PERSONAL CARE

KERAVIS™

KERAVIS	**Aqua (and) Hydrolyzed Vegetable Protein PG-Propyl Silanetriol**
KERAVIS PE	**Hydrolyzed Vegetable Protein PG-Propyl Silanetriol (and) Water (aqua)**

Hair Strengthening Active

Designed to deliver optimal anti-breakage benefits, Keravis is a multifaceted protein based complex which acts on all of the three fundamental parameters of hair strength: - tensile properties, bending modulus and cuticle abrasion.

The unique chemical composition of Keravis allows it to penetrate into the hair cortex, building strength from within, while providing film forming effects to reinforce, lubricate and protect the surface of the hair. These properties combine to strengthen the hair, thus, helping to reduce the degree of damage hair sustains from chemical treatments, environmental stresses or styling practices.

Benefits

- Increases the strength of virgin and damaged hair
- Able to triple the strength of damaged hair compared to a control conditioner
- Demonstrates strengthening and anti-breakage properties when compared to D-Panthenol
- Strengthens hair to resist combing damage
- Increases hair strength proportionally to usage level
- Continues to strengthen hair even in extreme humidity
- Consumer perceivable improvement in hair strength
- Proven efficacy from rinse-off systems

Features

- Copolymer of hydrolyzed vegetable protein and silicone
- Vegetable derived
- 15% active
- Easy-to-use liquid
- Suitable for use in all hair care applications

**KERAVIS and its use in personal care applications are covered under US Patent 8,048,846.*

Claim Substantiation Studies

The performance benefits of Keravis have been evaluated using a series of laboratory and sensory tests.

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Information on formulating, regulatory matters and health and safety is given on page 13.

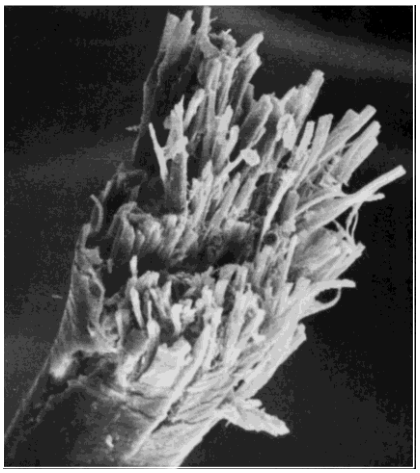
The Science of Hair Strength

Hair strength is commonly referred to as the tensile force required to snap the hair fiber, as measured by break-point analysis. However, this definition is irrelevant to the consumer experience, because it takes less force to pull a hair out of the scalp than it does to break it. A more accurate and consumer-relevant description of hair strength is the resistance of the hair to breakage under normal grooming practices. This is influenced by:-

- Cross-sectional diameter of the hair fiber
- Moisture content and flexibility
- Lubricity / resistance to frictional forces
- Structural damage

Three important factors[1] lead to hair fracture during mechanical manipulation:

- Incremental bending & straightening leads to local fatigue fracture
- Tensile forces lead to hair extension and cuticle decementation
- Frictional interactions of hairs upon each other lead to localised abrasion and cuticle loss



Weakening of the hair due to these factors ultimately can lead to breakage in the form of “brush ends” (Figure 1) or “split ends” (Figure 2).

Figure 1: Brush end formation

Repetitive movement leads to localised weakening within the hair shaft and ultimately brush end fractures

Figure 2: Split end formation

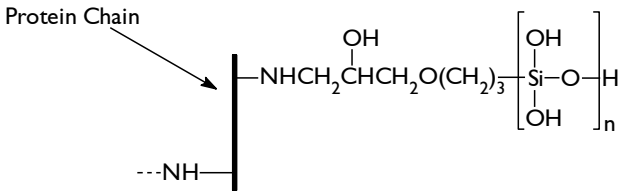
Cuticle and cortex damage leads to weakening at the end of the hair shaft, resulting in splitting and ultimately fiber fracture

The unique chemical composition of Keravis enables it to act on all three of the hair breakage mechanisms identified above. Cuticle penetration builds strength from within to increase resistance to tensile forces and withstand bending stresses. Meanwhile, film formation reinforces, lubricates and protects the hair surface, preventing cuticle abrasion and loss.

