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Light scattering and shine measurements of human hair: A sensitive probe of the hair surface

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Synopsis

In this paper, an instrumental method for measuring hair shine is presented and shown to exhibit excellent correlation with a large series of subjective evaluations of shine. In addition to providing shine values, the light-scattering methods developed are shown, in many cases, to provide a sensitive means of following changes to the hair surface, including deposition (soiling), particle removal (cleaning), and even interactions on the fiber surface. Employing the developed methods, the effect of washing hair tresses with a series of commercial shampoos is investigated. The effects examined are shown to fall into three classes: Shampoos without highly substantive ingredients left hair in its cleanest and shiniest state. Shampoos containing ingredients substantive to hair (polycationics and soap) left deposits on the fiber surface and dulled the hair. The worst dulling was observed when particles deposited on the hair from one shampoo formed a complex with particles contained in a second product.

INTRODUCTION

Shiny hair is consistently cited by consumers in panels and surveys as one of the most desirable of cosmetic attributes. As a result, much effort has been expended in recent years to understand the physical phenomena that give rise to shine and also to quantitate this desirable hair attribute.

The most effective means described in the literature to study hair shine has been the use of a goniophotometer to measure light scattering by hair fibers (1–4). Goniophotometric techniques have been used by the authors of references 1–4 to relate light-scattering patterns to hair morphology and also to develop formulas relating goniophotometric data to hair shine.

Unfortunately, the bulk of the effort in the above studies has been concerned with explaining the light-scattering patterns; only cursory efforts were made to relate calculated shine values to panelists' subjective evaluations. In addition, little effort was made to systematically apply the methods developed in these papers to problems related to hair care.

In this paper, a large series of subjective evaluations of hair shine is reported and shown to exhibit excellent correlation with a formula developed to calculate hair shine using goniophotometric measurements. In addition to providing a quantitative measure of shine, the light-scattering methods developed in this work are shown to provide a means of following changes to the hair surface, including deposition (soiling and buildup), removal of particles (cleaning), and even interactions at the fiber surface.

Single-fiber techniques are presented and shown to provide a rapid, qualitative means of determining the effects on hair fibers of various treatments. The results of shine measurements of tresses exposed to various treatments of interest are then shown to confirm and make quantitative the foregoing single-fiber results.

Finally, the shine of hair tresses treated with various shampoos is measured and shown to be a function of the nature of the substantive ingredients found in particular shampoos.

The results in this paper show that light scattering can be used not only to obtain quantitative information about shine but that it can also serve as a sensitive probe of the hair surface, providing information difficult or impossible to obtain using other surface techniques.

EXPERIMENTAL

SHAMPOO EXPERIMENTS

All tresses for shampoo experiments were prepared using three grams of human hair purchased from DeMeo Brothers, New York. In order to minimize scattering from the rear cuticle, only dark brown Oriental hair was used (1).

For each comparison series, a minimum of three tresses was prepared for each shampoo treatment. In addition, three control tresses, treated only with 20% sodium lauryl sulfate (SLS) and representing "clean hair," were prepared for each series. In comparison experiments, succeeding tresses in a series received different treatments so that any particular treatment was staggered throughout the entire series.

All tresses were subjected to five pretreatment washes with SLS, followed by five treatments with a test shampoo. In those cases where shampoo combinations were employed, five treatments with the first shampoo were followed by three treatments with a second.

Pretreatment cycles were performed by immersing tresses for five minutes in 250 ml of 5% SLS in a graduated cylinder. The tresses were then removed, rubbed by hand for two minutes, and then rinsed twice for one minute each in graduated cylinders containing 500 ml of deionized water.

Treatment cycles were performed in the same manner as the above, except that a 2:1 tap water, shampoo solution was substituted for SLS, while tap water rather than deionized water was employed in rinses. Note that the tap water used in these experiments had a water hardness level of 80 ppm.

SUBJECTIVE SHINE EVALUATIONS

Subjective evaluation of tress shine was performed on groups of six tresses each, with three tresses mounted for each treatment. Figure 1 shows a photograph of the frame used for mounting tresses for subjective comparisons. Each tress is clamped at the root end, stretched over the cylinder, and secured at the tip. This setup insures that all tresses are oriented in the same manner toward the viewing light and also minimizes orientation differences among the individual hairs comprising the tresses.

Tresses were viewed under two lamps, each of which contained two cylindrical, 60-watt tungsten bulbs. The lamps were placed side by side so that all four bulbs were in a single row approximately ten inches above the tresses. This configuration maximizes the chances that each tress receives the same amount of light.

