WHITE PAPER

Determining the Effect on Caucasian Hair When Glycolic Acid Is Used in Hair Care Products

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Overview

Glycolic acid is among the most popular actives in anti-aging formulations and has strong consumer recognition as an ingredient in many anti-aging skin care products. Extensive clinical data show that glycolic acid penetrates the skin effectively and stimulates cellular activity, supporting its use in a wide range of skin care products, including: cell turnover/exfoliation products; moisturizing skin lotions and creams; wrinkle reduction creams; and anti-aging creams.

Although the positive effects of using glycolic acid in skin care products are well documented, similar research did not exist for the effects of using glycolic acid in hair care products. That's why DuPont, the manufacturer of Glypure®, a cosmetic-grade glycolic acid used in many cosmetics and personal care products, investigated the role glycolic acid could play in the formulation of hair care products.

This paper provides background information about glycolic acid and a brief explanation of the complex structure of hair before discussing the experiments that were conducted by researchers at DuPont and at a third-party testing lab. Data from each of the five classes of experiments are presented.

Based on the results from the experiments described in this paper, it is clear that glycolic acid serves several functions in hair care products. Specifically, the addition of glycolic acid in a conditioning formulation can aid in conditioning hair and moisturizing both hair and scalp by penetrating the hair shaft and skin.

As shown by the data, glycolic acid in a conditioning formulation provides moisturizing-like effects such as softness, and helps prevent hair breakage, giving hair overall better manageability.

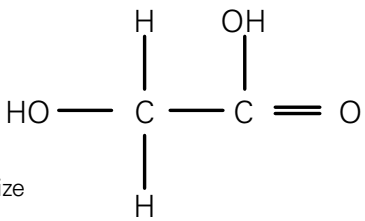
It is also important to note that because glycolic acid has such a positive effect on skin, hair care products with glycolic acid in the formulation will not only benefit the hair, but also moisturize the scalp and reduce flaking, resulting in an overall healthy look and feel.

To assist readers who wish to further explore this topic, a list of supplemental references is provided.

Glycolic Acid

Glycolic acid is a carboxylic acid with alcohol functionality. It is the simplest alpha hydroxy acid (AHA) and smallest of the fruit acids. Highly soluble in water, it has low odor, low corrosiveness, very low vapor pressure and low toxicity. In addition, glycolic acid is an efficient pH adjuster, biodegradable (~90% in seven days) and non-volatile.

Figure 1. Chemical structure of glycolic acid



Well known for its use in skin care products, glycolic acid has been shown to normalize the process of keratinization and therefore improve the look of the skin. Specifically, it can readjust moisture in the epidermis allowing for smoother, softer, more radiant skin.

Glycolic acid improves the appearance of sun damaged skin and fights skin aging by improving elasticity and firmness; increasing the production of dermal matrix compounds such as glycosaminoglycans (GAGs); and helping to increase collagen synthesis. It can also help to reduce the appearance of fine lines and wrinkles; improve skin texture; reveal smoother, brighter skin with less pigmentation; and promote the cycle of cell shedding and re-growth.

Hair—A Complex Structure

Hair is a complex structure that can be broken down into four parts: the follicle, hair bulb, hair root and hair shaft. The hair shaft consists of the cuticle, cortex and medulla.

The Cuticle

The protective layer of the hair shaft, the cuticle is made up of four keratinaceous layers, each with a different level of the amino acid cystine in the hair protein. The outer layer of the cuticle, which is known as the epicuticle, has high cystine content; the middle layers, which are known as the exocuticle, contain lesser amounts; and the innermost layer, which is known as the endocuticle, contains a low amount.

Under the microscope, a healthy hair shaft appears to be covered in smooth, unbroken scales, as shown in figure 3. The cuticle layer is responsible for visual attributes of hair such as shine and smoothness. It is also responsible for the friction effect of hair, which determines manageability (i.e., how easy it is to comb or brush hair). The cuticle layer can be damaged by daily grooming, such as combing and brushing, as well as by chemical treatments, such as bleaching.

