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Spatially resolved combing analysis

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Synopsis

A new technique, termed *spatially resolved combing analysis,* was employed to study the conditioning properties of a cationic polymer on various types of hair. In this method, special frames are employed that allow the application of a treatment to selected areas of the fibers, while shielding the remaining portions as internal reference sections. The combing traces of hair treated in such a way, obtained by using a Diastron tensile tester, show positive or negative peaks depending on whether the treatment results in an increase or a decrease of friction against the hair surface. The method has higher sensitivity than previously employed procedures that involved the measurements of hair before and after conditioning of the whole tress.

The conditioning of polyquaternium-11 was evaluated on untreated, Caucasian brown hair as well as on fibers damaged by oxidative dyeing, bleaching, bleaching/dyeing, and perming. It has been shown that the conditioning effect depends on the type of hair modification. The effect of the polymer on untreated hair is relatively small and becomes apparent only after multiple shampooing, which roughens the untreated sections of hair. For dyed hair, which exhibits three to four times higher combing forces than the reference virgin hair, the adsorbed polyquaternium-11 reduces the combing forces to the level of untreated hair. The conditioning layer of the polymer can be removed from dyed hair by one to two shampooings. The polymer showed higher substantivity for bleached and permed hair, with the conditioning effect persisting after multiple shampooings.

INTRODUCTION

Quantitative combing measurements are widely employed in the evaluation of hair care products. The technique was first described by Newman *et al.* (1), and further developed by Tolgyesi *et al.* (2) and Garcia and Diaz (3). Detailed discussion of various factors affecting the quantitative combing analysis of hair, including the application of two combs, was reported by Kamath and Weigmann (4). In all these publications the procedure consisted of passing a comb through a hair assembly in the form of a tress and measuring the forces as a function of distance. To evaluate the conditioning effect, the measurements had to be performed first on untreated fibers, and then, after the treatment, on modified hair. The error of combing work data was ±20% and ±50% for wet and dry hair evaluations, respectively. This relatively high error, related to variability in relative placement or entanglement of the fibers in a tress, precludes the possibility of unequivocal detection of small changes in the frictional properties of hair. This report presents a new approach to combing measurements, termed *spatially resolved combing analysis,* which consists of applying the modifying treatments to only selected areas on a tress rather than to the whole fiber assembly. This was accomplished by the use of special treatment frames that allowed the application of polymer, surfactant, or formulation solutions to selected areas of the fibers, while shielding the remaining portions as internal reference sections. The combing traces of hair (usually presented as combing force as a function of distance or position of a comb in a hair tress) treated in such a way show positive or negative peaks depending on whether the treatment results in an increase or a decrease of the lubricating properties of the hair surface. Since treated and control areas are scanned in one combing measurement, the new technique has higher sensitivity than the previously employed procedures and also allows a more graphic representation of the data.

The experimental data presented in this report refer to polyquaternium-11, which is a cationic copolymer frequently employed in fixative and conditioning hair care products. Previously, the results of qualitative analysis of conditioning attributes of cationic copolymers of vinyl pyrrolidone were reported by Murphy (5). Recently, a reference to conditioning properties of polyquaternium-11 was made by Hossel and Pfrommer (6).

EXPERIMENTAL

INSTRUMENTATION

Spatially resolved combing analysis. The formulations were applied to selected areas of hair tresses by the use of a two-window treatment setup (Figure 1). It consists of two plexiglas and two silicone rubber frames. A hair tress is compressed between the rubber and plexiglas frames with stainless steel screws and bolts. A typical procedure consists of (a) treating the exposed sections of the fibers with an appropriate formulation, (b) extensive rinsing with water to remove any residues of the treatment solution, and (c) opening the frame to perform the wet combing analysis of the treated tress.

The combing measurements were performed by employing a Diastron miniature tensile tester with the following instrument settings: range 500 G; gauge 2 G; sample size 50 mm; phase 1 extension 350%; phase 2, 0%; phase 3, 0%; and phase 4, 0%. The instrument was operated by Rheopac software (revision 3.0A). The data calculations and plotting were performed by using QuattroPro for Windows or Lotus 1-2-3 for Windows spreadsheets.

The combing measurements were done on wet hair (3). In all experiments hair was combed several times to remove entanglements, and then mounted in the Diastron with hair evenly distributed across the 1.25-inch length of a comb (12 teeth/inch). The combing force was continuously recorded as the tress was combed at a crosshead speed of 12 cm/min. The data were analyzed either by comparing the features of the combing curves (combing force as a function of distance) or by calculating the total combing work as an integrated value of force over the length of the tress.

MATERIALS

Samples of hair. Experiments were performed on dark brown hair purchased from DeMeo Brothers, New York. Hair samples were precleaned with 3% ALS solution and thor-