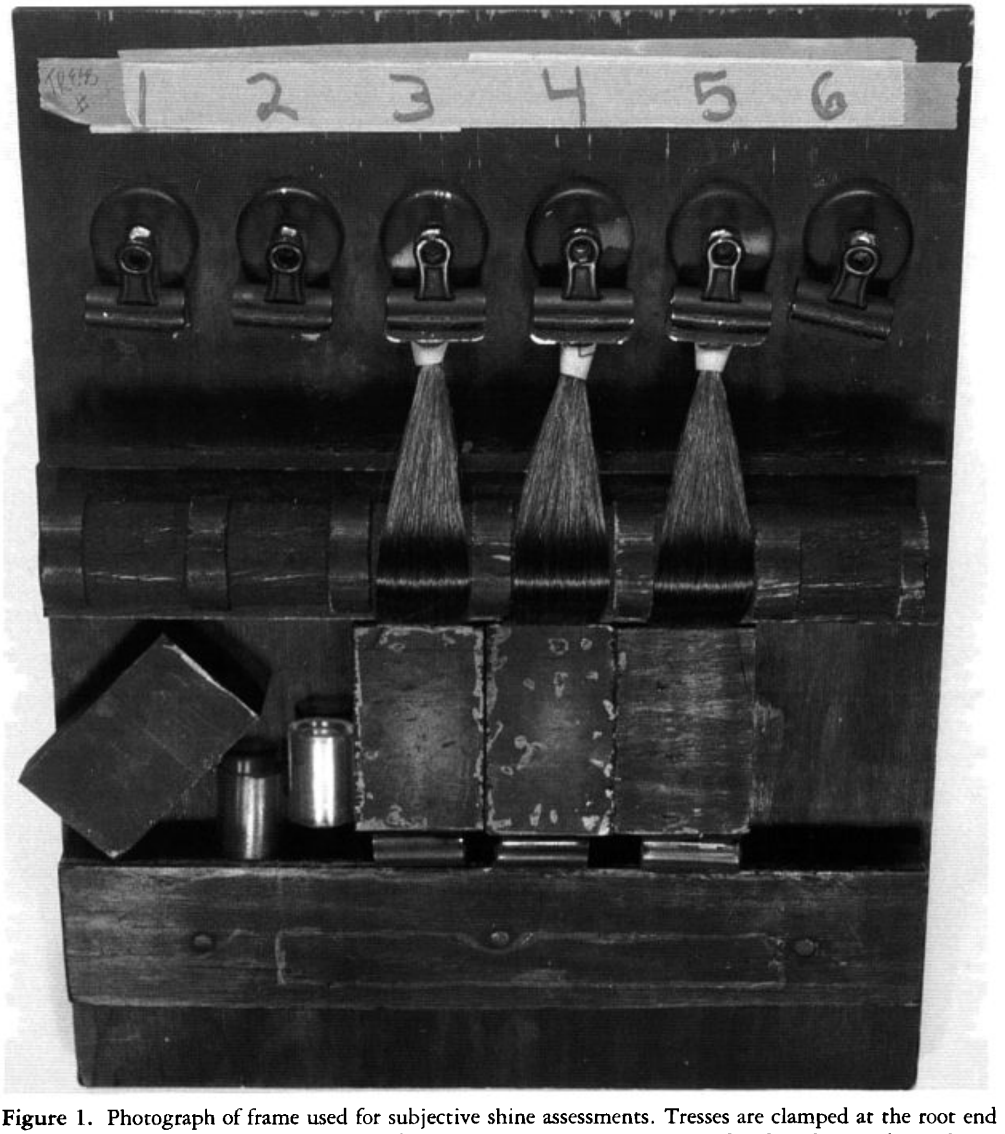

Figure 1. Photograph of frame used for subjective shine assessments. Tresses are clamped at the root end and stretched over the cylinder. A wooden block is then placed over the tip end and held secure by wedging a small piece of flexible tubing between the frame and the block.

Following mounting, tresses were evaluated by sixteen to twenty panelists, each of whom was asked to rank the tresses in order of relative shine. After evaluation, tress positions were interchanged and a new set of evaluations performed. This was done in order to minimize any positional biases.

The data from the above rankings were evaluated statistically by the Friedman test (5). This is a non-parametric test and has several advantages over the corresponding parametric methods since it does not require a normal distribution and makes no assumptions concerning variance of the data.

GONIOPHOTOMETRIC MEASUREMENTS

The bulk of the goniophotometric measurements were performed using a Brice-Phoenix light-scattering photometer (Virtis Co., Gardiner, NY), modified by attaching a recorder motor to the graduated disc in order to automate scanning of receiving angles.

Subsequent to the research described in this paper, a Murakami GP-1R automatic goniophotomer was purchased (Hunter Associates Laboratory, Inc., Reston, VA). This instrument is not subject to the scanning limitations experienced with the Brice-Phoenix photometer. The sample light-scattering curve presented in Figure 2 was produced with the GP-1R photometer.

