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# Multiple Bleaching of Hair: Fatigue Failure Investigations and Weibull Analyses

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**Abstract**

The fatigue failure (F/F) test is an important method for evaluating hair strength. In this study, we evaluate commercial European hair after up to four bleaches. F/F-data were directly fitted using a two-parameter cumulative Weibull distribution (CWD) function, described by lifetime index ($\lambda$) and shape factor ($\beta$). Fibre samples taken from treated tresses ($T$) exhibited stable F/F behaviour across multiple treatments, while treated single fibres ($S$) showed early breakage if treated more than twice. We attribute this effect mainly to the liquor ratio during treatment. In this context, we suggest a strategy to estimate a treatment-related apparent decay constant for $\lambda$. The constant for $T$-samples was determined as $k_n = 0.095$. This is consistent with DSC results in the literature for the denaturation enthalpy. This may indicate an important role for intermediate filaments in fatigue failure. To possibly establish a link with hair breakage during combing, we investigated the properties of the hazard function for $T$-samples based on $\beta = 0.92$. The model calculations indicate that early failure risks are low but increase exponentially with repeated bleaches. The correspondence with the literature results leads to the hypothesis of a relationship between F/F and combing tests.

**Keywords:** hair; fatigue failure; bleaching; Weibull analysis; multiple bleach kinetics; liquor ratio; hazard function

## 1. Introduction

Bleached hair is a standard material used when assessing the efficacy of ingredients, formulations and processes to improve hair properties such as strength and to repair damage [1]. An important tool to support related claims is the fatigue failure (F/F) test on single hair fibres [2–4].

In a recent F/F-investigation [5], we applied a wide range of constant dynamic strains (4–30%) to untreated hair. As an alternative to the standard linearisation approach [2–4], we showed that the direct fit of the cumulative Weibull distribution (CWD) function to the F/F-data was a consistent analytical approach. For the current investigation, we analysed the F/F-behaviour of hair that was bleached multiple times, either in the form of single fibres or as tresses. F/F-tests were carried out at an experimentally feasible, low level of strain (3.5%) to stay close to practical conditions. The low strain range provided stable shapes of CWD functions, namely, when using a logarithmic scale for the fatigue cycles [5].

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It is well established that the effects of bleaching depend heavily on the processing conditions. To evaluate the impact of the bleach-paste-to-hair ratio (liquor ratio) during the bleaching process, we compared the performance of individually treated fibres, as an extreme case, to that of hairs taken from tresses after each of the multiple treatments. This method enabled us to obtain an impression of the principal changes in CWD-function parameters that may be expected with drastic changes of treatment conditions.

Several authors have suggested or assumed a specific relationship between hair breakage during mechanical grooming (combing, brushing) and F/F performance e.g., [2,4,6,7]. A recent investigation on machine-based hair combing called this view into question [8]. To further investigate the potential relationship between the test methods, we examined the hazard functions related to the CWD fits and the changes to these caused by bleaching. We speculate that the hazard function may be a suitable tool for specifically assessing the risk of early hair breakage, which can occur during combing and brushing.

## 2. Experiment

### *2.1. Materials and Treatments*

The study utilised European Natural Hair 7/0 sourced from Kerling International (Bagnang, Germany). This hair is a blend of untreated hairs from a large number of donors and is generally accepted as a reference material for European hair. The hair came as a long (20 cm free hair length), flat tress. Small (1 cm wide) sub-tresses were taken from the master tress, as required. Due to the nature of the commercial hair, each sub-tress is assumed to represent a random subsample of the hair fibres in the collective of the master tress.

For preparation, the sub-tresses were gently massaged with a 12.5% solution of SLES at pH 4.5 for 1 min. The surfactant solution was then rinsed off under tap water for 2 min. Subsequently, tresses were dried and stored under standard laboratory conditions for at least 24 h.

In the treatments involving single fibres, a random selection of hairs ($N \approx 50$) was taken from the tress. The middle part of each of these hair was crimped using the Dia-Stron Auto-Assembly System AAS 1600 (Dia-Stron, Andover, UK). The crimped fibres were then placed on a plate holder and bleached for 45 min (pH 9.5) using a standard commercial bleaching powder and a 9% hydrogen peroxide developer (mixing ratio 1:2) (Henkel, Hamburg, Germany). Subsequently, the fibres were rinsed under tap water for 2 min and dried under laboratory conditions. After 30 min, the procedure was repeated according to the intended number of treatments. We acknowledge that the brass crimps may have affected the oxidation process when treating single hairs. However, in view of the overall focus of this investigation, the potential effects were not further considered.

Single fibre samples were coded as *Sx_y*, where *S* signifies the sample formed by the fibres for single-hair treatments, *x* gives the number of bleaches and *y* is a running number given to the individual set of fibres tested by F/F (see Supplementary Materials Table S1).

An equivalent bleaching procedure was applied for 1 cm wide tresses, with a liquor-to-hair ratio of 2:1. After the intended number of treatments, approximately 50 fibres were taken from a tress at random. These fibres were crimped for F/F-testing. The remaining tress was subjected to further bleaching, as required. Similar to the *S*-samples, fibre samples taken from tresses (*T*) were coded as *Tx_y*, analogously to the *S*-samples (see Supplementary Materials Table S2). Untreated fibre sets were coded as *UT_y*, where *y* identifies a set of fibres taken from the master tress for testing.

Then, 48 h after each treatment, fibres were placed in the F/F-machine sample cassette and conditioned in a climate chamber containing the test machine, at 22 ± 2 °C and 55 ± 5% RH for at least 6 h.

### 2.2. Test Method

All tests were conducted on a fibre fatigue failure instrument (CYC802, Dia-Stron, Andover, UK). The machine was positioned in a climate chamber (5533, Ets Inc., Perkasie, PA, USA) achieving 22 ± 2 °C and 55 ± 5% relative humidity. The first analysis of the data, using the traditional Weibull linearization approach, has been presented previously [9].

Due to the crimping process, fibre length was ≈30 mm. The actual initial length $l_o$ for each fibre is defined as the length at a small load of 2 g. For our experiments, we applied a cyclic engineering strain setting of 5% (nominal strain, $\varepsilon_N$). As has been shown [5,9], the nominal strain $\varepsilon_N$ deviates for each fibre from the achieved strain $\varepsilon_A$, where the average deviation depends on the actual experiment. For $\varepsilon_N$ = 5%, we expected a mean achieved strain of $\varepsilon_A \approx 3.5\%$. This strain is in a range wherein the shape of the CWD function becomes largely independent of strain [5]. We confirmed the consistency of the achieved strain for the current investigation, namely, for *S*-sets of samples (*S1_1*, *S2_1*, *S3_1*; see Supplementary Materials Table S1) and found it to be $\varepsilon_A = 3.45 \pm 0.082\%$. These results reflect the overall consistency of our testing process. The cyclic strain was applied to the fibres at a rate of 40 mm/s, translating into a frequency of ≈30 Hz. As has been consistently shown [5,10], constancy of F/F-testing conditions, whether for constant strain or constant stress, is a prerequisite to ensure the reproducibility and comparability of results across samples. Experiments were conducted up to a maximum of 100k cycles.

