Determination of the Feel of Hair after Cosmetic Treatment – Sensory and Objective Test Methods

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Abstract

This work was performed in the period from 2002 to 2006. The feel of natural and damaged hair was studied after treatment with classical conditioning formulations.
A cetrimonium chloride/cetyl alcohol conditioner and shampoos based on sodium lauryl ether sulfate and polyquaternium-10 or dimethiconol, respectively, were used. In addition to a broad-based round robin test to determine the hair feel in 12 test centers, suppleness measurements, atomic force microscopy and scanning electron microscopy, combing force measurements and shine measurements were performed.
The cetrimonium chloride conditioner and the silicone shampoo produced the best hair feel. There was a correlation between hair feel and the suppleness measurement. The coating of the hair surface after shampooing with polyquaternium-10 and dimethiconol could be seen with atomic force microscopy and scanning electron microscopy. There was no correlation of the hair feel with shine or ease of combing.

Keywords: Human hair, feel, handle, conditioner, conditioning shampoo, combing, measurement, suppleness, microscopy, round robin test

INTRODUCTION

The feel or haptic properties of hair are, like the shine and smell of hair, so-called subjective perceptions that play an increasingly important role in the quality of cosmetic hair formulations. Consciously or subconsciously, the feel of hair determines whether or not we like cosmetic formulations and is therefore of central importance. So far this interaction between the hands and fingers of both hands with the hair could be detected reliably only in in-use tests and has been difficult to correlate with test methods. Therefore this study was designed to obtain a subjective assessment of the feel of normal and damaged hair and to correlate this with the results of objective test methods. In round robin tests in test centers at various companies trained panelists determined the feel of damaged and undamaged hair after different cosmetic pretreatments. Besides the assessment of the hair feel according to a scoring scale, the semantics of the panelists for positive and negative assessment of the feel was determined. The assessment of feel was then correlated with the results of objective test methods like wet and dry combing, gloss, surface analyses of the hair with electron microscopy and atomic force microscopy, as well as the suppleness of hair.

EXPERIMENTAL

Hair

Used for the studies were

a) natural hair tresses from Kerling (Backnang, Germany), (color 7/0) 20 cm long, ca. 4 cm wide (u – for undamaged) and
b) hair tresses identical to a) and then damaged (d – for damaged).

The hair was damaged by Kerling as follows:

- Bleached 1 x 4 h 3% H_2O_2
- Permed 1 x 3 min (5.5% thioglycolate; pH 8.8)
- Bromate fixation 1 x 10 min (pH 4).

Pretreatments

The following cosmetic pretreatments were used for the studies on natural and damaged hair:

1. Sodium lauryl ether sulfate (SLES) (blank)

2. Shampoo formulation containing SLES and polyquaternium-10 (SLES + PQ10)
3. Shampoo formulation containing SLES and the silicone emulsion dimethiconol/sodium dodecylbenzenesulfonate (SLES + silicone)
4. Conditioner containing cetrimonium chloride and cetyl alcohol (CTAC).

Two series of tests with undamaged (u) and damaged (d) hair were performed. Always run with each test series for comparison was the blank of the other series.

Test formulations

1. Sodium lauryl ether sulfate SLES (blank)

Sodium lauryl ether sulfate (28%)	35.7%
Water	ad 100%
Citric acid	pH 5.5 to 6.0

2. Shampoo formulation with polyquaternium-10 (SLES + PQ 10)

Sodium lauryl ether sulfate (28%)	37.5%
Polyquaternium-10 (Amerchol)	0.3%
Water	ad 100%
Citric acid	pH 5.5 to 6.0

3. Shampoo formulation with dimethiconol/sodium dodecylbenzenesulfonate; 50% silicone (SLES + silicone)

Sodium lauryl ether sulfate (28%)	37.5%
Dimethiconol/sodium dodecylbenzenesulfonate (General Electric)	4.0%
Water	ad 100%
Citric acid	pH 5.5 to 6.0

4. Conditioner with cetrimonium chloride (30%) and cetyl alcohol (CTAC)

Cetrimonium chloride	1.7%
Cetyl alcohol	3.0%
Water	ad 100%
Citric acid pH 4.5	

Description of hair treatment (DWI)

The tresses were precleansed by rinsing for 15 min with water (ca. 35°C) and then wetting with sodium lauryl ether sulfate (0.5 ml for each g hair). After massaging in for 5 min the tresses were rinsed for 3 min with water (ca. 35°C) and dried under a hood dryer for 60 min at 50°C while combing repeatedly.
For treatment with formulations 1 to 4, the tresses were rinsed for 15 min with water (ca. 35°C) and then treated with one of the test formulations 1 to 4 by wetting with the respective test formulation (0.5 ml for each g hair). After massaging in for 5 min the tresses were rinsed for 3 min with water (ca. 35°C) and dried under a hood dryer for 60 min at 50°C while combing repeatedly.

Round robin test procedure

The round robin test for assessment of the feel was performed from 2002 to 2003. A total of 12 test centers participated with a total of 69 panelists. Each hair tress was tested just once and the test performed double blind. The feel was assessed »blind«, *i.e.* with the eyes closed. Two test series were performed, one with undamaged hair and one with damaged hair. Because the blank of one test series was always also run with the other test series, each test series consisted of 5 hair samples.
The test series on undamaged (u) hair tresses consisted of the following: SLES blank u, SLES + PQ10 u, SLES + silicone u, CTAC u, and SLES blank d.
The test series on damaged (d) hair tresses consisted of the following: SLES blank d, SLES + PQ10 d, SLES + silicone d, CTAC d, and SLES blank u.
The assessment was performed as follows: The tresses with the best feel always received 4 points, those with the worst feel 0 points. The remaining tresses received 1, 2 or 3 points. Each number of points was assigned once only, *i.e.* every tress received a different number of points. The panelists were also asked which adjectives they associated with good or poor feel.

