



# Model-based analysis of the torsional loss modulus in human hair and of the effects of cosmetic processing

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# 1 **Model-based analysis of the torsional loss modulus in human hair**

## 2 **and of the effects of cosmetic processing**

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9

### 10 **Synopsis**

11 Torsional analysis of single human hairs is especially suited to determine the properties of the  
12 cuticle and its changes through cosmetic processing. The two primary parameters, which are  
13 obtained by free torsional oscillation using the torsional pendulum method, are storage ( $G'$ )  
14 and loss modulus ( $G''$ ), respectively. Based on previous work on  $G'$ , the current investigation  
15 focuses on  $G''$ . The results show an increase of  $G''$  with a drop of  $G'$  and *vice versa*, as is  
16 expected for a viscoelastic material well below its glass transition. The overall power of  $G''$  to  
17 discriminate between samples is quite low. This is attributed to the systematic decrease of the  
18 parameter values with increasing fiber diameter, with a pronounced correlation between  $G''$   
19 and  $G'$ . Analyzing this effect on the basis of a core/shell-model for the cortex/cuticle-structure  
20 of hair by non-linear regression leads to estimates for the loss moduli of cortex ( $G''_{co}$ ) and  
21 cuticle ( $G''_{cu}$ ). While the values for  $G''_{co}$  turn out to be physically not plausible, due to  
22 limitations of the applied model, those for  $G''_{cu}$  are considered as generally realistic against  
23 relevant literature values. Significant differences between the loss moduli of the cuticle for the  
24 different samples provide insight into changes of the torsional energy loss due to the cosmetic  
25 processes and products, contributing towards a consistent view of torsional energy storage and  
26 loss, namely, in the cuticle of hair.

27

28 **Keywords:** human hair, dynamic torsion, loss modulus, cortex, cuticle, chemical damage,  
29 repair

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## 31 INTRODUCTION

32 The behavior of human hair under torsional stresses and strains is an important contributing  
33 factor for the formation and maintenance of a hair style (1). Due to the nature of torsional  
34 deformation the results for a fiber are biased towards contributions from its outer regions (2).  
35 For human hair the method is thus especially suited to investigate the properties of the cuticle.  
36 In a recent publication (3) we presented a set of data from investigations on untreated and  
37 cosmetically treated human hair fibers using the torsional pendulum technique. For that  
38 investigation we concentrated on considerations of the storage modulus  $G'$ , which is derived  
39 from the frequency of the free torsional oscillation. A basic core-shell model of cortex and  
40 cuticle was applied to model the observed decrease of  $G'$  with fiber diameter or rather polar  
41 moment of inertia. This analysis enabled to obtain estimates for the torsional storage moduli of  
42 cuticle and cortex through non-linear curve fitting and extrapolation. The results of the analysis  
43 supported the hypothesis that the torsional storage modulus of the cuticle is significantly higher  
44 than that of the cortex. Though the absolute value for the modulus of the cortex was too low  
45 compared to literature values, plausible changes of cuticle and cortex moduli were determined  
46 after cosmetic treatments.

47

48 This part of the investigation now is focused on the logarithmic decrement  $\mathcal{A}$ , as a measure of  
49 energy loss in the fiber and as one of the primary variables from a torsional pendulum  
50 experiment. The loss modulus  $G''$ , as primary physical variable, is determined indirectly from  
51 the logarithmic decrement  $\mathcal{A}$  and the torsional storage modulus  $G'$  for an individual  
52 measurement.  $G'$  is proportional to the energy stored and  $G''$  to the energy lost during a  
53 torsional oscillation. The objective is to investigate whether the structure-based, basic  
54 core/shell model approach for  $G'$  (3) is also applicable for  $G''$ . This includes estimates of the

55 loss moduli of cuticle and cortex as well as of effects of cosmetic treatments. The potential as  
56 well as the specific limitations of the approach are discussed.

57

58

## 59 MATERIALS AND METHODS

60

### 61 THEORETICAL BACKGROUND

62

63 Free torsional oscillation, e.g., of a fiber in a torsional pendulum apparatus (2, 4, 5), yields the  
64 complex torsional modulus  $G^*$  as:

65

$$66 \quad G^* = G' + iG'' \quad (1)$$

67

68 where  $G'$  and  $G''$  are the storage and loss modulus, respectively.

69

70  $G'$  is given by:

71

$$72 \quad G' = 4\pi^2 \frac{Jl}{IT^2} \quad (2)$$

73

74 where  $J$  is the moment of inertia of the pendulum,  $l$  the length of the fiber,  $I$  the polar moment  
75 of inertia of the fiber, and  $T$  the time taken for one oscillation.