Due to factors outside the scope of our current investigations, the number of fibres tested varied between 20 and 74 (see Supplementary Materials Tables S1 and S2). With the exception of sample *UT_2*, all fibres failed before the chosen maximum number of cycles (100k). Fibres were omitted from the set only if there were clear signs of experimental problems, such as crimping issues, which are not uncommon in tensile testing, or if there were technical artefacts related to data collection by the F/F-instrument. No data were subsequently excluded from the data sets.

### 2.3. Data Analysis

For reasons outlined previously [5], we directly fitted the cumulative Weibull function to the data rather than using the standard linearization method. This approach satisfies the requirements to minimise the deviations of the data points from the theoretical curve and to ensure that these deviations are normally distributed.

The experiment determined the number of cycles ($C_i$) at which each fibre *i* failed. At each of these points of the experiment, the fraction of broken fibres was calculated as follows:

$$F_i = n_i/N \tag{1}$$

so that the fraction of surviving fibres is given as follows:

$$S_i = 1 - n_i/N \tag{2}$$

where $n_i$ is the number of broken and $N$ the total number of fibres in a testing set.

The change of $F_i$ with the testing cycles may be modelled using the cumulative Weibull distribution (CWD) as the failure function $F$, as follows:

$$F = 1 - exp[-(C/\alpha)^{\beta}] \tag{3}$$

or equivalently with Equation (2), for the survival function $S$, as follows:

$$S = exp[-(C/\alpha)^{\beta}] \tag{4}$$

Continuing with the established parameterisation [5], we refer to $\alpha$ as the *specific lifetime* and to $\beta$ as the *shape factor*.

The Weibull probability density function $f$ underlying Equations (3) and (4) is given as follows:

$$f = \beta/\alpha\ (C/\alpha)^{\beta-1}\ exp[-(C/\alpha)^{\beta}] \quad (5)$$

Given the nature of the CWD function [5], we consider the survival function on the $ln(C)$-scale, thus making $ln(\alpha)$ the relevant parameter, referred to as *lifetime index* ($\lambda$). The direct fit of Equation (4) to the data sets was conducted using suitable software (Excel, Microsoft, Redmont, WA, USA, 2016; Statistica, V13, Tibco Software Inc., Palo Alto, CA, USA, 2017) [5].

## 3. Results

Figure 1A,B summarize survival curves for selected $S$ sets of fibres repeatedly treated by bleaching. Samples are identified by their specific codes (see Supplementary Materials Table S1).

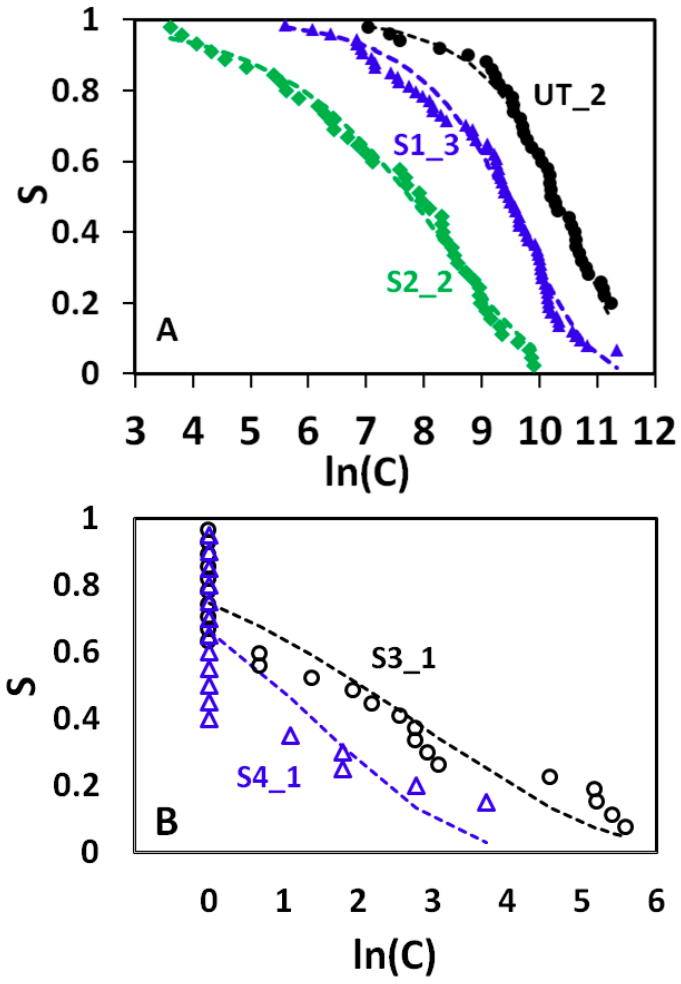



**Figure 1.** Graphical representation of survival data for a specific $S$ set of samples against $ln(C)$. (**A**) Untreated (*UT_2* ●), 1× (*S1_3* ▲), and 2× bleached hair (*S2_2* ♦). (**B**) Data for 3× (*S3_1* o) and 4× bleached hair (*S4_1* Δ). The dashed lines are the fitted curves according to Equation (4) with the parameter values applying for a specific set of data (see Supplementary Materials Table S1).

The curves for untreated (*UT_2*) and bleached (*S1_3*, *S2_2*) hair follow Weibull curves of roughly similar shape, systematically shifted to lower $ln(C)$-values (see Figure 1A). However, for *S3_1* hair, there was a significant change in the F/F-behaviour. The data

dramatically shifted to lower *ln*(*C*)-values and a large number of fibres had already broken during the first straining cycle [*ln*(*C*) = 0; *C* = 1]. As expected or at least anticipated, this effect became increasingly pronounced with further bleaching (*S4_1*, see Figure 1B). Though fitting of the CWD function is possible for both the *S3* and *S4* data (see Table S2), we conclude that the accumulation of failures at *C* = 1 formally invalidates the results. The related F/F-parameter values are thus considered for qualitative purposes only.

Supplementary Materials Table S1 summarises the $\lambda$- and $\beta$-results for all tests on the *S* sets of fibres together with their standard errors (*SE*) and the coefficients of determination ($R^2$), as a measure of the goodness of fit of Equation (4).

$R^2$ values are in all cases in the range of 0.95 and well above, indicating excellent fits of the CWD function for all samples *UT* to *S2*. As expected [5], residuals are in all cases normally distributed. Plots of experimental against theoretical values give no indications of specific early breakage of subsets of fibres. These observations are documented for the *S2_3*-set of data in the Supplementary Materials Figure S1. Due to the pronounced early breakage of fibres (see Figure 1B), the $R^2$ value drops for the *S3* (0.890) and even more for the *S4* sample (0.674) (see Table S1).

Data from hair samples from treated tresses *T1* to *T4* are all well described by Equation (4) (see Table S2). Typical survival curves are summarised in Figure 2. Though the individual curve shapes are somewhat different, all fits show strong linear regressions between theoretical and experimental values. This implies that all data for a sample belong to a homogeneous set without obvious indications for early breakage. Also, all fits show normally distributed residuals. This statement is supported by the example of sample *T4_2* in Supplementary Materials Figure S2. Table S2 summarises the results of the fits of Equation (4) to the *T* sets of data, analogous to Table S1. The $R^2$ values are all around 0.98 and above.