Suppleness measurements (DWI)

In a self-constructed instrument a previously damaged hair tress (weight ca. 2 g, length 19 cm) was subjected to continuous three-point bending. This was done by sliding the tool along the hair tress from top to bottom. The force was measured versus the resistance of the tress at a tensile rate of 100 cm/min. Five dried tresses (22°C, 55% rel. humidity) were measured for each product, with repeated measurements, a total of 12 x. The difference between the values before and after treatment was analyzed. A reduction in the suppleness work due to the treatment corresponded to an improvement in suppleness.

Atomic force microscopy measurements (BASF SE)

The atomic force microscopy measurements were performed with a Digital Instruments Nanoscope Dimension 3000 SPM (Veeco, Mannheim, Germany) in the tapping mode [1,2]. The surface topography of the hair and hard-soft contrasts were measured.

Scanning electron microscopy measurements (Wella)

The scanning electron microscopy images were generated with a Zeiss Ultra 55 microscope (Zeiss, Leipzig, Germany) at a working distance of 7 mm, a tilt angle of 45° and a magnification of 2000. The hair samples were coated in a sputter device with a thin, 10 nm layer of platinum before analysis. The cuticle edges and deposits could be visualized very well.

Combing force measurements (Wella)

The combing force measurements were performed with an automated robot (moving single comb model, self-constructed, Darmstadt, Germany) on wet and dry hair tresses (weight ca. 2 g, length 16 cm). The comb was moved through the whole tress at a rate of 10 s per run and the combing force generated recorded in N by a force dynamometer. As the relevant data, the mean force over the length of the hair was analyzed for wet hair and the maximum force at the tips of the tresses for dry hair.

Gloss measurements [3]

Thirty hairs were taken randomly from the treated hair tresses and glued in groups of 10 on aluminum frames for the gloss measurement. Each hair was measured at five equidistant points. The measurement was performed with a green light laser (532 nm) with an incident angle of 40° and simultaneous detection of the re-

flected light in an angle range of 50 - 170° (Laser Multiangle Goniophotometer, Fiantec, Hamburg, Germany). Irradiation was in the so-called root-tip direction. The fractions of specularly (S), diffusely (Ds) and internally (Di) reflected light were determined by fitting the three normal distributions to the reflection profile. From these the gloss index (GL) was calculated as the amount of specular gloss:

$$GL = S/(D + S) \text{ where } D = DS + Di$$

GL describes the contrast gloss as a measure of the hair shine. This approach corresponds to earlier definitions and subjective descriptions referring to the »brilliance of reflected light« and the »brilliance of gloss reflections«. The fractions of diffuse reflection and internal reflection were calculated analogously as the measure of silky shine.

All goniophotometric spectra and data sets were checked for their physical plausibility and obvious outliers eliminated.

RESULTS

In-use and round robin tests

In the »undamaged hair u« test series the tresses treated with conditioner (CTAC u) clearly differed from those subjected to all other treatments ***(Figure 1)***. At the 92% level (p = 0.08) and the 95% level (p = 0.05) bleached hair (blank d) differed significantly from all other hair tresses including undamaged hair (blank u).

The distribution of scores is clearly positive (score 4) for the CTAC treatment and clearly negative (score 0) for the blank run with damaged hair. The assessment of all other treatments was highly variable ***(Figure 2)***.

In the »damaged hair d« test series there were strong differences among the individual treatments overall. As with the »undamaged hair u« test series, the hair tresses after CTAC treatment received the most positive assessment ***(Figure 3)***. The hair tresses treated with conditioner (CTAC d) were assessed even better than the undamaged hair used as the blank (blank u). Shampooing with the silicone-containing formulation (SLES + silicone d) resulted in an assessment that was nearly the same as the blank value for undamaged hair. Shampooing with PQ 10

