J. Cosmet. Sci., **58**, 393–404 (July/August 2007)

Evaluation of hair humidity resistance/moisturization from hair elasticity

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Synopsis

Average water regain and hair elasticity (Young's modulus) of virgin dark brown and bleached hair fibers under different relative humidity (RH) were determined. It is observed that hair water regain increases linearly with an increase in RH in the range of 40–85%; and the remaining percent of hair elasticity decreases linearly with an increase in RH in the range of 50–80%. Therefore, measurements of average hair elasticity at 50% and 80% RH, respectively, under various equilibrium times before and after cosmetic treatments can be used to evaluate effects of cosmetic treatments on water adsorption behavior of hair—improvement in hair humidity resistance or enhancement in hair moisture uptake.

A Hair Humidity Resistance Factor (H_2RF) has been defined. If $R_2HF > 1$, the product improves hair humidity resistance—anti-frizz; if $R_2HF < 1$, the product enhances hair water adsorption; when $R_2HF \sim 1$, the product has no significant effect on hair water adsorption behavior. This method was applied to evaluate anti-frizz performance of several shampoo formulations containing Polyquaternium-10, or Polyquaternium-70, or Polyquaternium-67, or Guar Hydroxypropyltrimonium Chloride. It was found PQ-70 shampoo showed the highest H_2RF value and the best anti-frizz performance among these tested shampoos. The results were consistent with those obtained from Image Analysis.

INTRODUCTION

It is well known that the tensile mechanical properties of hair fibers, with respect to fiber extension under small deformations (<2%), are related to the moisture level within the hair fiber. Water molecules are capable of penetrating into hair and plasticizing the components of hair fibers, and as such the force required for a given extension length is reduced with an increase in moisture content of hair.

The exhibited behavior of hair fibers in low and high humidity environment is markedly different. At low humidity the hair fiber demonstrates low plasticity/flexibility. Mechanical agitations such as combing, styling etc. can lead to significant fiber damage and hair breakage. Conversely, at high humidity the hair fiber may suffer the alternate extreme, that is the fiber becomes over plasticized and *limp* leading to poor hold/style retention. Especially, untreated naturally curly hairs are in frizzy condition and become unmanageable—"frizz" at high relative humidity. Therefore, reduction of the differences in mechanical properties of hair fibers at low and high relative humidity, i.e. remaining higher moisture content at low RH environment and reducing moisture uptake at high RH environment should lead to significant improvements in hair strength, texture, style retention and reduction of split ends.

A new test methodology has been developed to determine "Hair Moisturization/ Humidity Resistance." By measuring changes in hair elasticity with different equilibrium period at a constant RH, we are able to evaluate the penetrating rates of moisture into hair fibers. Comparing the average ratios of hair elasticity of a set of hair fibers at 50% and 80% RH before and after cosmetic treatments, we can define and determine the "Hair Humidity Resistance Factor" (HHRF).

EXPERIMENTAL

MATERIALS

Shampoo samples

1a. SH-1 Shampoo (formulation is listed in Table I)
1b. SH-1 Shampoo with additional 1% active Polyquaternium-70 (Croda Inc)
1c. SH-1 Shampoo with additional 0.5% active Guar Hydroxypropyltrimonium Chloride (Hercules)
1d. SH-1 Shampoo with additional 0.5% active Polyquaternium-67 (Dow)
1e. SH-1 Shampoo with 0.2% active Polyquaternium-10 (Dow)

Hair samples. Virgin dark brown and naturally curly hairs were purchased from International Hair Importers, Inc., New York [Lab bleached hair (30 minutes and 60 minutes bleaching time)].

TEST METHODS

Measurement of hair elasticity. Forces at 1% extension of single hair fibers were measured using a Dia-Stron MTT-670 (Dia-Stron Limited, UK) attached with an Autosampler, which was placed in an environmentally controlled chamber with a constant temperature of 23°C and a fixed relative humidity. The cross-sectional area of each tested hair fiber was determined by a Laser Scanning Micrometer LSM-5000 (Mitutoyo, Japan) and the data were transferred into MTTWIN software for calculations of elasticity of each hair fiber.

Image analysis. Hair tresses were placed in an environmentally controlled chamber at room temperature of 23°C and a fixed relative humidity for at least four hours for equilibration. Digital images of hair tresses were taken using a Sony DSC-717 digital camera.