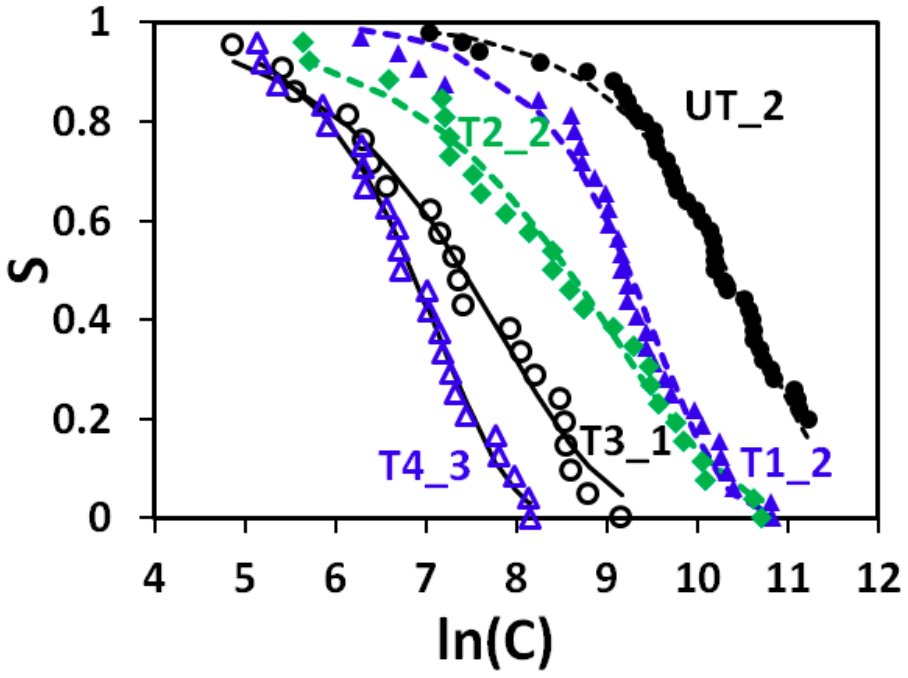



**Figure 2.** Graphical representation of survival data from the *T*-set of samples. Untreated: *UT_2* (●), 1× bleached: *T1_2* (▲), 2× bleached: *T2_2* (♦), 3× bleached: *T3_1* (o), 4× bleached: *T4_3* (Δ). The dashed lines are the fitted curves according to Equation (4) with the parameter values applying for a specific set of data (see Table S2).

## 4. Discussion

Figures 1 and 2 summarise the F/F-results for treated single fibres as well as for fibres from treated tresses for the various bleach levels. For the *S* sets of fibres (see Figure 1A) only two bleaches were experimentally feasible before a large number of fibres broke during the first fatigue cycle (see Figure 1B). Data for bleach levels 0–2 are well fitted by Equation (4) (see Table S1). In contrast, all *T* data sets are 'well behaved' (see Figure 2) in that they show no early breakage. All *T* data sets are well fitted by Equation (4) (see Table S2). *S* as well as *T* data consistently shift to lower values on the *ln*(*C*)-scale with bleaching (see Figures 1 and 2).

The repeated bleaching of single fibres and tresses under defined conditions provides an ordinal scale against which these results may be considered. Building on previous work [11,12] and as corroborated by recent investigations [1,13,14], we furthermore assume that repeated bleaches have equivalent and additive effects on the F/F variables. On this basis, we hypothesise a pseudo-absolute scale for the bleach treatments, with the untreated material representing a genuine zero-state (Bleach Level: 0).

Figures 3 and 4 summarise the results for the Weibull parameters for all experiments for single-fibre and tress treatments, on the bleach level scale. The related data are summarised in Supplementary Materials Tables S1 and S2.

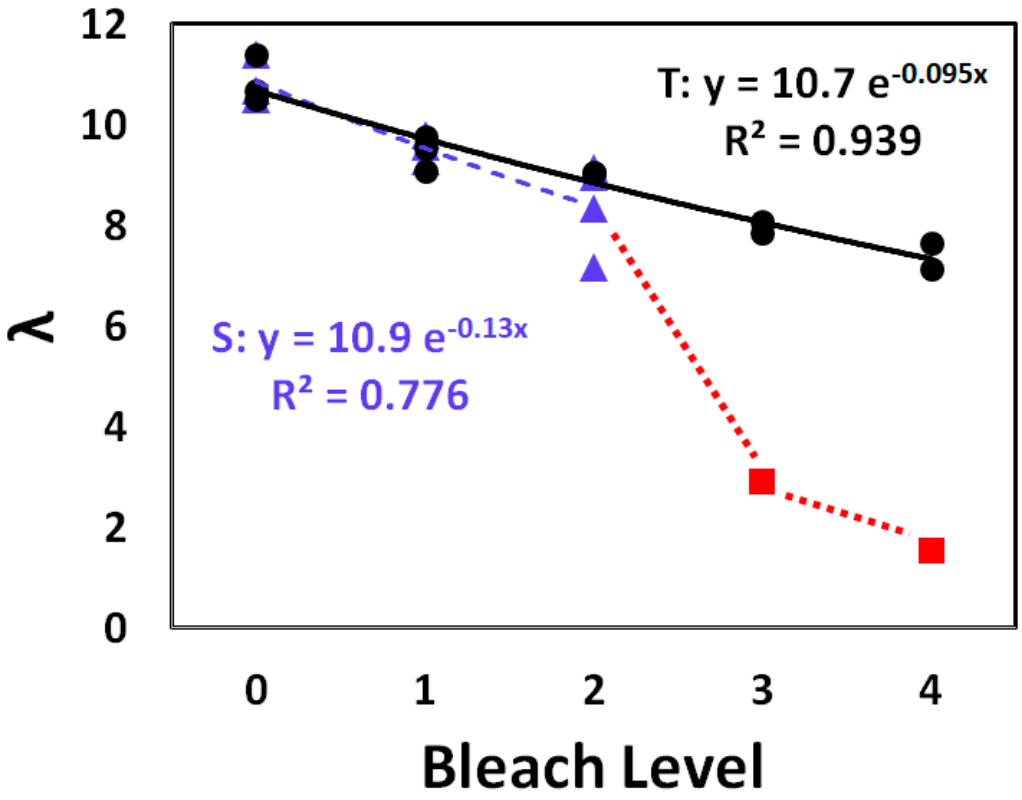



**Figure 3.** Summary of the $\lambda$ values for the *S* (▲) and *T* sets (●) of samples against bleach levels. The standard errors of the variable values are not given, because the related whiskers would be equivalent or even smaller than the symbol sizes. The lines through the data points (—, - - -) are exponential fits according to Equation (7). The respective equations are given on the graph. This approach does not apply to samples *S3* and *S4*, where we observed a discontinuity for more than two bleaches, indicated by the change of symbol (■) and the empirical lines connecting the data (. . .).