The Cortex

The cortex makes up the largest portion of the hair shaft. It is made up of three types of cells: orthocortical, paracortical and mesacortical. Amino acids in the form of proteins and keratin are found in this inner layer of the hair shaft. This area of the hair shaft contains pigment and is responsible for structural integrity, strength, swelling and stretching of the hair.

Figure 2. Anatomy of hair

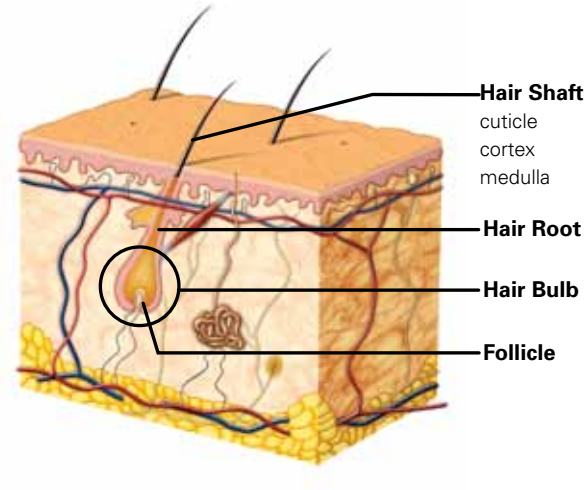
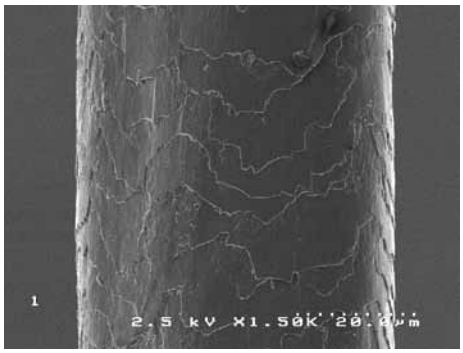


Figure 3. Scanning Electron Micrograph (SEM) of a healthy hair shaft at 1,500X magnification

The smooth, scale-like structures are the cuticle, the outermost protective layer.



Experiments

Five classes of experiments were conducted to evaluate the performance of glycolic acid in hair care applications. Because consumer research shows that manageability, enhanced softness, protection and conditioning are key attributes for hair care products, the experiments focused on these factors.

Differential scanning calorimetry (DSC) and analysis of solids at atmospheric pressure-mass spectrometry (ASAP-MS) experiments were conducted by researchers at DuPont. The other experiments—tensile strength, repeated brushing and wet combing studies—were conducted by a third-party testing lab.

All testing was performed on Caucasian hair. Both healthy and chemically damaged (bleached) hair samples were used. The results from similar experiments conducted on Asian hair samples were reported in a white paper published by DuPont in April 2010.[1]

For the DSC experiment, DuPont researchers tested sets of hair samples (healthy and bleached), each treated with one of the main ingredients found in a hair conditioning treatment that is based on DuPont™ Glypure® (see appendix for details). The ingredients in this complex formulation were tested separately to determine which, if any, would cause an increase in denaturation temperature, resulting in a positive effect on hair.

For the ASAP-MS experiment, DuPont researchers tested hair tresses (healthy and bleached) that were either treated with a 5% DuPont™ Glypure® solution or left untreated as a control.

In the tensile strength experiments, researchers evaluated the effects of a 5% DuPont™ Glypure® solution on chemically damaged (bleached) hair.

In the repeated brushing experiment, researchers compared the effects of a hair conditioning treatment based on a DuPont™ Glypure® starting point formulation to a commercial brand hair conditioner and a high-quality commercial brand hair conditioner on bleached hair samples.

In the wet combing experiment, researchers compared the effects of a hair conditioning treatment based on a DuPont™ Glypure® starting point formulation—one with DuPont™ Glypure® included and one without—to a commercial brand hair conditioner and to a high-quality commercial brand hair conditioner on bleached hair samples. For the version that did not include DuPont™ Glypure®, citric acid was added to obtain a pH level of 4.0.