Table I
SH-1 Shampoo Formulation

Ingredient	Weight (%)
Sodium lauryl sulfate (30.0% active)	23.33
Sodium loureth sulfate (30.0% active)	10.00
Cocamidopropyl hydroxysultaine	3.00
Cocamide DEA	2.00
Propylene glycol (and diazolidinyl urea (and) methylparaben (and) propylparaben	1.00
Deionized water	60.67

RESULTS AND DISCUSSION

CORRELATION BETWEEN WATER GAIN PERCENT AND RH

The water regain percent in hair samples was determined using a gravimetric method. Around 1.5 grams of hair sample were equilibrated at various RH levels for 8 hours and then weighed using an electronic analytical balance (precision 0.00001 gram) in a sealed weighing bottle. Plots of water gain (%) of hair samples vs. the environmental RH are presented in Figure 1. It is observed that the water regain percent of human hair increases with an increase in the environmental relative humidity (RH). The bleached hair showed more water regain than the virgin hair at the same level of RH and the water regain slightly increases with an increase in the bleaching time. This can be attributed to the increased hair hydrophilicity after oxidation. It is found that the hair water regain increases linearly with an increase in RH in the range of 40–85%, and the water regain increases more rapidly after 85% RH due to capillary condensation in the swollen fiber.

CHANGE IN HAIR ELASTICITY

It has been known that hair elasticity decreases with an increase in the environmental relative humidity. The plots of hair elasticity vs. environmental RH are presented in Figure 2. It is seen that the hair elasticity decreases linearly with an increase in the range of 50–80% RH and drops quickly after 80% RH. This is consistent with the results showed in Figure 1, which exhibits a rapid increase in water gain after 85% RH. Since the absorbed water molecules break and replace hydrogen bonding inside hair, which is responsible for approximately 50% of the hair elasticity. More water content inside hair, less hydrogen bonding between the coils of the alpha helix, smaller the elasticity of hair should be.

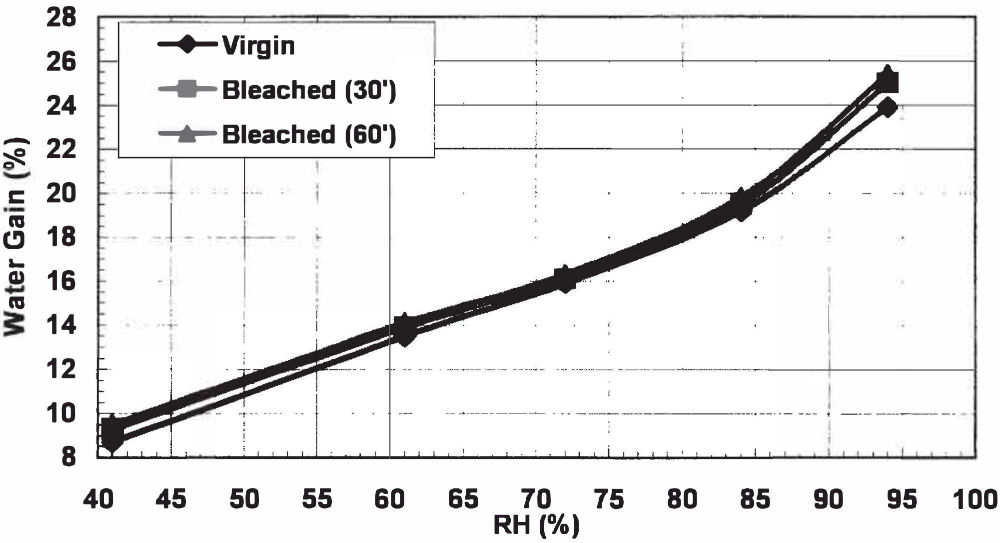


Figure 1. Correlation between water gain and RH for human hair.