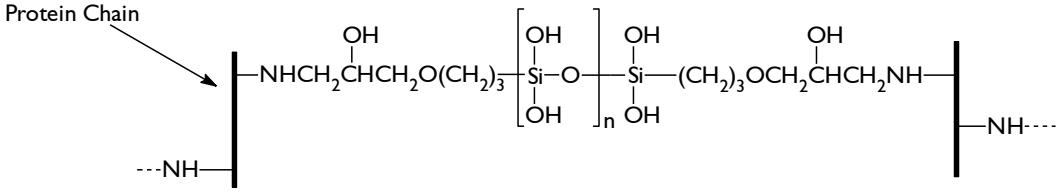
Structure and Composition

The silicone groups are covalently attached to the protein amino groups by reactive link groups present as part of the organofunctional silane or siloxane. The siloxane chains are capable of forming cross-links, which may be inter- or intra-molecular protein links. The structure may be then further extended by the condensation of silanol groups to yield a polymeric network.

Within Keravis there is a low level of silicone copolymerisation of the protein, which in the final product delivers a mix of hydrolyzed vegetable protein and hydrolyzed vegetable protein copolymerised with the silicone. This allows the unique multifunctional benefits of Keravis.



Silanol Terminated Copolymer



Siloxane Cross-Linked Copolymer

Figure 3: Structure of Keravis

Measuring Hair Strength – The Flexabrasion Technique

As mentioned above, there are three components that effect the fracture of hair fibers, namely tensile extension, flexure and abrasion. Flexure of hair fibers is thought to be of most importance in the mechanism of fiber fracture. Flexure is the associated forces of bending that are generated when fibers interact with other fibers and grooming tools. Tensile forces also play a role and are present in normal grooming practices. Frictional interactions are involved through cuticular abrasion which can lead to the cortex becoming exposed, thus reducing the fibers ability to withstand any further chemical or physical damage and also through increasing the longitudinal and bending forces the fiber experiences.

In the past, hair-strengthening claims have been supported only by tensile measurements and have ignored the important frictional and flexure forces that also impact on hair fiber strength. For a more consumer relevant measurement, it is important to evaluate the effect of all three of the components of hair fiber breakage, flexure, tensile and frictional forces.

The Flexabrasion technique[2] provides such a measurement. Hair fibers are cyclically drawn, under load, across a tungsten wire simulating the movement and mechanical forces experienced by hair during everyday grooming and styling practices (see Figure 4). When a brush is pulled through the hair, the hair strands are entwined and move against one another causing longitudinal shear within the fiber shaft, abrasion and extension of the hair fiber, which will eventually cause longitudinal splitting and premature fracture.

The Flexabrasion instrument allows the testing of 20 hair segments at once. It is positioned in a humidity-controlled cabinet, which allows monitoring of the relative humidity under which the hair is being tested. The number of cycles required for the hair to break allows assessment of cosmetic actives in terms of hair strength benefits. Application of this methodology for the assessment of human hair fibers was first reported in 1995 by Leroy et al[2].

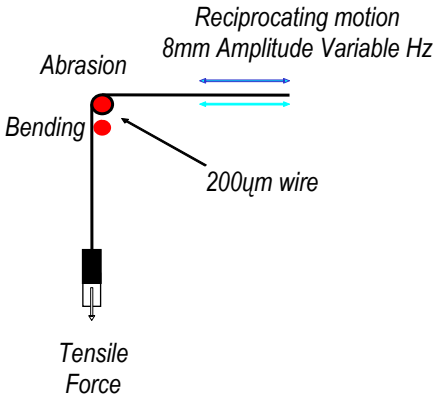


Figure 4: Schematic of flexabrasion test

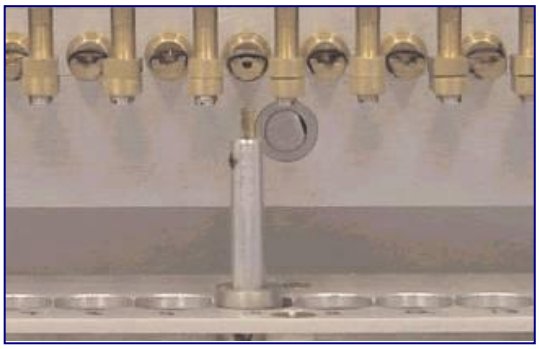


Figure 5: Flexabrasion Apparatus: Close-up of tungsten wire over which the hair fiber is drawn

A large amount of data has been generated substantiating the hair strengthening/anti breakage performance of Keravis from a wide range of formulations/systems. Most of them have been led with the first generation of Keravis, except for the consumer preference test that has assessed the new Keravis PE.