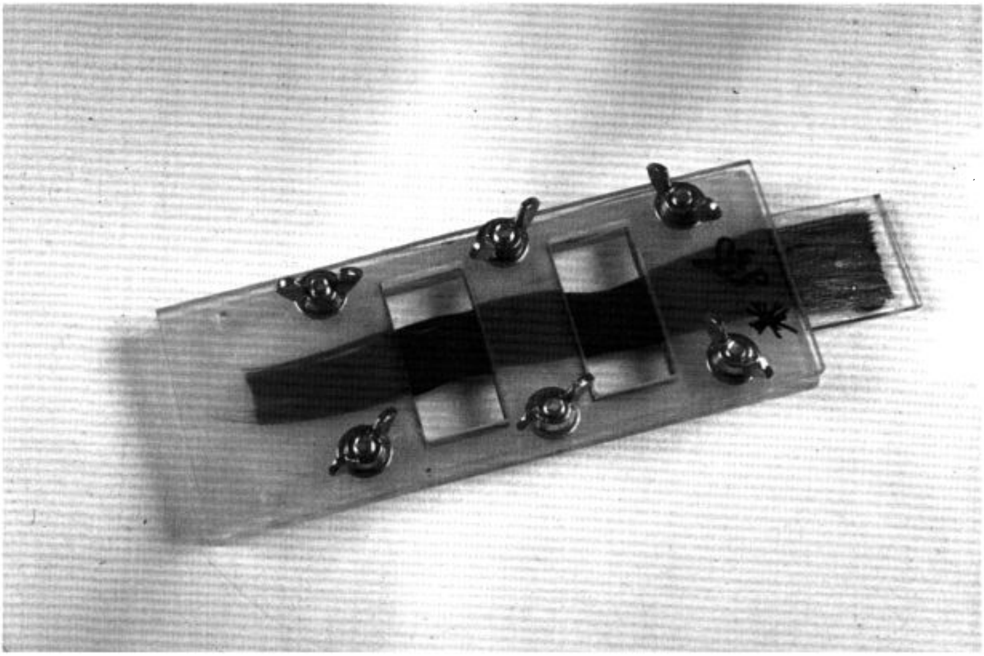

Figure 1. A two-window treatment frame for treating the fibers in two sections.

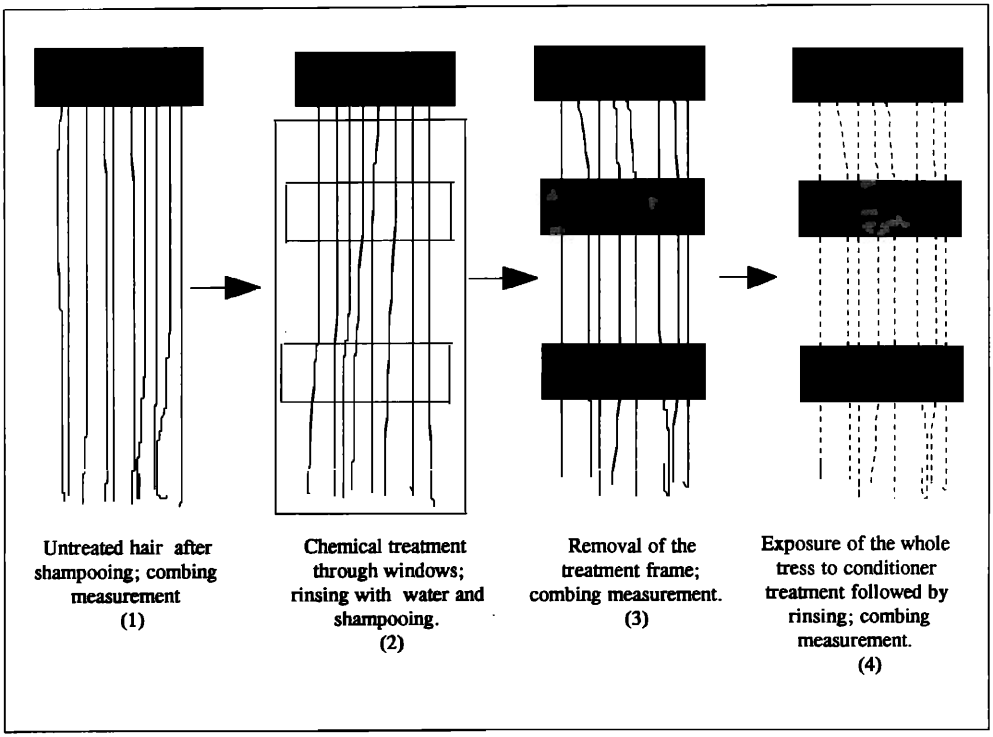

Figure 2. Experimental protocol of procedure A.

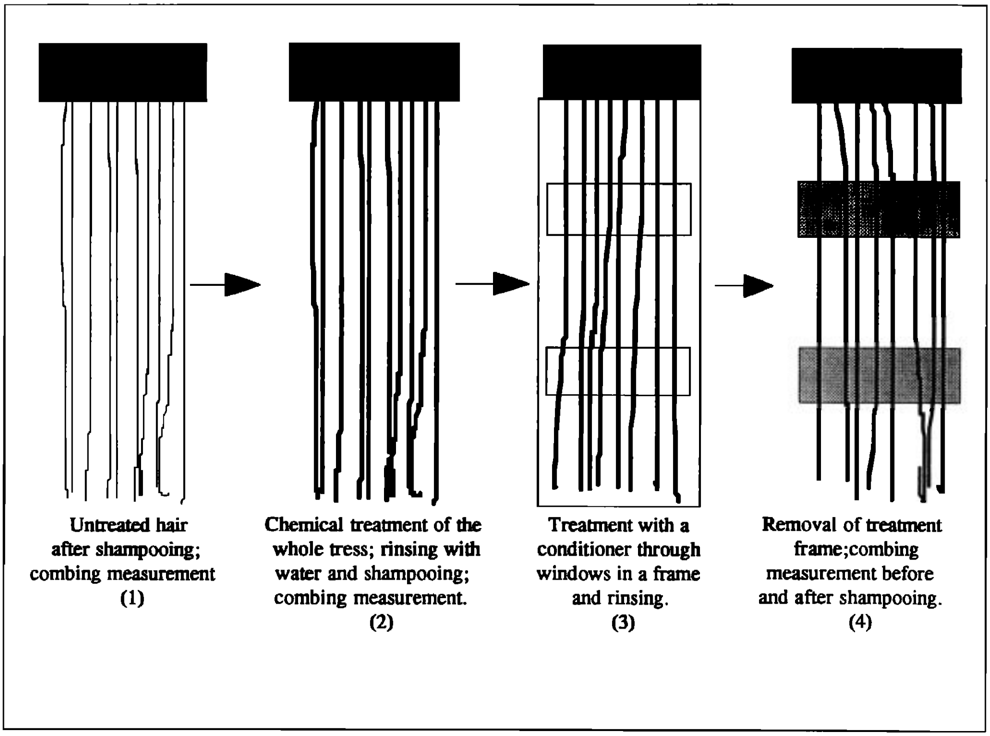

Figure 3. Experimental protocol of procedure B.