76

77 The cross-section of a hair fiber is generally assumed to be best described as elliptical so that  
78 the polar moment of inertia is given by:

79

80

$$I = (\pi/4) (a^3b + b^3a) \quad (3)$$

82

83 where  $a$  and  $b$  are the semi-axes of the ellipse.

84

85 The use of the polar rather than the torsional moment of inertia (6) assumes the limiting case  
 86 that no warping of the test specimen occurs (7), which is plausible for small deformations and  
 87 low resonance frequencies (8), as realized in this study. The situation is certainly different for  
 88 combinations of high tensile and torsional strains (9). The approach was furthermore chosen to  
 89 provide better comparability of data with previous investigations (4, 10, 11) including those,  
 90 which are based on the assumption of circular hair cross-sections (1, 12, 13).

91

92 Arithmetic means for oscillation time  $T$  were determined from five successive oscillations.  $G'$   
 93 values were determined from the mean oscillation times for five-fold measurements for a given  
 94 fiber.

95

96 From the continuous decrease of the torsional amplitude due to damping, the logarithmic  
 97 decrement  $\Lambda$  is determined through:

98

$$\Lambda = \frac{1}{n} \sum_{i=1}^n \ln \frac{A_i}{A_{i+1}} \quad (4)$$

100

101  $A_i$  and  $A_{i+1}$  are the amplitudes of successive oscillations.  $n$  is the number of oscillation from  
 102 which the value for  $\Lambda$  is calculated. For the current investigation  $n = 5$  generally applies. Values  
 103 are based on five-fold determinations for a given fiber.

104

105 For low degrees of damping, the connection between logarithmic decrement  $A$  and the  
 106 torsional phase angle  $\delta$  as  $\tan \delta$  is given by:

107

$$108 \quad A = \pi \tan \delta \quad (5)$$

109

110 With the loss factor:

111

$$112 \quad \tan \delta = G''/G' \quad (6)$$

113

114 this yields:

115

$$116 \quad A = \pi G''/G' \quad (7)$$

117

118 so that

119

$$120 \quad G'' = A G' / \pi \quad (8)$$

121

122 Equation 8 enables to determine the value for  $G''$  from the related values of  $G'$  and  $A$  for a  
 123 given experiment.

124

125 In view of the fact that hair is not an uniformly isotropic, viscoelastic material, as may in  
 126 principle be required, a core/shell-model is suggested, which enables to estimate the separate  
 127 contributions of cortex and cuticle to  $G''$ , in analogy to  $G'$  (3) as:

128

$$129 \quad G'' = (G''_{co} I_{co} + G''_{cu} I_{cu}) / I \quad (9)$$

130

131 with

132

133 
$$I = I_{co} + I_{cu} \quad (10)$$

134

135 where subscripts *co* and *cu* relate to cortex and cuticle, respectively.

136

137 In accordance with the experimental evidence for the material used, the cuticle is treated for  
138 each fiber as a hollow, elliptical shaft with a constant wall thickness of 3  $\mu\text{m}$ . This relates to  
139 about six layers of cuticle in the cross-section, which are assumed to be constant along fiber  
140 length and independent of fiber diameter.

141

142 Equation 9 was fitted to the  $G''$ -data using the established non-linear regression method (3).  
143 This approach accounts for a certain fraction of the variance of the data and also yields  
144 estimates for the torsional loss moduli of cortex and cuticle together with their 95%-confidence  
145 limits. The justification of this model-based approach, the applicability of which is considered  
146 as independent of the actual scatter of the data, is, namely, based on the observation that the  
147 torsional storage modulus of hair fibers drops significantly after the removal of the cuticle (10).  
148 Further considerations are given elsewhere (3).

149

150

## 151 EXPERIMENTAL

152

153 All experiments on hair fibers were conducted on a Single Fiber Torsion Pendulum apparatus  
154 (TRI/Princeton, NJ, USA) as described by Persaud and Kamath (4). Effective hair fiber length  
155 was 3 cm, frequency about 0.1 Hz, and environmental conditions 22 °C and 22% relative  
156 humidity. All tests and treatments were conducted on dark brown, commercial, Caucasian hair  
157 (International Hair Importers & Products Inc., Glendale, NY, USA). For each fiber tested the  
158 smallest and largest diameters were determined at five equidistant points and through 360°  
159 (Laser Scan Micrometer, LSM-500, Mitutoyo, Kanagawa, Japan). Hair tresses were taken from  
160 a collective of virgin hair (V) and subjected to a permanent waving treatment (7% thioglycolic  
161 acid, pH 9.5, 30 min) followed by re-oxidation (2.2% H<sub>2</sub>O<sub>2</sub>, pH 4). This was followed by  
162 bleaching (8% H<sub>2</sub>O<sub>2</sub>, pH 9.4, 30 min). The perm-waved and bleached sample is referred to as  
163 *WB*. A group of fibers already prepared for torsional testing was furthermore treated with a  
164 commercial ‘repair’ shampoo (30 min & 30 s rinse). The sample is referred to as *WBS*. For  
165 further, specific details the reader is referred to Wortmann et al. (3). Data analysis and non-  
166 linear curve fits were conducted using Statistica (Version 13, Dell) and SPSS (Version 20,  
167 IBM). Homogeneity or in-homogeneity of data sets were determined by Analysis of Variance  
168 (ANOVA) and non-conservative, post-hoc LSD-tests, as implemented in the statistics  
169 programs.