Differential Scanning Calorimetry (DSC) Experiment

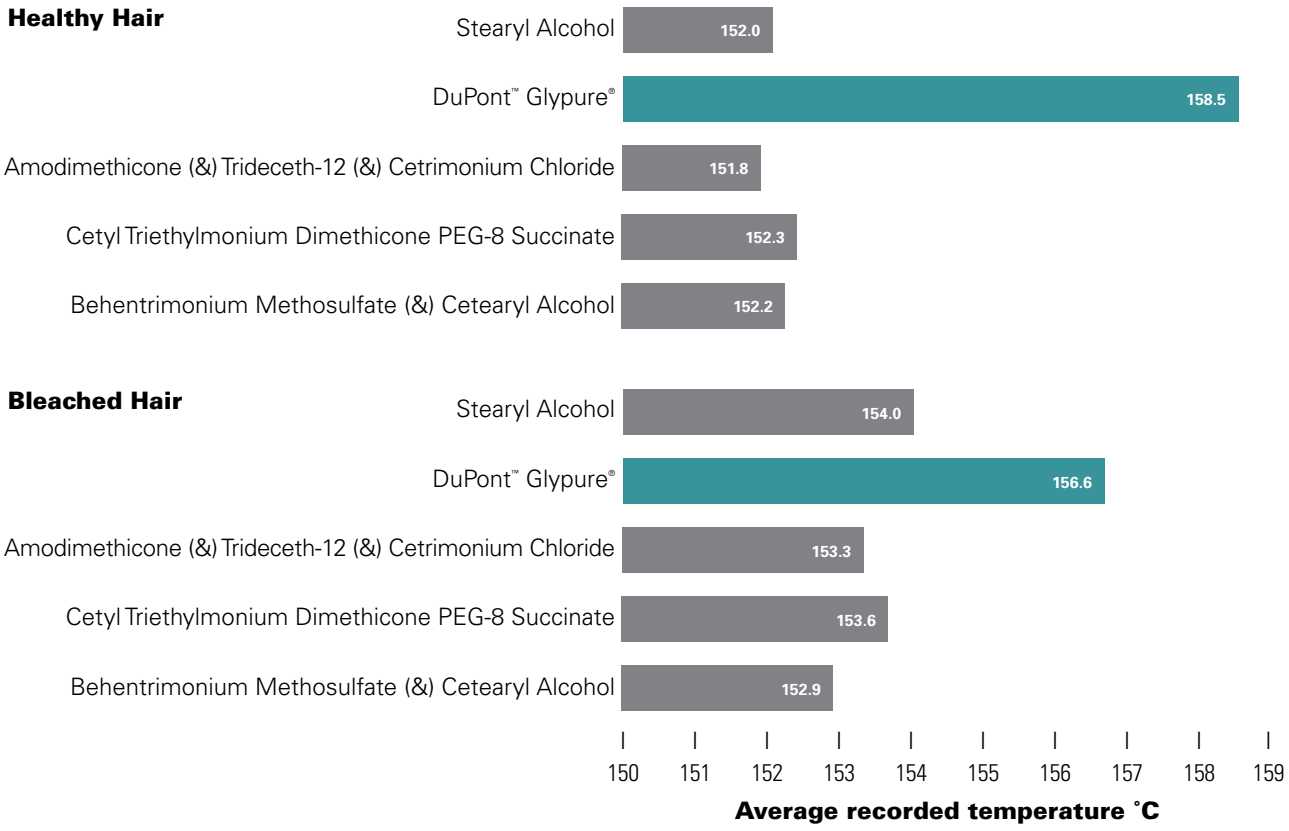
Researchers at DuPont used DSC to study the denaturation of keratin in hair according to a method well established in the industry. During this experiment, high-pressure DSC was performed to determine the temperature at which the keratin in hair denatures. The test method used was based on that referenced in *Characterizing Keratins Using High-Pressure Differential Scanning Calorimetry (HPDSC)* by Wortmann and Deutz.[2]

