Hair breakage—How to measure and counteract

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Synopsis

A system to determine the efficacy of hair treatments in terms of anti-breakage and split end prevention was developed which involves the repeated combing of hair strands. The device allows ten hair strands to be combed simultaneously. First, the influences of chemical hair treatments like bleaching on hair breakage were examined. In a next step, the protective effects of benchmark products from the market were studied. Since nearly all commercial products with anti-breakage claims contain silicones combined with cationic polymers, alternative actives were searched. In a test series with different waxes in shampoo formulations with a variable number of parameters, the particle size was found to be the factor with the strongest influence on the amount of wax deposited on the shampooed hair. Therefore, a targeted development was started, resulting in a combination of several ethers dispersed in sodium laureth sulfate. Excellent conditioning, anti-breakage and split ends protection properties of the compound were found, showing also a dosage dependency. The latter could be explained by analyzing the amounts of waxes applied on treated hair. In these experiments, a dependency on the concentration in the shampoo was found.

INTRODUCTION

Anti-hair breakage is one of the most popular claims made for modern shampoos and conditioners. Nearly any brand offers anti-breakage or anti-damage shampoos and conditioners, or even some other products like masks.

Literature describes several methods to test anti-breakage properties. The most laborious way reported was an in-vivo determination of broken hair fibers gathered from 15 panelists (1). Hair breakage was also tested, applying a tensile strength protocol (2), which may be questioned since the force to pull out hair from the bulb is significantly smaller than the break forces (3) and also depends on the phase of the growth cycle of each hair. Another means to determine the anti-breakage properties of cosmetic products is a repetitive combing of treated hair strands (4–6).

We developed a set-up for such a testing protocol, allowing for an automated parallel combing of up to 10 hair strands. Broken hair fibers are collected in separated drawers for each strand. Besides hair breakage, the generation of split ends can also be observed and quantified.

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We tested the influence of hair status on the hair breakage by combing virgin and chemically damaged hair (bleached and permanently waved). Several anti-breakage shampoos from the market containing silicones were examined and found to be efficient in terms of hair breakage protection.

As silicones are often regarded less sustainable and biodegradable compared to oleochemical ingredients and the usage of silicones in shampoos is very much limited by patents, silicon-free alternatives for anti-breakage efficacy are of considerable interest. Therefore, several waxes were tested for their deposition on the hair from shampoo formulations. This approach is straight forward also because waxes are often used for shampoos to give them a nice milky or even pearlescent appearance. Several parameters of wax dispersions were tested for their influence on the wax amounts deposited on hair.

EXPERIMENTAL

HAIR STRANDS TREATMENT

All experiments are performed using dark brown European hair (from International Hair Importers, New York). If not stated otherwise the strands were bleached applying 5% of hydrogen peroxide (pH 9.4) for 15 min followed by intensive rinsing. Treatment of the hair with shampoo formulations was performed as follows: 0.25 g formulation per 1 g hair was applied on the hair using dyeing brushes for hair dressers. After 5 min incubation time the strands were rinsed with warm tap water (38°C) using a special rinsing device ensuring a water flow of 1 l per minute and strand. The whole procedure was repeated once. For wet combability, the hair strands were taken directly after the second rinsing, for all other methods the strands were dried. For conditioners a similar procedure was applied, but with a single application and only 3 min incubation time.

Perming of hair strands was done applying first a perming solution (7% thioglycolic acid, pH 9.5) for 30 minutes, followed by extensive rinsing and the application of fixation (2.2% hydrogen peroxide, pH 4) for 30 minutes. Subsequent to extensive rinsing the hair strands were dried with hot air for 30 minutes.

The persulfate bleaching was performed by a 30 minute treatment of the hair strands with a bleaching solution (6% hydrogen peroxide, 15% ammonium peroxide sulfate, pH 9.4) followed by extensive rinsing and 60 minutes of hot air drying.

HAIR BREAKAGE DETERMINATION

A customized system was developed, allowing the parallel combing of up to ten hair strands (see Figure 1). Combing was performed by two combs per strand mounted on a motor-driven axis. The broken hair fibers were collected in drawers made from stainless steel, one for every hair strand. The device was set in a box allowing the control of temperature and relative humidity. Standard conditions were 40% relative humidity and 30°C (to avoid an active cooling system).