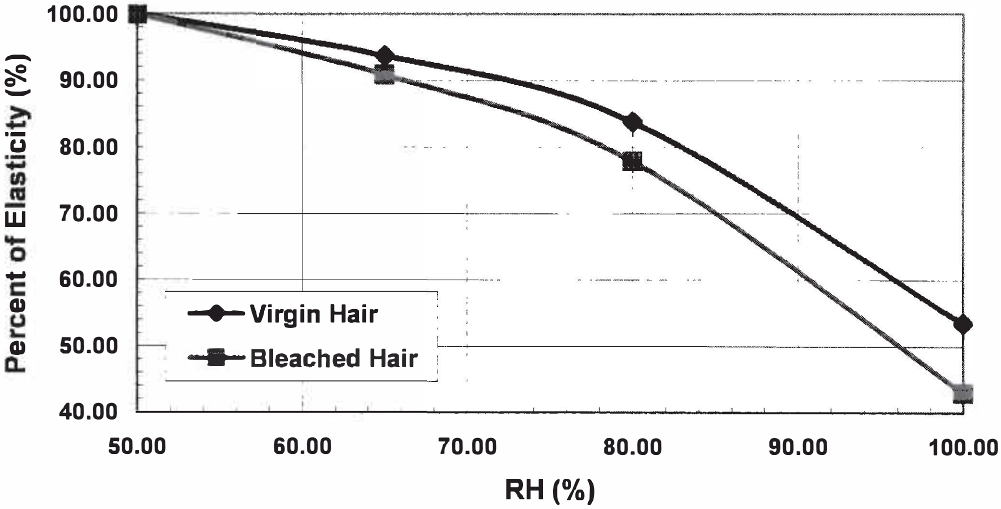


Figure 2. Percent changes in Young's modulus of human hair with RH.

Combining Figures 1 and 2, we find that the differences in hair elasticity at 50% and 80% RH can provide information about the change in water contents inside hair fiber. Larger the difference, more sensitive to the environmental RH the hair sample is. Smaller the difference, more resistant to the environmental RH the hair sample should be. Comparing these differences before and after cosmetic treatments on the same hair sample, we are able to evaluate effects of cosmetic treatments on hair water adsorption behavior—improvement in humidity resistance or enhancement in hair moisturization.

EFFECTS OF COSMETIC TREATMENTS ON HAIR ELASTICITY

Cosmetic treatments usually modify the hair surface and sometimes hair cortex as well. A lot of cosmetic treatments make hair more hydrophilic and improve hair moisturization—more water regain under the same RH than that before the treatment. Some cosmetic treatments make hair more hydrophobic and improve hair humidity resistance—less water regain under the same RH than that before the treatment. Few treatments may not show any significant effects on hair water adsorption behavior.

Typical force-extension curves of a single fiber at 50% and 80% RH, respectively, before and after a treatment with a personal care formula containing hydrolyzed wheat protein are presented in Figure 3. It can be seen that at 50% RH, the slope of the line, which corresponds to the hair elasticity, E(50), decreased from 18.35 to 16.49 after the treatment. The lower E(50) value of the treated hair fiber indicated higher water content inside the hair compared to that of the same fiber before the treatment. This suggests that the treatment protected the hair fiber from loss of moisture under 50% RH. It is also found that at high RH of 80%, hair elasticity, E(80) increased from 10.05 to 12.87 after the treatment. This implied that the treated hair fiber absorbed less water under 80% RH compared to that of the same untreated hair fiber, and the treatment protected the hair from gain of moisture under high humidity. The differences in elasticity of the

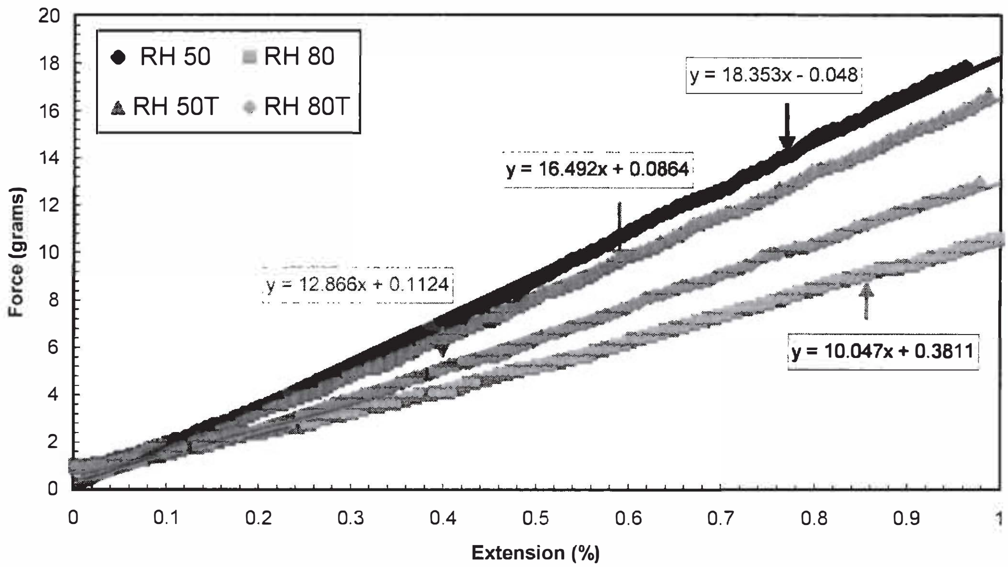


Figure 3. Change in Young's modulus of a hair fiber after a cosmetic treatment.