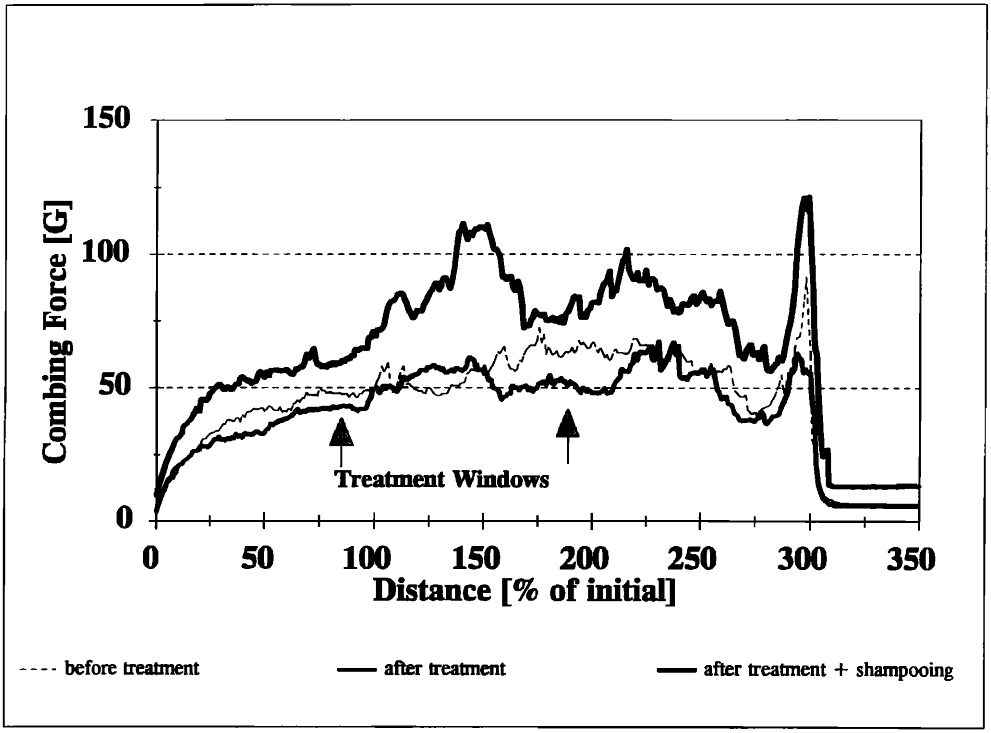

Figure 4. Combing curves of virgin, dark brown hair treated according to procedure B (no chemical treatment) after exposure to 0.2% polyquaternium-11.

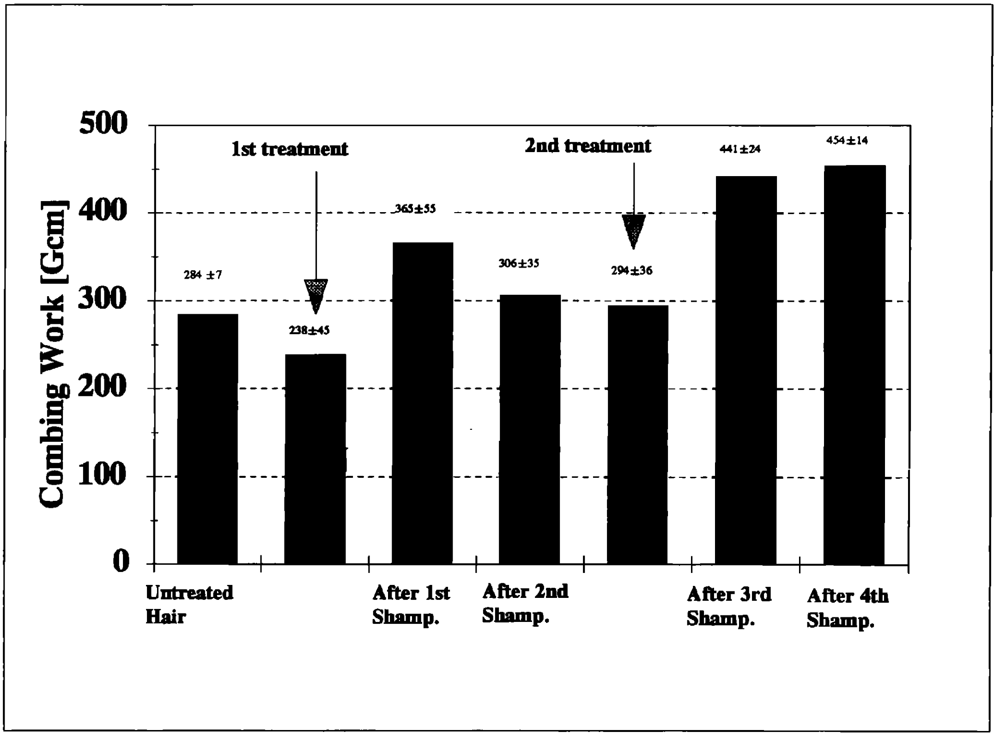

Figure 5. The combing work of virgin, dark brown hair after one, two, and three treatments with 0.2% polyquaternium-11 followed by shampooing.

oughly rinsed before experimentation. For nonviscous treatment products, the fibers were immersed in their solution for a specified amount of time, followed by rinsing with water. For viscous creams, a spatula was employed to ensure a uniform distribution of a product in hair, and the treatment was followed by prolonged rinsing with water to remove any formulation residues. Hair tresses were prepared by gluing 2-g fibers to 1.5 × 1.5-in plastic tabs with Duco cement. The length and width of a hair tress was 6.5 in and 1.25 in, respectively.