The amount of broken hair was determined gravimetrically after sorting out fibers longer than 9 cm (which are most probably not broken but pulled out of the glued part of the
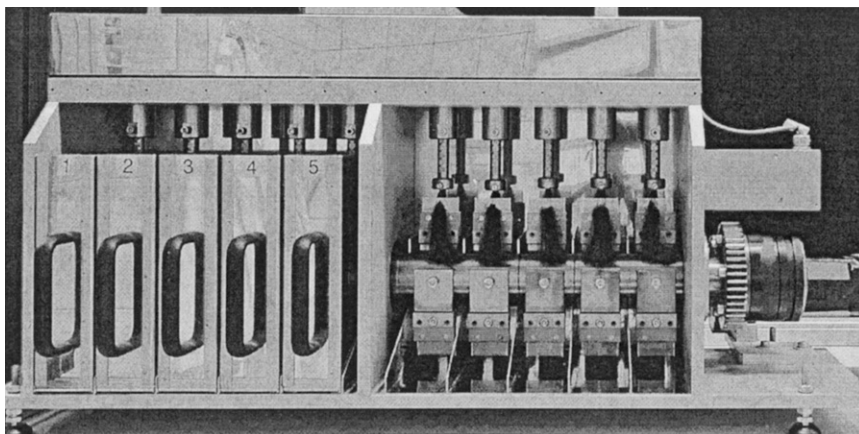
Figure 1. Set-up for the parallel combing of 10 hair strands to determine hair breakage.

strand). The mean and standard deviations of all ten strands were calculated to determine the hair breakage of strands treated with the same formulation.

Differences in the means were evaluated statistically calculating a heteroscedastic Student's t-test. The difference is regarded significant for p-values <0.05.

SPLIT ENDS EVALUATION

To determine the amount of split ends, 1.5 cm of the tip section of each strand was cut. The fibers with split ends were then separated from the intact fibers by using a special device. It consists of a sieve with a pore size of 200 μm equipped with a cylinder to avoid loss of hair fibers. For the sieving procedure the sieve was rotated and a counter air flow was applied to bring the fibers in a vertical position. Within 15 min all intact fibers had passed the sieve, whereas the fibers with split ends stuck in the pores. The amount of split ends was determined weighing the sticking fibers and dividing their weight by the weight of all fibers before the sorting procedure. The mean and the standard deviations of all ten strands were calculated to determine the amount of split ends treated with the same formulation.

WET AND DRY COMBABILITY

Wet and dry combing performances were determined using a robotic system, combing 10 bleached strands per formulation. The combing work was determined by integrating the force versus distance curve. After determining the baseline values of the strands, they were treated according to the protocol given above. The residual combing work was calculated as ratio of (work after shampoo application) / (work before shampoo application) for each strand.

DETERMINATION OF THE AMOUNTS OF WAXES DEPOSITED ON THE HAIR

Hair strands were treated with shampoo formulations as described above. Samples were taken out after 1, 3 and 5 treatments. The hair strands were extracted with appropriate

solvents using an ASE (automated solvent extraction) system. The extracts were analyzed by GC/MS using flame ionization detector (FID) and mass spectrometry (electron ionization (EI) mode).

RESULTS AND DISCUSSION

INFLUENCE OF COSMETIC TREATMENTS ON HAIR BREAKAGE

After setting up the machine, hair strands treated in various cosmetic ways were tested. In Figure 2 the amount of hair breakage is given for virgin, bleached, bleached and permed, and persulfate bleached hair strands. It can be seen that the amount of broken hair fibers increases by chemical treatments. While a bleaching with hydrogen peroxide increases the amount of broken hair by 54%, the combination of bleaching and perming more than doubles the level of hair breakage (213% related to virgin hair). By applying a persulfate bleach to the hair the amount of hair breakage is increased by 244%. All further examinations have been performed using bleached hair, as such hair is a good model for damaged hair.