Keravis PE differs from Keravis only by its preservative system, so we are confident that this minor change does not affect the performances of Keravis.

Effect of Keravis in a shampoo and a conditioner system on Damaged (Bleached) Hair

Hair segments that had been damaged by bleaching were treated with a shampoo or conditioner containing Keravis, or with the same shampoo / conditioner without Keravis. Control hair segments were bleached and then treated with water only. The flexabrasion lifetimes of the different samples were measured and compared to virgin hair.

The results are expressed as a percentage change in the mean fatigue lifetime of the hair fiber in comparison to the untreated control virgin hair fiber. The formula used is detailed below:

Percentage Difference = ((B-A)/A) x 100

where:-

A = mean fatigue lifetime of control hair segment

B = Mean fatigue lifetime of test hair segment

Keravis was incorporated into a shampoo system at 4% inclusion (1% active) to evaluate its efficacy in a surfactant system on bleached hair. Each test shampoo was applied at a 10% dilution, to mimic a consumer's usage level.

This test showed (figure 6) that Keravis delivered up to three times the strengthening benefits when delivered from a shampoo system whereas the shampoo alone provided little benefit.

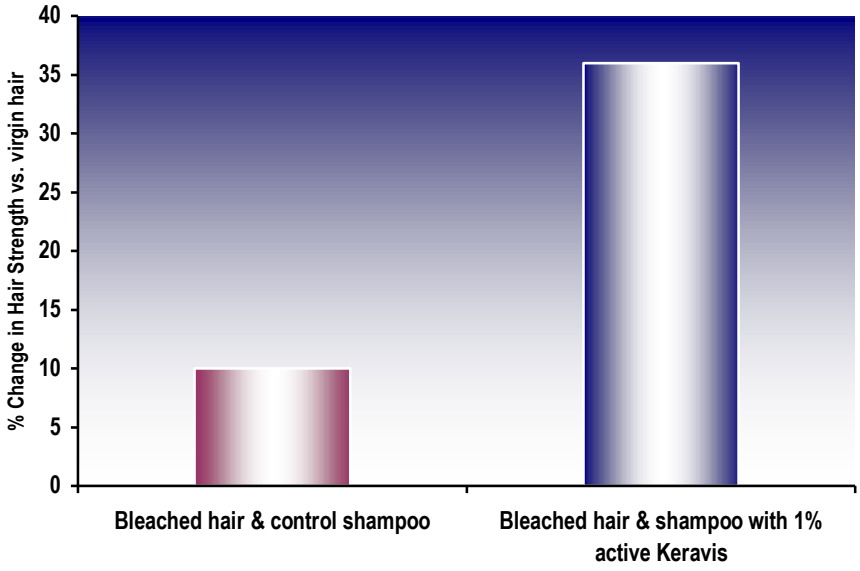


Figure 6: Effect of Keravis, from a shampoo, on bleached hair

Figure 7 shows that the conditioner containing Keravis (0.25% active) delivered three times the strengthening or anti-breakage benefits compared to the same conditioner without Keravis. It is interesting to note that bleached hair treated with the Keravis conditioner is 175% stronger than the virgin hair fibers.

The conditioner alone increased hair strength by 60% because of its ability to lubricate the hair fiber surface and reduce the frictional component of flexabrasion.

