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Quasi-Static Torsional Deformation of Single Hair Fibers: Application of a Modeling Approach and Results from Cosmetic Treatments

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Synopsis

To date, most single hair fiber mechanical testing publications in the literature have focused on tensile deformation with torsional measurements receiving far less attention. However, there is much to be gained from the measurement of torsional properties of a single hair fiber such as providing an insight into the shear stiffness changes that are associated with cuticle damage. This study outlines the potential use of torsional measurements to differentiate between cosmetic treatments where other modes of deformation do not. A core/shell modeling approach has also been applied to separate out the potential contributions of the cuticles and the cortex on the fiber torsional modulus and the effect of relative humidity on hair fiber structural components.

INTRODUCTION

Human hair is a highly complex material, biologically engineered to favor load bearing in a longitudinal direction while still preserving fiber bending and twisting flexibility. This is a result of the hair being an anisotropic material, exhibiting a change in the mechanical properties with direction. To gain a fuller understanding of hair fibers' mechanical properties, it is paramount not only to consider tensile deformations, but also to measure bending and torsional deformations. A more thorough understanding of the mechanical behavior of hair fibers across the multiple modes of deformation could aid the development of hair care products, which may result in benefits to the consumer, such as improved manageability, ease of styling, and less breakage.

The multiple substructural components of human hair influence the mechanical behavior of the fiber to differing extents dependent on the mode of deformation. The tensile stiffness of hair fibers arises from the crystalline intermediate filaments (1,2) which are embedded within a sulfur-rich, cross-linked amphorous matrix. These filaments are aligned in a parallel orientation to the longitudinal axis of the fiber and along with the inter- and intramolecular interactions of the keratin-associated proteins within the matrix provide

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resistance to tensile deformation. When hair fibers are hydrated, the hydrogen bonds within the matrix are disrupted causing radial swelling of the fiber and a weakening of the matrix. The intermediate filaments are much less affected by the presence of water and therefore dominate the tensile stiffness (3,4). Under small levels of strain, typically less than 2%, the hair behaves as an elastic or "pseudo-elastic" material (5). Therefore, hair cuticles account for a low fraction area/volume of the whole fiber and that the tensile deformation occurs solely in the longitudinal direction, the Young's modulus can be assumed to be almost entirely associated with the cortex (6). Bending is a complex mode of deformation, and a number of theories describing the role the various substructures play have been proposed including the cuticles (7,8) and the cortex (9). The bending stiffness of a fiber has a fourth power relationship with the fiber diameter, and consequently, material further away from the fiber center has a greater impact on the bending stiffness than material at the core of the fiber.

The torsional deformation of hair relates to the twisting of the fiber around the longitudinal axis (Figure 1). When a fiber is twisted, the shearing stress is not equally distributed throughout the fiber (10) but increases from the central point to a maximum shear stress

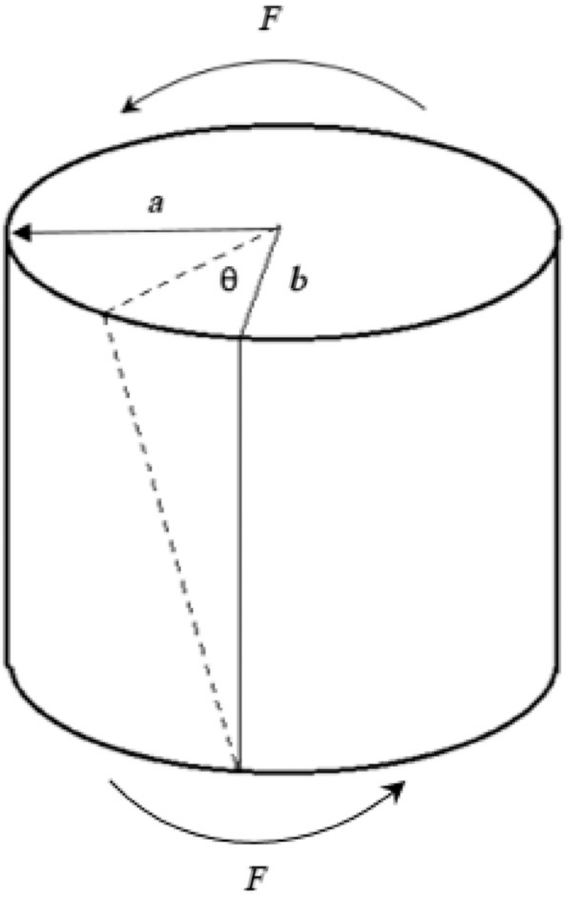

Figure 1. Shear deformation in an elliptical cylinder, where F is the force applied, θ is the deformation angle, and a, b are the major and minor radii, respectively.