In order to maximize the accuracy of the diffuse scattering measurement (D in Figure 2), polarizers having directions of polarization perpendicular to the plane of incidence were

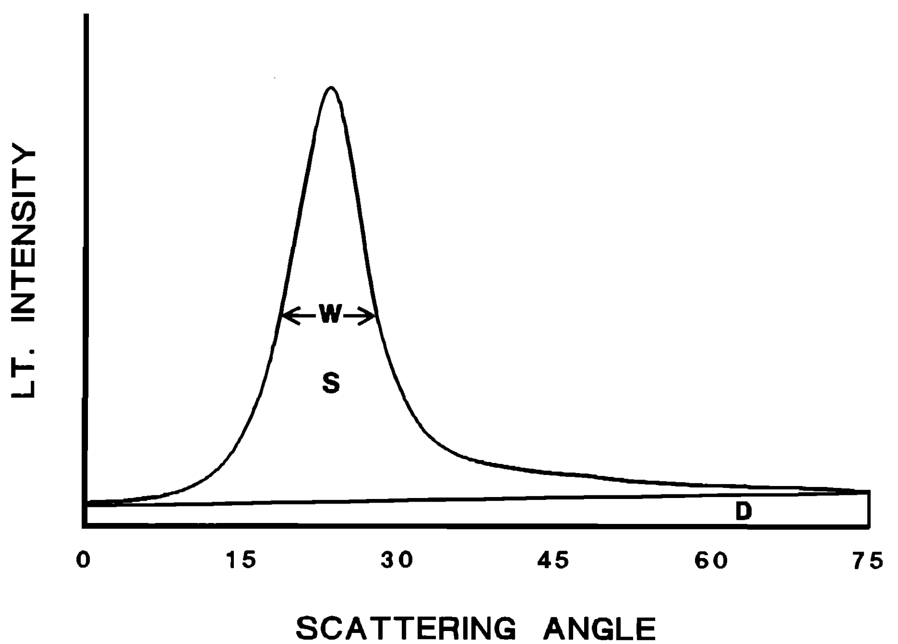

Figure 2. A typical light-scattering curve for a virgin Oriental hair. D is the diffuse reflectance and is calculated by measuring the area under the straight line connecting the light intensities at 0 and 75 degrees. S is the specular reflectance, and is the total area under the curve minus the diffuse region. W is the width of the curve at half height.

placed before the samples and photomultiplier tubes of the goniophotometer in all light-scattering experiments (2).

The sample holder for the Brice-Phoenix photometer consisted of two supports, indented at the top and 4 cm apart, across which a single hair could be laid. A sample hair fiber was secured at one end and kept under constant tension by attaching a 2-gram alligator clip to the other end. Hair fibers had to be at least four inches long to conveniently fit on the sample holder.

Because of the short distance between sample and photomultiplier tube in the Brice Phoenix photometer, samples run at an incident angle of 30° with respect to the perpendicular to the hair could only be scanned between 15° and 75°. This proved adequate for qualitative work, but for quantitative measurements, the incident angle was changed to 37.5° so that scans could be run between 7.5° and 75°. The scanning time for the latter interval was 72 seconds.

For all experiments, hair fibers were oriented so that the direction of the incident light was toward the tip end of the hair (RER orientation in reference 1).

The output from the goniophotometric measurements was sent to a Bascom-Turner 3120T electronic recorder (Bascom-Turner Instruments, Norwood, MA) that digitized each scan into 500 points. These points were then sent to an IBM PC computer for processing.

SINGLE-FIBER SCREENING TESTS

For single-fiber screening tests, single hair fibers were mounted on the goniophotometer sample holder and held taut with a 2-gram alligator clip. Treatments were then applied by dropping one or two drops of a test solution onto a glass microscope slide supported under the hair by a platform high enough so that the slide just missed touching the hair.

After application of test solutions, the slides were moved back and forth under the hair for 60 seconds. Fibers were then rinsed with tap water in the same fashion. Following this, light-scattering curves were run, and the fiber was then either treated again or discarded.

Note that some fibers are sufficiently irregular in cross section that movement of the sample holder from the instrument to the lab bench could cause the fiber to twist and change the light-scattering curve. A change in a measured curve was thus only considered to be real if it could be reproduced after movement of the fiber holder.

DYE-STAINING EXPERIMENTS

Dye-staining experiments were performed using Sirius Red F3BA New, C. I. No. 35780. This is a high-molecular-weight (1372) anionic dye and was obtained from Mobay Chemical Co., Rock Hill, SC.

Experiments were performed by wetting a 3-inch by 4.5-inch wool swatch (wool challis, Test Fabrics Inc., Middlesex, NJ) with water, applying 3 ml of a test shampoo, rubbing for one minute, and then rinsing under 100°F running tap water for one minute. Following this, swatches were either treated with dye solution or treated with 3 ml of a second shampoo, rinsed, and then treated with dye.

Dye treatments consisted of placement of a swatch in 40 ml of a 0.5% solution of dye for 30 seconds. The swatches were then rinsed under 100°F running tap water for 30 seconds and hung up to dry.

MEASUREMENT OF SHINE

Shine, or luster, is perceived when an object scatters much more light at a particular angle or in a particular direction than in other directions. Under these conditions, surface highlights or brightness contrasts appear and the object is seen to be shiny (see, for example, references 6–8).

In order to measure shine, therefore, one needs to be able to measure scattered light intensity as a function of angle. This is accomplished with a goniophotometer. Figure 3 shows, in rudimentary fashion, the operation of such an instrument with a hair fiber sample.

