

A comparison of dyeing efficacy between hair-oxidation-based and hair-coating-based shampoos for the treatment of gray hair

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Abstract

Background: The process of hair dyeing causes hair damage, and periodic re-dyeing is required for newly grown hair. To avoid these hassles, hair color shampoos have been developed and are widely used. In this study, we compared the effects of two hair color shampoos with different dyeing principles to analyze the function of hair color shampoos. We analyzed hair tresses treated by hair-oxidation- and hair-coating-based shampoos.

Materials and methods: We measured the color, tensile properties, softness, elasticity, gloss, moisture content, and protein content of the hair tresses dyed with color shampoos. The hair structures were analyzed by scanning and transmission electron microscopies (SEM and TEM) and a hydroxy radical-based method.

Results: The shampoo based on hair coating enhanced the hair dyeing effect and roughness, whereas that based on hair oxidation improved the color retention and moisture content in the hair tresses. Frictional resistance, gloss, and elasticity of the hair tresses were similar for the two products. However, according to the results of the protein loss test, TEM, and hydroxyl radical staining, the shampoo based on hair oxidation showed a longer dyeing retention compared to that based on hair coating but caused cuticle damage.

Conclusion: These results show that the two shampoos with different dyeing principles exhibit different hair dyeing abilities and hair health indices. Therefore, we recommend that hair color shampoos should be used according to the requirements of an individual.

KEYWORDS

cuticle damage, dyeing effect, hair health, hair-coating-based shampoo, hair-oxidation-based shampoo

You Na Jang and Joon Seok contributed equally to this work.

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1 | INTRODUCTION

Artificial treatment of hair, including dyeing, bleaching, straightening, and perming, enhance the quality of life and enhance beauty.¹ However, artificial hair treatments (such as chemical and physical treatments) cause significant hair damage,^{2,3} which leads to the breakdown of the keratin protein and loss of elasticity and strength in the hairs. As a result, the hairs eventually become dry, rough, firm, and brittle as well as lose color.^{4–6}

A majority of the population uses permanent dyes to cover gray hairs.⁷ However, in this case, re-dyeing is required periodically and repeatedly after 4–6 weeks for newly grown hair⁸, indicating that dyeing is a time-consuming and costly process. Moreover, permanent dyes based on chemical reactions penetrate the cuticle and cortex of the hair and cause contact dermatitis, hair damage, scalp irritation, and a pungent odor.^{9,10} As a result, the demand for a safe procedure to restore damaged hairs is growing along with the requirement for alternative products for permanent dye.

Shampoos are likely the most commonly used cosmetic products for hair and scalp cleansing in our daily lives. Shampoo is a solution of a detergent containing appropriate additives for benefits, such as hair conditioning improvement, and medication. Currently, research is ongoing to develop shampoos for dyeing that are time-saving and relatively safe. As a result, many companies have launched various versions of hair color shampoos that can cover gray hair and reduce dyeing time.

In this study, we characterized and compared the effects of two hair color shampoos and follow different dyeing principles using visual, mechanical, and chemical indicators.

2 | MATERIALS AND METHODS

2.1 | Hair samples

For this study, mixed source Chinese hair tresses in ivory and black colors were obtained. Note that, 5 g of 24-cm-long ivory-colored hair tresses were purchased from CROWN HAIR (Korea) and used to measure the dye and retention capacities. Further, 2 and 6 g of 24-cm-long black-colored hair tresses were purchased from CROWN HAIR (Korea) and used to assess hair health. The samples were stored in the dark at room temperature prior to the analyses.

2.2 | Shampooing

The hair tresses were shampooed with a hair-coating-based shampoo (Double Effector Black, Amore Pacific, Korea) and a hair-oxidation one (Pro-change Black, Modamoda, Korea); a hair-coating shampoo contains ingredients such as black soybean extract, ginseng root cell extract, and black yeast fermentation product to the coat hair surface, and a hair-oxidation shampoo contains ingredients such as tannic acid, caffeine, and green tea extract to induce oxidation of hair surface. The shampooing was performed according to the manufacturer's

protocol. First, we applied a coin-sized amount of shampoo to the wet hair tresses. After foaming, the hairs were left undisturbed for approximately 3 min and subsequently rinsed with lukewarm water.