Using this type of pretreated hair the influence of the relative humidity was checked. In Figure 3 hair breakage of bleached hair is given for 40%, 60% and 75% relative humidity. It can be seen that the amount of hair breakage increases when going to more humid conditions by 14.5% (40% RH -> 60% RH) and 20.3% (40% RH -> 75% RH) Since all other tests in our lab are performed at 40% RH, this condition was kept throughout the work described in this paper.
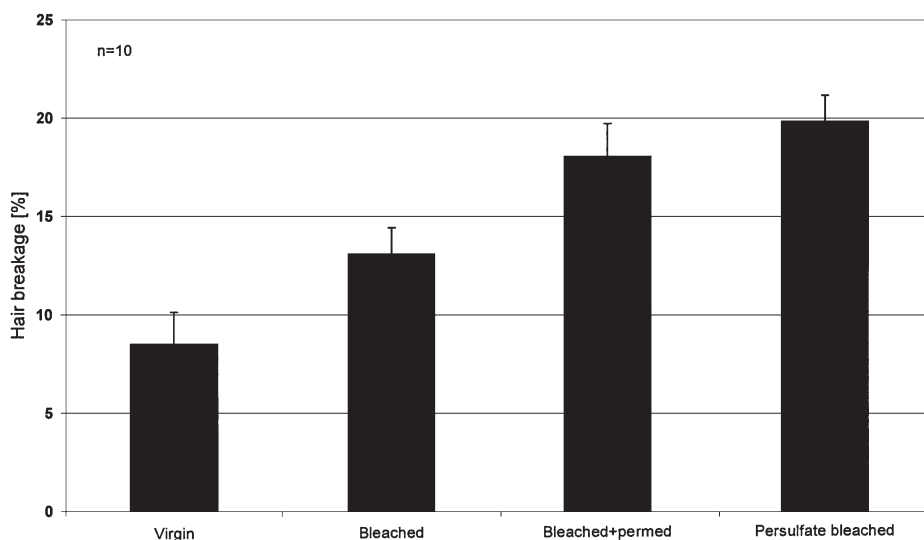

Figure 2. Hair breakage for hair strands of different cosmetic treatments. All results except for the difference of the last two columns are significantly different ($p<0.05$).

DETERMINATION OF THE ANTI-BREAKAGE EFFICACY OF COMMERCIAL HAIR CARE PRODUCTS

Several shampoos from major brands were tested for their anti-breakage efficacy. The results of two shampoos from different brands, both containing dimethicone and cationic polymer guar hydroxypropyltrimonium chloride, are given in Figure 4. Since the producers suggest using first the anti breakage shampoo and then the related anti breakage conditioner from the same product line, this was also tested. It can be seen from
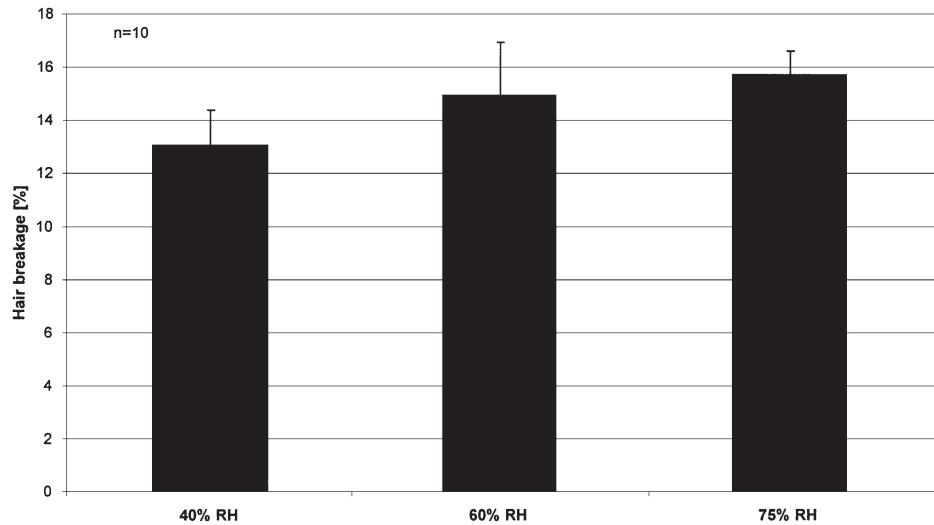

Figure 3. Hair breakage of bleached hair at relative different humidities. The result for 40% is significantly different from the data found for 60% and 75% RH ($p<0.05$).
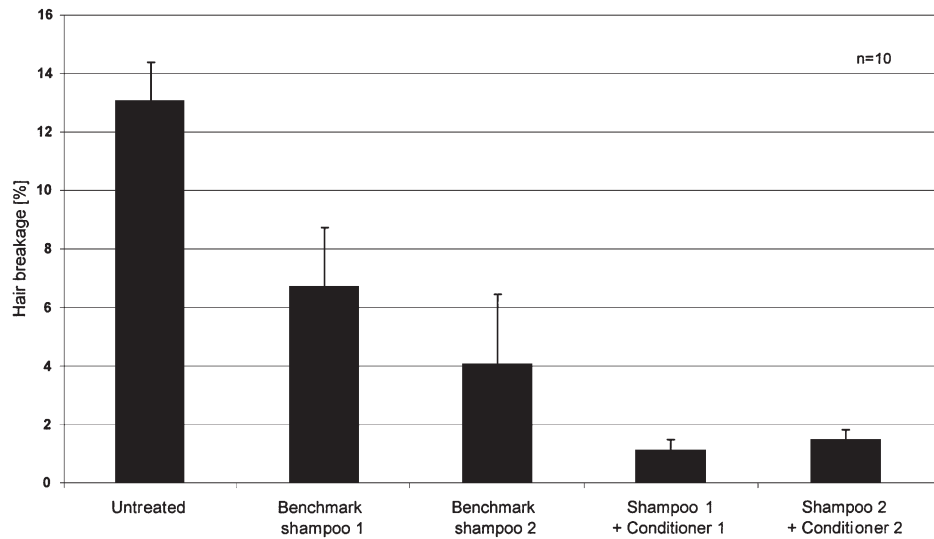