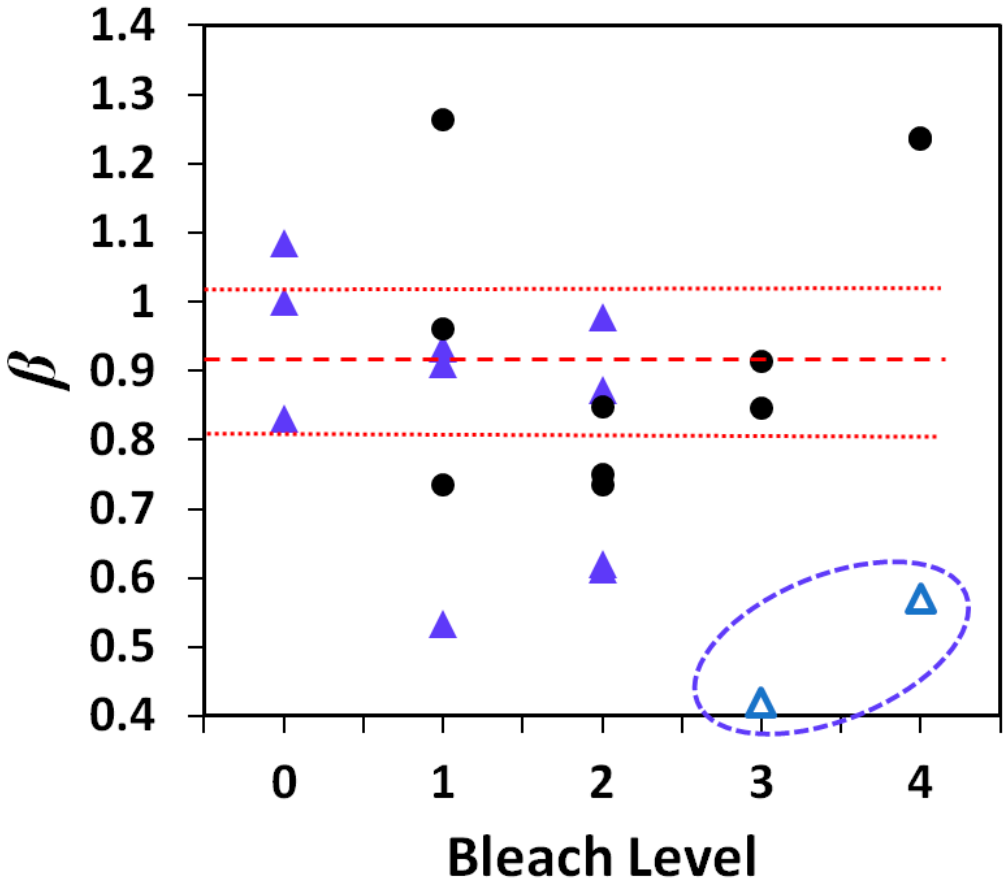



**Figure 4.** $\beta$ values for all *S* (▲) and *T* (●) samples (see Tables S1 and S2). The mean across all samples is marked (- - -) together with the 95–confidence range (. . .). Level 0 data (*UT*) are necessarily identical for both data sets. The results for the *S3* and the *S4* sample (Δ) are not included in the considerations of the data set (see text) and are marked by the ellipse (- - -).

Figure 3 summarises the results for the lifetime index $\lambda$ for both *S* and *T* samples. The results for untreated (*UT*) conditions are necessarily equal for both sets. After one bleach, results for $\lambda$ were still very similar for *S* and *T* samples. This changed after two bleaches, when $\lambda$ values for the *S* fibres dropped significantly. A pronounced discontinuity developed for the *S* samples when proceeding from two to three bleaches (see Figure 1B). The fits of Equation (4) are excellent for the *S* samples after one and two bleaches, as reflected by $R^2 > 0.95$ (see Supplementary Materials Table S1). Fits of the CWD function are also possible for the data of the *S3* and *S4* samples (see Table S1).

However, as mentioned above, these results are only qualitatively discussed due to the significant accumulation of failures during the first straining cycle, which in our view formally invalidates the application of Equation (4) for cases *S3* and *S4*. The low $\lambda$ values for three and four bleaches for the *S* samples corroborate the view that high bleaching levels lead to very brittle fibres with a strong tendency for early failures.

We attribute the difference between the $\lambda$ values for the *S* and *T* sets in Figure 3 primarily to the ratio of liquor to solid (liquor ratio) during the bleaching process. The reaction between bleaching liquid/paste and hair is, in fact, very complex [15]. The concentration of $H_2O_2$ in the bleach paste is comparatively stable, and any changes are caused only by its natural decomposition. However, the concentration quickly decreases once in contact with hair through the chemical reaction between the peroxide and melanin as well as proteins in hair [16–18]. With regard to the proteins and on the molecular level,

it is well established that bleaching leads to the oxidation of the disulfide bonds to form cysteic acid [15,16,19]. On the structural level, oxidation leads to substantial changes in the properties of the main morphological components of hair, such as the intermediate filaments and matrix [1,20,21]. This, as a result, negatively affects, for example, the thermal [11,12] and mechanical properties of hair [4,22–25].

Due to chemical reactions, the concentration of the active species in the liquor decreases with time [15] through a diffusion-reaction mechanism [20]. This decrease is more pronounced at a lower liquor ratio. In this study, the liquor ratio was effectively infinite for single fibres, resulting in comparatively little change in the active species. For the tresses, liquor ratio was 2:1, leading to a decrease in the oxidative species while bleaching progressed. Consequently, bleaching damage following equal treatment times was more severe in single fibres compared to fibres from tresses.

As expected, a very high degree of brittleness and early failures were observed for *S* samples after three and four bleaches. This aligns with the observation that $\beta$ was substantially lower than unity for these samples (see Supplementary Materials Table S1).

Based on findings of earlier DSC studies on systematically bleached hair strands [11], and considering the pseudo-absolute x-scale presented in Figure 3, we examined whether apparent first-order kinetics could be applied to the change of $\lambda$ with repeated bleaching. Accordingly, we formulate the following:

$$-d[\lambda]/dn = k_n\,[\lambda] \tag{6}$$

where $n$ is the number of bleaches and $k_n$ the apparent decay constant for $\lambda$. The integration of Equation (6) leads to a simple exponential equation, expressed as follows [26]:

$$\Lambda = \lambda_0\, exp(-k_n\, n) \tag{7}$$

where $\lambda_0$ is the estimated start value of the decay constant.

Due to the pronounced early failures of fibres for the *S* samples after multiple bleaches (see Figure 1B), a discontinuity of $\lambda$ occurred for Bleach Levels > 2 (see Figure 3). Accordingly, only the results for *S* data up to two bleaches are considered.

As shown in Figure 3, the change in $\lambda$ with the number of bleaches ($n$) is well described by Equation (7) for both the *S* and *T* data sets. This leads to apparent decay constants for the *S* data of ${k^S}_n = 0.13 \pm 0.024$ and of ${k^T}_n = 0.095 \pm 0.008$ for the *T* data, respectively.

When comparing the results for the *S* and *T* datasets (Figure 3) based on the equations provided, we conclude that on a linear C scale, the *S* curves generally shift to lower *C* values by about a factor of 3.2 [≈exp(1.17)] with each bleach. For the *T* data, the curves shift only by about a factor of 2.4 [≈exp(0.88)] to lower *C* values per bleach. Overall $\lambda$ values for *T* are higher than for the *S* sets for equal bleach levels, reaching $\Delta\lambda = 0.45$, that is, a factor of about 1.5, after two bleaches. These considerations corroborate the above discussion of the effects of the liquor ratio and especially emphasise the importance of defined treatment conditions to enable sample comparison.