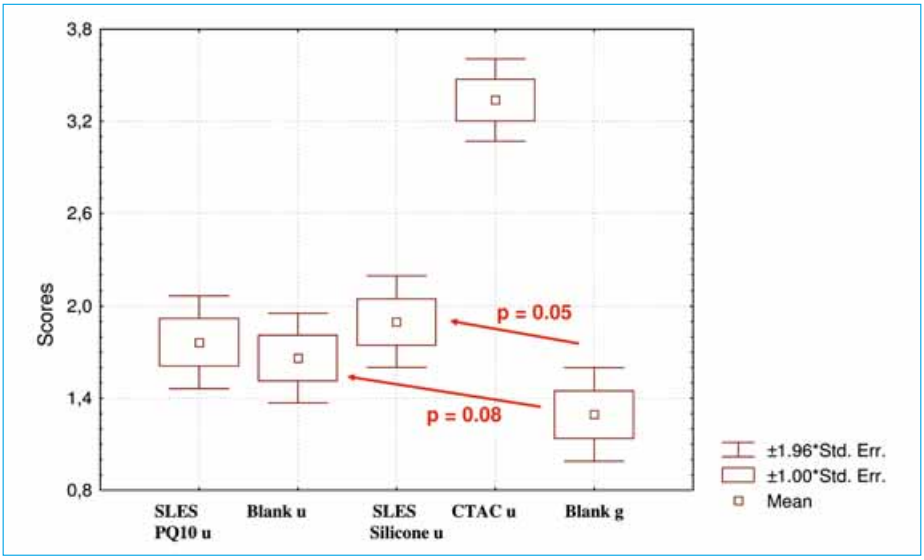

Figure 1: *Statistical analysis of assessment of the feel of undamaged hair.*

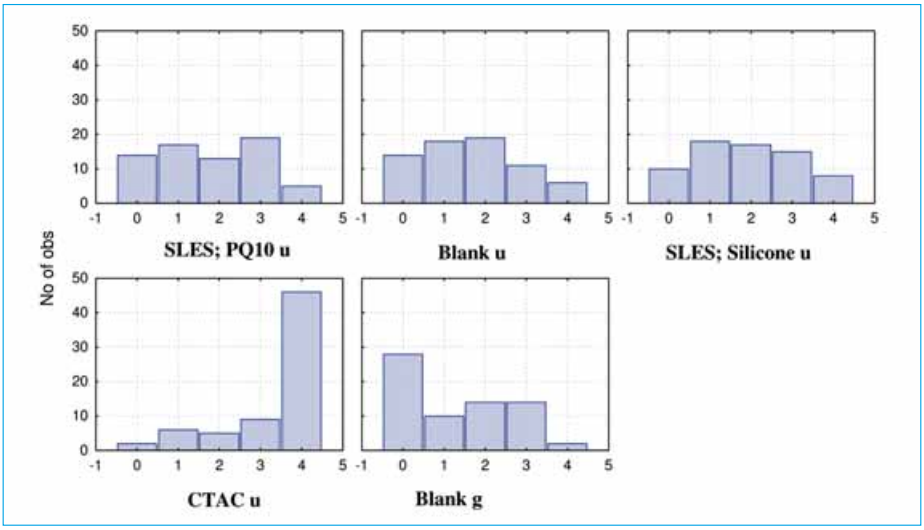

Figure 2: *Statistical analysis of assessment of the feel of undamaged hair – distribution of scores.*

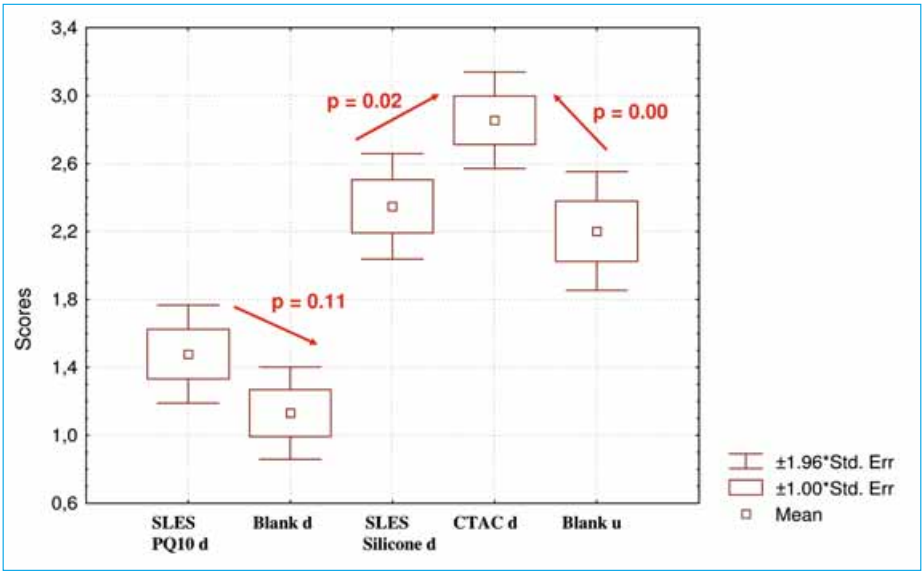

Figure 3: *Statistical analysis of assessment of the feel of damaged hair.*

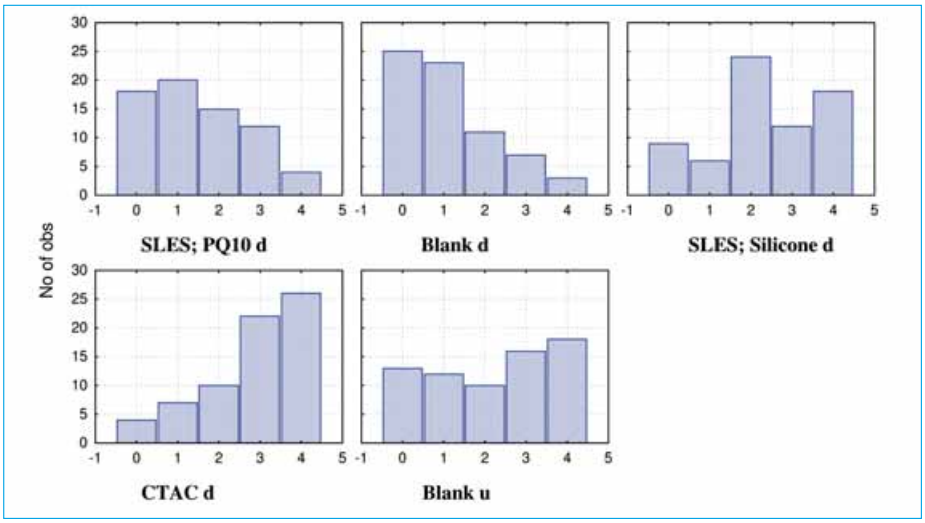

Figure 4: *Statistical analysis of assessment of the feel of damaged hair – distribution of scores.*