In this case, the fiber is assumed to be held taut and is irradiated with light at an incident angle, i, that is prechosen and is measured with respect to the perpendicular to the fiber.

The intensity of that portion of the incident light that is scattered by the hair is measured by rotating a photomultiplier tube, or light detector, from 0° to 75°. The illustration shows two of the positions of the photomultiplier tube.

Typical results from a goniophotometer measurement are shown in Figure 2 for an undamaged Oriental hair. The light-scattering scan is presented as a plot of scattered light intensity as a function of angle.

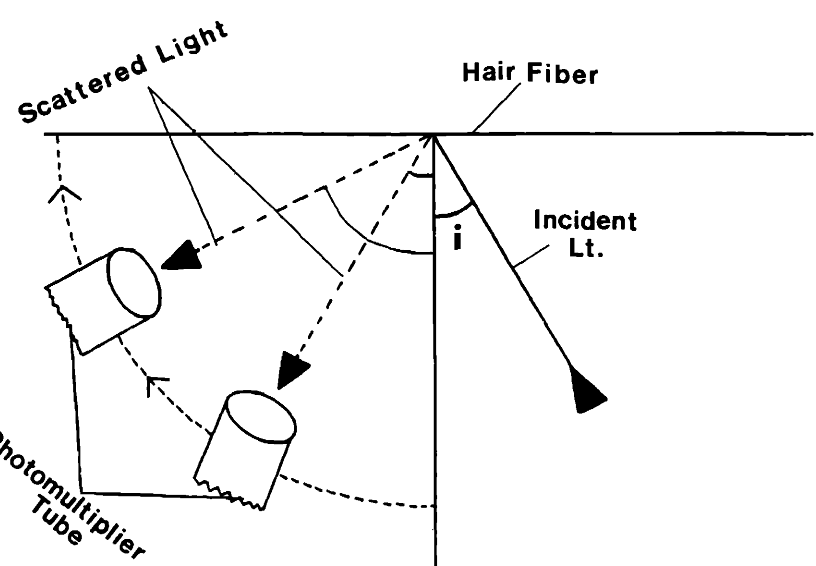

Figure 3. Rudimentary diagram of a goniophotometer experiment. A hair fiber is held taut and struck by incident light at angle i. Scattered light is then detected by rotating a photomultiplier tube from 0 to 90 degrees with respect to the perpendicular to the fiber. Two of the photomultiplier positions are shown.

The incident light in this experiment was set at 30°, and one can see a maximum in the measured curve near this angle. This is termed *specular reflection*. Light is also scattered at angles other than the specular; this is termed *diffuse reflection*. This latter type of scattering is caused by light hitting the scale edges of the hair. It can also be caused by small imperfections on the hair surface and, in addition, by deposited particles. An excellent discussion of the morphological features of hair that give rise to observed light-scattering patterns can be found in reference 1.

The question now arises as to how one can use the goniophotometric results to measure shine or luster. From the definition of shine, it appears obvious that luster increases with increasing specular reflection and decreases with increased diffuse scattering. Any functions used to estimate shine must therefore take these two relationships into account.

Several workers have developed shine functions, employing these relationships for applications such as textile fibers, polymer surfaces, etc. [see, for example, (2,6–10)].

In this work, several functions were tested both from the literature and also devised by ourselves. The best agreement with subjective evaluations was found using the relationship

$$L = S/DW(\tfrac{1}{2}) \qquad \text{(Eq. 1)}$$

where L equals luster or shine. D in this expression is the integrated diffuse reflectance and is obtained, as in reference 2, by connecting the scattered light intensities at 0° and 75° and measuring the area under the resulting line. S in equation 1 is the integrated specular reflectance and is obtained by measuring the area of the specular peak, while $W(\tfrac{1}{2})$ is the width of the specular peak at half-height. All three of these quantities are illustrated in Figure 2.

It has been pointed out (2) that use of expressions such as equation 1 with D in the denominator are valueless for cases where diffuse reflectance goes to zero. For most cases involving hair, however, scattering off the scale edges insures a minimum value for D, so that equation 1 is broadly applicable.

In the current experiments, hair fibers from treated tresses were scanned one at a time in the goniophotometer. There is tremendous variation from hair to hair, even from a single head of hair, so that in order to obtain meaningful shine values for a particular treatment, an average of many hairs must be taken. In the current case, 21 hairs were taken from each tress, while three tresses were employed for each treatment. Each shine value, therefore, represents an average taken from 63 hairs.

RESULTS AND DISCUSSION

SINGLE-FIBER SCREENING TESTS

Figures 4–6 show typical light-scattering scans taken after a series of shampoo treatments of single hair fibers. These types of single-fiber experiments are useful as a means of rapidly screening the effects of various treatments on hair.