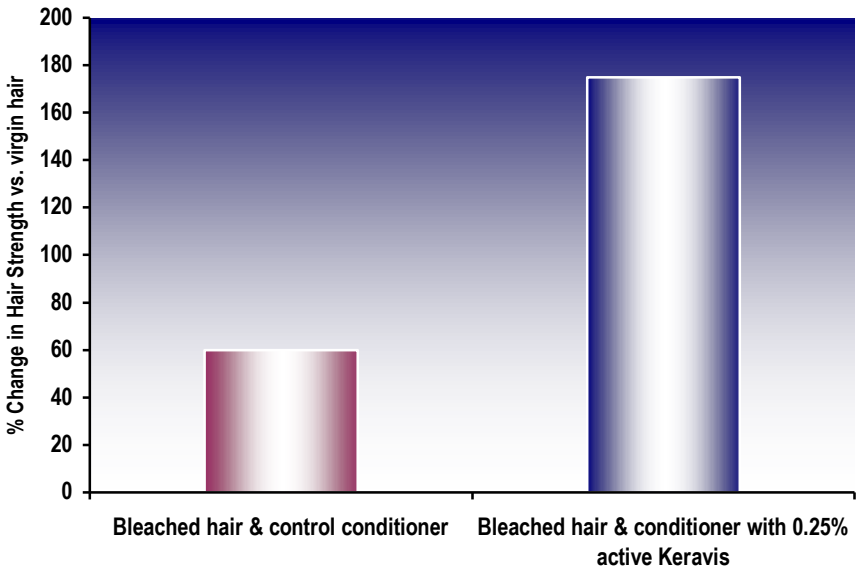


Figure 7: Effect of Keravis, from a rinse-off conditioner, on bleached hair

The test was conducted at 60% Relative Humidity (RH) and the conditioners were undiluted.

Evaluation of KERAVIS versus D-Panthenol

D-Panthenol, or Pro Vitamin B5 as it is commonly known, is used extensively in the personal care industry for its moisturization benefits and claims of adding hair strength. The following studies evaluated D-Panthenol and KERAVIS in an active-to-active comparison using the Flexabrasion test method[3].

- The relative hair strengthening effect of Keravis vs. D-Panthenol when applied from a rinse-off system
- The effect of active concentration on strengthening performance of Keravis and D-Panthenol
- The relative strengthening performance of Keravis and D-Panthenol, applied from a conditioner at various active levels, at various relative humidities

Figure 8 shows the comparison between Keravis and D-Panthenol when applied from a conditioner at 0.25% active. The conditioners were diluted to 20% and tested at 60% RH, representing realistic consumer use. Based on this data, Keravis delivers more than three times the strengthening performance of Panthenol.

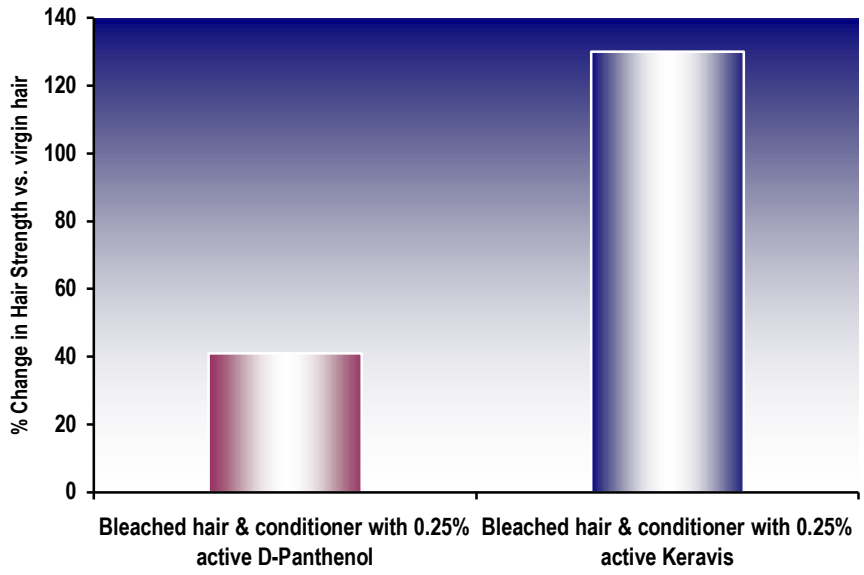


Figure 8: Strengthening effect of Keravis vs. D-Panthenol, from a conditioner

Keravis was also compared to D-Panthenol from a shampoo system where both were added at 1% active. Each was tested at a 10% dilution on hair fibers to simulate consumer usage conditions (figure 9).