at the outer surface because of a fourth power relationship between torsional stiffness and fiber diameter. For a circular, symmetrical cylinder, there is no change in cross-sectional shape as a torque is applied, which results in the shear stress being uniformly distributed along the fiber's outer surface (11). In reality, hair fibers do not exhibit a perfect circular cross section but a more elliptical cross-sectional shape, which complicates matters. This elliptical nature results in a warping effect of the fiber cross-sectional plane as it is twisted with the maximum shear stress acting on the outer edge of the fiber, where the maximum arc length displacement of twist angle exists (10,12). This produces a maximum peripheral shear strain at the minor axis and points along the fiber's outer surface (10,12). Since the shear stress is at a maximum at the periphery of the fiber, the torsional modulus may be significantly influenced by the outer layers of the fiber, particularly by the cuticles (6).

This has been experimentally demonstrated by Masaaki et al. (13), where the torsional modulus was measured on hair fibers with intact cuticles and where the cuticle layers had been removed. With the intact cuticle fraction, the fibers were found to have a torsional modulus of 0.795 GPa, whereas when the cuticles were removed, the modulus decreased to 0.430 GPa, concluding that the cuticle makes a significant contribution to the torsional modulus. A similar deduction was made by Harper and Kamath (14), who observed a substantial reduction in the torsional modulus when the surface of the hair was abraded as a means of removing the cuticles.

Early work investigating the torsional deformation of keratin originated within the wool industry and focused on using variations of the pendulum method (15–19). These approaches have since been adopted in the cosmetic industry to measure torsional properties of hair fibers (12,14,20–23). The pendulum method involves suspending a weighted bob from one end of a fiber, twisting the fiber, usually through 360°, and then setting it off into free rotational oscillations. The frequency and the magnitude of the oscillations are measured, from which both the storage and the loss torsional moduli can be calculated.

Despite the simple design of the pendulum method, there are a number of shortcomings. The method lacks automation and the pendulum bob needs to be manually attached to each fiber specimen, resulting in a time-consuming and labor intensive operation, which limits the sample sizes that can be run. In addition, the pendulum method is an indirect measurement of the torsional storage modulus. Bell et al. (6) conducted studies to compare the torsional storage modulus of nylon and hair fibers using both a pendulum method and the Dia-Stron FTT950 automated system (Dia-Stron Ltd., Andover, UK), which directly measures the torsional modulus. The results indicated that whereas both the indirect pendulum and direct automated methods provided both a consistent and reproducible measurement of the torsional modulus, the direct measurement method appeared to offer improved repeatability and higher testing throughput.

The Dia-Stron FTT950, Fibre Torsion Tester (Dia-Stron Ltd.) is an automated system which enables the direct measurement of the torsional properties and subsequent stress relaxation of fibers. The system is used alongside the Dia-Stron FDAS770 (Dia-Stron Ltd.) to measure fiber dimensional properties and in conjunction with the automated loading system, allowing high-throughput measurements of the torsional properties of fibers.

Fibers are mounted between two plastic tabs, giving a gauge length of 30 mm. Either before or after dimensional measurements, a central plastic torsion paddle is attached. This paddle has a length of 14 mm, which generates two equal test sections of the fiber with lengths of 8 mm. The two ends of the fiber are rotated at a user-defined angle, synchronously at a constant angular rate so that the central paddle moves against a fixed microbalance, which is incorporated into the measurement module (Figure 2). Based on that instrument principle, the resulting torque τ can be calculated by equation (1):

$$\tau = FL, \tag{1}$$

where F is the force (N) applied onto the stainless steel pin of the microbalance and L is the arm length of the pivot paddle from the fiber to the pin (m).

The Dia-Stron UvWin software (Dia-Stron Ltd.) determines the torsional modulus G from the cross-sectional measurements of the fiber. The UvWin software analysis function calculates the torsional rigidity constant D using equation (2):

$$D = \pi \frac{a^3 b^3}{a^2 + b^2}, \tag{2}$$

where a and b are the radii of the major and minor axes, respectively. The torsional modulus G can be calculated as follows by using equation (3):

$$G = \frac{FLl}{\theta D}, \tag{3}$$

where G is the shear modulus (Pa), F is the force measured by microbalance (N), L is the distance of fiber pivot from microbalance (m), D is the torsional rigidity constant (m^4), θ is the angular rotation (radians), and l is the fiber length (m).