Keratinous fibres are essentially filament/matrix composite materials [27]. Investigating a range of keratins via DSC (wet), it was established that the denaturation enthalpy ($\Delta H_D$) relates to the $\alpha$-helical content of the intermediate filaments (IF) [28]. When DSC (wet) was used to investigate hair from tresses after repeated bleaching, it was observed that the change in $\Delta H_D$ is well described through a version of Equation (7) with an apparent decay constant of $k_n(\Delta H_D) = 0.069 \pm 0.0103$ [11]. This agrees surprisingly well with the value of ${k^T}_n$ for $\lambda$, given above.

The good agreement between the apparent decay constants for λ and $\Delta H_D$, given that they relate to similar multiple-bleach experiments, may suggest a special role for the filaments and their bleaching-induced damage in the F/F performance of chemically treated hair. This is in line with observations for polymeric fibre/matrix composites [29]. We hypothesise that this is due to the specific chemical damage of bleaching to the structure of the IFs [12,18,25,30].

These considerations do not contradict the evidence for an important role of the IF-associated matrix in the F/F-performance of hair. Against the background of the fatigue/failure behaviour of fibre/matrix composite materials [29,31,32], the effects of the matrix are reflected by the positive effects of repairing/filling of cracks and holes in the fibre structure, e.g., through polymers and peptides [33,34]. Through patching microscopic defects, the progression of planes of failure is delayed [35,36], thus increasing the lifetime of a fibre [3,4,37–39]. A positive effect on fatigue failure of hair also appears to be induced by a shift of fibre pH, e.g., through citric acid [40].

Similar to Figure 3, Figure 4 summarises the $\beta$-values obtained from fitting Equation (4) to all data sets. The details are given in Table S2. For the *S* samples, the restrictions discussed above apply for Bleach Levels 3 and 4. In Figure 4, the related $\beta$ values are marked by an ellipse. The variability of the $\beta$ results is essentially attributed to dependence on the individual specific strain of an experiment to which the fibres of the samples were subjected during testing [5]. For the current investigation, the strain level is overall consistent with $\varepsilon_A \approx 3.5\%$, as discussed above. The results for $\beta$ are in line with expectations [5] and largely consistent with those in the literature for straight European hair [2–4,39,40].

In contrast to the results for $\lambda$ (see Figure 3), there was no consistent trend for $\beta$ with Bleach Levels. Also, there was no consistent difference between the sample groups (*UT*, *S*, *T*), excluding *S* samples for levels 3 and 4. This is shown by Analysis of Variance ($p = 0.236$). The mean across all data is $0.92 \pm 0.10$ (95% confidence range). The levels for the mean and the confidence limits are marked in Figure 4.

In line with the expectations from the graph, the mean is not significantly smaller than unity (95–level, *t*-test, $p = 0.1$). $\beta = 1$ signifies a process for which the failure rate remains constant while the fatiguing process progresses [5]. The failure rate does not significantly change with the structural and chemical damage imparted by bleaching until a certain threshold is reached, e.g., for *S* samples at levels > 2. In contrast, $\beta$ values in the range of $\beta \approx 0.3$–$0.7$ have been observed for textured hair [41], indicating a tendency for early failure for this type of hair [42]. This leads us to conclude that there seem to be significant differences in the mechanisms of fatigue failure for straight compared to textured hair.

Welzel et al. [43] investigated the F/F break morphology of hairs after repeated bleaches. They observed a strong prevalence of angled fractures regardless of the type of hair treatment. They noted that the length of the 'tongues' decreased with $\lambda$ and thus with the number of bleaches. The results confirm that bleaching increases hair brittleness.

Figure 5, adapted from Welzel et al. [43], shows images of two typical fibres with 'tongue-like' fractures broken under equal F/F-conditions, corresponding to recent observations by Samoylenko et al. [44] when applying the moving loop test. It is worthwhile noting that certain fracture morphologies (smooth, step, fibrillated), as reported in various references [45–49], were rarely observed in the F/F tests [36]. Ngoepe et al. [41] demonstrated that, while failure rates differ between straight European hair and textured African hair, as indicated by variations in $\beta$, both types of ethnic hair exhibit similar tongue-type failure patterns.

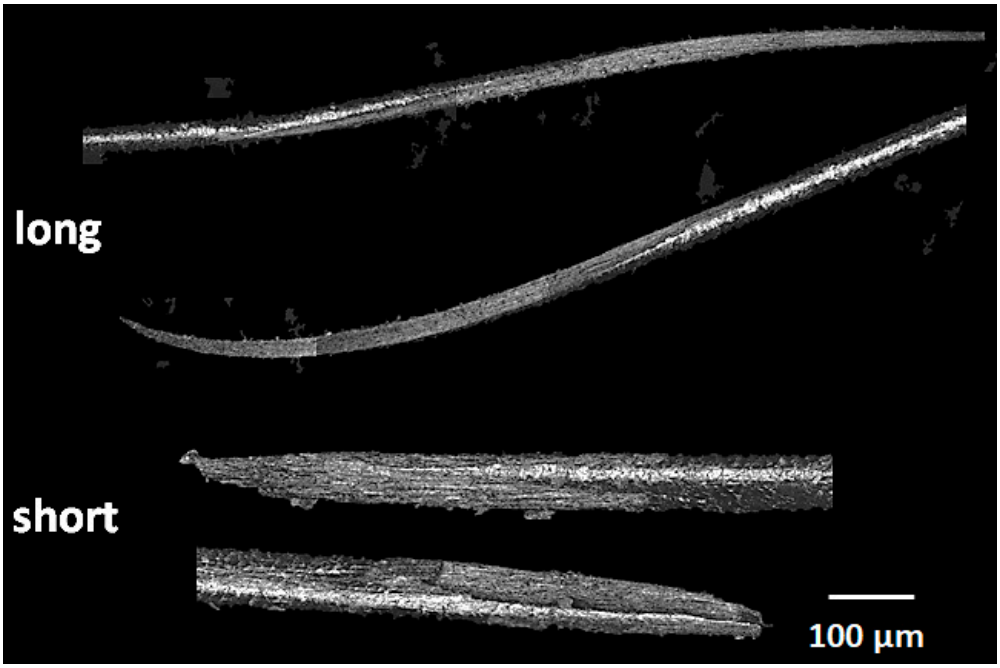



**Figure 5.** Two hair fibres broken by fatigue failure, showing the pairs of associated broken ends. The fibres were chosen to represent the predominant tongue-like fractures. (Adapted from Ref. [43]).

For hair-grooming practices such as combing and brushing, it is important to understand the risk of hair failure in the early stages of the fatigue process. To assess this risk, the hazard function $h(C)$ is a suitable tool (see Equation (8)). $h(C)$ indicates the likelihood of fibre failure at a specific fatigue cycle $C$, based on the fibres which have survived up to that point. The hazard rate is given by the ratio of the Weibull distribution [$f(C)$] over the survival function $S(C)$ [42,50]:

$$h = f(C)/S(C) \tag{8}$$

With $S(C)$ being defined by Equation (4), we obtain:

$$h = \beta/\alpha\ (C/\alpha)^{\beta-1} \tag{9}$$

Figure 6 summarises the $h(C)$ curves for both untreated and bleached $T$ samples. It is important to note that a principal change in the shape of the hazard function occurs when the $\beta$ value is either below or above unity, as occurred with the experimental results (see Figure 4). To keep the discussion straightforward at this stage, we decided to focus on the results for $h(C)$ based on the theoretical $\lambda$ values derived from the exponential equation in Figure 3 and using the mean experimental value of $\beta = 0.92$. In Figure 6, to emphasise results for risks related to the early stages of hair grooming, such as hair breakage caused by combing, we utilise a linear $C$ scale for the initial phases of the F/F test ($C \leq 100$).