2.3 | Color measurement

The color was determined using a colorimeter (CR-10; Konica Minolta Sensing Inc., Osaka, Japan). The measurements were performed in triplicate at the top, middle, and bottom of each strand. The total change in the color of the hair samples was determined as a difference in brightness (ΔL ; + lighter, –darker).

2.4 | Tensile properties

The tensile properties of the hair tresses, before and after using the test products, were measured using the Generic Tensile Tester MTT175 (DIA STRON, UK) system. For the tensile test, a force was applied on the hair fibers at a constant speed, and their resistance to this applied force was measured in grams mass force (gmf). An increase in the gmf value indicated improved hair strength (i.e., minimization of breakage).

2.5 | Hair softness

The softness of the hair tresses was measured using the Generic Tensile Tester MTT175 (DIA STRON, UK) system before and after using the test products. First, we fixed one bundle of the hair tresses on a fixed plate and subsequently applied a load (50 g) on the hairs using the device. The measurements were performed three times with a certain applied force, and the average gmf of the first to the third cycle was obtained as the measurement result. A decrease in the gmf value indicated an effective improvement in hair softness.

2.6 | Hair elasticity

The elasticity of the hair tresses was measured using the Generic Tensile Tester MTT175 (DIA STRON, UK) system before and after using the test products. A round-shaped bundle of hair fibers was fixed on the fixed plate in the system, and a certain force was applied to it; the force data were subsequently evaluated to assess the elasticity of the tresses. The measurements were performed thrice (from the first to the third cycle), and the resulting gmf values were averaged. An increase in the gmf value indicated an increase in hair elasticity, which revealed the effectiveness of the test product.

2.7 | Hair gloss

The hair gloss measurements were performed using SAMBA Hair (Bossa Nova Technologies, USA) on the same hair tresses before and after using the test products. For the analysis, a built-in program was

used to measure the luster parameter (L_{BNT}) value, which represents hair gloss, using the following equation:

$$L_{\text{BNT}} = \frac{S_{\text{in}}}{(D + S_{\text{out}}) \times W_{\text{visual}}}$$

where L_{BNT} is the luster parameter (BNT: Bassa Nova Technologies), S_{in} is the peak of the specular light and increases the luster, S_{out} corresponds to the wings of the specular light (large angles) and decreases the luster, D is the total amount (integral) of diffused light, and W_{visual} represents the visual width of the distribution. An increase in the L_{BNT} value indicated an improvement in the glossiness of the hair tresses.

2.8 | Hair moisture

The moisture content in the hair tresses was measured using an infrared moisture meter (FD-660; Korea Kett Co. Ltd., Japan) before and after using the test products. The human hair tresses were cut at 2 cm intervals, and 0.5 g of each cut bundle was dried by heating at 105°C for 30 min using an infrared moisture meter. An increase in the moisture content (%) indicated that the test product was effective in improving the moisture content of the hairs.

2.9 | Protein loss test

Exactly 100 mg of the hair tresses were placed in a tube with 2.0 ml of the radioimmunoprecipitation assay (RIPA) buffer. The soluble protein was extracted from the hair fibers by centrifuging the tube at 125 rpm for 10 s. Further, protein quantification using the Bradford assay was performed on 0.1 ml of the supernatant.

2.10 | Scanning electron microscopy

The surface of the hair tresses was investigated by scanning electron microscopy (SEM; SIGMA 300). For the SEM analysis, the hair tresses were glued to specific support using carbon tape and then coated with platinum. All the micrographs were collected at an accelerating voltage of 5 kV.

2.11 | Roughness of hair surface

The roughness of the hair tresses was quantified by SEM image analysis using the Image J 1.52 program (NIH, Bethesda, MD, USA), which calculated the arithmetical mean roughness (R_a) value.

2.12 | Transmission electron microscopy

The hair tresses were further analyzed by transmission electron microscopy (TEM; FEI Tecnai F20 G2) at an accelerating voltage of

50–200 kV. For the TEM analysis, a hair strand was immersed in propylene oxide for 15 min and processed with propylene oxide and Epon mixture (1:1 ratio) overnight. Subsequently, the hair strand was embedded in an Epon mixture and analyzed by TEM.