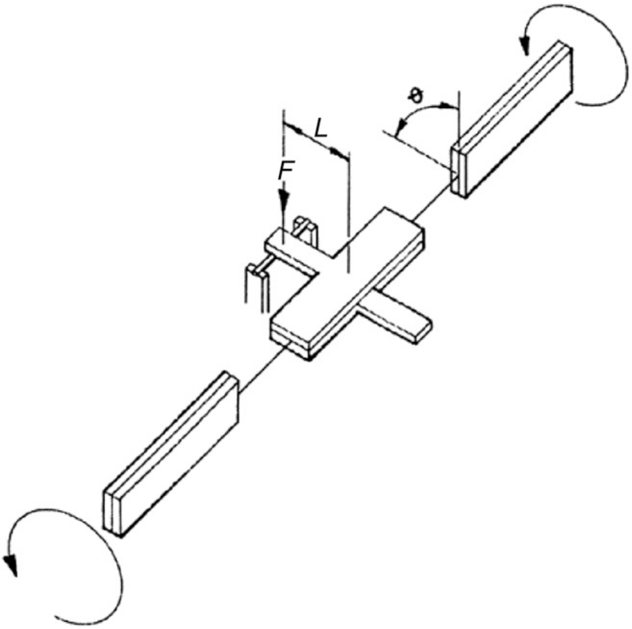

Figure 2. Principle of operation of direct torsion method, where F is the applied force at a moment arm length L and θ is the angle of twist.

MATERIALS AND METHODS

PREPARATION OF THE HAIR

Dark brown European hair was obtained from International Hair Importers and Products (Glendale, NY). Before any treatments, the swatches were washed with a 14% solution of sodium laureth sulfate (SLES), rinsed, and allowed to dry under controlled environmental conditions. For the studies requiring bleached hair, the swatches were bleached using a basic commercial bleach powder and 12% hydrogen peroxide for 60 min at 30°C before rinsing with warm water and dried with a hair drier set to 55°C. The swatches were allowed to equilibrate at 20°C and 50% relative humidity (RH) for 2 h in between bleaching cycles. The swatches were either bleached for two or three cycles dependent on the study.

Individual fibers were selected at random from the swatch and mounted between two 2-part plastic tabs to give a gauge length of 30 mm. A plastic torsion tab of length 14 mm was mounted and attached to the central section of the fiber leaving two 8-mm test length sections.

For the studies carried out on double bleached hair treated with a market leading commercial shampoo and conditioner system, the swatches were first washed with 0.2 g of shampoo per gram of hair and rinsed for 30 s. This was followed by applying 0.1 g of the conditioner per gram of hair for 60 s and rinsed for 30 s. For the studies carried out on double bleached hair treated with Abyssinian oil (Crambe Abyssinica Seed Oil, Elementis), the premounted fibers (without the central torsion tab) were immersed in the oil for 5 min taking care not to immerse the end tabs in the oil. The fibers were then rinsed for 60 s in deionized water and then washed with a 5% SLES solution to remove any excess oil. For both treatments, the specimens were allowed to dry overnight under controlled environmental conditions.

FIBER DIMENSIONAL ANALYSIS

After sample preparation at equilibration at 50% RH and 20°C, the fiber dimensions were measured with an automated Dia-Stron FDAS770. Six data slices, one rotation per slice, were collected for each specimen with three slices being taken on either side of the centrally mounted torsion tab. The maximum and minimum fiber diameters per slice are obtained, with the averages across each slice being used in the calculations.

TORSIONAL EXPERIMENTS

The torsional properties of each fiber were measured using an automated Dia-Stron FTT950. The fibers were subjected to a pretension force of 10 g at a linear extension rate of 10 mm/min. From this, it is possible to obtain the tensile elastic modulus, although this was not recorded for all of these studies. A torsional gauge force of 2 mg was applied and the fiber specimens were rotated through an angle of 90° at a rate of 5°/s. All fibers were equilibrated at the required environmental conditions for at least 2 h before the measurements were taken.

RESULTS AND DISCUSSION

TORSIONAL MODULUS RELATIONSHIP WITH FIBER GEOMETRY

Torsional modulus measurements on European virgin hair fibers with cross-sectional areas ranging between 2,000 and 6,500 µm^2 were recorded (Figure 3). This large data set, enabled by the use of the automated system, provided a diverse range of dimensions. Our data confirms the findings in previous publications (12,23) that the torsional modulus decreases with increasing fiber dimensions or polar moment of inertia. The polar moment of inertia of an ellipse is described by equation 4, where *a* and *b* are the major and minor axis radii, respectively.