hair fiber at 50% and 80% RH were 7.85 (18.35–10.05) before the treatment and 3.62 (16.49–12.87) after the treatment, respectively. This difference was reduced after the treatment, and it indicated that the treatment improved hair moisture balance and provided hair humidity resistance.

It is assumed that the determined difference in hair elasticity under the same RH at a fixed equilibrium time period (from 15 minutes to 4 hours) before and after cosmetic treatments is mainly attributed to the change in hair moisture content. Therefore, by comparing differences between the average hair elasticity at 50% and 80% RH before and after cosmetic treatments, we are able to evaluate effects of cosmetic treatments on the hair: hair moisturization or hair humidity resistance (anti-frizz). If the average difference between hair elasticity at 50% and 80% RH increases after a cosmetic treatment, this indicates that the difference in moisture contents inside hair fibers becomes larger, and the cosmetic treatment enhances hair moisturization. If the difference between hair elasticity at 50% and 80% RH decreases after a cosmetic treatment, it implies that the difference in moisture contents inside hair fibers turns smaller, and the cosmetic treatment improves hair humidity resistance.

We may use another way to measure these changes in hair elasticity. As seen in Figure 3, the remaining percent (ratio) of hair elasticity (the slope of the force-extension curve) at 80% RH to the one of the same hair fiber at 50% RH (10.5/18.35 = 57.2%) was greatly reduced after the cosmetic treatment (12.87/16.49 = 78.0%). The value of this ratio reflects the effect of the environmental RH on hair elasticity at a fixed equilibrium time period. Higher the ratio, less effect of the environmental RH on hair elasticity, and better the humidity resistance. Lower the ratio, stronger effect of environmental RH on hair elasticity, and easier the hair moisturization. Therefore, comparing these ratios of hair elasticity at 50% to that at 80% RH before and after cosmetic treatments of hair samples, we are able to evaluate the effects of cosmetic treatments on hair water adsorption behavior—Hair Humidity Resistance/Moisturization

HAIR HUMIDITY RESISTANCE FACTOR (H_2RF)

Definition of experimental parameters

1. RE_0—The average remaining percent (ratio) of hair elasticity of 25 single fibers at 80% RH compared to that at 50% RH before cosmetic treatment at a fixed equilibrium time:

$$RE_0 = \text{Average } [100\% \times E_{0i}(80)/ E_{0i}(50)] \qquad i = 1, 2, 3 \ldots 25$$

2. RE_1—The average remaining percent of hair elasticity (ratio) of the same 25 single fibers at 80% RH compared to that at 50% RH after cosmetic treatment at the same equilibrium time

$$RE_1 = \text{Average } [100\% \times E_{1i}(80)/E_{1i}(50)] \qquad i = 1, 2, 3 \ldots 25$$

3. HHRF (H_2RF)—Hair Humidity Resistance Factor

$$H_2RF = 100\% \times RE_1/RE_0$$

H_2RF is a measure of changes in the remaining percent of hair elasticity before and after cosmetic treatment. A t-test should be performed to determine if two mean values of a pair of experiments (RE_0 and RE_1) are significantly different.

When $H_2RF > 1$, it means that cosmetically treated hair samples, which were equilibrated under an environmental 80% RH for a fixed time, remained higher percentage of the original elasticity (under 50% RH) compared to that before the treatment during the same equilibrium time period. This indicates that less moisture was capable of penetrating into hair fibers under 80% RH in the same equilibrium time—the treatment improved the humidity resistance.

When $H_2RF < 1$, it indicates that cosmetically treated hair samples, which were equilibrated under 80% RH for a fixed time, remained lower percentage of the original elasticity (under 50% RH) compared to that before the treatment during the same equilibrium time period. This implies that more moisture was capable of penetrating into hair fibers under 80% RH in the same equilibrium time—the treatment enhanced the moisture penetration (hair moisturization). When $H_2RF \sim 1$, it shows that after the cosmetic treatment and during the same equilibrium time, no significant change in hair moisture adsorption behavior was observed.