2.13 | Detection of hydroxy radicals on the hair surface

The generation of hydroxy radicals on the surface of the hair tresses was examined using a hydroxy-radical-detection reagent hydroxyphenyl fluorescein. Hair tresses after shampooing were cut into a length of 0.5 cm and hair tresses for positive control were exposed to ultraviolet (UV) radiation for 20 min using BIO-SPECTRA (Vilber Lourmat, Marne-la-vallet'e France). After the procedure, 20 μl of 1 $\mu\text{mol/L}$ hydroxyphenyl fluorescein (HPF) acid was added, and the hair tress was observed under a fluorescence microscope (NIBA; excitation (Ex.) wavelength: 470–490 nm; emission (Em.) wavelength: 510–550 nm; Olympus Corporation, Tokyo, Japan). Next, 20 μl of the 1 $\mu\text{mol/L}$ HPF acid was added to the shampooed hair tresses, without performing the UV irradiation step, and these shampooed tresses were then observed under the fluorescence microscope.

2.14 | Statistical analysis

All the statistical analyses were performed using the GraphPad Prism 7.0 software (GraphPad Software Inc., San Diego, CA, USA), and the statistical significance was assessed by Student's *t*-test analysis of the variance; the statistical significance was set as a two-tailed *p*-value < 0.05.

3 | RESULTS

3.1 | Color-changing capacities of the hair-oxidation and hair-coating-based shampoos

To investigate the hair dyeing effect of the two hair color shampoos, the hair tresses were shampooed with them, and the dyed tresses were imaged with a DSLR camera and folliscope for visual evaluation. The tresses were shampooed one, five, and 10 times, and the dyeing effect was evaluated for each of these cases. According to the DSLR and folliscope image, the darkening effects of the two hair-oxidation and surface-coating-based shampoos depend on the shampooing times (Figure 1A,B). To quantify the hair dyeing effect, a colorimeter was used, and the dyeing effect was determined by the ΔL color scale, which indicates the light or dark level of the hair color. The ΔL values decrease as the darkening progresses. Further, the ΔL values of the shampoos based on hair oxidation and surface coating decrease depending on the shampooing times. Especially, the ΔL values of the hair tresses treated with the shampoo based on hair coating are lower than those of the tresses treated by the shampoo based on hair

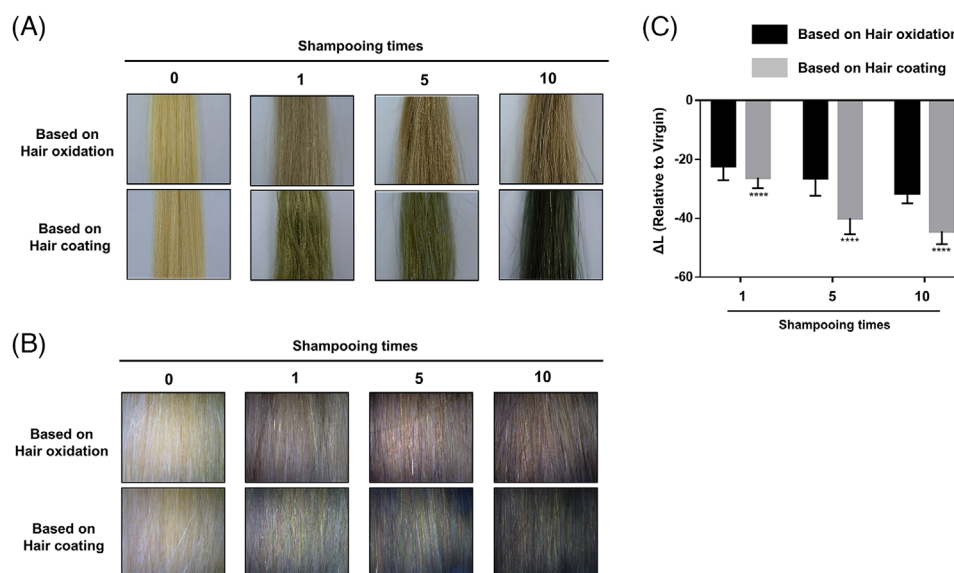


FIGURE 1 Hair dyeing effect of the hair color shampoos. (A) DSLR image, (B) folliscope image, and (C) ΔL values of the hair tresses shampooed one, five, and 10 times with the hair-oxidation and hair-coating-based shampoos. * $p < 0.05$, ** $p < 0.01$, and *** $p < 0.001$ are determined from an unpaired t -test.

oxidation (Figure 1C). These results demonstrate that the hair dyeing effect of the hair-coating-based shampoo is stronger than that of its hair-oxidation-based one.