A total of 10 hair tresses—five healthy and five bleached samples—were used in this experiment. One healthy hair sample and one bleached sample were treated with a 5% DuPont™ Glypure® solution. One each of the remaining four healthy hair samples and four bleached samples was treated with either Stearyl Alcohol; Amodimethicone (&) Trideceth-12 (&) Cetrimonium Chloride; Cetyl Triethylmonium Dimethicone PEG-8 Succinate; or Behentrimonium Methosulfate (&) Cetearyl Alcohol in the percentages found in the hair conditioning treatment that is based on DuPont™ Glypure® (see appendix for details). The ingredients in this complex formulation were tested separately to determine which, if any, would cause an increase in denaturation temperature, resulting in a positive effect on hair. All samples were treated for 5 minutes.

As shown in figure 4, there was a 6.5˚C increase in denaturation temperature for the healthy hair sample treated with DuPont™ Glypure® and a 2.5˚C increase in denaturation temperature for the bleached hair sample treated with DuPont™ Glypure®. Healthy hair samples treated with the other ingredients showed either virtually no change or a slight increase in denaturation temperature. Bleached hair samples treated with the other ingredients all showed a decrease in denaturation temperature.

Simply stated, this proves that hair—both healthy and bleached—is better able to withstand heat when treated with DuPont™ Glypure® in a conditioning formulation, an important benefit for hair that is styled with high-wattage blow driers, hot curlers, curling irons and straightening irons, or subjected to other conditions that are stressful to hair.

Figure 4. Results from DSC experiment using high-pressure method[2]



Analysis of Solids at Atmospheric Pressure-Mass Spectrometry (ASAP-MS) Experiment
Researchers at DuPont used ASAP-MS to determine the presence of glycolic acid in hair samples. ASAP-MS is a test method that couples a modified Atmospheric Pressure Chemical Ionization (APCI) source with an extremely sensitive, high-resolution mass spectrometer. Hot nitrogen gas is used to vaporize materials, while a corona discharge ionizes these volatile components. The high vacuum atmosphere in the mass spectrometer pulls the ions into a trap where their mass is analyzed with exceptional accuracy.

In this experiment, one healthy hair sample and one bleached hair sample were treated with a 5% DuPont™ Glypure® solution, while one healthy hair sample and one bleached hair sample were left untreated as a control. All hair samples were then rinsed and dried.

A section of hair from the middle of each tress (measuring approximately 2.54 cm long by 5 mm in diameter) was introduced directly into the ion source at atmospheric pressure. Nitrogen was then blown directly onto the hair samples beginning at 100˚C and slowly ramping up to 275˚C. Volatiles were then drawn into the mass spectrometer and measurements were taken from 50 to 1,000 Daltons.

The resulting mass spectra showed that glycolic acid is retained in both healthy hair and bleached hair samples treated with DuPont™ Glypure® long after drying, but not in the untreated samples. This test proves that DuPont™ Glypure® penetrates throughout the hair shaft.

Consistent with the DSC results, ASAP-MS provides further evidence of the penetration and retention of glycolic acid in hair.

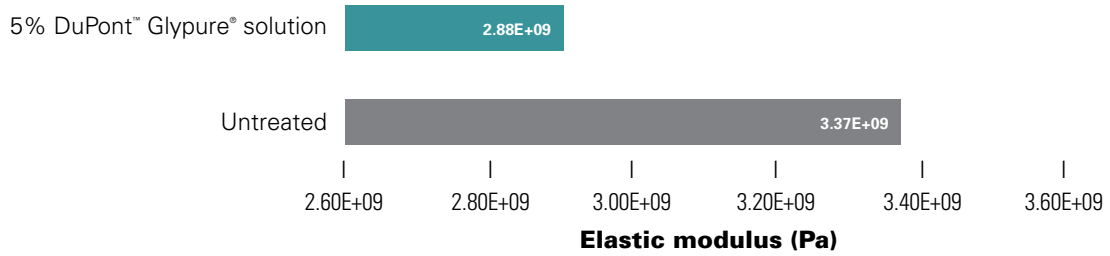
Tensile Strength Experiments
Researchers at a third-party testing lab conducted tensile strength experiments on bleached hair tresses using a Diastron Mini Tensile Tester, which is an industry standard.