Evaluation of H_2RF of hair samples washed with different shampoos. Figure 4 shows the average remaining percent of elasticity of hair fibers before and after treatment with a Croda standard shampoo SH-1 at various equilibrium times. No significant change in the remaining elasticity of hair fibers was observed before and after the shampoo washes during different equilibrium times. The hair lost about 20% of their initial elasticity after 15 minutes under 80% RH environment, and the hair elasticity remained at a constant value between 15 and 60 minutes of equilibrium time. The results indicate that the moisture content inside hair might reach a saturated level in about 60 minutes under 80% RH. The similarity of two curves in Figure 1 indicates that no significant change occurred in hair water adsorption behavior after hair was washed with SH-1 shampoo.

Figure 5 explains the change in remaining elasticity of hair washed with SH-1 shampoo containing additional 1% active Polyquaternium-70. It can be seen that the average remaining percent of hair elasticity under 80% RH after the treatment with the Polyquaternium-70 containing shampoo is always higher than that before treatment in all different equilibrium time periods. This indicates that after the shampoo treatment

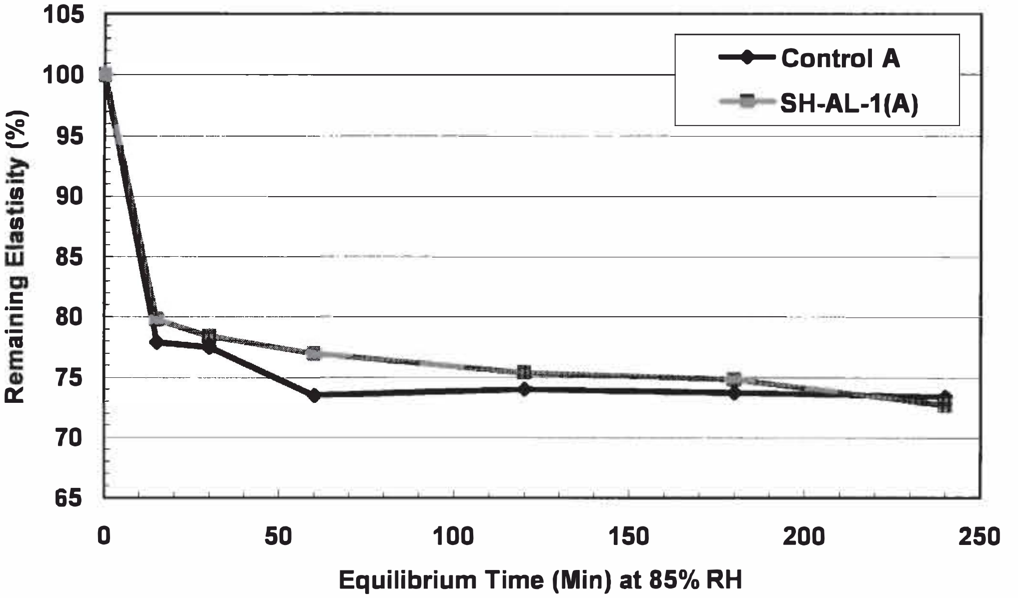


Figure 4. Remaining elasticity of hair washed with SH-AL-1 shampoo.