Solutions of polyquaternium-11. The conditioning treatments were prepared as 1% polyquaternium-11 (Gafquat 755N, International Specialty Products) solutions (0.2% active). They were applied to hair samples for two minutes by saturating the exposed sections of fibers in the excess of polymer solution, followed by extensive rinsing with water.

Bleaching. Hair was bleached by using a powder lightener based on ammonium persulfate/potassium persulfate blended with 20 volume hydrogen peroxide. The paste was applied to hair in the window area for 60 minutes, followed by extensive rinsing with water and shampooing with 3% ammonium lauryl sulfate.

Perming. Hair perming was performed by using a formulation based on ammonium thioglycolate with 2% hydrogen peroxide as a neutralizer. In order to test the conditioning properties of polyquaternium-11, a neutralizer lotion containing 0.2% active

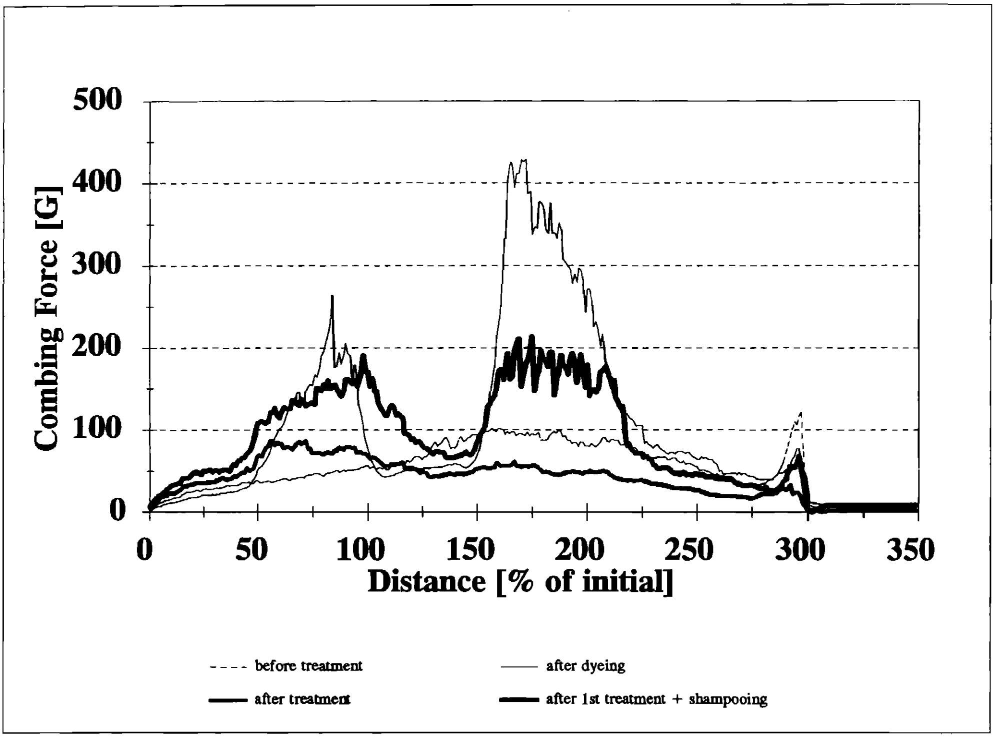

Figure 6. The effect of oxidative dyeing and treatment with 0.2% polyquaternium-11 (procedure A) on the combing curves of virgin, dark brown hair.

polyquaternium-11 and 2% hydrogen peroxide was employed. For comparison, a commercial conditioning formula of a neutralizer including quaternium-52 was also used.

Dyeing. Hair was dyed by employing a black shade of a conventional oxidative haircolor that did not contain conditioning agents. A dye lotion, after mixing with an equivolume amount of 20 volume hydrogen peroxide, was left on the hair for 30 minutes, followed by extensive rinsing with water and shampooing with 3% ammonium lauryl sulfate.

RESULTS AND DISCUSSION

The properties of hair surface can be affected by both chemical and physical modification. Reactive chemical treatments usually increase the hydrophilicity of hair because of dissolution of surface lipids, deposition of dyes, breakage of disulfide bonds, and keratin oxidation, which leads to the formation of hydrophilic groups. These changes in the chemical composition of hair surface result in an increase in fiber-to-fiber or fiber-comb adhesion, which is further reflected in higher combing forces, especially in the wet state. Physical modification of hair, through adsorption of conditioning agents such as cationic surfactants, polymers, or oils, is usually designed to alleviate the damaging effects of reactive chemical treatments by lowering the combing forces.