3.2 | Color retention capacities of the hair-oxidation and surface-coating-based shampoos

To evaluate the hair-dyeing retention effect of the two hair color shampoos, the hair tresses that were shampooed 10 times and then washed the same number of times were visually evaluated using the DSLR and folliscope images. The evaluation results presented in Figure 2A,B show that the shampoo based on hair oxidation maintains its dyeing effect on the hair tresses, whereas that based on hair coating increases their brightness. Further, the brightness of the hair tresses, treated by the hair-oxidation-based shampoo, decreases by $-2.5 \Delta L$, whereas that of the tresses treated by the coating-based shampoo increases by $8.3 \Delta L$ (Figure 2C). These results suggest that the hair-dyeing retention capacity of the shampoo based on hair oxidation is higher than that of the hair-coating-based one.

3.3 | Hair health effect caused by the hair-oxidation and coating-based shampoos

Hair health is affected by several factors, such as deficiency of proper nutrients, UV exposure, and artificial treatments (drying, perming, and dyeing). Damaged hair shows a decrease in gloss, strength, elasticity, moisture capacity, and hair protein, while its frictional resistance increases significantly. Therefore, we measured the tensile strength, softness, elasticity, glossiness, moisture, and protein content of the hair

tresses to determine the damage after 10 times of shampooing. Evidently, both the shampoos (one based on hair oxidation and the other based on surface coating) increase the tensile strength (Figure 3A) and reduce the frictional resistance, although this effect is insignificant (Figure 3B). Further, as shown in Figure 3C,D, both increase hair elasticity and glossiness; however, the increase in glossiness is statistically insignificant. Moreover, the shampoo based on hair oxidation increases the moisture content but decreases the protein content in the hair as evidenced by the results presented in Figure 3E,F. These results collectively show that dyeing with shampoos based on hair oxidation and those based on surface coating have different characteristics.

3.4 | Comparative structural analysis of the shampooed hair tresses

SEM and TEM imaging were performed to confirm the hair surface, cuticle, and cortex damage. In the SEM analysis, we examined the surface of the hair tresses that were shampooed 10 times. In the virgin hair tresses, the cuticles were regularly spaced, overlapped with multiple layers, and exhibited a round shape with almost no surface damage (Figure 4A). The side of the hair tress also maintained a straight shape. In addition, when both shampoos were used, the distance between the hair cuticles decreased, and the surface appeared undamaged. The SEM image reveals that the shampoo based on hair coating reduces the roughness of the hair tresses (Figure 4B). Furthermore, TEM analysis was conducted to assess the damage to the cuticle and cortex of the hair tresses after 10 times of shampooing with both products. Compared to virgin hair tresses, no noticeable changes are observed in the tresses treated by shampoo based on hair coating. However,