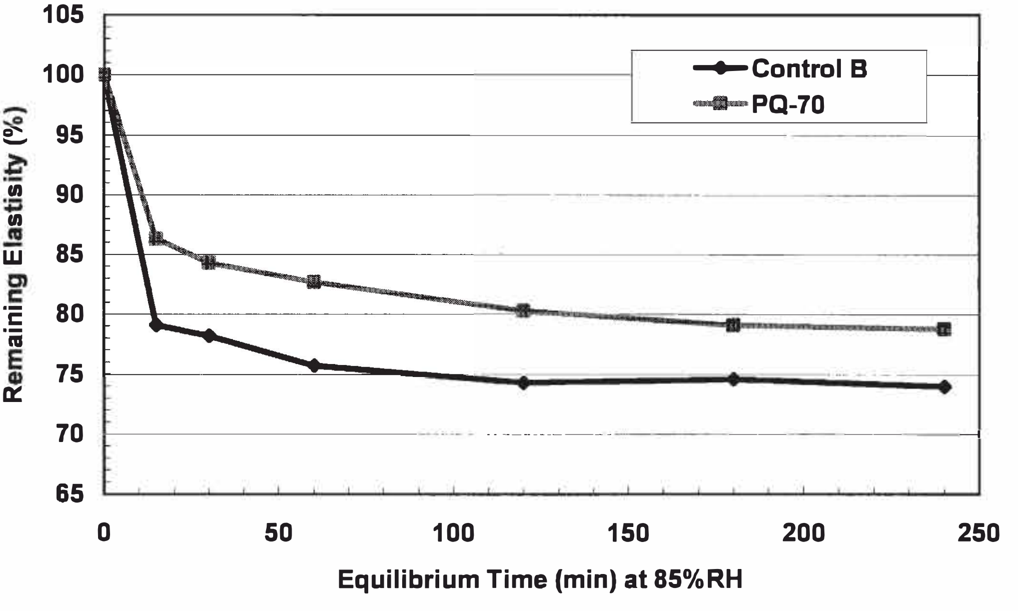


Figure 5. RE of hair washed with PQ-70-containing shampoo.

less moisture is capable of penetrating into hair under 80% RH. Based on the experimental results it may be concluded that addition of 1% of Polyquaternium-70 into SH-1 shampoo improved its humidity resistance performance.

Five shampoo samples were evaluated and their hair humidity resistance factors at different equilibrium time were calculated and presented in Figure 6. It is seen that only

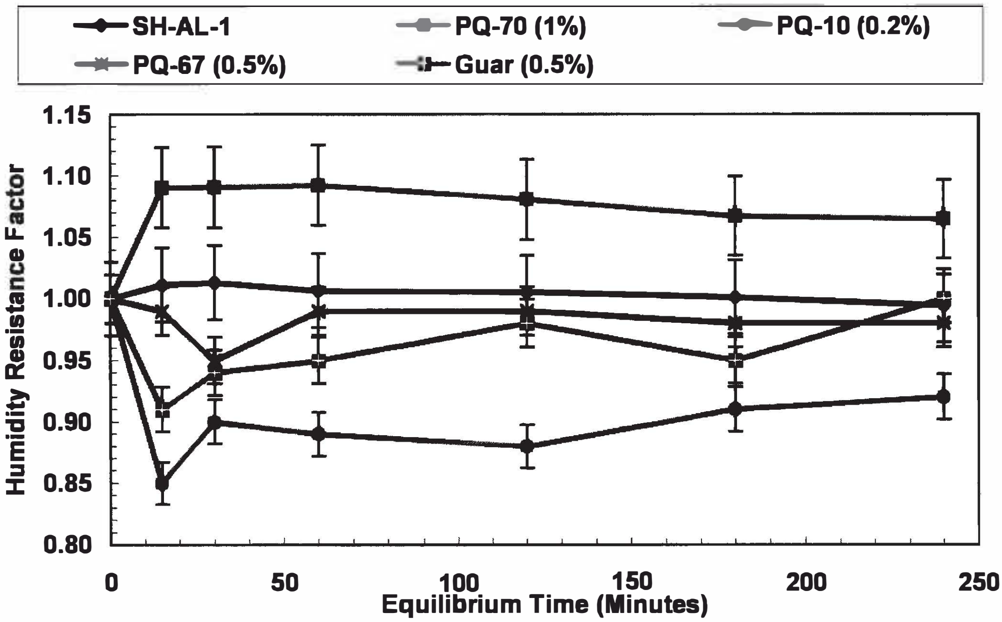


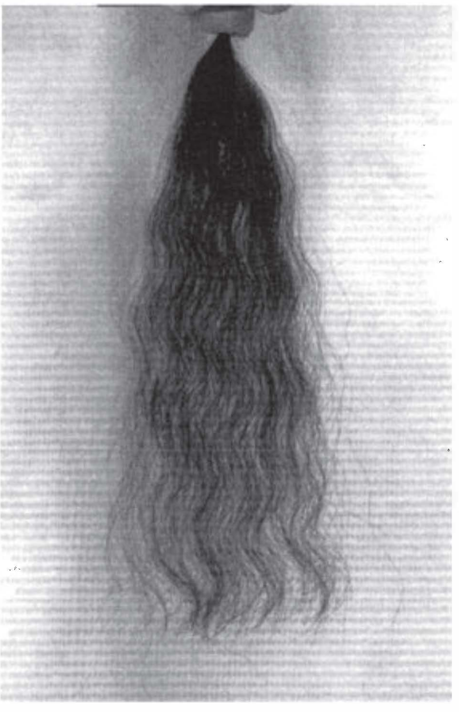
Figure 6. H_2RF of different shampoos at various equilibrium times.