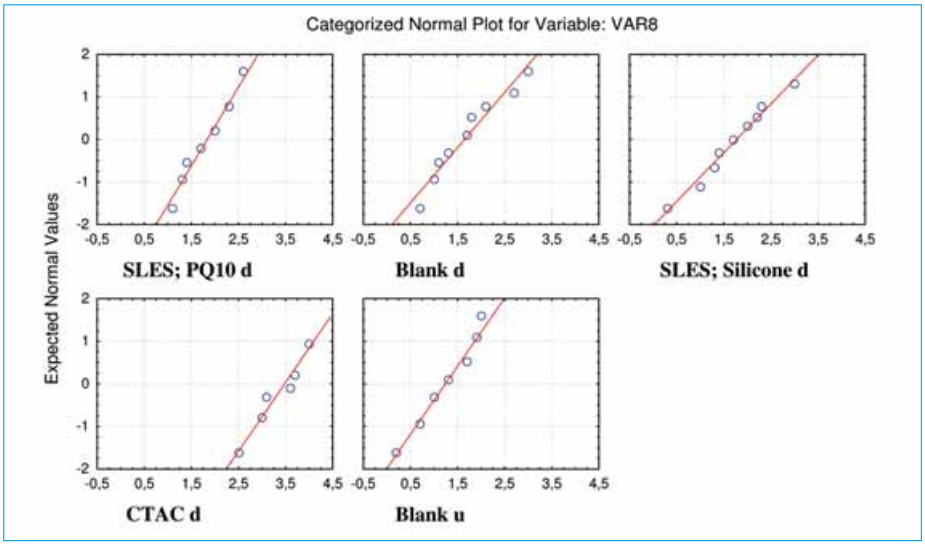

Figure 5: *Statistical analysis of company results in the probability network.*

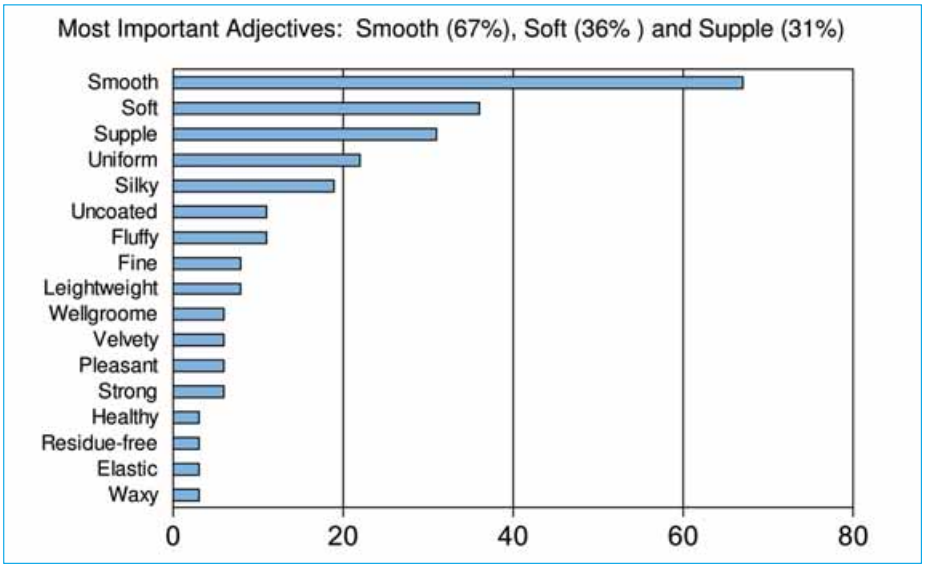

Figure 6: *Semantic analysis of the understanding of »good feel«.*

(SLES + PQ10 d) was evaluated only slightly better than the blank for damaged hair (blank d).

Not only the scores for the treatments with the two shampoos containing PQ 10 (SLES + PQ10 d) and silicone (SLES + silicone d), respectively, but also those for the blank with undamaged hair (blank u) showed a fairly broad distribution ***(Figure 4)***. In contrast, the distribution of scores after CTAC treatment was clearly positive and for the blank with damaged hair clearly negative.

Statistical analysis of company results in the probability network revealed no differences among companies. Plots of the results of all companies (data points) versus the expected normal values gave straight lines, *i.e.* the data field over all companies was homogeneous. The slope of the lines corresponds to the standard deviation.

The adjectives most often used for a »good feel of the hair« were: smooth (67%), soft (36%) and supple (31%), uniform (22%) and silky (19%) ***(Figure 6)***.

The words used most often to describe a »poor feel of the hair« were: rough (76%) and dull (50%) ***(Figure 7)***.

Objective test methods

Suppleness measurements

The suppleness measurements were performed with the damaged hair group (Group d). The test results correlated with the feel assessments in the round robin test ***(Figure 3)***. The least suppleness work was required by the hair tresses treated with cetrimonium chloride conditioner followed by the tresses treated with the shampoos in the order »SLES + silicone« and »SLES + PQ10«. The »blank« hair tress showed an increase in suppleness work after treatment with SLES without conditioning additives ***(Figure 9)***.

Atomic force microscopy

Surface analyses were performed using atomic force microscopy and electron microscopy. These analyses focused on the shampoo treatment of damaged hair with »SLES + PQ 10« and »SLES + silicone«. A significant difference in the feel had been found in the round robin test between these two treatments of the same shampoo formulation type ***(Figure 3)***. Therefore