Figure 4. Hair breakage for hair strands treated with benchmark products. All results except for the difference of the last two columns are significantly different ($p<0.05$).

Figure 4 that both shampoos reduce the amount of broken fibers significantly by 49% and 69%. These effects are even improved when using both, shampoo and conditioner reaching a nearly 90% hair breakage reduction. Interestingly, the shampoo containing 3% dimethicone was less effective when compared to the other shampoo with only 1.4% dimethicone.

DEVELOPMENT OF AN ALTERNATIVE ANTI-BREAKAGE SHAMPOO INGREDIENT

In a screening test, several parameters were tested on their influence on the deposition of waxes on hair from a shampoo:

- Chemistry
- Hydrophilicity/hydrophobicity
- Particle size
- Hair state (virgin/bleached)
- Cationic polymer in the shampoo
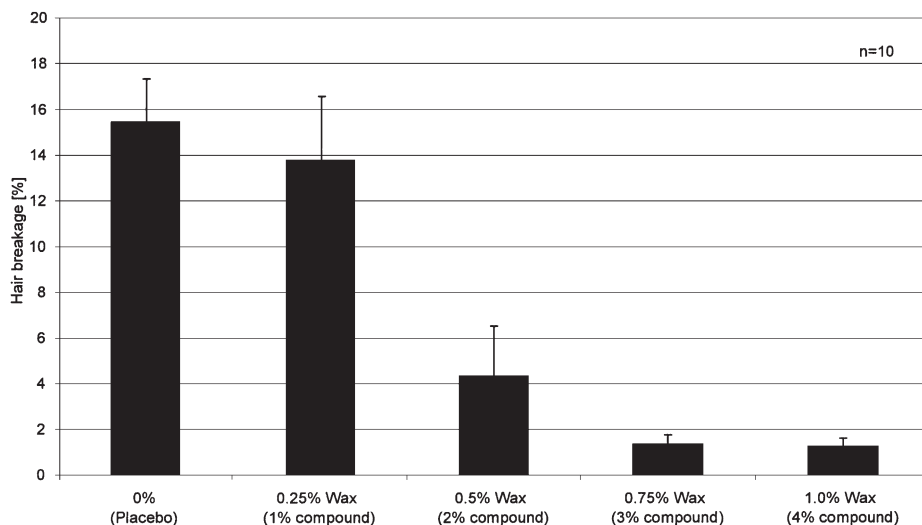- Concentration of the wax in the shampoo

Out of these series, the particle size was found to be the most significant parameter. Therefore, our objective was to develop a wax dispersion with particles smaller than 1 μm. Ether chemistry was chosen to ensure hydrolytic stability over a wide pH range, allowing the use in different fields like bleaching, perming or hair straightening. Finally, a compound consisting of PEG-4 distearyl ether, distearyl ether and dicaprylyl ether dispersed in sodium laureth sulfate was developed.

The efficacy of this ingredient was tested in shampoos with increasing concentrations of the wax dispersion combined with guar hydroxypropyltrimonium chloride. The composition of the shampoos can be found in Table I.