the shampoo containing PQ-70 showed higher humidity resistance factors. Even after 4 hours of equilibrium time, the remained elasticity is still 6% higher than that of the untreated hair. Shampoos containing PQ-67 or Guar did not show any benefits on hair humidity resistance. Hair treated with the shampoo containing PQ-10 showed decrease in hair remaining elasticity; this is an indication of hair moisturization. Unfortunately, the moisturized hair exhibited higher wet combing force due to the absorption of extra water.

HAIR IMAGES OF FRIZZY HAIR AT DIFFERENT RH

Digital images of frizzy hair tresses were taken at 50% and 80% RH and presented in Figure 7. It is clearly shown that there were significant changes in hair volume and curvature of a fizzy hair sample due to an increase in the environmental humidity. In order to understand the mechanism of hair frizz, the average ellipticity (the ratio of maximum diameter to minimum diameter of a hair fiber) of different types of hair fibers was determined using a Laser Scanning Micrometer LSM-5000. It is observed that naturally curly and kinky hairs have higher average ellipticity than other types of hair such as oriental hair and Caucasian hair. Data are presented in Table II. Due to the large differences in two main diameters on hair cross-section, moisture has pronounced effects on hair curvature and volume, and therefore, naturally-curly and kinky hair is the frizziest hair among all types of hairs.

Image Analysis is a very useful tool to see the difference of hair under various environmental RH and used for evaluations of anti-frizz performance of hair care formulation by comparing images of hair samples at different humidity before and after cosmetic treatments. Figure 8 shows images of a frizzy hair tress at 50% and 80% RH before and



7-1 Untreated frizzy hair at 50%RH

7-2 Untreated frizzy hair at 80%RH

Figure 7. Change in the appearance of a frizzy hair sample.

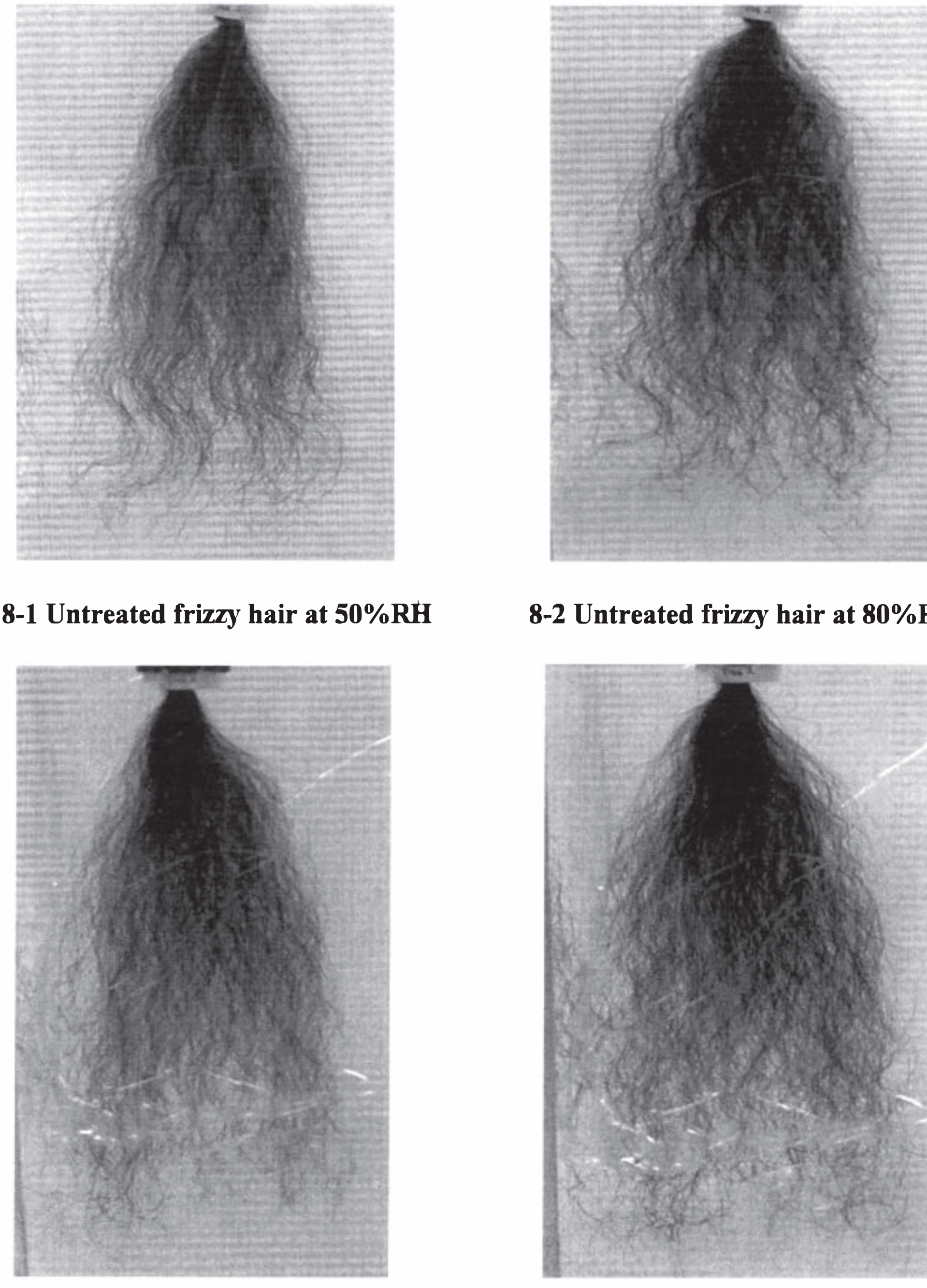
Table II
Shape and Ellipticity of Different Types of Hairs