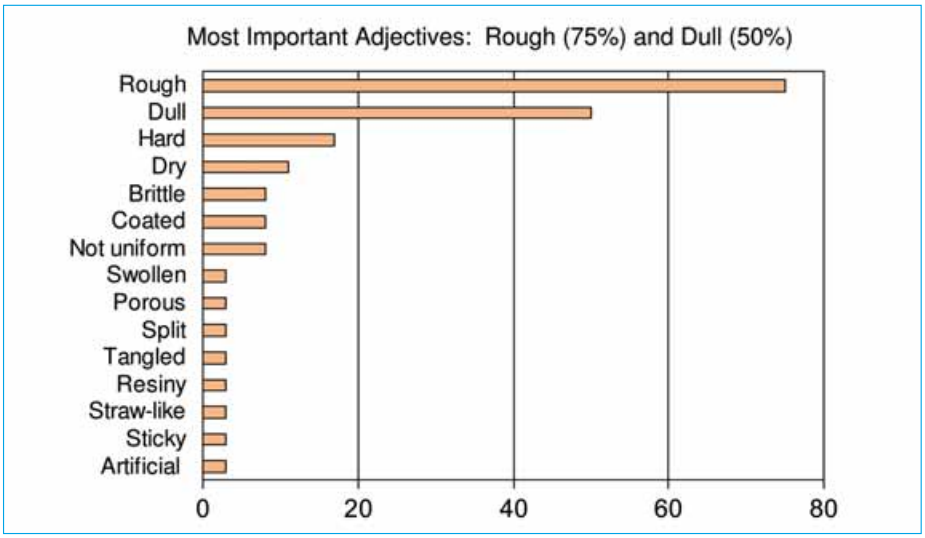

Figure 7: *Semantic analysis of the understanding of »poor feel«.*

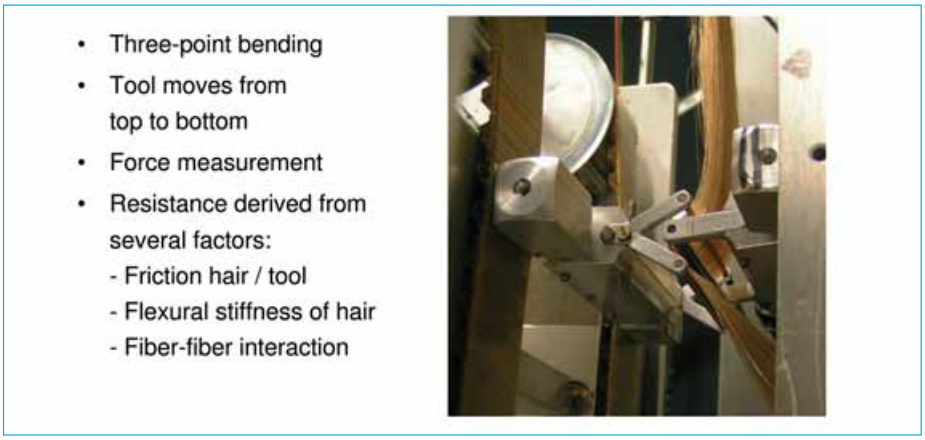

Figure 8: *Experimental setup for suppleness measurements.*

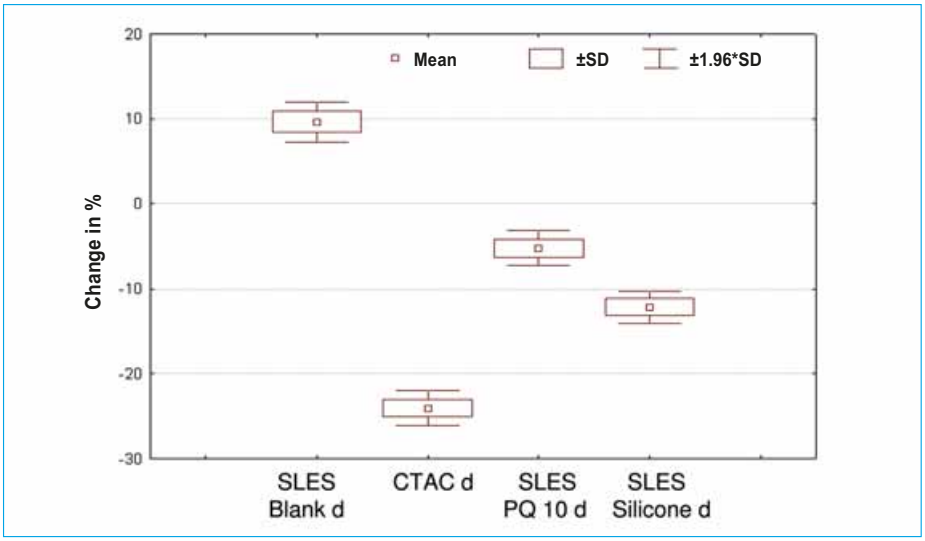

Figure 9: *Suppleness measurements on hair tresses.*

it was especially interesting to see here whether this difference could be measured. For comparison, the blanks for damage and undamaged hair were also measured.

Figure 10 presents an overview of all measured hair tresses. Neither the blanks nor the treatment with »SLES + PQ10« is distinguishable. The surfaces look smooth and untreated. The amplitude image of damaged hair which had subsequently been exposed to the »SLES + silicone« treatment clearly shows some coating and irregular lumpy deposits on the scale faces.

Figure 11 shows the step heights and shapes for hair after different treatments. Undamaged hair had the highest step heights. Bleaching and perming significantly reduced step heights. Neither the height nor shape of the steps of the »SLES + PQ10« treatment changed. After treatment with the »SLES + silicone« shampoo a significant »rounding« of the steps could be seen, which is in agreement with complete coating.

Shown in ***Figure 12*** are unbleached and bleached hair at high resolution up to 0.4 µm. Both hair types have a smooth surface with negligible coating. The cuticle layers are clearly recognizable. At high resolution in the phase mode distinct »holes« can be seen in the surface structure of the bleached, damaged hair.

The AFM amplitude and phase images (SLES + PQ10 d) of damaged hair that had subsequently been exposed to the »SLES + PQ10« treatment clearly show random deposits on the scale faces which are especially heavy at the scale edges ***(Figure 13)***.