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

This effect was further exemplified by additional measurements that were conducted on finer hair fibers obtained from a European juvenile. These results clearly demonstrate that the storage modulus of hair is not a material property but is highly dependent on fiber dimensions, particularly for finer fibers (12).

As mentioned earlier, the torsional stress is the greatest at the periphery of the fiber, where the cuticle layers are found. The cuticle thickness does not substantially change as the whole fiber diameter increases but the cuticle to cortex ratio does change. As the whole fiber diameter increases, the cuticle to cortex ratio decreases. This observed relationship between torsional storage modulus and diameter, as shown by our data and others, is suggestive that the outer layers of the cuticle are dominating the measurement (12).

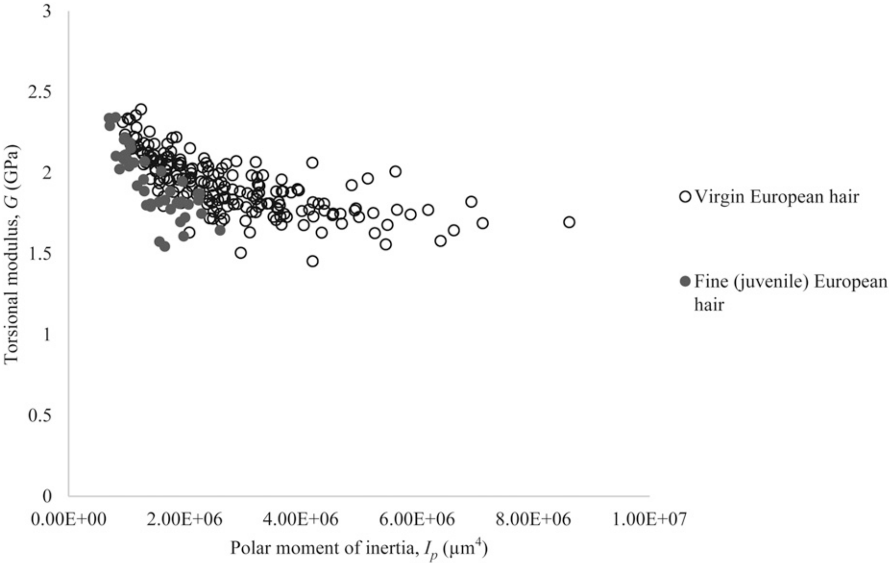

Figure 3. Plot of torsional modulus *G* as a function of the polar moment of inertia, I_p, for both virgin normal and virgin fine European hair.

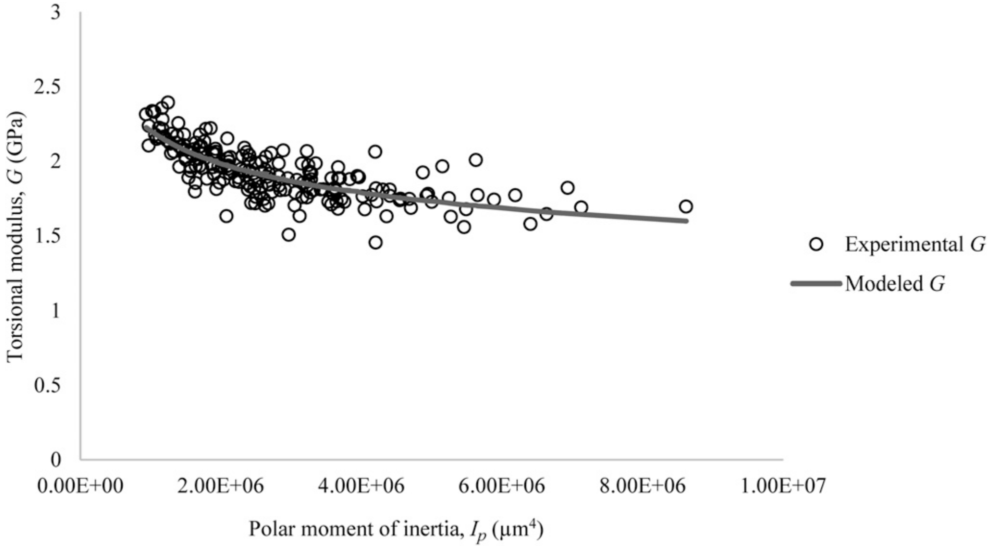

Figure 4. Plot of the torsional modulus G as a function of the polar moment of inertia, I_p, of 192 virgin European hair fibers. Line of best fit was obtained using the equation for the torsional modulus G' as described previously.