Hair type	Caucasian	Black	Oriental	Hispanic
*CSD, μm (Average)	29–96 (70)	46–120 (90)	36–125 (92)	30–120 (70–92)
Ellipticity	1.17–1.41	1.68–1.98	~1.25	1.17–1.98
Shape of hair shaft	Oval	Flat	Round	Oval-flat-round
Shape	Straight to wavy/curly	Wavy to very curly	Straight to wavy	Straight/wavy to very curly

* CSD: Cross-sectional diameter.

after treatment with a PQ-70-containing shampoo. Before the shampoo treatment, the hair tress became volume-expanded and curlier when the RH increased from 50% to 80%. After the shampoo treatment, the changes in hair volume and curvature were reduced.

Figure 9 shows images of a frizzy hair tress at 50% and 80% RH before and after treatment with a PQ-10-containing shampoo. Before the shampoo treatment, the frizzy hair tress became volume-expanded and curlier when the RH increased from 50% to 80%. After the shampoo treatment, the changes in hair volume and curvature with an increase in the environmental humidity did not show any improvement. Comparing theses images, it can be concluded that the PQ-70 shampoo did show better anti-frizz performance compared to other tested shampoo formulations.



8-1 Untreated frizzy hair at 50%RH

8-2 Untreated frizzy hair at 80%RH

8-3 PQ-70 treated hair at 50%RH

8-4 PQ-70 treated hair at 80%RH

Figure 8. Images of PQ-70-shampoo-treated frizzy hair at different RH.

9-1 Untreated frizzy hair at 50%RH

9-2 Untreated frizzy hair at 80%RH

9-3 PQ-10 treated hair at 50%RH

9-4 PQ-10 treated hair at 80%RH

Figure 9. Images of PQ-10 shampoo-treated frizzy hair at different RH.

CONCLUSIONS

- A new parameter—Hair Humidity Resistance Factor (H_2RF) has been defined and can be determined by measuring changes in the remaining percent of hair elasticity at two levels of RH (50% and 80%) at different equilibrium time.
- H_2RF can be used as indicators of hair anti-frizz behavior as well as hair moisturization degree.
- Addition of 1% active Polyquaternium-70 into SH-1 shampoo improved its hair anti-frizz performance and demonstrated the best performance on hair humidity resistance among all tested shampoo samples
- An Image Analysis method was used to evaluate anti-frizz performance of hair care products. The improvement in hair anti-frizz performance by addition of 1% active Polyquaternium-70 into SH-1 shampoo system has been validated by image Analysis method.

ACKNOWLEDGMENT

The author thanks the application group of Croda Inc. for preparations of shampoo samples.