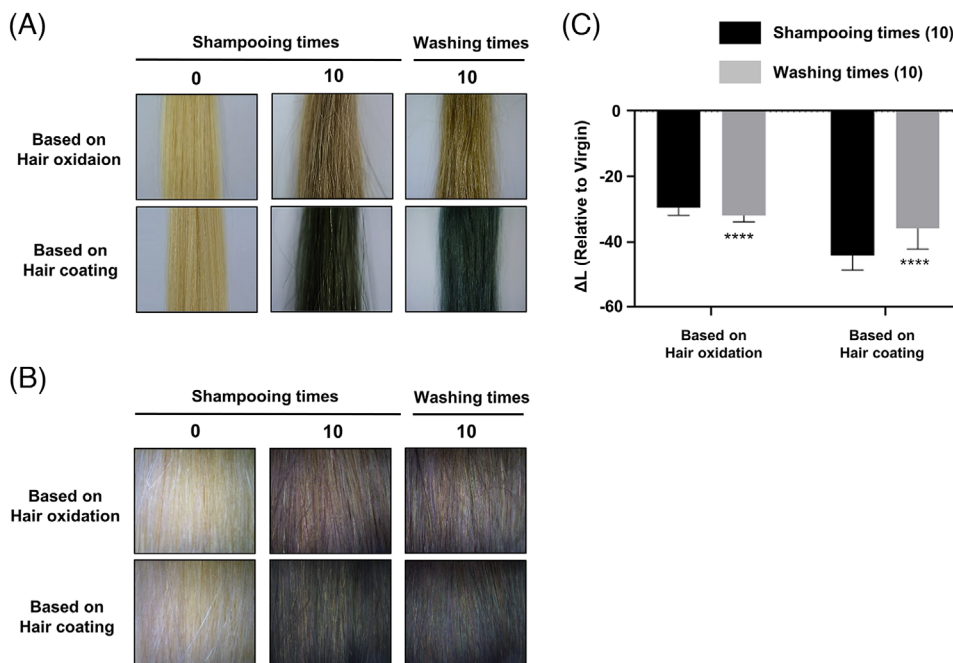


FIGURE 2 Hair dyeing retention capacity of the hair color shampoos. (A) DSLR image, (B) folliscope image, and (C) ΔL values of the hair tresses washed 10 times after 10 times of shampooing with the two hair-oxidation and coating-based shampoos. * $p < 0.05$, ** $p < 0.01$, and *** $p < 0.001$ are determined from an unpaired *t*-test.

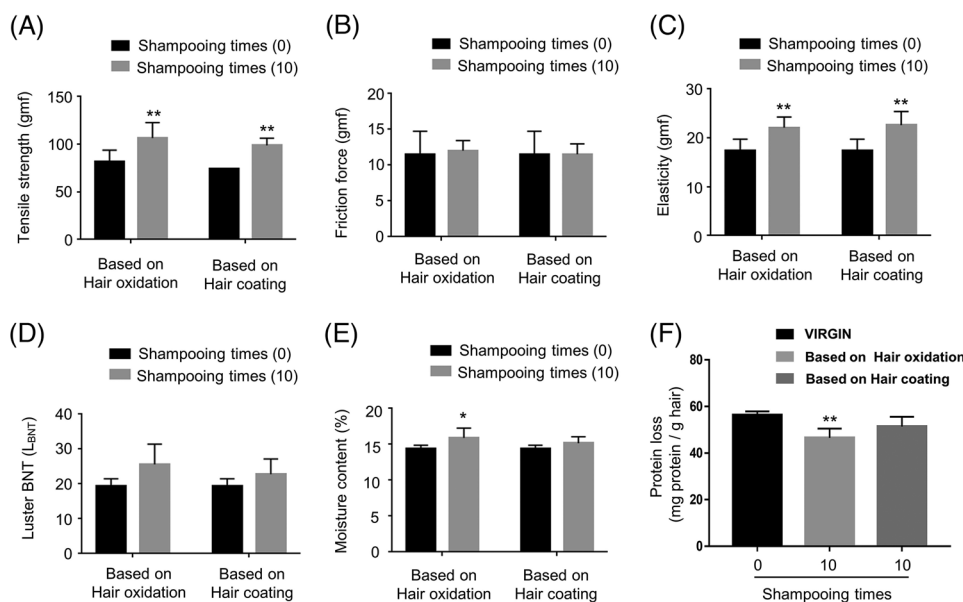


FIGURE 3 Hair health indicator analysis of the hair color shampoos. (A) Tensile strength, (B) frictional force, (C) elasticity, (D) L_{BNT} values, (E) moisture content, and (F) protein loss of the hair tresses that were shampooed 10 times using the shampoos based on hair oxidation and hair coating. * $p < 0.05$, ** $p < 0.01$, and *** $p < 0.001$ are determined from an unpaired *t*-test.

punched-out cuticles appear in the TEM image of the tresses treated by the hair-oxidation-based shampoo (Figure 4C).