170

171

172



173 **RESULTS AND DISCUSSION**

174

175 **BASIC OBSERVATIONS**

176

177 One of the primary experimental variables obtained from the free torsional oscillation test and  
178 in particular from the continuous decrease of the oscillation amplitude is the logarithmic  
179 decrement  $A$  (see Equation 4), as a measure of damping within the viscoelastic hair fiber.  
180 Figure 1 summarizes the results for  $A$  for the three samples.

181

182 Logarithmic decrement values at the chosen conditions (22% rh, 22 °C) are low compared to  
183 literature values for wool (14) and hair (12) at 65% rh. This is attributed to the humidity  
184 dependent glass transition of wool (15) and hair (16), where low humidity shifts the properties  
185 of a keratinous material further into the glassy region. The values show satisfactory agreement,  
186 however, with the values for wool at 25% rh and  $T < 30$  °C of  $A < 0.06$  (17). Also reasonable  
187 agreement is observed with the values for hair given by Persaud & Kamath (4) and Harper at  
188 al. (11) as well as from other sources on the basis of Equation 5 (18, 19, 20).

189

190  $A$ -values drop after the waving & bleaching treatment compared to virgin hair, signifying a  
191 decrease of internal energy loss. Values increase again after the shampoo treatment. In line  
192 with the qualitative impression from Figure 1 ANOVA as well as LSD-tests show that  
193 differences between all data sets are highly significant well beyond the 95%-level. The results  
194 for logarithmic decrement thus show a high discriminative power for the cosmetic treatments.

195

196  $G''$ -values are determined with Equation 8 from the individually obtained values for  $A$  (see  
197 Figure 1) and  $G'$  (see Figure 2B) and are summarized in Figure 2A.  $G''$ -values are roughly by  
198 a factor 50 – 100 smaller than  $G'$ , which is in agreement with observations by Dynamic  
199 Mechanical Analysis (DMA) (19) and attributed to the general properties of hair as a glassy  
200 polymer well below its glass transition (16) under the conditions of the measurements. In line  
201 with expectations for such a material  $G''$  increases when  $G'$  decreases and *vice versa* (21).

202

203 Moving from the experimental variable  $A$  to the primary, physical variable  $G''$  much of the  
204 discriminative power of the measurement of energy loss is lost. The insets in Figures 2A and  
205 B summarize the significance of the differences between the samples, as determined through  
206 the LSD-test. Compared to  $G'$  the number of significant differences is smaller for  $G''$ , leaving  
207 only  $V > WB$  as significant on the 95%-level.

208

209 This difference of performance and loss of discriminative power are attributed to compensation  
210 effects between values for the storage and the loss modulus, respectively. Plotting  $G''$  and  $A$   
211 against  $G'$ , as is done for the virgin sample in Figure 3, shows that the correlation between  $A$   
212 and  $G'$  is only faint though significant ( $r^2 = 0.08$ ), while it is quite pronounced for  $G''$  ( $r^2 =$   
213  $0.69$ ). Similar observations were made for  $G''$  vs.  $G'$  for the WB- ( $r^2=0.74$ ) and the WBS-  
214 sample ( $r^2=0.77$ ), respectively.

215

216 Underlying the analysis for  $G''$  above is the assumption that the data are essentially normally  
217 distributed. This assumption seems to be apparently correct, when inspecting the cumulative  
218 probability plots of the data, which all provide adequate straight lines.

219

220

## 221 APPLICATION OF THE CORE/SHELL-MODEL

222

223 When plotting  $G''$  against the moment of inertia for all samples systematic decreases are  
224 observed (see Figure 4), similar as for  $G'$  (3, 4). These observations are generally in line with  
225 data by Leray & Winsey (22) from torsional stress relaxation for both modulus and relaxation  
226 gradient. As for  $G'$ , this highlights that  $G''$  for hair is not a material constant. The decrease as  
227 such is in line with the core/shell-model (Equation 9) and implies that the cuticle has a higher  
228  $G''$ -value than the cortex, as related to the limiting values for  $G''$  at low and high values of  $I$ ,  
229 respectively.

230

231 The observation that  $G'$ - and  $G''$ -values both decrease with increasing moment of inertia (3)  
232 implies that both storage and loss modulus are higher for the cuticle than for the cortex. For the  
233 current cases the correlated changes of  $G''$  and  $G'$ , as shown in Figure 3 lead to the  
234 compensation effects for  $A$ , as mentioned above.