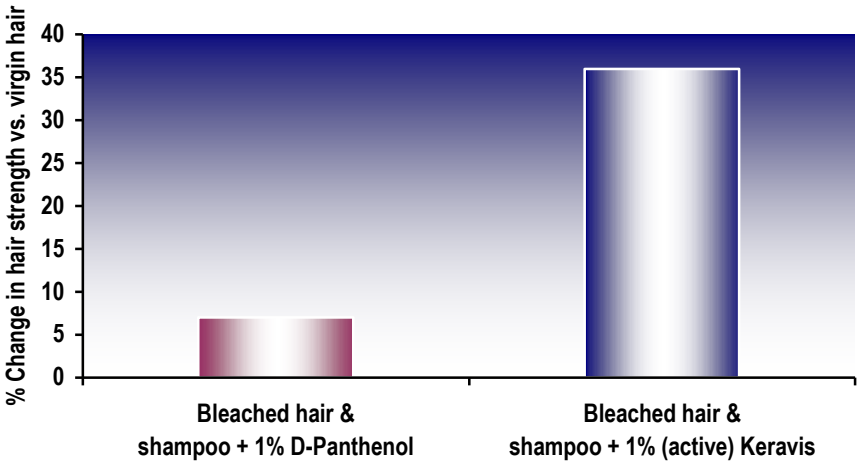


Figure 9: Strengthening effect of Keravis vs. D-Panthenol, from a shampoo

Figure 9 shows that addition of Keravis at 1% prolongs the fatigue lifetime of the hair fiber by 36%, whereas D-Panthenol at 1% active did not show a significant improvement.

The effectiveness of an ingredient is usually dependent on the level of activity. Therefore, Keravis and D-Panthenol were compared by increasing the level of each ingredient from 0.25% to 0.50% active to understand if the dosage of the active is related to its degree of functionality (see Figure 10).

Whereas increasing the concentration of D-Panthenol has no effect on its strengthening performance, there is an improvement when the concentration of Keravis is increased.

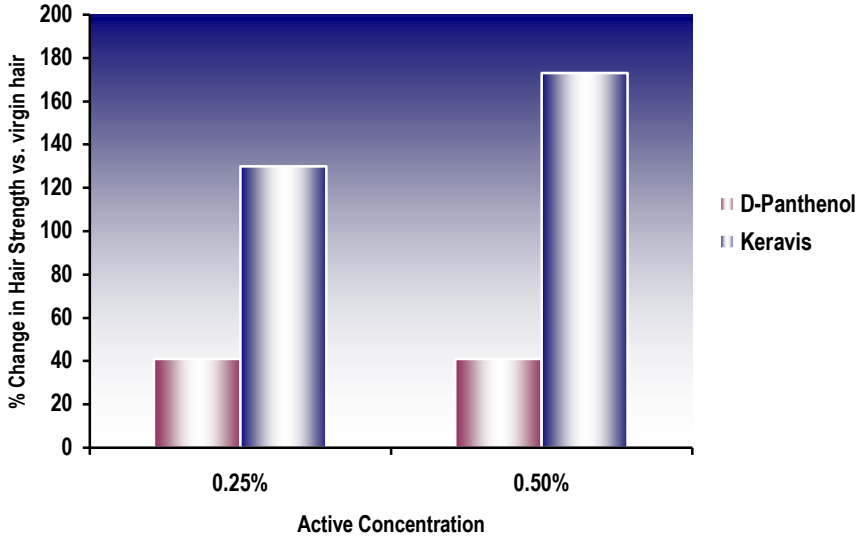


Figure 10: KERAVIS vs D-Panthenol in conditioner at 0.25% and 0.50% active

Relative Humidity (RH) has an influence on flexabrasion hair strength, whereby if the hair contains moisture and becomes plasticized with water, the fatigue lifetime of the hair increases. Relative humidity greater than 80% frequently weakens the hair due to excessive water content, which causes interruption of salt bridges and hydrogen bonding.

