torsional properties of a hair fiber, similar as in bending (25), are controlled to a significant extent by cuticle properties (2). The large difference between the moduli values for the two morphological components is in agreement with observations by Parbhu *et al*. (23) and with *a priori* expectations, regarding the high degree of cross-linking, namely, in the exo-region of the cuticle (13).

The storage modulus of the cortex G'_{co} drops from 0.61 GPa for virgin hair by about 1/3 to 0.40 GPa with permanent waving and bleaching. The relative drop as such is largely

Table I.

Arithmetic means of the storage modulus G' for samples V, WB, WBS (see text) at 22°C and 22% RH. N is the number of fibers tested in each case. s_T^2 is the total variance of the data and s_R^2 the residual variance after subtracting the amount accounted for by the fit of the data using Equation 4. G'_{co} and G'_{cu} are the storage moduli for cortex and cuticle determined on the basis of those fits. The goodness of fit is characterized by the coefficient of determination r^2, giving the fraction of data variance explained by the fitted regression lines (Equation 7). Fits are conducted on the assumption of a diameter-invariant thickness of the cuticle of 3 μm, thus assuming six layers of cuticle cells (see text). All values are based on the cross-sectional dimensions of the fibers at 22°C and 55% RH.

Sample (N)	G', GPa	$s_T^2 \times 10^2$	$s_R^2 \times 10^2$	G'_{co}, GPa	G'_{cu}, GPa	r^2
V (69)	1.58	3.81	2.07	0.61	3.60	0.457
WB (56)	1.81	4.47	1.39	0.40	4.84	0.689
WBS (23)	1.76	3.65	0.88	0.37	4.63	0.758

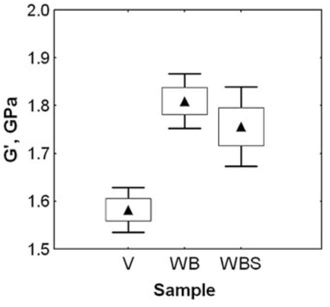

Figure 2. Graphical summary of the G′-data for the three samples, giving the mean (▲), the standard error (S.E., as box), and the limits of the 95% confidence range (±1.96 × S.E., as whisker).

consistent with the changes observed for filaments and matrix in the cortex by differential scanning calorimetry (26) of hair in water after reduction (27) and oxidation (28). Since the shampoo treatment will affect the surface only, the cortex properties stay largely unchanged, accordingly. Since G'_{co} is determined by extrapolation to I→∞, the absolute values in Table I have to be considered with some care and are subject to further investigations.

The cuticle modulus increases with the reduction/oxidation treatment (see Table I). This can be expected to lead to increased stiffness of the hair surface structures, in agreement with frictional measurements (29) and also general observations by consumers of "dry" hair after such harsh cosmetic processing. The slight drop of cuticle modulus with the shampoo treatment may be associated with a trend towards cuticle "softening", possibly through the surface deposition of a relatively soft layer of polymer.

However, apart from these general considerations, no attempt is made at this stage to assess the quantitative quality of the moduli of cuticle and cortex, thus determined. This would require to take into account the complex composite structure of cuticle and cortex as well as the relationship between moduli from different types of testing geometries and is thus well beyond the scope of these investigations.

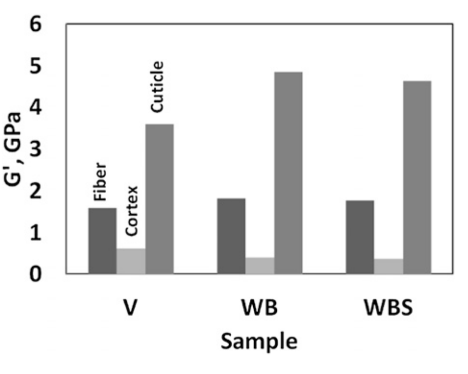

Figure 3. Torsional storage moduli for the whole fiber (G′) and separately for cortex (G'_{co}) and cuticle (G'_{cu}) for the various samples (see Table I).

Apart from gaining insights into the mechanics of the hair fiber and the role of cortex and cuticle, the primary emphasis of the investigation is to account, in a traceable manner, for the systematic fraction of the overall variance in order to improve the discriminative power of the torsional method.

Table I shows that the residual variance s_R^2 for G' after the fit is substantially smaller than the total variance s_T^2. On this basis, a t-test was conducted on the G′-values for samples WB and WBS, in order to assess the significance of the difference between the two samples. The results (t = 1.060) show that this specific difference is significant above the 80% level for a one-sided test (p = 0.15) and well below that level for a two-sided test (p = 0.29), when using the raw data. The significance levels increase to close to 95%, (t = 1.953, p = 0.054) when using the residual variance only and for a two-sided test. The significance level exceeds 95%, when a one-sided test is applied (p = 0.027). The ability to separate the random and the systematic component of G′-variance thus leads to a very significant increase of the discriminative power of the torsional test.

CONCLUSIONS

The torsional storage modulus of hair exhibits, irrespective of pretreatment, a distinct decrease with the moment of inertia. This shows that the modulus is not a material constant and that its measurement carries a significant systematic component, which impacts on data variance. The fit of a morphology-based two-component model, featuring cortex and cuticle, enables to separate the systematic component of data variance. The ability to thus determine the random (residual) component of data variance enables to significantly increase the sensitivity of the torsional method, so that even effects on hair, which would be expected to be very subtle, may be detected. As additional information, specific moduli data are obtained for cortex and cuticle. Their general changes with cosmetic treatments are in line with expectations. Further quantification of the moduli of the morphological components is currently subject to further and detailed analysis.

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