235

236 Equation 9 was fitted to the data applying non-linear regression. The free optimization showed  
237 that the estimate for  $G''_{co}$  gave slightly negative values in all cases ( $G''_{co} \geq -0.005$  GPa), which  
238 is physically not reasonable. For this reason  $G \geq 0$  was introduced as a boundary condition for  
239 the fit. Table I summarizes the results of the fits for  $G''_{co}$  and  $G''_{cu}$  together with the associated  
240 95%-confidence ranges and the coefficients of determination  $r^2$ . The solid lines through the  
241 data in Figure 4 are based on Equation 9 and the parameter values in Table I.

242

243 Coefficients of determination for the fits of the core/shell-model through Equation 9 to the  $G''$ -  
244 data (see Table I) are substantial and comparable to those for  $G'$ . They may be used to reduce  
245 the unexplained variance of the data and thus to improve the discriminative power for  $G''$ ,

246 similarly as for  $G'$  (3). However, in the present case this would need to be implemented with  
247 added caution in view of the boundary condition for the loss modulus of the cortex ( $G''_{co} \geq 0$ ),  
248 which is expected to increase the risk of Type I errors, when identifying significant differences  
249 between samples.

250

251 The application of Equation 9 is justified by the observation that the torsional moduli are not  
252 material constants of hair, but rather change with fiber diameter or rather moment of inertia  
253 (see Figure 4). This is attributed to differences of properties of cortex and cuticle in the  
254 core/shell structure of hair. The observed limitation of the model, as reflected by the  $r^2$ -values  
255 for the fit of Equation 9, may be attributed to the fact that the torsional moduli of the cuticle  
256 are not true material constants. This may be related to the layered structure of the cuticle, which  
257 in practice is subject to damage (23, 24), namely, by thermal stresses as, e.g., reflected in  
258 delamination (25). Changes of structural integrity are expected to generate substantial and  
259 overriding contributions, namely, to frictional interactions within the cuticle layers, which will  
260 impact on  $G''$ . This may be considered as an explanation for the apparent lack of fit, namely,  
261 for the WB-sample at low values of  $I$  (see Figure 4), that is for comparatively high volume  
262 fractions of cuticle. Further complications are expected to arise from the limitations of the  
263 assumptions of constant cuticle thickness with fiber diameter as well as along fiber length, as  
264 well as the simplifications underlying Equations 3 and 9 (7).

265

266 For all three samples the boundary condition  $G''_{co} \geq 0$  needed to be applied for the fits, where  
267 the necessity for this condition may be attributed to some extent to the required extrapolation  
268 to  $I \rightarrow \infty$ . Given this restriction, the upper 95% confidence limit for the loss modulus of the  
269 cortex in virgin hair is  $G''_{co} = 0.005 \text{ GPa}$ . With the corresponding value of  $G'_{co} = 0.61 \text{ GPa}$   
270 (see Table I) this yields with Equation 6 a maximum value of  $\tan \delta_{co} = 0.008$ . This value may

271 be compared to  $\tan \delta \approx 0.022$  of rhinoceros horn perpendicular to the growth direction under  
272 not too dissimilar conditions (110 Hz, 5.2% regain) (18). For this testing geometry specifically  
273 the properties of the matrix are determined, analogous to torsion. The comparison of the data  
274 shows that even the calculated maximum value for  $\tan \delta_{co}$  is too low by a factor of about 3  
275 using the extrapolation of the data in Figure 4. The fits on the basis of the core/shell-model thus  
276 turn out to not be suitable to estimate the torsional loss modulus of the cortex.

277

278 In contrast  $\tan \delta$  – values for the cuticle with the applicable values for  $G''_{cu}$  and  $G'_{cu}$  (see Table  
279 I) yield a range of  $\tan \delta = 0.01 - 0.02$ , in acceptable agreement with expectation values for  
280 keratins for roughly comparable conditions (18, 19, 20, 26, 27, 28). This gives some support  
281 for the overall validity of the estimated  $G''_{cu}$ -values in the absence of reference values.

282

283 Due to the systematic decrease of  $G''$  with  $I$ , the estimates for  $G''_{cu}$  are substantially higher  
284 than the  $G''$ -means, though they follow the same pattern for all samples. The overall behavior  
285 for  $G''_{cu}$  is as to be expected for a material below the glass transition, in that  $G''_{cu}$  decreases  
286 with an increase of  $G'_{cu}$  for a sample and *vice versa*.