In this work, two experimental protocols are employed to quantify the effect of modifying treatments on hair. Both employ treatment frames with two windows to produce

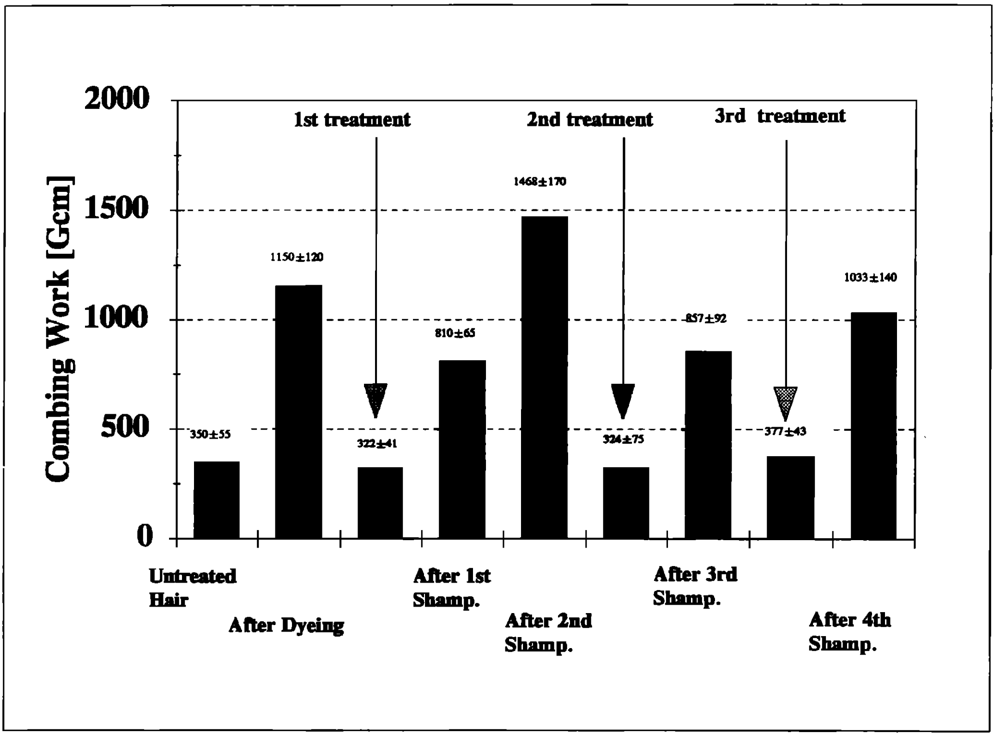

Figure 7. The combing work of dyed hair after consecutive treatments with polyquaternium-11 followed by shampooings.

spatially resolved modification of the fiber surface. In procedure A (Figure 2), a reactive composition, such as an oxidative dyeing or bleaching system, is applied through the windows, while the subsequent conditioning treatment is performed on the whole tress. After chemical modification, combing curves usually show two peaks corresponding to modified areas of hair. The height of the peaks can further decrease as a result of the application of a conditioning agent, and the extent of this reduction can be used as a measure of the efficacy of this treatment. It should be also noted that, in this case, the reference portion of the fibers is chemically unchanged, but modified with an adsorbed layer of a conditioning agent. In procedure B (Figure 3), the whole tress is chemically modified, and the conditioning treatment is applied through the windows. The combing curves, after the conditioning treatment, display two valleys, and their depth can be related to the efficacy of a conditioning agent. In this experiment, the combing curves allow for the assessment of a difference in friction resulting from adsorption of a conditioning agent on a substrate with uniform surface properties.

The effectiveness of a conditioning agent on virgin hair can be assessed by applying the treatment through the windows (procedure B). The combing trace of hair treated in such a way reflects a difference in friction between untreated control sections of the fibers and those modified by an adsorbed polymer or surfactant. The relative lowering of the combing force in the treated areas indicates a conditioning effect. For chemically modified hair, on the other hand, the conditioning effects can be studied by adopting both experimental protocols, procedures A or B.

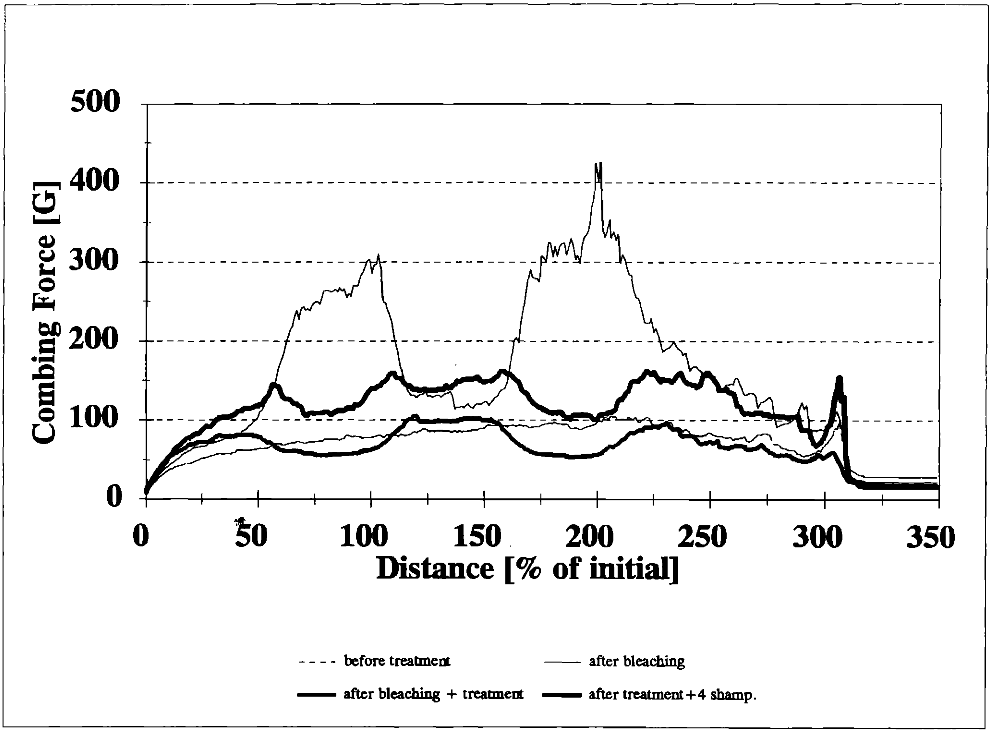

Figure 8. The effect of bleaching and treatment with 0.2% polyquaternium-11 (procedure A) on the combing curves of virgin, dark brown hair.