As was to be expected, Figure 6 (inset) shows that the risk of failure at the first testing cycle ($C = 1$) increases with the number of bleaches about 20-fold from $4.9 \times 10^{-5}$ ($UT$) to $109 \times 10^{-5}$ (4×). However, despite this strong increase, the actual risk of early failure is still quite low even for 4×-bleached hair from tresses ($T$ samples). This aspect of the results may well be related to the preparation of the fibres, wherein broken fibres were discarded, as well as to the machine filter, where fibres showing atypical loading curves were excluded from the final data set. Overall, the results for $h(C)$ suggest that F/F-experiments are more reflective of the long-term strength of hair rather than its early failure.

In a recent investigation, it was observed that hair breakage after a high number of combing cycles was a strong predictor of early breakage [8]. In this context, it is noteworthy

that the increased risk of failure associated with bleaching (Figure 6), though consistently low, corresponded to the rise in the number of fragments released during the early stages of combing ($N_0$) [8]. The ratio of the number of early released fragments ($N_0$) during combing for bleached versus untreated hair was 1.8 [8]. The ratio for $h(C = 1)$—values for 1× bleached (11.9) vs. untreated hair (4.9), as given above, was 2.4.

Given the differences in materials and methods, we find this agreement quite remarkable. With this observation, we speculate that $h(C = 1)$ may serve as a link between F/F- and cyclic combing testing. Given the significance of both tests for product development and claim support, we want to emphasise that this hypothesis requires further evaluation before it can be considered practically relevant.

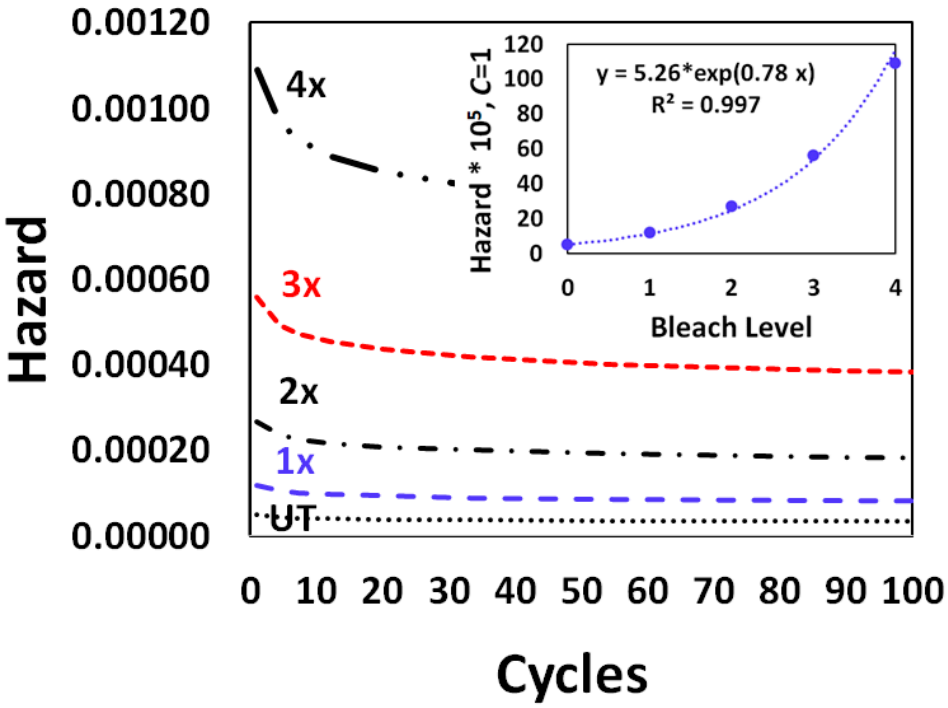



**Figure 6.** Hazard functions for *T* samples, derived from the experiments (see text), for untreated (*UT*), 1×-, 2×-, 3×-, and 4×-bleached hair. Calculations are based on arithmetic means for $\lambda$ for each bleach level and $\beta = 0.92$ for all samples. The x-scale is limited to a relatively small number of cycles ($C = 100$) to correspond to early stages of mechanical grooming. The inset gives the hazard values for the first F/F cycle ($C = 1$) for all samples and the applicable exponential trendline.

## 5. Conclusions

As anticipated from a prior investigation [5] and supported by extensive literature [2,3,38,41], our study demonstrates that the cumulative Weibull distribution (CWD) function offers excellent fits for the F/F results of hair. This holds true for samples comprising treated single fibres (*S* samples) and fibres randomly selected from treated hair tresses (*T* samples).

In the case of the *T* samples, all fibre sets showed consistent behaviour (see Figure 2), for at least up to four bleaching treatments. We attribute the differences observed between the *S* and *T* samples to significantly different liquor ratios during the treatment process. It may be argued that the *S* samples represent the ultimate, 'true' effect of bleaching on hair. Along with emphasising the need for strictly controlled testing conditions, the differences between *S* and *T* samples highlight the importance of maintaining consistent treatment conditions, e.g., for bleaching. This is crucial to ensure that results from F/F or other tests for evaluating hair treatments and actives are comparable.

The results given in Figure 3 for the *T* samples support the notion that repeated bleaching sets up an apparent absolute scale for the analysis of the F/F-parameters. This facilitates the determination of an apparent decay constant for the lifetime index $\lambda$. For the *T* samples, this decay constant was found to be $k^T{}_n = 0.095 \pm 0.008$. This value is in satisfactory agreement with the constant determined by DSC (wet) for the bleaching-induced decrease of the helix in the intermediate filaments (IF) [11]. This leads to the hypothesis that damage and defects in the IFs play an important role in the fatigue-induced failure of hairs.

In contrast, the shape factor of the CWD function, denoted as $\beta$, remained largely unchanged by the bleaching processes. With a mean of $\beta = 0.92 \pm 0.10$, it tended to remain slightly below $\beta = 1$. This value is significantly higher than that observed for textured hair ($\beta = 0.3$–$0.7$) [41], indicating significant differences in the fatigue failure of straight versus textured hair. The consistency of $\beta$ in our study indicates that the failure process involving constant strain remains fundamentally unchanged by bleaching, only shifting to shorter lifetimes. It is notable that $\beta = 1$ corresponds to the width of the Kelvin–Voigt equation, which was recently utilised [8] to model hair fragment formation during combing.

The prevalence of continuous and angled fracture planes (see Figure 5) across bleaching levels [43] corresponds to the overall invariance of the shape factor. We suggest that the consistent form of the fracture planes calls into question whether the 'weakest link' hypothesis, associated with the CWD function to fit the data, is in fact the most plausible model to characterise hair fatigue failure, at least for the current framework of samples.