In Figure 5 the anti-breakage efficacy of shampoos containing 1, 2, 3 and 4% dispersions (equal to 0.25–1% active matter of the wax) is compared with the effect of a placebo containing a styrene/acrylates copolymer as turbidity ingredient. The shampoo composition is given in Table I. A clear dependence on the dosage was found. By comparing the levels of

Table I
Tested Shampoo Compositions

	Placebo	Shampoos with active
Sodium laureth sulfate	10	10
Cocoamidopropyl betaine	3	3
PEG-4 distearyl ether (and) sodium laureth sulfate (and) distearyl ether (and) dicaprylyl ether		1–4 (equals 0.25 to 1% wax)
Styrene/acrylates copolymer (and) cocoglucoside	0.4 (equals 0,12% wax)	
Guar hydroxypropyltrimonium chloride	0.2	0.2
Laureth-2	1	1
Citric acid	pH adjustment to pH 5	
Sodium chloride	Viscosity adjustment	
Perfume	0.3	0.3
Preservative	0.5	0.5
Water	Add to 100%	

Figure 5. Hair breakage for hair strands treated with shampoos containing varying concentrations of an ether-based wax dispersion. All results except for the difference between 3% and 4% of the wax dispersion are significantly different ($p<0.05$).

hair breakage with those of strands treated with market shampoos (Figure 4), it can be seen that a concentration of 2% in the shampoo shows a similar protection compared to the benchmarks. The shampoos with concentrations of 3 and 4% wax dispersion even reached the same protection level as the combination of shampoos and conditioners (90% reduction of hair breakage compared to only bleached hair without further treatment).

The wet and dry hair conditioning performances of the shampoos containing the wax dispersion was tested in comparison to the benchmark shampoos. The results are shown in Figure 6 and Figure 7. Interestingly, while the wet combing performance of the shampoos with higher contents of wax reaches those of the market shampoos, the latter are a little more efficient in terms of dry combing. This is in contrast to the anti-breakage performance. Obviously, low hair breakage is not completely in line with a good dry combability. This might be due to additional effects in the anti-breakage test which will be further discussed in the conclusions of this paper.

To have a closer look to the dosage dependency of the anti-breakage performance in shampoos the amount of wax deposited on the hair after several shampooing cycles have been determined by GC analytics of hair strands extracts. The results are shown in Figure 8. A clear dosage dependency can be seen in these data, as well as some build-up effects.

CONCLUSIONS

We were able to show that tailor-made wax dispersions formulated in shampoos with a cationic polymer are at least equally efficient as silicones in reducing hair breakage and split ends. The results of combability experiments and hair breakage tests are completely parallel with the silicone containing shampoos and the shampoos containing the wax dispersion. Most probably the reason can be found in a different underlying mechanism.
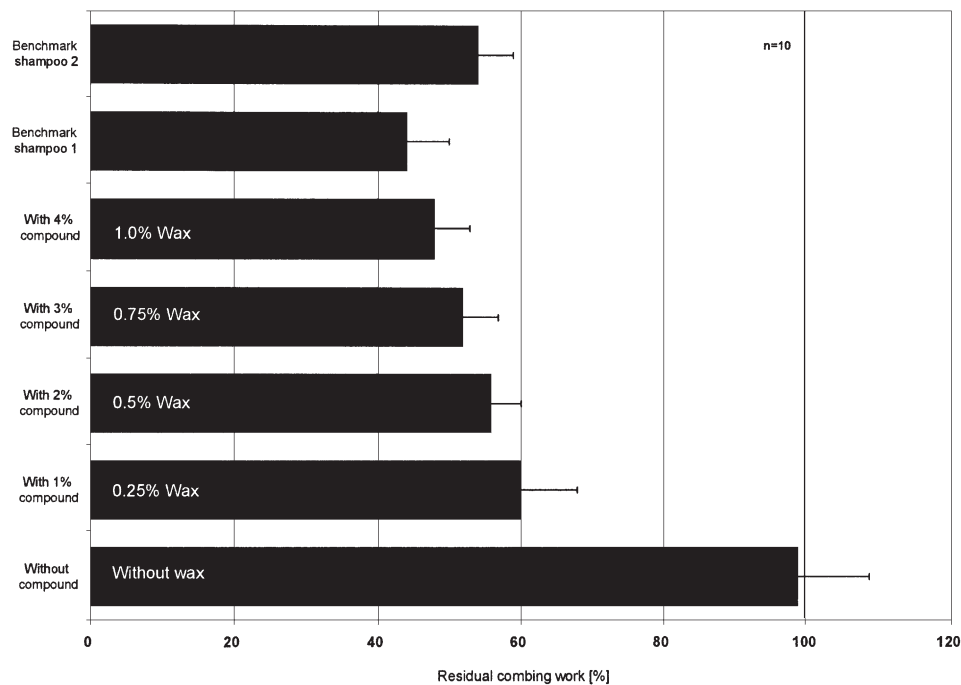
Figure 6. Residual wet combability for shampoos with an ether-based wax dispersion in comparison to benchmarks.
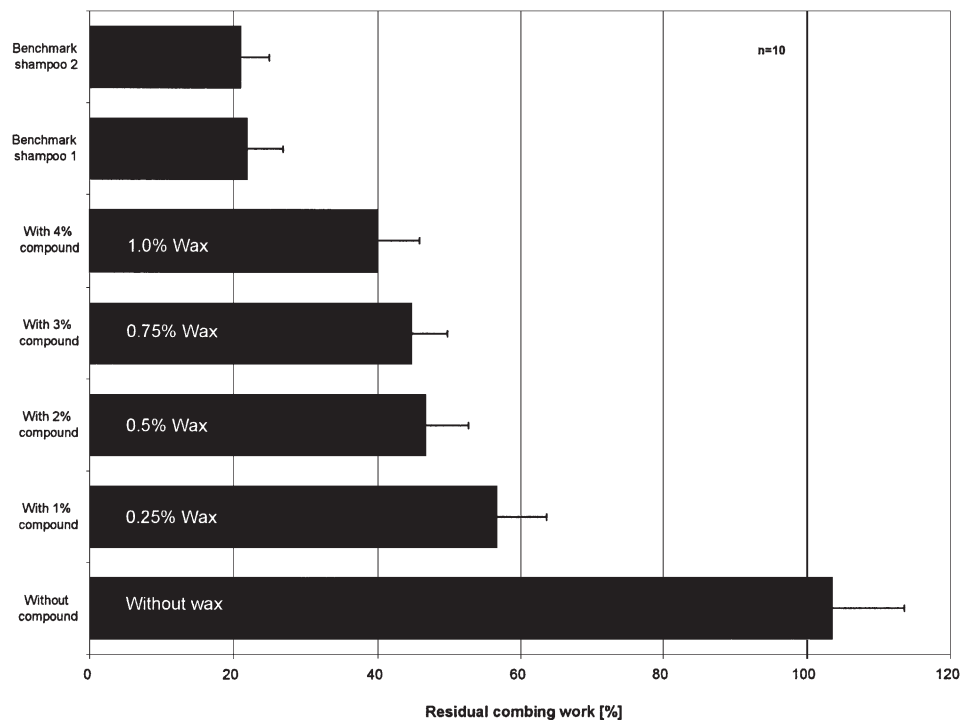