287

288 The  $G''$ -value is reduced by a factor of about two compared to the virgin hair through the  
289 chemical processing of reduction and oxidation (WB), in line with considerations of increased  
290 stiffness and brittleness of the cuticle (3). While the effect of the additional ‘repair’ treatment  
291 (WBS-sample) is small for  $G'_{cu}$ , the corresponding value of  $G''_{cu}$  increases well beyond the  
292 value for virgin hair. This not only indicates that the ‘repair’ agent improves the overall  
293 structural integrity of the cuticle but also introduces through its components, possibly, namely,  
294 through the polymer content a strong viscous component, which contributes to the increase of  
295  $G''_{cu}$ .

296

297

298 **CONCLUSIONS**

299

300 Using the values for the storage modulus  $G'$  and the logarithmic decrement  $A$  as parameters  
301 obtained from the free torsional oscillation experiment on hair the values for the loss moduli  
302  $G''$  were determined. The raw data show a rather low discriminative power between the  
303 different samples, despite their rather strong chemical pre-treatment. This can be attributed to  
304 a strong component of variance due to the systematic decrease of  $G''$  with fiber moment of  
305 inertia. This decrease is associated with a decrease of the area fraction of the cuticle in the fiber  
306 cross-section, when the fibers get thicker. The effect is accounted for by a core/shell-model for  
307 the cortex/cuticle-structure of hair, yielding satisfactory coefficients of determinations. These  
308 model fits may be used, with due caution with respect to Type I errors, to improve the  
309 discriminative power for  $G''$ -measurements, when investigating hair samples with different  
310 processing histories. The more speculative aspects of the investigation relate to the  
311 determination of the loss moduli for cortex and cuticle. While the determination of  $G''_{co}$  proved  
312 to be unsuccessful, values for  $G''_{cu}$  show overall consistency. The distinct and plausible  
313 differences between the loss moduli for the cuticle for the samples support previous suggestions  
314 (3) that torsional measurements in the appropriate model context are a very sensitive tool to  
315 assess changes of the properties of the hair cuticle through cosmetic processes and ingredients,  
316 in line with expectations by Robbins (13). In conclusion and in agreement with Bogaty's (1)  
317 considerations, it is suggested that imparting the appropriate balance of torsional storage and  
318 loss moduli in hair by cosmetic processes and products will make a major contribution to their  
319 ability to control the dynamic movement of a hair style in line with consumer expectations.

320

321

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326 Personal Care and Nutrition GmbH), Duesseldorf (GER).

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## REFERENCES

- (1) H. Bogaty, Torsional properties of hair in relation to permanent waving and setting. *J.Soc.Cosmet.Chem.*, **18**, 575-589 (1967).
- (2) BS EN ISO 6721-1 *Plastics – Determination of dynamic mechanical properties. Part 1: General principles*. BSI (British Standards Institution), London, UK (2011).
- (3) F.J. Wortmann, G. Wortmann, H.-M. Haake, and W. Einfeld, Analysis of the torsional storage modulus of human hair and its relation to hair morphology and cosmetic processing, *J.Cosmet.Sci.*, **65**, 59-68 (2014).
- (4) D. Persaud and Y.K. Kamath, Torsional method for evaluating hair damage and performance of hair care ingredients, *J.Cosmet.Sci.*, **55**, S65-S77 (2004).
- (5) BS EN ISO 6721-2 *Plastics – Determination of dynamic mechanical properties. Part 2: Torsion-pendulum method*. BSI (British Standards Institution), London, UK (2008).
- (6) F.I. Bell, P. Carpenter, and S. Bucknell, Advantages of a high-throughput measure of hair fiber torsional properties, *J.Cosmet.Sci.*, **63**, 81-92 (2012).
- (7) R.J. Roark, *Formulas for Stress and Strain* (McGraw-Hill Book Co., NY, USA, 1965)
- (8) B.E. Read and G.D. Dean, *The Determination of Dynamic Properties of Polymers and Composites* (Adam Hilger Ltd., Bristol, UK, 1978).
- (9) T.A. Dankovich, Y.K. Kamath, and S.B. Ruetsch, Tensile properties of twisted hair fibres, *J.Cosmet.Sci.*, **55**, S79-S90 (2004).
- (10) D.L. Harper and Y.K. Kamath, The effect of treatments on the shear modulus of human hair measured by the single fiber torsion pendulum, *J.Cosmet.Sci.*, **58**, 329-337 (2007).