Hydroxyl radicals are a form of active oxygen, which is intensely toxic for cells. These radicals are reportedly produced on hair and skin surfaces by the reaction between ferrous oxide (cuprous oxide)

and hydrogen peroxide and that between tyrosinase and phenol compounds.¹¹ In our results, only a negligible number of hydroxyl radical production sites were detected on the surface of the virgin and shampooed (using the shampoo based on hair coating) tresses. In contrast, the surface of the hair exposed to UV radiation and the shampoo

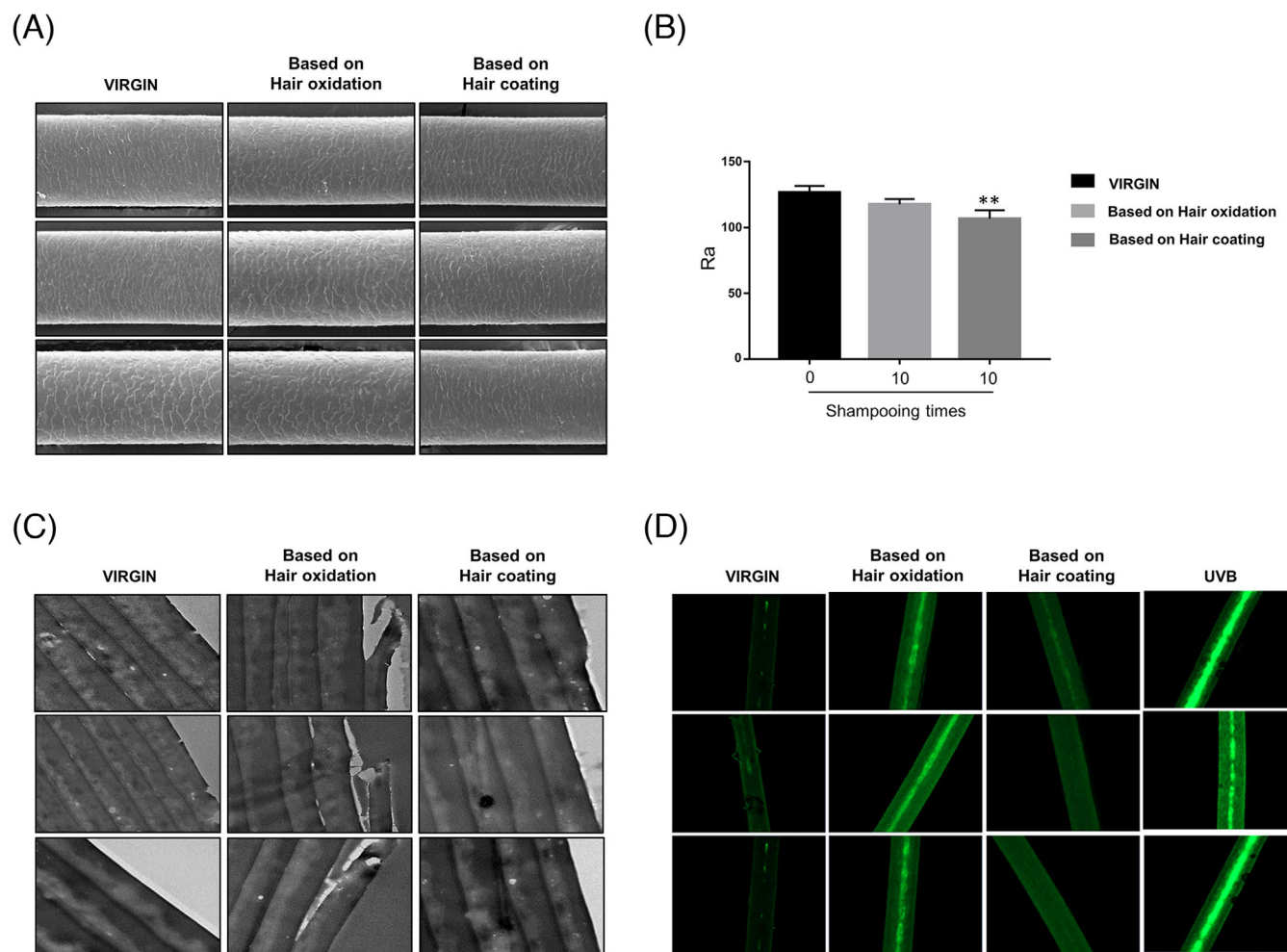


FIGURE 4 Structural evaluation of the shampooed hairs. (A) Hair surface damage and roughness measured by scanning electron microscopy (SEM), (B) The arithmetical mean roughness (R_a) value was calculated, (C) Cuticle layer damage measured by transmission electron microscopy (TEM), (D) Fluorescence microscopic images showing the fluorescence emission caused by the hydroxy radicals in the hair tresses. The hair tresses used in the SEM, TEM, and fluorescence spectroscopy analyses were shampooed 10 times with the two hair oxidation and coating-based shampoos. * $p < 0.05$, ** $p < 0.01$, and *** $p < 0.001$ are determined from an unpaired t-test.

based on hair oxidation showed distinct hydroxy radicals (Figure 4D). These results indicate that the hair-oxidation-based shampoo damages the hair surface by releasing hydroxy radicals.

4 | DISCUSSION

Hair graying, resulting from the complex regulation of melanogenesis, is a common sign of aging and occurs irrespective of gender or race. Since no medical treatment is available for hair re-pigmentation,¹² hair dyeing is the only alternative method of covering gray hair. However, permanent hair dyes cause numerous side effects. To overcome these problems, color shampoos are manufactured and marketed as viable alternatives.

Among the various hair color shampoos, those based on hair coating or oxidation are widely used. The detailed mechanism of the two shampoos can be determined from their respective manufacturer's

methods. The shampoos based on hair coating contain black toning ingredients such as black ginseng, black beans, and arrowroot, which adhere to the hairs (like a magnet) and gradually cover the gray hairs, thereby darkening them. Contrarily, the hair-oxidation-based shampoos contain a complex antioxidant, which reacts with oxygen and sunlight during the shampooing process and forms a film on the cuticle layer of the hairs; this gradually darkens the gray hairs to a natural dark brown color similar to the browning phenomenon.