VIRGIN, BROWN HAIR

The effect of a cationic polymer, applied through the windows in the treatment frame, on virgin, brown hair is presented in Figure 4. The analysis of the combing traces suggests that after the first treatment and rinsing, the conditioning effect of the adsorbed layer of the polymer is relatively small. The lowering of combing forces, in comparison to untreated hair, is evident in tress sections corresponding to both windows, and is of the order of 10–20 G (Figure 4). Subsequent shampooing leads to an increase in combing forces in the areas that were not treated with the polymer. One possible explanation of this increase can be a progressive removal of sebum and/or hair lipids that form a lubricating layer on the fiber surface. Another contributing factor could be a progressive damage to hair, such as the abrasion of cuticle cells, incurred in the process of shampooing as a result of rubbing and handling the hair. This phenomenon is well documented in the literature (7,8). The combing forces in treated sections of the fibers increase to a smaller extent, and the two combing force minima are clearly evident after shampooing. This suggests that the adsorbed polymer is not removed completely by exposure to anionic surfactants. Also, the formation of a polymer–surfactant complex may enhance the lubricating properties of the treated sections of the fibers. The subsequent, second, treatment of the same tress with the polymer solution (through the windows; combing traces not shown) reduces the combing force values again, to a level characteristic for untreated hair. The traces obtained after shampooings following the second treatment display the same pattern as those obtained after the first treatment,

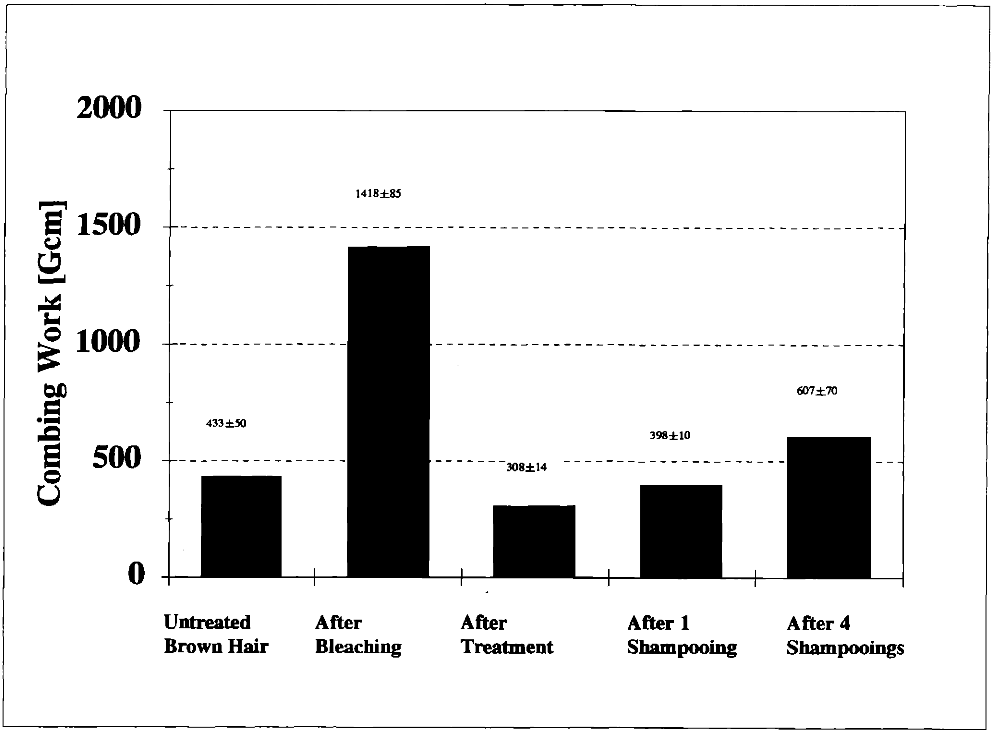

Figure 9. The combing work of bleached, virgin, dark brown hair after each, consecutive treatment or shampooing. The figure summarizes the data presented in Figure 8.

with an evident increase in combing forces in untreated sections of hair. The data are summarized in a bar graph plot illustrating the variation of combing work after treatments and shampooings in relation to untreated hair (Figure 5). The results presented in Figure 5 emphasize again a small conditioning effect of polyquaternium-11 and an increase of combing forces after repeated shampooings.

DYED HAIR

To assess the effect of the cationic polymer on oxidatively dyed hair, untreated brown hair was first dyed through windows with a black shade of an oxidative hair dye (procedure A). This results in a significant increase in combing forces in the dyed sections of hair (Figure 6). Exposure of the whole tress modified in such a way to a 0.2% solution of polyquaternium-11 results in an adsorption of the polymer and a decrease in the combing force values to a level similar to that characteristic for untreated hair. However, subsequent shampooings desorb the polymer and produce a significant increase of friction, especially in the damaged portions of hair. A conditioning layer of the polymer is partially removed by one shampooing and completely stripped by two shampooings, as shown by the data in Figure 6. The ensuing, second, treatment of the same tress with a solution of the conditioning agent reduced the combing forces again, and a subsequent shampooing partially removed a conditioning layer of the polymer