With regard to the practice of hair grooming, it is of interest to gain information about the risk of hair failure at a very early stage of the fatigue process. In this context, we investigated the hazard function and found, as expected, that the risk of early failure of fibres was low but increased with the bleaching level. We find it reasonable to conclude that the F/F test emphasizes the resilience or long-term strength of hair, while combing failure provides a more effective measure of short-term strength.

Analysing the values of the hazard function, especially during the first fatigue cycle, and exploring the similarities between the shapes of the CWD (F/F) and the Kelvin–Voigt (combing) function, leads us to suggest a potential connection between these two methods through the hazard function. We consider this potential relation as quite unexpected in light of a previous study [8]. This matter will certainly require further and comprehensive investigation.

**Supplementary Materials:** The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/cosmetics13050231/s1, Figure S1: Normal probability plot of the residuals for the fit of the CWD-function to the data for sample *S2_3*; Figure S2: Normal probability plot of the residuals for the fit of the CWD-function to the data for sample *T4_2*; Table S1: Values for the Weibull-parameters together with their standard errors (*SE*) for untreated hairs (*UT*) and fibres bleached individually. Specific codes are given for all samples. The coefficients of determination ($R^2$) are given together with the total number of fibres tested for each sample (*N*). Samples for 3× and 4× bleached fibres are marked in grey. These results are for information only because of the formal invalidity of the fit, due to the pronounced early breakage of fibres (see text); Table S2: Values for the Weibull-parameters together with their standard errors (*SE*) for fibres taken from tresses. Specific codes are given for all samples, as in Table S1. The coefficients of determination ($R^2$) are given together with the total number of fibres tested for each sample (*N*). UT-values are the same as in Table S1.

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## References

1. Martins, E.; Castro, P.; Ribeiro, A.B.; Pereira, C.F.; Casanova, F.; Vilarinho, R.; Ramos, Ó.L. Bleached hair as standard template to insight the performance of commercial hair repair products. *Cosmetics* **2024**, *11*, 150. [CrossRef]
2. Evans, T.A. Fatigue testing of hair-a statistical approach to hair breakage. *J. Cosmet. Sci.* **2009**, *60*, 599–616. [PubMed]
3. Everaert, E.; Tran, D.; Zhang, S.; Tran, D.; Kroon, B.; Zhang, G.; Thompson, B.; McMullen, R.L. Strengthening the hair fiber from within: Repairing the cortex of damaged hair. *IFSCC Mag.* **2015**, *3*, 111–117.
4. Marsh, J.M.; Felts, T.J.; Whitaker, S.; Cowans, C.; Ritter, A.; Husen, K. Fatigue Strength of Panelist Hair and Correlation with Hair Damage Measures. *J. Cosmet. Sci.* **2021**, *72*, 600–610.
5. Berriche, L.; Welzel, J.; Sirenko, S.; Wortmann, G.; Vill, V.; Wortmann, F.J. Fatigue failure testing of human hair: Weibull-analysis for constant strain experiments. *J. Mech. Behav. Biomed. Mater.* **2025**, *163*, 106845. [CrossRef] [PubMed]
6. Evans, T.A.; Park, K. A statistical analysis of hair breakage. II. Repeated grooming experiments. *J. Cosmet. Sci.* **2010**, *61*, 439–455. [PubMed]
7. Kamath, Y.K.; Robbins, C. Hair breakage by combing and brushing—A comment on: TA Evans and K. Park, A statistical analysis of hair breakage. II. Repeated grooming experiments, J. Cosmet. Sci., 41, 439–456 (2010). *J. Cosmet. Sci.* **2011**, *62*, 579–585.
8. Davies, T.; Wortmann, G.; Wortmann, F.J. Cyclic combing of untreated and bleached human hair: Analysis of the time-dependent breakage of hair through recording the formation of fibre fragments. *Int. J. Cosmet. Sci.* **2025**, *48*, 201–210. [CrossRef] [PubMed]
9. Berriche, L.; Grosjacques, C.; Wortmann, G.; Bayersdoerfer, R.; Vill, V.; Wortmann, F.J. Failure testing of humanhair: Model-based analysis of constant strain experiments. In *Proceedings of the 10th International Conference Applied Hair Science, Online, 6–10 June 2022*; TRI: Princeton, NJ, USA, 2022.
10. Lunn, R. Factors Influencing the Fatigue Behaviour of Hair in Strain Controlled Experiments. In *Proceedings of the 11th International Conference on Applied Hair Science, Online, 12–13 June 2024*; TRI: Princeton, NJ, USA, 2024.
11. Wortmann, F.J.; Springob, C.; Sendelbach, G. Investigations of cosmetically treated human hair by differential scanning calorimetry in water. *J. Cosmet. Sci.* **2002**, *53*, 219–228. [PubMed]
12. Wortmann, F.J.; Popescu, C.; Sendelbach, G. Nonisothermal denaturation kinetics of human hair and the effects of oxidation. *Biopolymers* **2006**, *83*, 630–635. [CrossRef] [PubMed]
13. Gasparin, R.M.; Botelho Lourenço, C.; Leonardi, G.R. Porosity and Resistance of Textured Hair: Assessing Chemical and Physical Damage Under Consumer-Relevant Conditions. *Cosmetics* **2025**, *12*, 93. [CrossRef]