The results from these experiments can only be treated qualitatively, however, since they represent treatments on single hairs and there is too much variation among hairs for

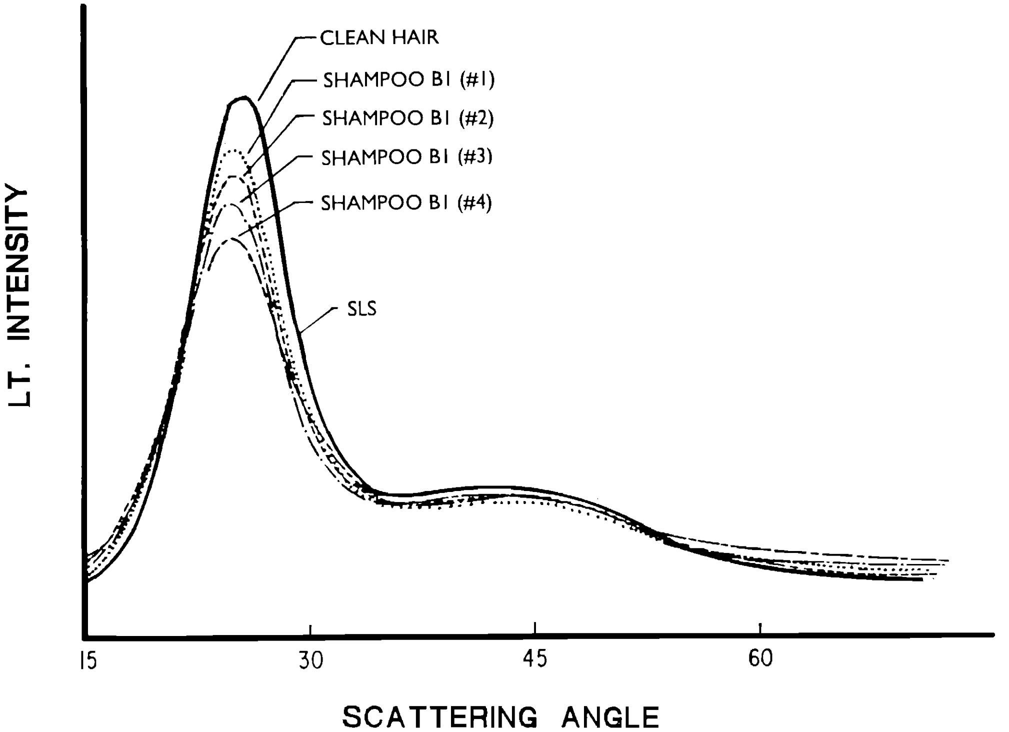

Figure 4. A single-fiber experiment testing the effect on hair of shampoo B1. The hair was washed with SLS at the beginning of the experiment (clean hair) and at the end. The same curve was obtained in both cases.

quantitative results from a single hair to be meaningful. A particular result must therefore be repeated several times on different hairs in order to be considered real.

Bearing this in mind, and also the fact that single-fiber treatment conditions are very different from tress and in-shower treatments, one can use single-fiber screening tests as a convenient means of determining the possible effects of many different products on hair. This method is especially useful when hairs are undergoing a series of treatments, since after each particular step in the series, a light-scattering scan can be run in order to determine the effect of that particular step. This can be very helpful in elucidating the mechanism of a particular effect.

Figure 4 shows the results of a series of treatments of an Oriental hair with shampoo B1. This is a commercial product that, at the time these experiments were performed, contained Polyquaternium-10, a polycationic well known to be substantive to hair, in a trideceth-7 carboxylic acid detergent system.

The hair was first washed with 20% sodium lauryl sulfate (SLS). The resultant light-scattering curve is considered that of a clean hair. Following this, successive treatments with shampoo B1 (followed by water rinses) caused dulling, indicated by decreases in the peak height along with increases in diffuse scattering. This loss of shine was caused by deposition of shampoo residue, probably Polyquaternium-10, on the hair surface.

After four treatments with B1, the hair was again treated with 20% SLS. The resulting light-scattering curve was congruent with the original, clean hair curve, implying that