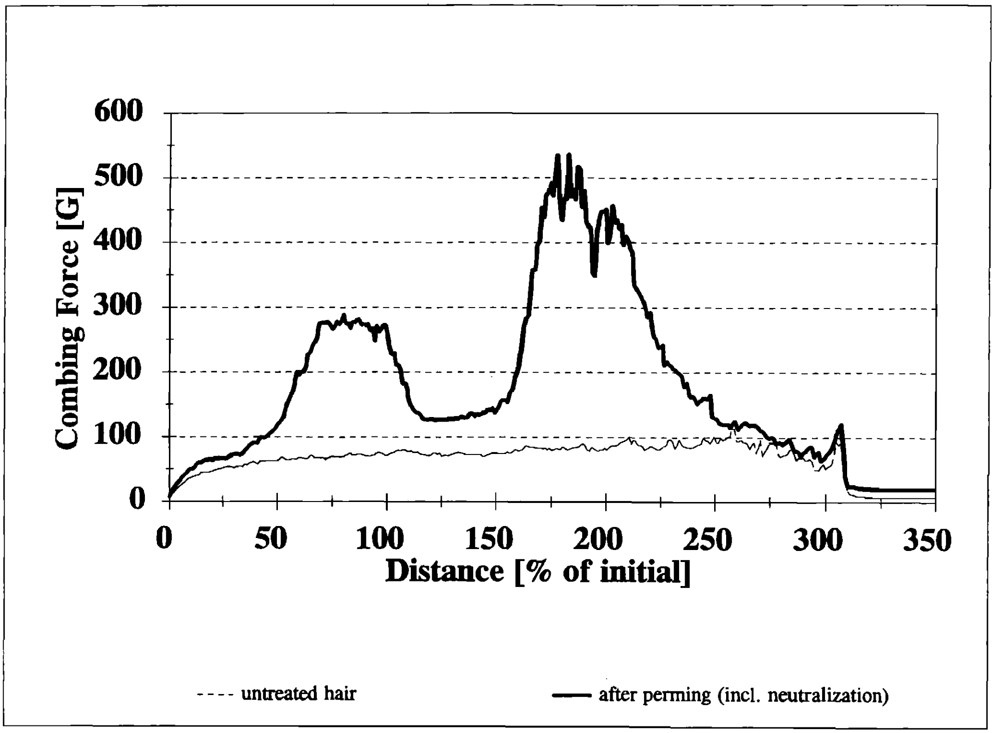

Figure 10. The effect of perming with a nonconditioning formulation and shampooing (procedure A) on the combing curves of virgin, dark brown hair.

from the hair. The same sequence of conditioning and shampooing repeated for the third time yielded the same result.

The combing data are summarized in Figure 7, which shows the combing work of the dyed sections of the tress after each treatment. It demonstrates the conditioning effect of the polymer and its removability from hair as a result of one or two shampooings. The data also provide indirect evidence of the lack of polymer buildup after consecutive applications. This presumption is supported by the fact that no decrease in combing values is evident after the second and third polymer treatment, which suggests no additional deposition of polymer on hair.

BLEACHED OR PERMED HAIR

The conditioning of bleached hair is illustrated by the data presented in Figure 8. One hour bleaching of hair results in a three- to fourfold increase in combing forces as compared to the untreated control. Subsequent treatment with a 0.2% polymer solution brings about a reduction of combing forces, especially pronounced in the bleached sections of a tress. The shape of the combing trace after polymer treatment, with two minima corresponding to the bleached section of hair, suggests that the cationic polymer is more substantive to damaged hair, and its adsorption reduces the combing friction to a level below that characteristic for untreated hair. In addition to this, a modifying layer of polymer cannot be removed by subsequent multiple shampooings. While combing

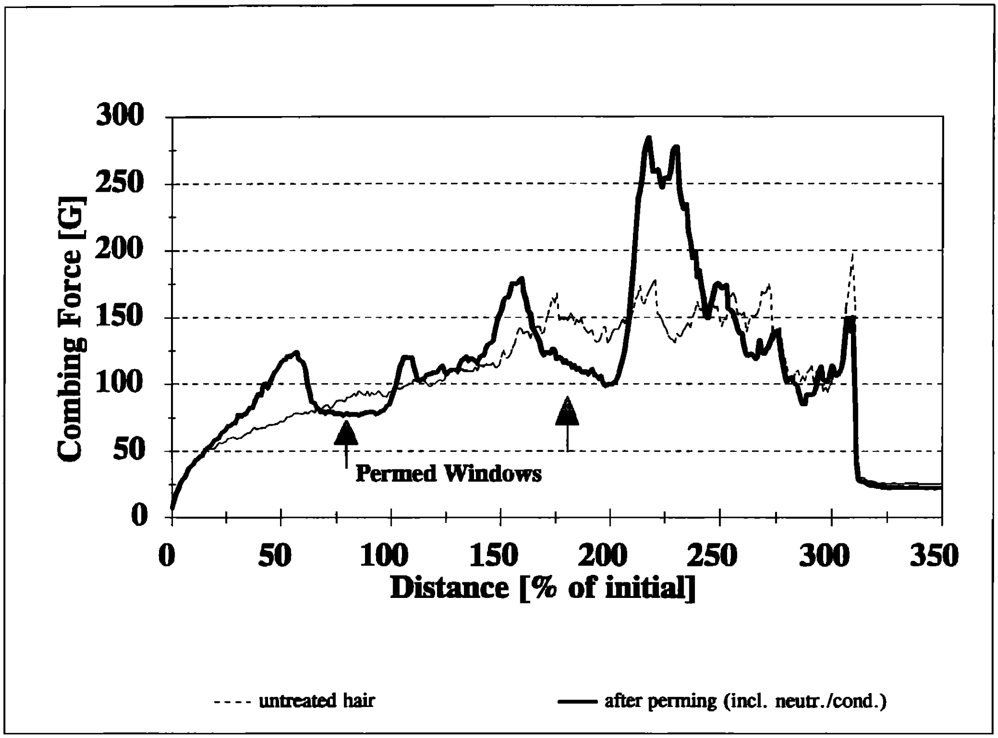

Figure 11. The effect of perming with a conditioning formulation (0.2% polyquaternium-11 in a neutralizer; procedure A) on the combing curves of virgin, dark brown hair.

forces increase after one and four shampooings, they remain at a relatively low level, close to that measured for untreated hair, as evident from both combing force traces (Figure 8) and from the calculated combing work values (Figure 9).