Because several factors need to be considered while coloring, we evaluated the effects of the hair color shampoos by different analytical methods.¹³ The results indicated that the hair-coating-based shampoos enhance the hair-dyeing effect and roughness, whereas those based on hair oxidation improve the color retention in the hair tresses. Furthermore, both shampoos protect hairs by preventing moisture loss, but only the hair-oxidation-based shampoo enhances hair moisturization. The hair tresses treated by both products showed

TABLE 1 Changes in the hair health indices of the hair tresses treated by the shampoo based on hair oxidation and that based on the surface coating.

Hair Index	Shampoo based on hair oxidation	Shampoo based on hair coating
Dyeing power (ΔL)	1 time : -22.14	1 time : -26.09
	5 times : -26.53	5 times : -39.96
	10 times : -31.46	10 times : -44.30
Color retention (ΔL)	+2.45	-8.27
Tensile strength (gmf)	+16.57	+18.61
Frictional resistance (gmf)	+0.49	+0.00
Hair elasticity (gmf)	+4.69	+5.18
Glossiness (LBNT)	+6.29	+3.50
Moisture content (%)	+10.84	+5.63
Protein content (%)	-17.38	-8.75
Surface roughness (Ra)	-8.91	-19.91
Fluorescence intensity (A.U.)	+35.30	+19.66

similar frictional resistances, gloss, and elasticity. However, the TEM and protein-loss test results reveal that the hair damage caused by the hair-oxidation-based shampoo is more evident than that caused by its coating-based one. The results of the hydroxyl radical staining experiments confirm that the shampoo based on hair oxidation can induce radicals on the hair tresses and cause hair damage. The hair health indicators of the shampooed tresses are summarized in Table 1.

5 | CONCLUSION

These two hair color shampoos based on different dyeing principles have different advantages and disadvantages related to their dyeing effects and retention capacities. Thus, based on the results of this study, we recommend that the users should use a hair color shampoo according to their specific requirements.

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CONFLICT OF INTEREST STATEMENT

The authors declare no conflict of interest.

DATA AVAILABILITY STATEMENT

None.

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