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- (11) D.L. Harper, C.J. Qi, and P. Kaplan, Thermal styling: Efficacy, convenience, damage tradeoffs, *J.Cosmet.Sci.*, **62**, 139-147 (2011).
  - (12) L.J. Wolfram and L. Albrecht, Torsional behaviour of human hair, *J.Soc.Cosmet.Chem.*, **36**, 87-99 (1985).
  - (13) C.R. Robbins, *Chemical and Physical Behavior of Human Hair*, 5<sup>th</sup> Ed. (Springer Verlag, Heidelberg, GER, 2012).
  - (14) D.G. Phillips, Effects of humidity, ageing, annealing, and tensile loads on the torsional damping of wool fibres, *Text.Res.J.*, **57**, 415-420 (1987).
  - (15) F.J. Wortmann, B.J. Rigby, and D.G. Phillips, Glass transition temperature of wool as a function of regain, *Text.Res.J.*, **54**, 6-8 (1984).
  - (16) F.J. Wortmann, M. Stapels, R. Elliott, and L. Chandra, The effect of water on the glass transition of human hair, *Biopolymers*, **81**, 371-375 (2006).
  - (17) P. Nordon, A damping maximum in the free torsional oscillation of wool fibres, *J.Appl. Polym.Sci.*, **7**, 341-346 (1963).
  - (18) M. Druhala and M. Feughelman, Dynamic mechanical loss in keratin at low temperature. *Colloid & Polym.Sci.*, **252**, 381-391 (1974).
  - (19) M. Jeong, V. Patel, J.M. Tien, and T. Gao, DMA study of hair viscoelasticity and effects of cosmetic treatments, *J.Cosmet.Sci.*, **58**, 584-585 (2007).
  - (20) F.J. Wortmann, M. Stapels, and L. Chandra, Humidity-dependent bending recovery and relaxation of human hair, *J.Appl. Polym.Sci.*, **113**, 3336-3344 (2009).
  - (21) R.J. Young and P.A. Lovell, *Introduction to Polymers*, 3<sup>rd</sup> Ed. (CRC Press, Boca Raton, FL, USA, 2011)
  - (22) Y. Leray and N. Winsey, Torsional properties of single hair fibres in relation to ethnicity, damage and other modes of deformation, 6<sup>th</sup> *Int.Conf.Appl.Hair Sci.*, (Princeton, NJ, USA, 2014)
  - (23) J. Jachowicz, Hair damage and attempts to its repair, *J.Soc.Cosmet.Chem.*, **38**, 263-286 (1987).

- 
- (24) M. Gamez-Garcia, Cuticle decementation and cuticle buckling produced by Poisson contraction on the cuticular envelope of human hair, *J.Cosmet.Sci.*, **49**, 213-222(1998).
- (25) M. Gamez-Garcia, Cracking of human hair cuticles by cyclical thermal stresses, *J.Cosmet.Sci.*, **49**, 141-153 (1998).
- (26) J.I. Dunlop, Dynamic mechanical properties of rhinoscerous horn keraton in the frequency range 2-20 KHz, *Text.Res.J.*, **42**, 381-385(1972).
- (27) G. Danilatos and M. Feughelman, The internal dynamic mechanical loss in  $\alpha$ -keratin fibers during moisture sorption, *Text.Res.J.*, **46**, 845-846(1976).
- (28) G. Danilatos and R. Postle, The time-temperature dependance of the complex modulus of keratin fibres, *J.Appl.Polym.Sci.*, **28**, 1221-1234 (1983).

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335

336

337 **Table I:** Estimates for the loss moduli of cortex ( $G''_{co}$ ) and cuticle ( $G''_{cu}$ ), respectively,  
 338 together with their 95% confidence limits, as obtained by fits of Equation 9 to the data in Figure  
 339 4. The number of measurements for each sample is given in brackets.  $r^2$  are the coefficients of  
 340 determination for the fits. Furthermore, values for the storage moduli for cortex ( $G'_{co}$ ) and  
 341 cuticle ( $G'_{cu}$ ) are given (3) to aid the discussion.

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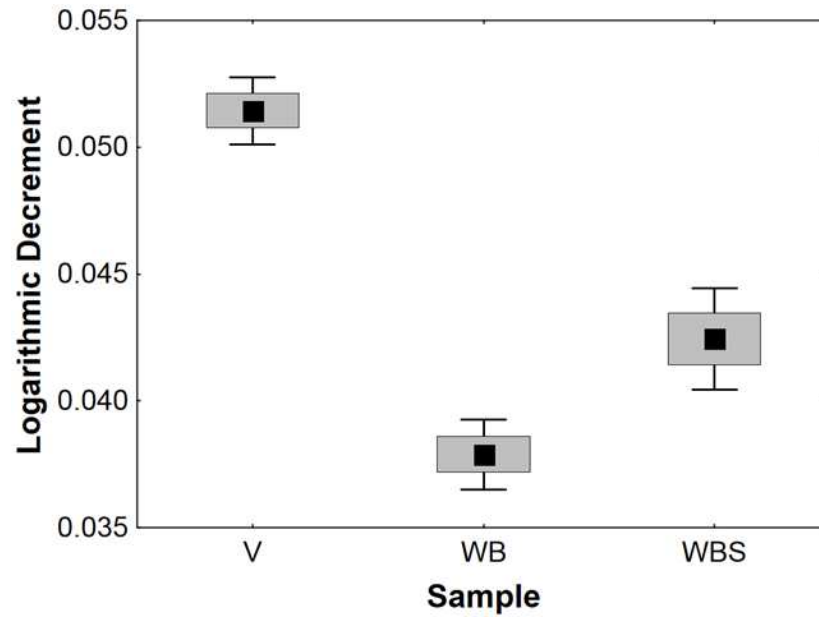
Sample	$G''_{co}$ , GPa	$G''_{cu}$ , GPa	$r^2$	$G'_{co}$ , GPa	$G'_{cu}$ , GPa
V (69)	$0 \pm 0.005$	$0.08 \pm 0.01$	0.722	0.61	3.60
WB (56)	$0 \pm 0.004$	$0.046 \pm 0.004$	0.600	0.40	4.84
WBS (23)	$0 \pm 0.009$	$0.11 \pm 0.08$	0.733	0.37	4.63