Perming, which consists of hair reduction with a mercaptan and its subsequent oxidation (neutralization) with hydrogen peroxide, also results in the damage of hair surface. Figure 10 shows a four- to fivefold increase in combing forces corresponding to the permed sections of fibers. Incorporation of a cationic polymer in the neutralizer prevents hair from becoming raspy by lowering the combing forces (Figure 11). Clearly, the treated sections are characterized by lower friction than the untreated portions of hair, which suggests high substantivity of polyquaternium-11 to perm-damaged fiber surface. The effect is permanent, and the conditioning persists through four shampooings. In contrast to this, when perming was performed by employing a neutralizer containing a low-molecular-weight conditioning agent such as quaternium-52, a smaller extent of combing force reduction was evident. In this case, the conditioning effect was also not durable, and disappeared completely after a single shampooing.

BLEACHED/DYED HAIR

The effect of the polymer on hair damaged by a combination of two bleachings and oxidative dyeing was also studied by employing procedure B. As with bleached hair, it

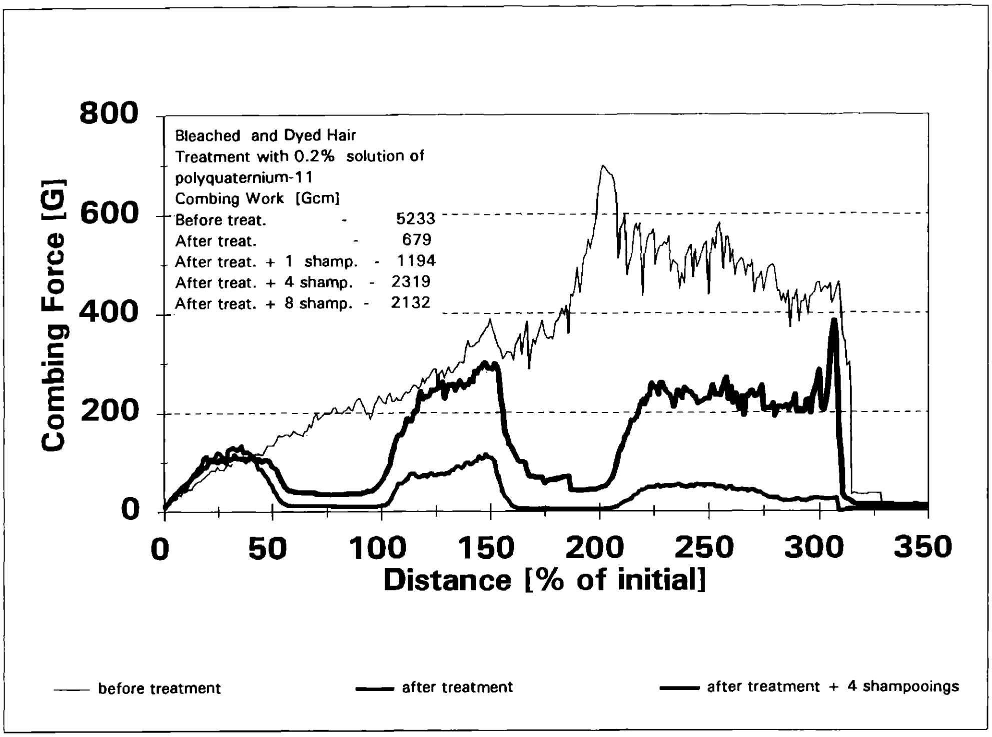

Figure 12. The effect of treatment with 0.2% polyquaternium-11 (procedure B) and subsequent shampooings on the combing curves of twice bleached and dyed hair.

was found that cationic polymer adsorbs on the fiber surface in an irreversible fashion and cannot be removed by repeated shampooings. As shown by the data presented in Figure 12, the combing forces are reduced in the treated sections by factors of 20 and 40, in the first and second windows, respectively. Combing forces corresponding to untreated sections of hair are also significantly reduced, an effect probably related to the mechanism of fiber disentanglement in the neighboring low and high friction areas of a tress. Four and eight shampooings result in an increase in the combing forces, especially in untreated sections of hair, although to a level significantly lower than that characteristic for unconditioned hair.

CONCLUSIONS

Spatially resolved combing analysis was applied to study the properties of hair damaged by the use of chemical treatments. The technique was capable of (a) quantifying the surface damage as a result of dyeing, bleaching, and perming, and (b) demonstrating the conditioning properties of a cationic polymer, polyquaternium-11. The substantivity of the polymer to hair could be assessed by the measurements performed after the conditioning treatment as well as after subsequent shampooings. The study demonstrates that the extent of hair conditioning by the polymer depends on the state of the hair surface. While undamaged hair was not significantly improved by the use of polyquaternium-11, its effect on chemically modified and raspy hair was very pronounced. In addition to this, the conditioning effect was found to be durable since an adsorbed layer of a cationic polymer could not be removed, from either virgin or damaged hair, by shampooing.

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