<u>0.25% and 0.5% w/w actives Panthenol and Keravis at various humidities</u>

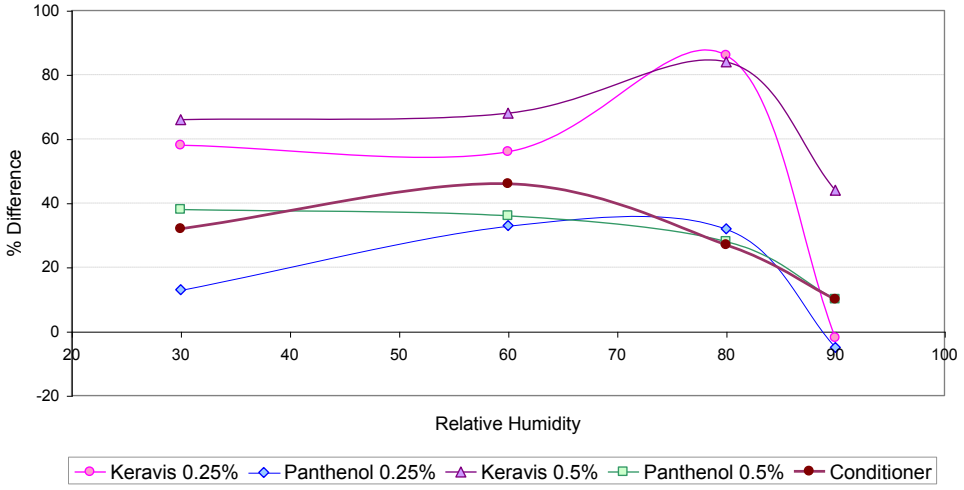


Figure 11: The effect of relative humidity and active content on the strengthening performance of Keravis and D-Panthenol in a conditioner

These results indicate that the performance of Keravis from a conditioner is improved as humidity increases, up to 80%RH. It also shows that increasing humidity slightly reduces the performance of a conditioner containing Panthenol with regard to effect on hair strength. The study also shows little difference between the conditioner base on its own and the base containing D-Panthenol, whereas including Keravis in the conditioner gives a substantial increase in strengthening performance throughout the range of humidity tested.

Combining Keravis with Panthenol

Keravis can also be used to improve the performance of products that already contain D-Panthenol, to deliver optimum benefits. Figure 12 shows Flexabrasion data for a commercial conditioner containing D-Panthenol and for the same conditioner with Keravis added also. Once again, the strengthening effect of the conditioner is improved by the addition of Keravis.

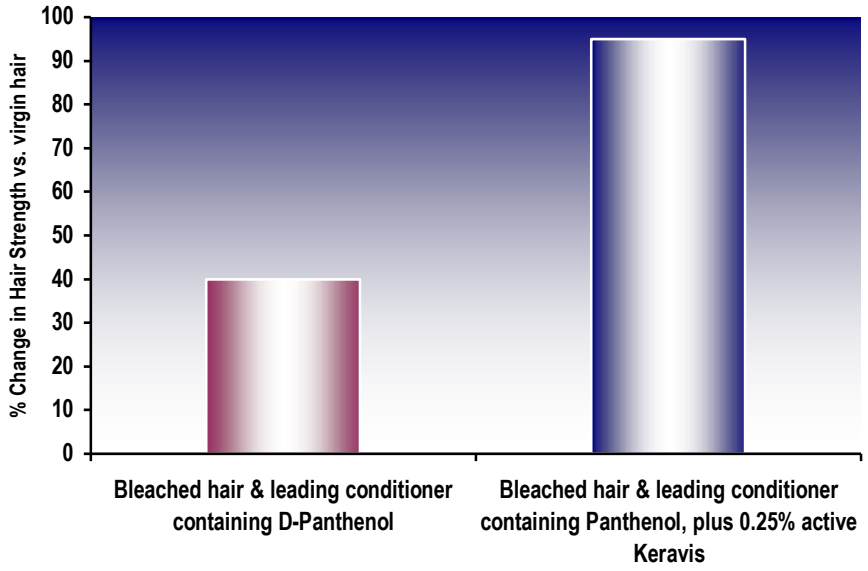


Figure 12: The effect of adding Keravis to a conditioner that contains Panthenol

Repetitive Combing Study

As already noted, grooming practices such as combing are a major cause of hair damage. Repetitive combing of the hair damages the fibers, making them weaker and gradually more and more difficult to comb. Knotting intensifies as the hair fibers are stretched and rubbed against one another and the comb teeth.

The Dia-Stron MTT 175 (Miniature Tensile Tester – see Figure 13) calculates the force required to comb a tress of hair. By measuring this before and after repetitive combing, the percentage change in force required to comb the hair can be measured. The more damaged the tress becomes, the more force is required to comb it.