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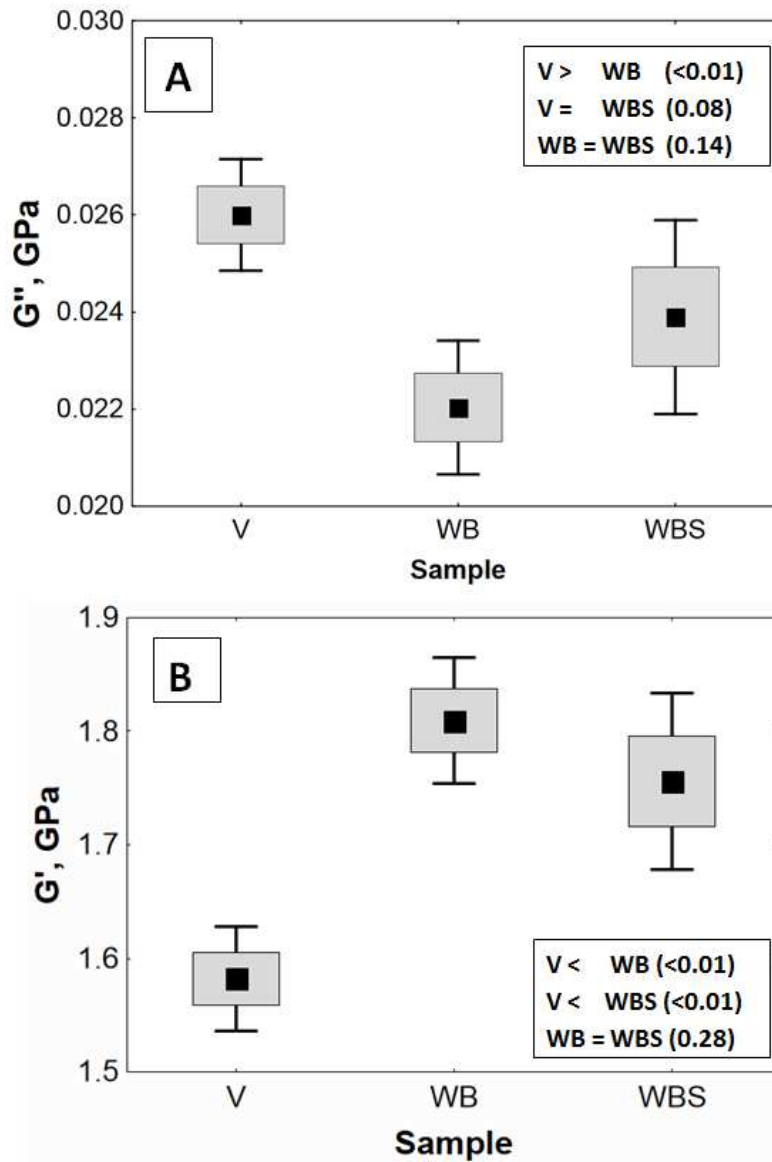
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348 **Figure 1:** Summary of data for the logarithmic decrement  $\lambda$  for the three samples. Data are  
349 given as means (■), standard errors SE (boxes), and limiting values for the 95% confidence  
350 range ( $1.96 \cdot SE$ : whisker). Differences between all data sets are highly significant on the 95%-  
351 level.