Experiments were performed by preparing and testing the properties of 50 individual hair fibers per sample. A variety of tensile parameters were measured, including elastic modulus, plateau load stress, break force and break extension.

One bleached hair sample was left untreated. The other bleached hair sample was treated with a 5% DuPont™ Glypure® solution for 5 minutes.

Figure 5 shows that when bleached hair samples are treated with a 5% DuPont™ Glypure® solution, there is a statistically significant decrease in elastic modulus. This decrease in stiffness, or softening effect, is consistent with the effect of moisture on hair. Although loss of water increases the elastic modulus,[3] results show that DuPont™ Glypure® reverses this effect because it penetrates throughout the hair shaft. This decrease in elastic modulus is consistent with consumer perception that hair feels softer after treatment with a hair conditioning formulation containing DuPont™ Glypure®.

Figure 5. Results from elastic modulus experiment



Repeated Brushing Experiment

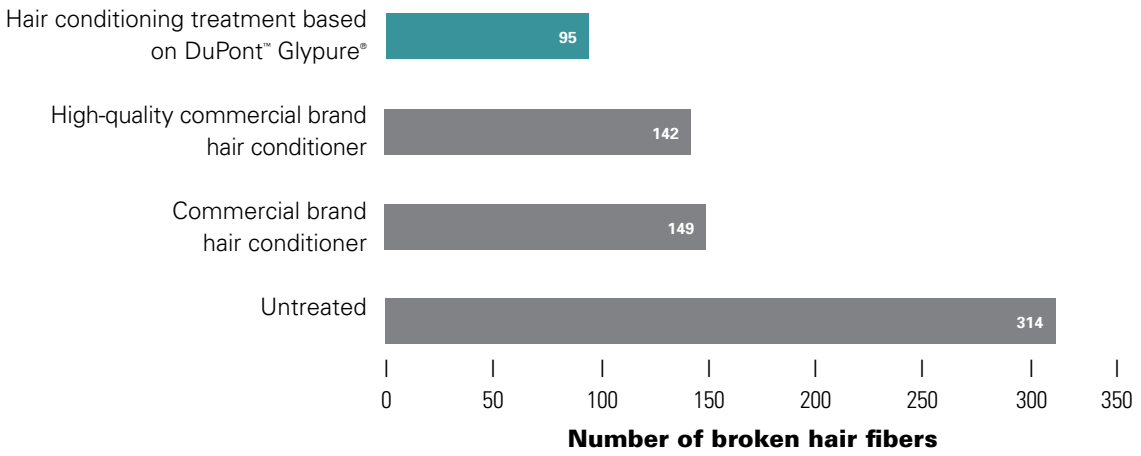
Researchers at a third-party testing lab conducted this experiment using 32 tresses of bleached hair. Eight were treated for 1 minute with a hair conditioning treatment based on a DuPont™ Glypure® starting point formulation. Eight were treated for 1 minute with a commercial brand hair conditioner and eight were treated for 1 minute with a high-quality commercial brand hair conditioner. The remaining hair tresses were left untreated to be used as a control.

The tresses were then brushed 1,000 times using a custom-made repeating brushing device and broken fibers were counted.

As shown in figure 6, the hair samples treated with the hair conditioning treatment based on a DuPont™ Glypure® starting point formulation showed a significant improvement over the untreated hair samples and the hair samples treated with either the commercial brand hair conditioner or the high-quality commercial brand hair conditioner.

The data indicate that DuPont™ Glypure® in a conditioning formulation provides superior lubrication to the hair, resulting in less friction and entanglement during brushing, which leads to significant reduction in hair breakage and improved manageability. This superior lubrication also benefits the scalp, reducing flaking and dryness.