The AFM amplitude and phase images clearly show heavy and more uniform coating of the scale faces and edges of damaged hair which had subsequently been exposed to the »SLES + silicone« treatment ***(Figure 14)***.

Scanning electron microscopy

The scanning electron microscopy studies gave results identical to those of the atomic force microscopy measurements ***(Figure 15)***. No deposits could be seen on untreated hair. Hair treated with the »SLES + PQ 10« shampoo showed only slight deposits. After treatment with the »SLES +

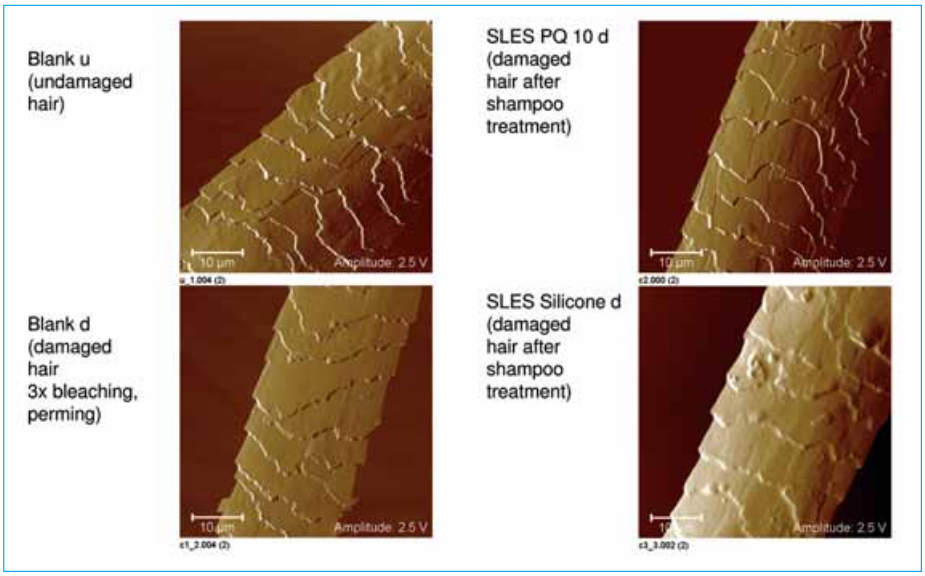

Figure 10: *Atomic force microscopy of hair after shampooing, amplitude images, 10 µm.*

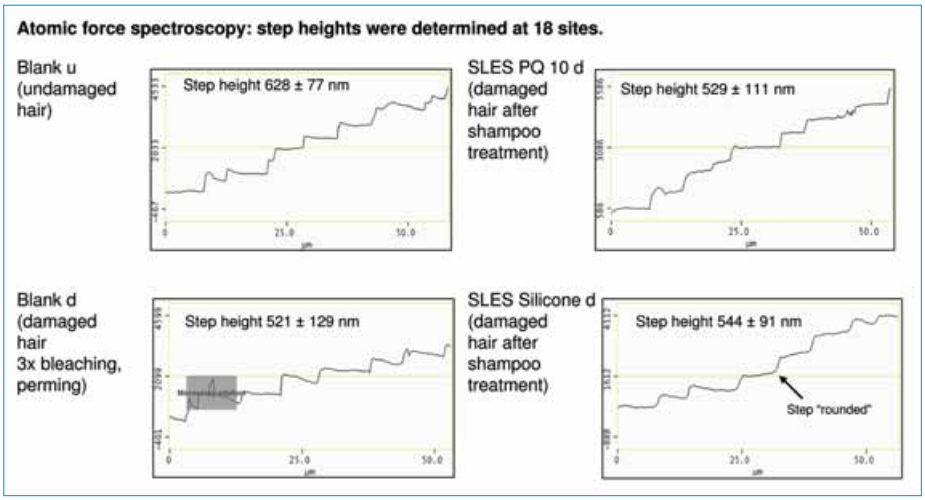

Figure 11: *Line profiles of the hair studied – atomic force microscopy.*

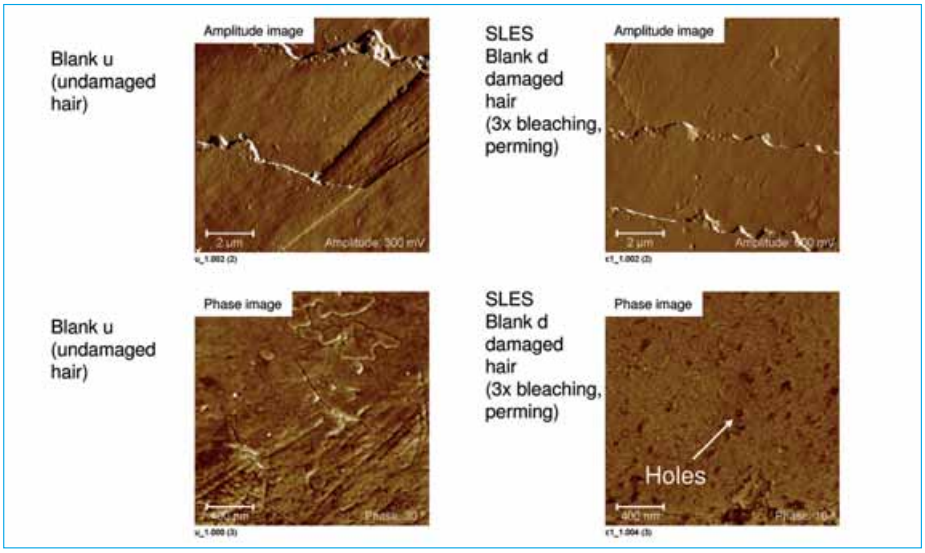

Figure 12: *Atomic force microscopy of unbleached and bleached hair (blank), amplitude and phase images, 2.0 and 0.4 µm.*

silicone« shampoo the hair was completely coated.