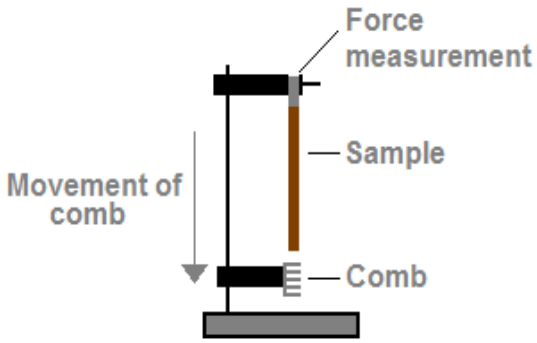


Figure 13: MTT Equipment

The ability of Keravis and D-Panthenol to protect the hair against the damage caused by repetitive combing was assessed as follows. Hair tresses were treated with either a basic conditioner, the same conditioner containing 0.25% active D-Panthenol, or the same conditioner containing 0.25% active Keravis. The MTT was used to measure the total work required to comb each tress. The tresses were then attached to a combing wheel and combed 5000 times, under controlled conditions of temperature and humidity. The work required to comb each tress was then re-measured on the MTT.

The results are expressed as the average percentage increase in the work required to comb the tress, calculated as:

Percentage Change = ((S-B)/B) x 100

where:-

S = the work required to comb the hair after 5000 combs

B = the baseline work required, before repetitive combing

10 repeats of the procedure were carried out and the average percentage change in total work required to comb the hair for each product was calculated. The results are shown in Figure 14.

It is clear that, while Panthenol has no effect on the damage caused by repetitive combing, Keravis does provide significant protection against this damage. For the hair treated with Keravis, the increase in the work required to comb the tress was 87%, compared to 140% for the base conditioner. This difference was found to be statistically significant where p=0.02.

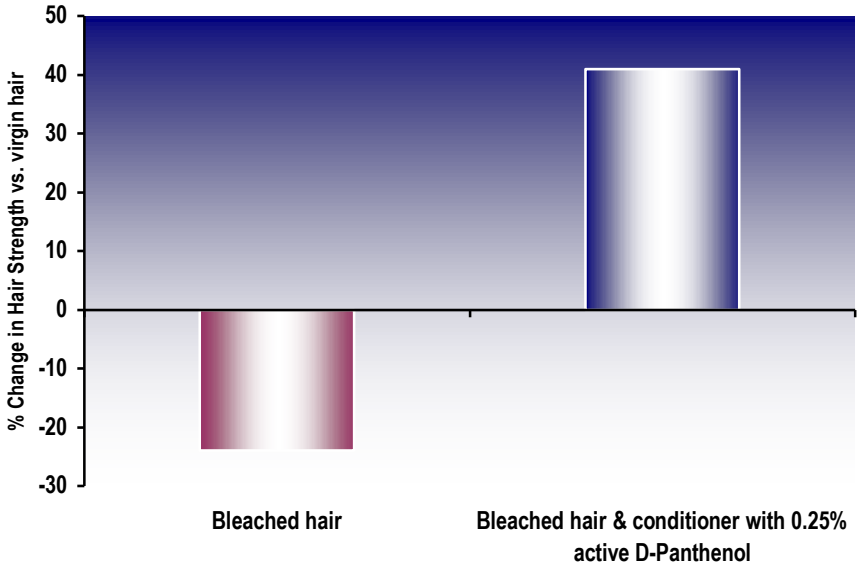


Figure 14: Effect of Keravis and Panthenol on the work required to comb tresses that have been damaged by repetitive combing

Use of Keravis in Hair Relaxer Systems

Hair relaxers represent one of the most important segments of the ethnic hair care market. There are a number of problems traditionally associated with relaxer systems, for example scalp irritation, dryness and itching. However the number one complaint among consumers after using a relaxer is hair breakage.

To combat these problems, Croda has developed The New Generation Relaxer System, combining Keravis with Keralenis™. Keralenis is a complete emulsifier system for relaxers, proven to minimise scalp irritation, deliver actives rapidly and protect the hair cuticle. The New Generation Relaxer System comprises a complete set of formulations covering all stages of a hair relaxer treatment regime:-

- Pre-treatment
- Relaxer and neutralising shampoo
- Conditioner
- Leave-in conditioner

The levels of Keravis in the various formulations are shown below. For these full formulations please refer to pages 18 through 23 of this document.