Given this apparent dominate contribution of the cuticles to the torsional modulus, it may be useful to use a model to separate out the respective contributions of the cuticles from cortex. To this end, we have used an approach based on the work conducted by Wortmann et al. (23). Applying this core/shell model, the following equation for the torsional storage modulus G' is defined (equation 5), where the polar moment of inertia, I, is the addition of the polar moments of inertia of the cortex and the cuticle, I_{co} and I_{cu}, respectively:

$$G' = \frac{\left(G'_{co}I_{co} + G'_{cu}I_{cu}\right)}{I} \tag{5}$$

Individual cuticle cells are generally accepted to have an approximate thickness of 0.5 μm (24) and the total number of cuticle cells in unaltered European hair is 6–10. Given that the hair used is not directly taken from the scalp, we have assumed that the hair used has six cuticle cell layers and therefore a thickness of 3 μm. Using this information, the theoretical polar moment of inertia of the cortex and the cuticle can be deduced by equation (6).

Table I

Means for the Experimental Torsional Storage Modulus of Virgin European Hair at 50% RH Based on the Analysis of 192 Fibers where s_R^2 is the Residual Variance between the Experimental Data and the Fit Data Based on equation (5) and s_T^2 is the Total Variance with a Value of 3.089×10^2

Cuticle layers	Thickness (μm)	G (GPa)	$s_R^2 \times 10^2$	$G_{cuticle}$, (GPa)	G_{cortex} (GPa)	r^2
4	2	1.923	1.201	6.747	0.655	0.611
6	3	1.923	1.205	5.043	0.592	0.610
8	4	1.923	1.209	4.191	0.523	0.609
10	5	1.923	1.213	3.680	3.680	0.607

The coefficient of determination, r^2, provides the goodness of fit based on an assumed number of cuticle layers. Refer to (23) for more details on calculations.

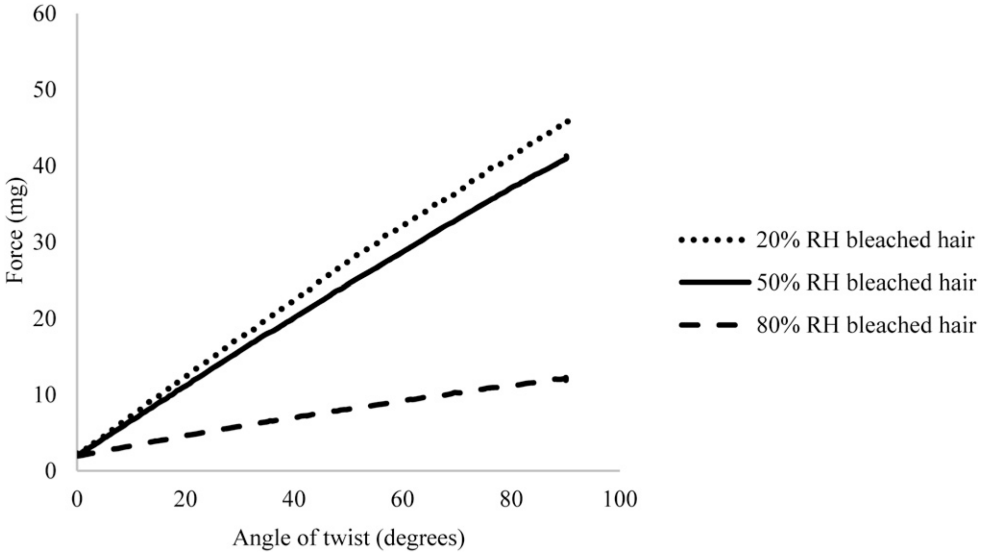

Figure 5. Typical plot of force measured by the microbalance (mg) as a function of twist angle (degrees) for bleached European hair at various humidity levels.

$$I = \frac{\pi}{4}\left(\left(a_o^3 b - a_i^3 b\right) + \left(a_o b_o^3 - a_i b_i^3\right)\right), \tag{6}$$

where the subscripts *o* and *i* denote the outer and inner axis radii, respectively, of the core/shell. Assuming that the cuticle thickness is constant along the length of the fiber, theoretical values of the torsional storage modulus for the cuticle and the cortex at 50% RH have been calculated (Figure 4, Table I). Estimations from our measurements on this particular set of virgin fibers suggest that the cuticle is 8–10 times more rigid than the cortex. Studies to measure the torsional modulus with the cuticles intact and after removal of the cuticles on virgin hair from the same source should be conducted to verify these theoretical predictions.