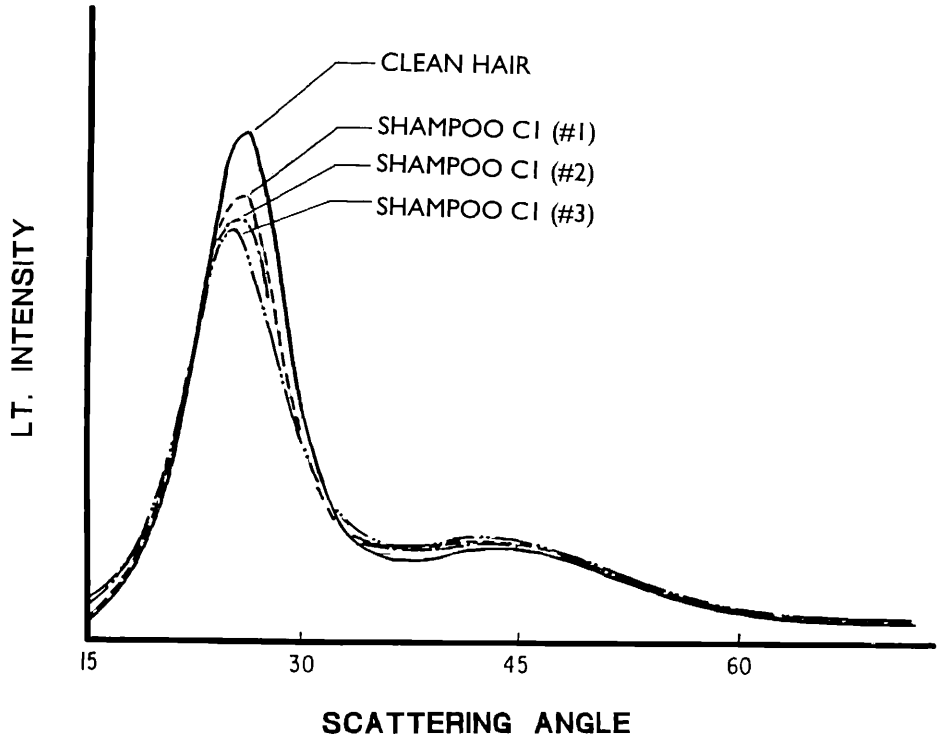

Figure 5. A single-fiber shampoo C1 experiment.

the dulling B1 residue was removed from the hair. These experiments demonstrate that light scattering can be used to follow both deposition (soiling) and removal of particles (cleaning) from the hair surface.

Figure 5 shows another single-fiber experiment using shampoo C1, a commercial product that at the time of sale contained sodium myristate, a component of soap that is well known to deposit on and dull hair (11). As with the B1 experiments, successive treatments with shampoo C1 caused dulling, in this case a result, probably, of deposition of sodium myristate particles on the hair. Again, as with the preceding experiment, after treatment of the dulled hair with 20% SLS, the original, clean hair curve was recovered.

Figure 6 shows the results of a single-fiber experiment in which a clean hair was treated twice with shampoo B1, resulting in dulling from shampoo residue. An attempt was made to clean this residue with shampoo C1, with the expectation that initially the C1 would remove accumulated B1 residue and that further applications would lead to accumulation of new C1 deposits.

Surprisingly, application of shampoo C1 did not lead to an initial increase in shine but, unexpectedly, to a great increase in dullness, much larger than the sum of the individual shampoo effects. This implies that the deposits from the two shampoos are interacting to form a new residue that is more dulling to the hair than the former deposits. That a new type of residue has formed is also supported by the observation that attempting to reverse the increased dullness by washing with SLS did not lead to a change in the

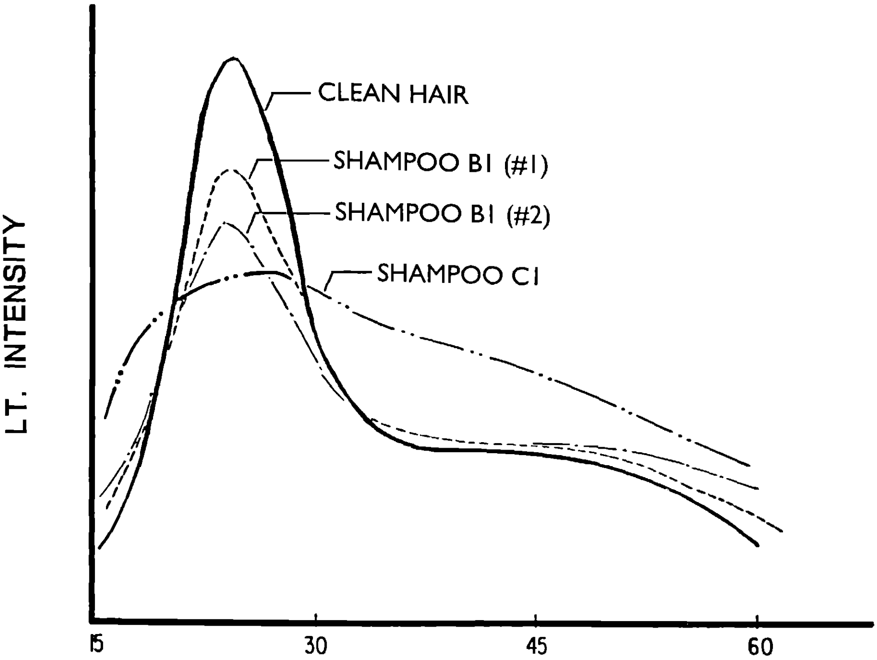

Figure 6. A single-fiber experiment measuring the effect of washing with two different shampoos. Note the huge increase in diffuse scattering and the great decrease in shine after treating the hair with shampoo C1.

light-scattering curve, indicating that, unlike the individual residues, the combination residue is resistant to removal by SLS.