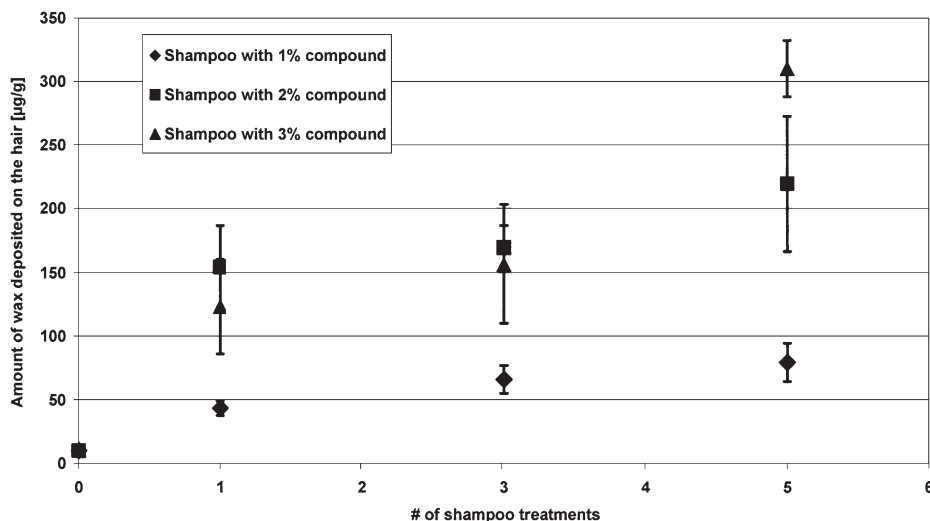**Figure 7.** Dry combability for shampoos with an ether-based wax dispersion in comparison to benchmarks.

Figure 8. Deposition of waxes from the shampoos containing an ether-based wax dispersion.

It is very likely that hair fibers break at regions with cracks in the cuticle and outer cortex area. Such cracks will reduce the cross sections of the fiber and thus increase the stress induced by combing. Wax particles acted not only as lubricants like silicones, but also fill these cracks, which in turn will reduce the stress by distributing the forces generated by the combing procedure. This hypothesis will be further explored in future work.

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