14. Câmara, Á.C.; Velasco, M.V.R.; Magnocavallo, N.; de Oliveira Pinto, C.A.S.; Dario, M.F.; da Gama, R.M. Impact of oxidative hair coloring and bleaching on hair fiber properties. *Braz. J. Hair Health* **2026**, *3*, BJHH44. [CrossRef]
15. Wolfram, L.J.; Hall, K.; Hui, I. The mechanism of hair bleaching. *J. Soc. Cosmet. Chem.* **1970**, *21*, 875–900.
16. Robbins, C.R. *Chemical and Physical Behavior of Human Hair*; Springer: New York, NY, USA, 2012.
17. Grosjacques, C.; Babiel, S.; Hodes, J.; Bobier, A.; Cavagnino, A.; Baraibar, M. Carbonylation of hair proteins: A robust biomarker of molecular and structural oxidative damage in hair fibres. *Int. J. Cosmet. Sci.* **2025**, *47*, 604–6025. [CrossRef] [PubMed]
18. Plowman, J.E.; Grosvenor, A.J.; Deb-Choudhury, S.; Bell, F.; Roberts, J.; Clerens, S. Key locations of oxidative damage in human hair keratins after heat and ultraviolet light exposure. *Int. J. Cosmet. Sci.* **2025**, *47*, 1104–1114. [CrossRef] [PubMed]
19. Suzuta, K.; Watanabe, K.; Maeda, T.; Ito, L. Evaluation of cysteic acid in bleached hair using infrared spectroscopy. *J. Fiber Sci. Technol.* **2016**, *72*, 1–8. [CrossRef] [PubMed]
20. Gillece, T.; Senak, L.; McMullen, R.L. Characterization of Bleached Hair: Vibrational Spectroscopy, Thermal Analysis, and Determination of Equivalent Damage Factor. *J. Cosmet. Sci.* **2021**, *72*, 519–546.
21. Kwon, S.; Lee, S.; Jang, J.; Lee, J.B.; Kim, K.S. Quantifying the effects of repeated dyeing: Morphological, mechanical, and chemical changes in human hair fibers. *Heliyon* **2024**, *10*, E37871. [CrossRef] [PubMed]
22. Jachowicz, J. Hair damage and attempts to its repair. *J. Soc. Cosmet. Chem.* **1987**, *38*, 263.
23. Beyak, R.; Meyer, C.F.; Kass, G.S. Elasticity and tensile properties of human hair. I. Single fiber test method. *J. Soc. Cosmet. Chem.* **1969**, *20*, 615–626.
24. Wortmann, F.J.; Quadflieg, J.M.; Wortmann, G. Comparing hair tensile testing in the wet and the dry state: Possibilities and limitations for detecting changes of hair properties due to chemical and physical treatments. *Int. J. Cosmet. Sci.* **2022**, *44*, 421–430. [CrossRef] [PubMed]
25. Wortmann, F.J.; Wortmann, G.; Popescu, C. Linear and nonlinear relations between DSC parameters and elastic moduli for chemically and thermally treated human hair. *J. Therm. Anal. Calorim.* **2020**, *140*, 2171–2178. [CrossRef]
26. Adam, G.; Läuger, P.; Stark, G. *Physikalische Chemie und Biophysik*; Springer: Berlin/Heidelberg, Germany, 2009.
27. Wolfram, L.J. Human hair: A unique physicochemical composite. *J. Am. Acad. Dermatol.* **2003**, *48*, S106–S114. [CrossRef] [PubMed]
28. Wortmann, F.J.; Deutz, H. Characterizing keratins using high-pressure differential scanning calorimetry (HPDSC). *J. Appl. Polym. Sci.* **1993**, *48*, 137–150. [CrossRef]
29. Bathias, C. An engineering point of view about fatigue of polymer matrix composite materials. *Int. J. Fatigue* **2006**, *28*, 1094–1099. [CrossRef]
30. Wortmann, G.; Marsh, J.; Wortmann, F.J. Latent damage in Keratin fibres. In *Proceedings of the 20th International Hair Science Symposium—HairS'17, Dresden, Germany, 6–8 September 2017*; DWI-Leibniz Institut Aachen: Dresden, Germany, 2017.
31. Dharan, C.K.H. Fatigue failure mechanisms in a unidirectionally reinforced composite materials. In *Fatigue of Composite Materials*; Hancock, J.R., Ed.; ASTM International: West Conshohocken, PA, USA, 1975; Volume STP569-EB.
32. Beaumont, P.W.R. The failure of fibre composites: An overview. *J. Strain Anal. Eng. Des.* **1989**, *24*, 189–205. [CrossRef]
33. Qu, W.; Guo, X.; Xu, G.; Zou, S.; Wu, Y.; Hu, C.; Chang, K.; Wang, J. Improving the mechanical properties of damaged hair using low-molecular weight hyaluronate. *Molecules* **2022**, *27*, 7701. [CrossRef] [PubMed]
34. Carvalho, J.P.; Costa, A.F.; Gonçalves, F.; da Costa, A.; Ribeiro, A.; Martins, M.; Tinoco, A.; Cavaco-Paulo, A. Interaction of polypeptides with hair in different reducing environments. *Int. J. Biol. Macromol.* **2026**, *340*, 150107. [CrossRef] [PubMed]
35. Lamb, G.E.R.; Weigmann, H.D. An Examination of Flaw-Dependent Theories of Fiber Strength. *Text. Res. J.* **1977**, *47*, 66–72. [CrossRef]
36. Berriche, L.; Sirenko, S.; Welzel, J.; Bayersdoerfer, R.; Vill, V.; Wortmann, G.; Wortmann, F.J. When hair strength and resilience diverge. In Proceedings of the Cosmetotest, International Symposium, 3rd Edition, Lyon, France, 29–30 May 2024.
37. Tate, M.L.; Kamath, Y.; Ruetsch, S.B.; Weigmann, H.D. Quantification and prevention of hair damage. *J. Soc. Cosmet. Chem.* **1993**, *44*, 347–371.
38. Marsh, J.M.; Brown, M.A.; Felts, T.J.; Hutton, H.D.; Vatter, M.L.; Whitaker, S.; Henry, I.D. Gel network shampoo formulation and hair health benefits. *Int. J. Cosmet. Sci.* **2017**, *39*, 543–549. [CrossRef] [PubMed]
39. Lourenço, C.B.; Gasparin, R.M.; Thomaz, F.M.; da Silva, G.C.; Martin, A.A.; Paiva-Santos, A.C.; Mazzola, P.G. Impact of hair damage on the penetration profile of coconut, avocado, and argan oils into Caucasian hair fibers. *Cosmetics* **2024**, *11*, 64. [CrossRef]
40. Zhang, D.; Baghdadli, N.; Greaves, A.J. Reinforcing chemically treated human hair with citric acid. *Int. J. Cosmet. Sci.* **2025**, *47*, 411–423. [CrossRef] [PubMed]
41. Ngoepe, M.N.; Cloete, E.; van den Berg, C.; Khumalo, N.P. The evolving mechanical response of curly hair fibres subject to fatigue testing. *J. Mech. Behav. Biomed. Mater.* **2021**, *118*, 104394. [CrossRef] [PubMed]
42. Modarres, M.; Kaminskiy, M.P.; Krivtsov, V. *Reliability Engineering and Risk Analysis: A Practical Guide*, 3rd ed.; CRC Press: Boca Raton, FL, USA, 2017.

43. Welzel, J.; Sirenko, S.; Berriche, L.; Vill, V.; Wortmann, G.; Wortmann, F.J. Fracture morphology of human hair after strain fatigue testing. In *Proceedings of the 11th International Conference on Applied Hair Science, Online, 12–3 June 2024*; TRI: Princeton, NJ, USA, 2024.
44. Samoylenko, D.; Whelan, L.S.; Yale, A.; Marty, G.; Santoprete, R.; Polacchi, L.; Taylor, D. Hair anisotropy and damage: Understanding hair cracking and fracture via the moving loop test. *J. Mech. Behav. Biomed. Mater.* **2025**, *175*, 107320. [CrossRef] [PubMed]
45. Kamath, Y.K.; Weigmann, H.D. Fractography of human hair. *J. Appl. Polym. Sci.* **1982**, *27*, 3809–3833. [CrossRef]
46. Kamath, Y.K.; Hornby, S.B.; Weigmann, H.D. Mechanical and fractographic behavior of negroid hair. *J. Soc. Cosmet. Chem.* **1984**, *35*, 21–43.
47. Swift, J.A. The mechanics of fracture of human hair. *Int. J. Cosmet. Sci.* **1999**, *21*, 227–239. [CrossRef] [PubMed]
48. Yang, W.; Yu, Y.; Ritchie, R.O.; Meyers, M.A. On the strength of hair across species. *Matter* **2020**, *2*, 136–149. [CrossRef]
49. Taylor, D.; Barton, E.; Duffy, I.; Enea-Casse, R.; Marty, G.; Teeling, R.; Santoprete, R. The biomechanics of splitting hairs. *Interface Focus* **2024**, *14*, 20230063. [CrossRef] [PubMed]
50. Lai, C.D.; Murthy, D.; Xie, M. Weibull Distributions and Their Applications. In *Springer Handbook of Engineering Statistics*; Springer Handbooks; Pham, H., Ed.; Springer: London, UK, 2006; Chapter 3.


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