EFFECT OF MOISTURE AND OXIDATIVE DAMAGE ON TORSION

It is well documented that water has the ability to significantly alter the mechanical properties of keratin fibers (25–28). In hydrated hair, the intermediate filaments retain the structural integrity of the fiber which is relatively unaffected by the presence of water, whereas within the matrix proteins, hydrogen bonds are broken leading to radial swelling between the protofilaments as opposed to along the fiber axis (12). This results in a lowering of the elastic modulus in the presence of water. The extent to which the modulus is changed is somewhat dependent on the extent to which the role the matrix plays in the deformation. Consequently, moisture has a much larger influence on the torsional modulus of fibers than on the tensile or bending moduli (29).

As the humidity level increases, the matrix and the endocuticle layers are increasingly plasticized rending the fiber more deformable. This is illustrated by a reduction in the

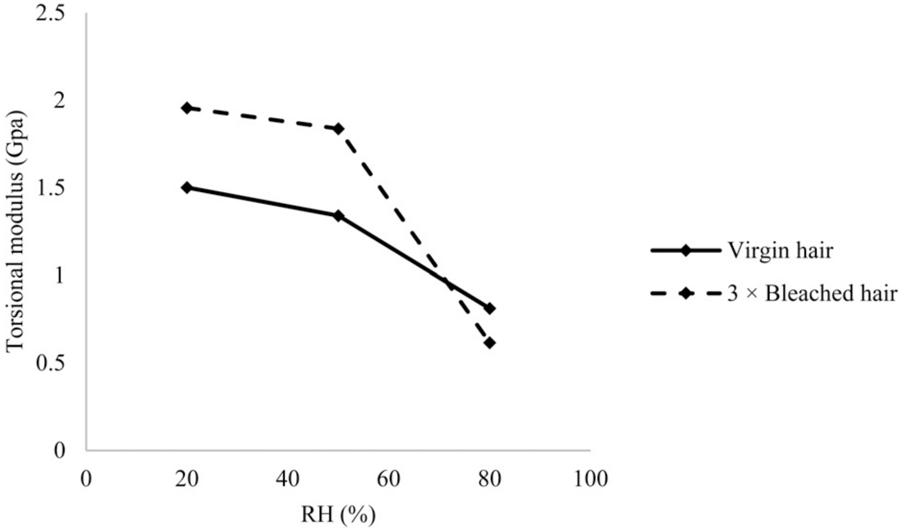

Figure 6. Plot of the torsional modulus G as a function of RH of virgin and 3 × bleached hair.

force required to twist the hair fibers with increased RH. The initially linear relationship between measured force and twist angle begins to plateau as the twist angle increases for both virgin and bleached (Figure 5) hair fibers. It is noteworthy that an increased moisture level results in the plateauing effect occurring at a smaller twist angle potentially indicative that the fibers are being twisted beyond the elastic region into the yield region. This apparent transition occurs between 40° and 50° which is considerably lower than the angle of twist which is often used in the pendulum methods. For this reason, an analysis angle of 45° was applied to ensure that only the elastic torsional deformation is analyzed.

When hair fibers undergo bleach treatment, the disulfide bonds of the cystine residues are oxidized diminishing the stability of the structure (30) compared with virgin fibers but

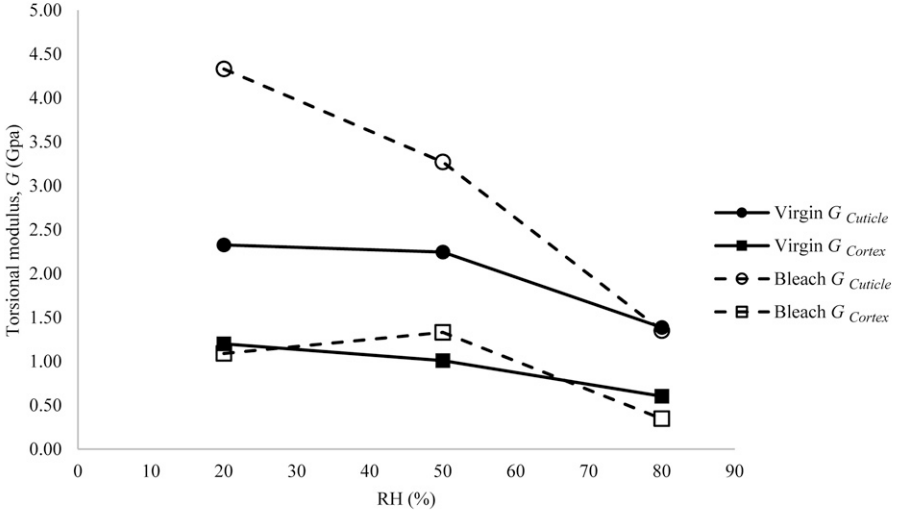

Figure 7. Plot of the torsional modulus as a function of RH for the calculated torsional modulus values for the cuticle and cortex components.