Figure 6. Results from repeated brushing experiment



Wet Combing Experiment

Researchers at a third-party testing lab conducted a wet combing experiment in accordance with a widely used method that was first published in the *Journal of the Society of Cosmetic Chemists* in 1976.[4]

For this experiment, researchers bleached 40 hair tresses. Eight were then treated for 1 minute with a hair conditioning treatment based on a DuPont™ Glypure® starting point formulation and eight were treated for 1 minute with the same hair conditioning treatment but without DuPont™ Glypure® in the formulation. For the version of the formulation that did not include DuPont™ Glypure®, citric acid was added to obtain a pH level of 4.0.

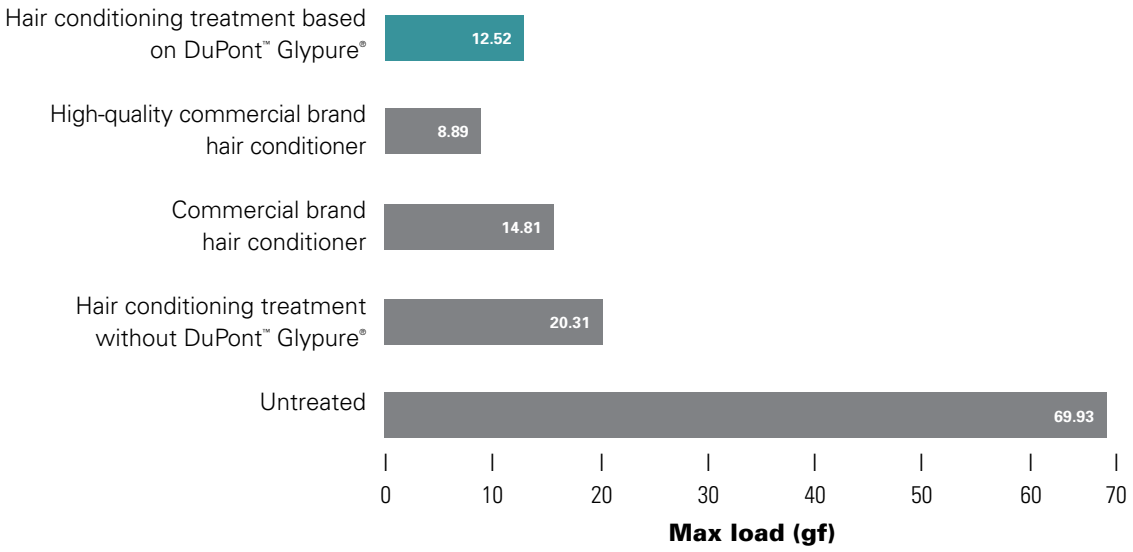
Of the remaining 24 hair tresses, eight were treated for 1 minute with a commercial brand hair conditioner, eight were treated for 1 minute with a high-quality commercial brand hair conditioner and eight were left untreated for use as a control.

The tresses were then subjected to wet combing per the test method previously cited. The amount of frictional force required to comb through the tresses was measured.

As shown in figure 7, the amount of frictional force required to comb through the treated hair samples is significantly less than that required to comb through the untreated samples. The high-quality commercial brand hair conditioner performed the best. The hair conditioning treatment based on a DuPont™ Glypure® starting point formulation performed better than both the same hair conditioning treatment without DuPont™ Glypure® in the formulation and the commercial brand hair conditioner.

This experiment further proves that the treatment containing DuPont™ Glypure® shows a significantly superior effect compared to the same treatment without this key ingredient, confirming that DuPont™ Glypure® helps make hair more manageable and easier to style.

Figure 7. Results from wet combing experiment



Conclusion

Glycolic acid is a popular anti-aging active that has been used in the personal care industry for years for skin care applications. It has strong consumer recognition as an ingredient in a wide variety of anti-aging skin lotions, creams and exfoliating products.

Results from experiments conducted on both healthy and bleached Caucasian hair samples show that the addition of glycolic acid in a hair conditioning formulation can deliver important benefits for hair care products by penetrating throughout the hair shaft, enhancing the softness, protection and manageability of hair by conditioning, moisturizing, allowing hair to better withstand heat and preventing breakage.