Formation of a B1:C1 complex, presumably between the Polyquaternium-10 cation and the myristate anion, was further tested by performing a dye-staining test, the results of which are shown in Figure 7. The wool swatch on the left in this figure was washed with shampoo B1 and exhibits a pink color as a result of subsequent treatment with Sirius Red. This dye binds to wool only in the presence of bound cationic (12); the color, therefore, indicates that some Polyquaternium-10 was deposited on the wool surface from shampoo B1.

The swatch on the right in Figure 7 was washed with shampoo B1, then shampoo C1, and then treated with Sirius Red. In this case, the swatch retained almost no red dye, indicating that after treatment with C1, very few cationic binding sites were left on the swatch. Since, from the single-fiber experiments, it is known that Polyquaternium-10 still remains on the surface, one must conclude that it is no longer available for binding to Sirius Red as a result of complexation with myristate anion. The conclusion from light scattering that a complex was formed on the hair surface is thus confirmed.

TRESS TREATMENT EXPERIMENTS

As stated previously, single-fiber results, such as those in the preceding section, cannot be treated quantitatively because of the tremendous variation among single hairs. In

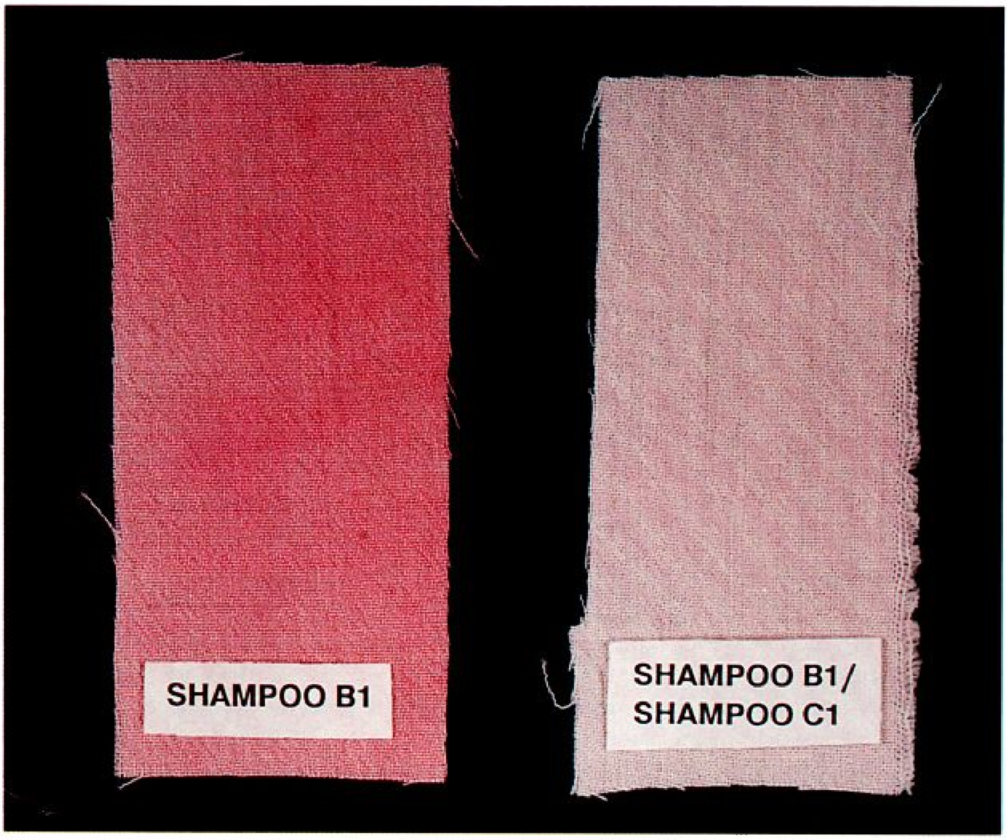

Figure 7. A dye-staining experiment testing the effects of washing with shampoos B1 and C1. The pink color of the swatch on the left indicates the presence of Polyquaternium-10 after washing with shampoo B1. The absence of a pink color for the swatch on the right indicates the lack of cationic binding sites as a result of complexation of Polyquaternium-10 from B1 and myristate anion from C1.

order to obtain meaningful instrumental shine values, therefore, it is necessary to treat tresses, rather than single hairs, and to then measure the shine values for a number of hairs from each tress and average the results.

Aside from the resultant quantitative data, there are two other advantages to treating tresses rather than single fibers. First, unlike the case with individual hairs, the shine of treated tresses can be subjectively assessed and, if desired, compared to instrumental data.

In addition, the conditions employed in treating tresses are much closer to actual in-shower conditions than is true for single-fiber tests. In the latter case, even though a hair is exposed to only one to three drops of treatment solution, this is still more exposure than it would receive buried in a head of hair.

Thus, although results from single-fiber tests are valid for the conditions employed, they would not necessarily be observed in normal usage. To confirm whether an observed effect on single hairs would actually occur under in-shower conditions, it is necessary to treat tresses (containing more than a thousand hairs) rather than single fibers.