Table II
Means for the Experimental Torsional Storage Modulus of Virgin European and 3 × Bleached Hair at 20, 50, and 80% RH

	Experimental	Predicted	
RH (%)	G (GPa)	$G_{Cuticle}$	G_{Cortex}
Virgin			
20	1.50	2.32	1.20
50	1.34	2.24	1.01
80	0.81	1.39	0.60
3 × Bleached			
20	1.96	4.33	1.09
50	1.84	3.27	1.33
80	0.62	1.35	0.35

The theoretical values of $G_{Cuticle}$ and G_{Cortex} have been calculated using the approach described in (23) based on a sample size of 45.

as a result also increases the potential for both inter and intramolecular hydrogen bonding. In high humidity environments, disruption of these hydrogen bonds in addition to the cleaved disulfide bridges results in a more deformable fiber and thus a lower elastic modulus than undamaged, virgin fibers. As the humidity decreases, the increase in hydrogen bonding stiffens bleached fibers to a greater extent than virgin fibers.

Studies by Harper and Kamath (14) showed that the torsional modulus of bleached hair at low humidity is higher than that of virgin hair. At high humidity this effect is reversed, with the crossover point occurring at around 55–60% RH. Our data show a similar relationship with a marked difference between virgin and 3 × bleached hair at the lowest humidity (Figure 6), most likely because of the increase in hydrogen bonding due to the harsh bleaching.

Separation of the cuticle and cortex contributions to the torsional modulus using the modeling approach described previously (Figure 7, Table II), hypothetically illustrates the increased stiffness of the cuticle at low humidity, particularly for the bleached hair. This is likely because of an increased amount of hydrogen bonding within the endocuticle and the matrix or an increase in electrostatic interactions. Although this modeling approach may account for the potential significant contribution of the cuticles to the overall torsional modulus, it does not make any allowance for the role in which the matrix plays in torsional

Table III
Means Values for the Tensile and Torsional Elastic Moduli for Virgin, 2 × Bleached and Treated Hair Fibers

Treatment	Tensile modulus, E (GPa)	Torsional modulus, G (GPa)
Virgin	4.43 (b)	2.58 (a)
2 × Bleached	4.86 (b)	3.07 (b)
2 × Bleached, shampoo + cond.	5.35 (a)	2.83 (c)
2 × Bleached + oil	4.79 (b)	2.18 (d)

Measurements were all conducted at 20% RH. A one-way analysis of variance was used to identify any significant differences between the tensile modulus groups. *Post hoc* analysis was conducted using the Tukey Honesty Significant Difference (HSD) test. Groups that do not share a letter are significantly different at the 95% confidence level. The same analysis was conducted for the torsional modulus test groups.