The ability of glycolic acid to penetrate throughout the hair shaft, allowing hair to better withstand heat, was clearly demonstrated by the DSC, tensile testing and ASAP-MS experiments. Glycolic acid penetrates the hair shaft and remains throughout the hair, similar to water, but the effect persists. It can be concluded that glycolic acid stabilizes keratin over time.

The tensile strength experiments show that DuPont™ Glypure® causes a statistically significant decrease in elastic modulus (stiffness), which is consistent with consumer perception of greater hair softness.

Adding glycolic acid to hair care formulations enables these products to provide hair with superior lubricity, which results in less friction and entanglement during combing and brushing, making hair more manageable, easier to style and significantly less prone to breakage.

Hair care products with glycolic acid also help make hair feel softer and help hair better withstand heat, enabling the use of high-temperature hair styling appliances without concerns about damaging the hair.

The data from these experiments clearly show that hair—whether healthy or bleached—can benefit from the multiple, positive effects that DuPont™ Glypure® delivers in hair care product formulations. With a long-proven reputation as an effective exfoliant and moisturizer for the skin, an important additional benefit of using glycolic acid in hair care products is that it helps the scalp by adding moisture and reducing flaking.

DuPont™ Glypure® is a cosmetic-grade glycolic acid that is used in many cosmetics and other personal care products. Manufactured in an ISO 9001:2000 certified facility located in Belle, W.Va., DuPont™ Glypure® is made in a continuous process 24/7. It is currently available in a wide variety of starting point formulations. New starting point formulations are introduced regularly to meet evolving marketplace needs.

In addition to reliable quality and supply, end users of DuPont™ Glypure® have access to a dedicated, professional staff of technical service representatives, formulating chemists and analytical chemists for help with technical questions and assistance with new product development.

Appendix

Note: For the wet combing experiment, one version of this hair conditioning treatment was formulated without the inclusion of DuPont™ Glypure®. For that version, citric acid was added to obtain a pH level of 4.0.

Hair conditioning treatment with DuPont™ Glypure®

Ingredient	Trade Name	Function	Manufacturer	Wt%
Purified Water	Purified Water			70.00
Deionized Water	Deionized Water			
Behentrimonium Methosulfate (and) Cetearyl Alcohol	Incroquat Behenyl TMS	detangling and conditioning agent	Croda	2.50
Behentrimonium Chloride	Incroquat Behenyl TMC-85	detangling benefits, reduce static, improve body, and improve wet/dry combability	Croda	0.75
Stearyl Alcohol	Lanette 18	viscosity regulation	Cognis	0.75
Ceteareth-20	Eumulgin B2	emulsifier	Cognis	0.25
Disodium EDTA	Dissolvine NA2-S	chelating agent, stabilizer	Akzo	0.05
Cetyl Triethylmonium Dimethicone PEG-8 Succinate	Bioplex Cetylsil S	conditioning agent	Biosil	0.50
Purified Water	Purified Water			10.00
Glycolic Acid (70%)	**DuPont™ Glypure® (70%)**	**Exfoliant, moisturizer, improves wet/dry combability, increases durability**	**DuPont**	**4.00**
Triethanolamine	Trolamine 99%	pH adjuster	Ruger	1.50
Amodimethicone (and)Trideceth-12 (and) Cetrimonium Chloride	Dow Corning 2-8194 Microemulsion	increases durability, shine, wet/dry combing, conditioning, reduced drying time, reduces fly aways	Dow Corning	1.50
Fragrance, Dye, Botanical Extracts, Vitamins	as desired		as desired	0.00
Caprylyl Glycol (and) Hexylene Glycol (and) Methylisothiazolinone (and) Water	Spectragard	preservative	Inolex	1.00
Triethanolamine	Trolamine 99%	pH adjuster	Ruger	pH 4.0-4.2
Glycolic Acid (70%)	**DuPont™ Glypure®**	**pH adjuster**	**DuPont**	**pH 4.0-4.2**
Purified Water	Purified Water			qs to 100%

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