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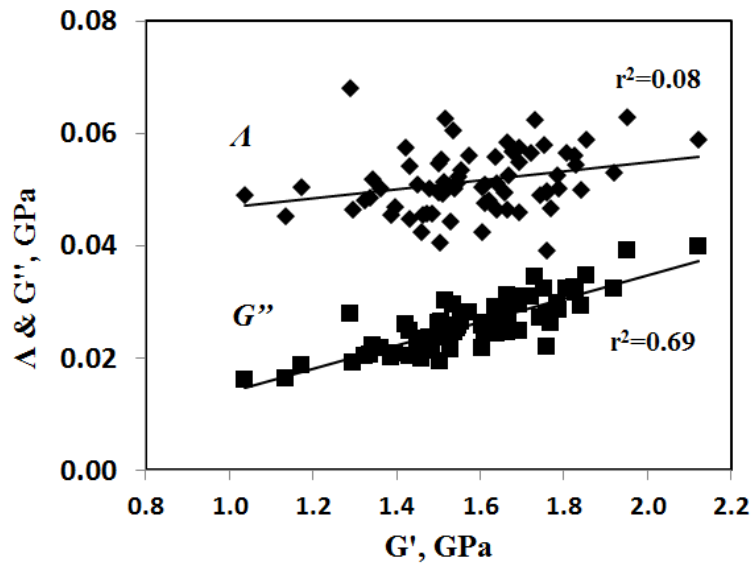
355 **Figure 2:** Summary of (A)  $G''$ - and (B)  $G'$ -data for all samples. Data are given as means (■),

356 standard errors SE (boxes), and limiting values for the 95% confidence range ( $1.96 \cdot SE$ :

357 whisker). Insets give the results of LSD multiple comparison of means tests with their levels

358 of significance (p-values). If  $p < 0.05$  effects are significant on the 95%-level.

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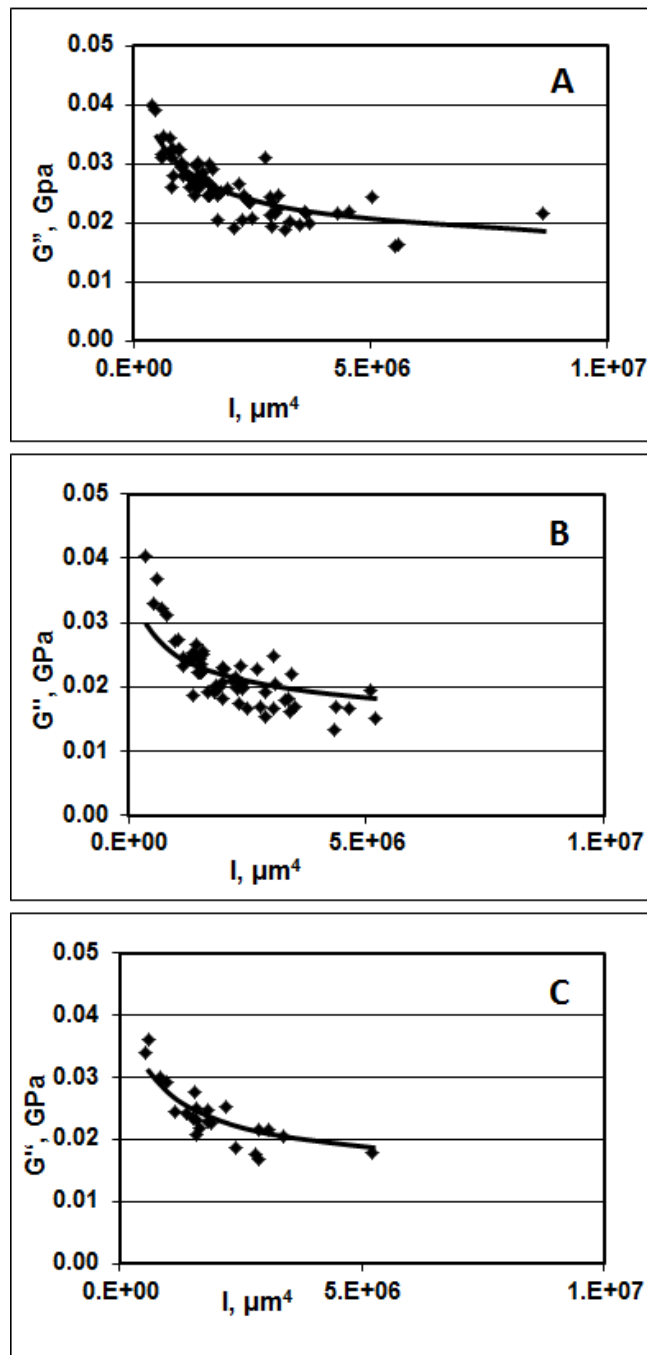


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361 **Figure 3:** Plot of  $\Delta$ - (◆) and  $G''$ -data (■) vs  $G'$  for virgin hair. Linear regression lines and the

362 coefficients of determination  $r^2$  are given.

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364

365 **Figure 4:**  $G''$  versus polar moment of inertia for virgin (A:V), perm-waved and bleached (B:  
 366 WB), and additionally shampoo-treated (C: WBS) hair. Solid lines are based on the fit of  
 367 Equation 9.

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