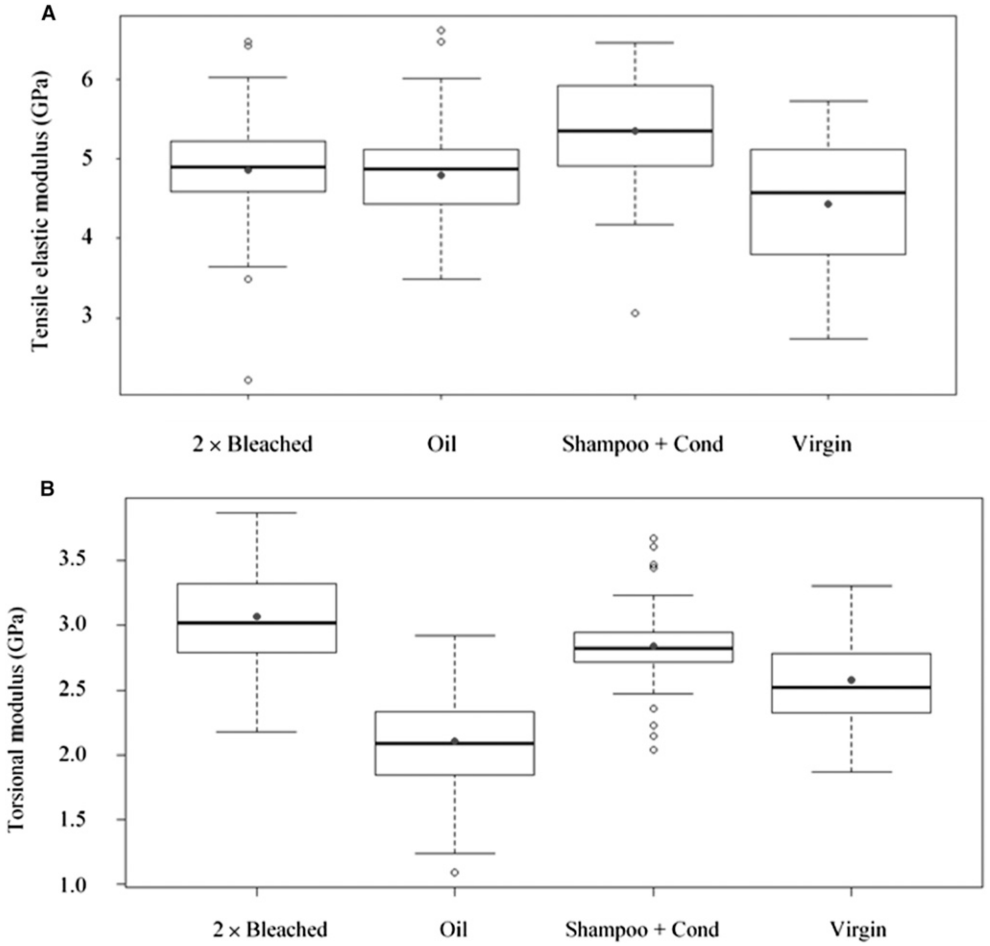

Figure 8. (A) Boxplot of the mean tensile elastic moduli for virgin, 2 × bleached and 2 × bleached hair treated with Abyssinian oil and treated with a commercial shampoo and conditioner. (B) Boxplot of the mean torsional moduli for virgin, 2 × bleached and 2 × bleached hair treated with Abyssinian oil and treated with a commercial shampoo and conditioner.

deformation nor the swelling of these substructures. Further exploration and optimization of this modeling approach would prove insightful.

PRACTICAL APPLICATIONS OF TORSION

A number of studies to illustrate the use of torsion in cosmetic applications have been conducted using the Dia-Stron FTT950 system. The exaggerated difference of the torsional modulus of bleached and virgin hair at low humidity prompted the decision to conduct the studies at 20% RH. Double-bleached fibers were treated with either an oil or a market leading shampoo and conditioner system. Abyssinian oil, a naturally derived oil rich in unsaturated fatty acids and long chain triglycerides which claims to improve hair softness and manageability, was selected for the study. A 5-min treatment with the oil resulted in a significant reduction in the torsional modulus compared with both virgin and untreated, bleached hair (Table III, Figure 8B). The lower modulus is indicative of a softening effect on the cuticles either by a plasticizing effect of the oil or through the formation of an occlusive barrier which retains and prevents moisture loss at lower humidity. Similarly, treatment of bleached hair with the commercial shampoo and conditioner regime reduced the torsional stiffness compared with that in the untreated, bleached hair, although to a lesser extent than the oil (Table III, Figure 8B).

The elastic tensile modulus was also measured on the same fibers using the FTT950 which reveals the different discrimination potential of the two modes of deformation. The torsional modulus identifies a significant difference between all of the treatments, whereas for tensile deformation only fibers treated with the product regime exhibit a significant change.

CONCLUSIONS

The Dia-Stron FTT950 allows for an automated and direct measurement of fiber torsional properties. The application of a theoretical two-phase model to single fiber torsional data showed that under a few assumptions, the cuticle layers may play a significant role in the torsional mode of deformation, whereas their contributions in tensile deformation can be assumed to be negligible.

We have also shown the impact of relative humidity on the torsional modulus of both virgin and bleached fibers. The differentiation between these treatments is maximized at low humidity levels; therefore, the sensitivity of the method could be increased by running experiments under low humidity conditions.

Finally, we have demonstrated the positive effect of an oil-based treatment and a market leading hair care system (shampoo and conditioner) on hair torsion behavior, whereas less discrimination was observed in the tensile measurements. Although there is much still to explore with such a high-throughput torsion testing technique, we have illustrated the ability of this mechanical method to highlight clear and significant effects from cosmetic treatments, opening the way to developing new ingredients and hair care formulations targeting the cuticle layers.

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