Gloss measurements

Several laser goniophotometric measurements on both hair groups u and d revealed few significant differences within the test groups. Analysis of the specular gloss showed the damaged tresses to be a homogeneous group of low gloss, as would be expected. In the group of unbleached tresses the gloss is clearly higher. However, in this group the hair treated with »SLES + silicone« was conspicuous and showed gloss that was significantly lower than that of the untreated blank ($p = 0.02$).

The diffuse reflectance showed the unbleached tresses (Group u) to be a homogeneous group. No influence of the products used could be detected.

The group of damaged tresses (Group d) showed a higher diffuse reflectance overall, as would be expected. Within this group the »SLES + silicone« treatment led to somewhat more and the CTAC treatment to clearly less diffuse reflectance.

The internal reflectance was similar for the unbleached tresses and somewhat higher for the bleached. The hair was apparently more permeable to light due to bleaching. The CTAC treatment gave the highest internal reflectance overall. The SLES + silicone-treatment was striking in that it showed an especially low reflectance.

Analysis of the peak width of the specular reflectance of damaged tresses revealed a narrower width than for unbleached hair. The »SLES + silicone« treatment resulted in a significantly smaller peak width within the unbleached group (u). Less specular reflectance is apparently associated with a smaller peak width.

Wet and dry combing

For undamaged hair (Group u) no differences in wet and dry combing were detectable.

The worst wet combing in test series d with damaged hair was determined for the »SLES + silicone« tresses. Rinsing with CTAC also gave combing values that were not as good as would be expected from the assessment of feel.

The combing force values for dry combing

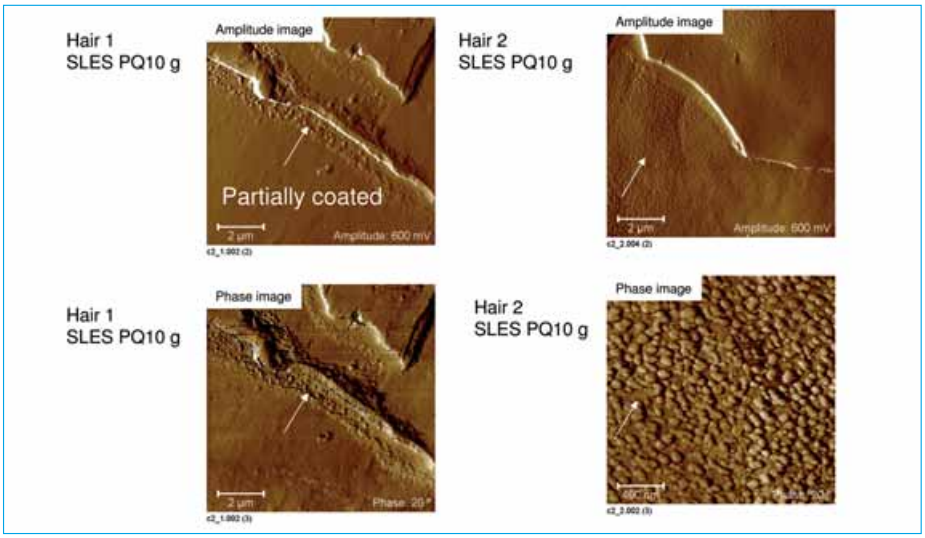

Figure 13: *Atomic force microscopy of bleached hair after shampooing (shampoo containing PQ10); amplitude and phase images, 2.0 and 0.4 µm).*

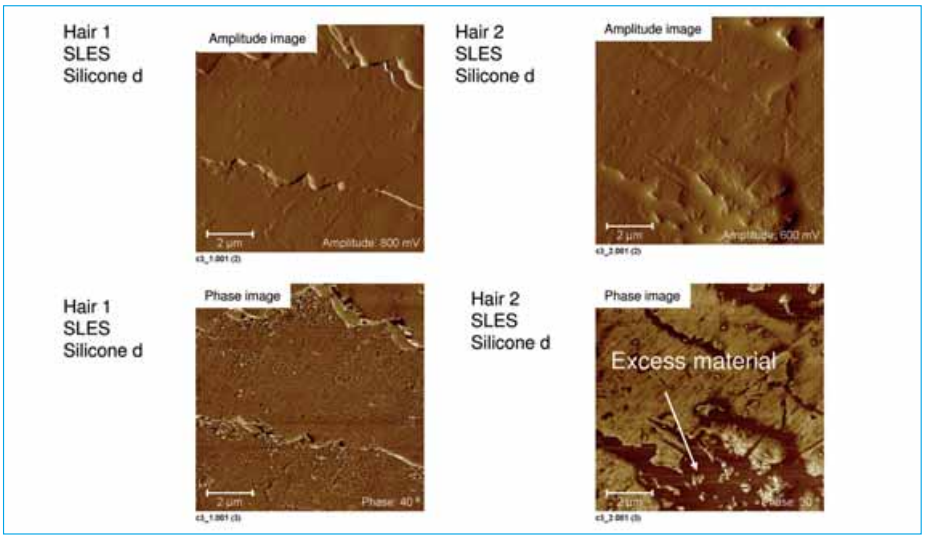

Figure 14: *Atomic force microscopy of bleached hair after shampooing (shampoo containing silicone); amplitude and phase images, 2.0 µm.*