In order to test the validity of equation 1, and also to confirm the preceding single-fiber results, a series of tresses was treated with shampoos B1 and C1 along with four other commercial shampoos and SLS. Table I lists the detergents contained in the shampoos employed, along with any ingredients substantive to hair that they contain.

Each of the tresses employed in the current experiments was washed at least five times with a particular shampoo in order to simulate buildup. Following this treatment, hair samples were taken from each tress for light-scattering measurements. Tresses were then mounted on the evaluation frame for assessment by panelists.

A large series of shine evaluation panels was run to determine treatment differences among all seven of the shampoos employed. The results are tabulated in Table II, where the rankings are listed in order of decreasing shine. Those treatments connected with a vertical line are not significantly different from each other.

Table II also lists the instrumental shine values measured for each treatment, along with the associated standard deviations. The numbers were obtained by calculating an average shine for each tress and then averaging the shine numbers for all tresses treated with the same shampoo.

The agreement between the subjective rankings and the instrumental values in Table II was tested using the Spearman rank correlation coefficient method (13). The value calculated for the Spearman rank correlation coefficient was 1.00, which is significant at a level greater than 99%. The agreement between panelists' assessments and instrumental measurements is thus excellent, and one can conclude, therefore, that the numbers calculated from equation 1 are a reliable measure of hair shine.

The results in Table II also indicate that the single-fiber effects reported in the previous section can be expected to be observed under actual usage conditions. Tresses washed with shampoo B1 were found to be duller than clean tresses, while tresses treated with shampoo B1 followed by C1 were found to be duller than those treated with B1 alone.

In general, the shampoo treatments in Table II were found to fall into three main groupings. In the first group, indicated by the first vertical line in Table II, none of the shampoos had ingredients highly substantive to hair. Treatments with these shampoos resulted in the shiniest hair observed, and we consider this hair to be essentially clean.

The second group of shampoos in Table II all contained Polyquaternium-10 (a cationic polymer) or fatty acid salts (soap). These ingredients are substantive to hair and can

Table I
Shampoos Employed in Shine Experiments[1]

Shampoo	Primary detergent	Substantive ingredients
A1	Ammonium lauryl sulfate	—
A2	Sodium laureth sulfate	—
SLS	Sodium lauryl sulfate	—
B1	Trideceth-7 carboxylic acid	Polyquaternium-10
B2	Sodium laureth sulfate	Polyquaternium-10
C1	Sodium laureth sulfate	Sodium myristate
C2	Sodium lauryl sulfate	Coconut acid (sodium salts)

[1] All shampoos except SLS and A2 were commercial products on sale in the United States. Shampoo A2 was a product available in England.

Table II
Subjective and Instrumental Assessments of Shampoo Treatments[1]

Subjective rank[2,3]	Instrumental shine[3]
Shampoo A1	0.762 (0.10)
Shampoo A2	0.714 (0.09)
SLS	0.696 (0.03)
Shampoo C2	0.602 (0.04)
Shampoo B2/shampoo C2	0.582 (0.02)
Shampoo B2	0.551 (0.08)
Shampoo B1	0.486 (0.03)
Shampoo B1/shampoo C1	0.427 (0.03)

[1] Assessments were made with a minimum of three tresses per treatment.
[2] Treatments are listed in order of decreasing shine.
[3] Vertical lines connect treatments that are not statistically different.

build up with repeated use. As a result of this particle deposition, the shampoos in group 2 all caused dulling.

The third type of treatment effect occurred when the negatively charged fatty acid salts in one shampoo formed a complex with the positively charged Polyquaternium-10 previously deposited on the hair by another shampoo. These types of particles caused the greatest degree of dulling on hair. Although only one example of Polyquaternium-10/ myristic acid dulling is shown in Table II (B1/C1), a second example of this type of interaction and shine loss was observed in single-fiber tests between shampoos B2 and C1. The former product contains Polyquaternium-10 in a sodium laureth sulfate detergent system.

CONCLUSIONS

In this paper, goniophotometric measurements of shine were presented and shown to exhibit excellent correlation with a large series of subjective assessments. In addition to providing a quantitative measure of hair shine, the light-scattering methods presented were also shown to serve as a sensitive probe of the hair surface, permitting one to monitor deposition, removal, and even interaction of particles on hair. Employing the light-scattering methods developed, a group of shampoos containing highly substantive ingredients was shown to dull hair as a result of deposition on the fiber surface, while even worse dulling was observed as a result of interaction on the fiber surface of incompatible particles from different shampoos.

It should be noted that the incidence and magnitude of dulling effects from deposition depends on the size of the particles deposited on the hair surface (14). The greatest degree of diffuse scattering and, consequently, the greatest degree of dulling, occurs from particles of the order of 0.4 to 0.7 microns. Particles of the order of 4 microns or larger, on the other hand, will reflect light specularly.

Thus, although the substantive ingredients in the shampoos tested in this work caused dulling, it is conceivable that substantive ingredients from other products might be deposited in the form of a film. Depending upon the orientation of its various parts, such a film might cause little or no dulling and, in fact, might even increase hair shine.

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