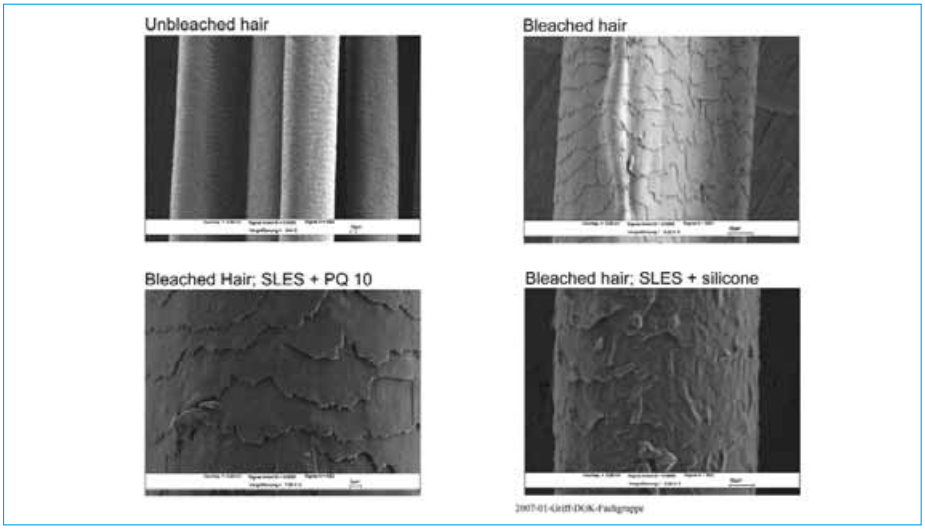

Figure 15: *Scanning electron microscopy of unbleached/bleached hair (blank) and bleached hair after shampooing (shampoo containing PQ10 or shampoo containing silicone).*

of damaged hair (Group d) decreased in the following order:

SLES+PQ10>blank>SLES+silicone>CTAC. However, the differences were slight and not significant.

DISCUSSION

The feel of undamaged hair after treatment with cetrimonium chloride conditioner was perceived as clearly improved in the round robin test. After shampooing with an »SLES + PQ10« shampoo and an »SLES + silicone« shampoo hardly any differences from damaged hair could be felt. The distribution of scores was especially wide here meaning that the panelists assessed the hair very differently ***(Figure 2)***. In contrast, every treatment led to a clear improvement in the feel of bleached hair in the round robin test ***(Figure 3)***. Here too the cetrimonium chloride conditioner improved the feel most. The »SLES + silicone« shampoo likewise resulted in a clear improvement in the feel to a level equal to untreated hair. The »SLES + PQ10« shampoo treatment, however, produced only a very slight but significant improvement in hair feel.

The semantics of the understanding of good and poor feel were surprisingly uniform. Nearly 70% of all panelists associated »smooth« with good feel and over 70% »rough« with poor feel of the hair.

The difference in the feel of hair after treatment of the damaged hair tips could be clearly demonstrated by the suppleness measurements ***(Figures 3 and 9)***. Treatment with CTAC resulted in the largest decrease in suppleness work followed by treatment with »SLES + silicone« and »SLES + PQ10«. These results show that the subjective perception of the hair feel by the panelists can be followed with the suppleness method. At the same time these tests confirmed that the suppleness method is suitable for assessing the feel of hair. It must still be determined whether this method is actually suitable in all situations for evaluation of the subjective feel of hair and further testing and comparisons with in-use results are definitely required to substantiate this.

No correlation between the shine and feel of hair could be found. On the contrary, the SLES/silicone treatment, which resulted

in the best feel and complete coating of the hair surface, gave a poorer shine. Although complete coating of the cuticle layer of the hair with the silicone treatment improved the feel of the hair, it did not improve the shine.
There was also no correlation of dry combing of hair and differences in the feel of hair. However, this result is probably due to a limited stock of hair tresses being measured. In addition, significant differences probably would have been obtained, if hair tresses were standardized as untreated blanks.
The results of the feel assessment in the in-use test correlated with the results of the surface analysis methods atomic force microscopy and scanning electron microscopy. Whereas untreated and bleached hair showed – apart from detectable damage like holes and traces of deposits a smooth – an uncoated surface, the partial coating after »SLES + PQ10« treatment and complete coating after »SLES + silicone« treatment correlated with the results for the subjective feel of hair. It should, however, be noted that such heavy silicone deposits are abnormal. We attribute them to use of extremely damaged hair for the tests performed here and treatment with conditioners that was likewise more intensive than is normally the case. Experience has shown that after standard damage to hair and normal shampooing considerably less silicone is deposited.

CONCLUSIONS

After subjecting natural hair to different treatments in a round robin test in 12 test centers, some differences and in the case of damaged hair clear differences could be detected in the feel of hair. Used for the treatments were a cetrimonium chloride/cetyl alcohol conditioner and shampoos containing sodium lauryl ether sulfate and polyquarternium-10 or dimethiconol. The best hair feel was achieved with the conditioner containing cetrimonium chloride and the shampoo containing silicone. There was no improvement in the feel of natural hair with the shampoo containing polyquaternium-10 and only a slight improvement for damaged hair.
Also sought were objective methods to enable a correlation with the subjectively perceived feel of hair. For this purpose atomic force microscopy and scanning electron microscopy, combing measurements, and gloss measurements were performed. There was a significant correlation of the hair feel with the suppleness measurement. The cetrimonium chloride conditioner had the lowest suppleness work followed by the shampoo treatments (SLES + silicone and SLES + polyquaternium-10). Coating of the hair surface after shampooing with polyquaternium-10 and dimethiconol was visible and distinguishable with atomic force microscopy and scanning electron microscopy. No correlation of the hair feel with the shine or combability of hair could be determined.

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