Formulation	Reference no.	% w/w Keravis (as supplied)
Pre-treatment	BW-50	0.50
Anti-breakage Relaxer	BW-41-1	1.00
Activator		1.00
Neutralizing Shampoo	SH-135	0.50
Conditioner	HP-300	1.00
Leave-in Conditioner	HP-301	0.50

To assess the hair strengthening benefits of these formulations, flexabrasion was used to measure the fatigue lifetime of African-American hair after each stage of the relaxer treatment. A leading brand relaxer system, which is claimed to reduce breakage, was used as a benchmark. Separate measurements were carried out for each step of the treatment (without applying any of the other steps). A further set of measurements assessed the change in hair strength after applying the complete treatment regime.

Figure 15 shows the results for the different stages of the relaxer treatment. Figure 16 shows the results for the entire system.

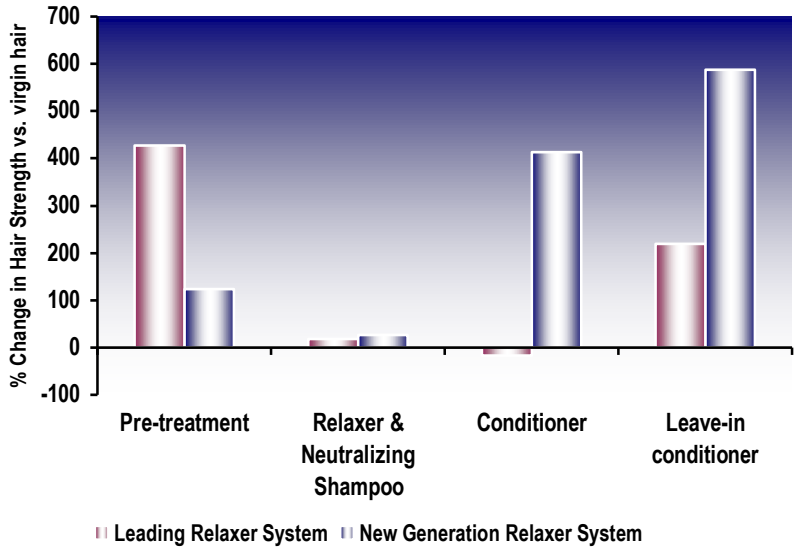


Figure 15: Comparison of the change in hair strength for different stages of relaxer systems

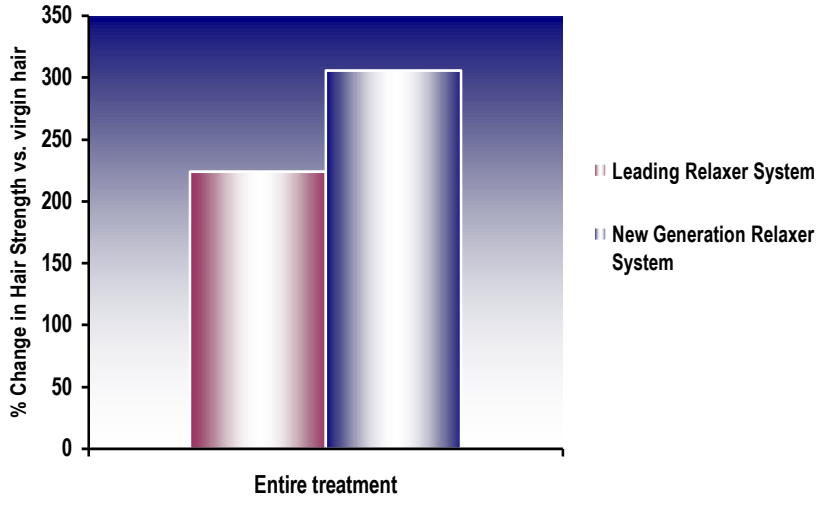


Figure 16: Comparison of the change in hair strength for the complete relaxer systems

For the pre-treatment, the New Generation system shows an improvement in hair strength, but the benchmark product performs better. However for each other stage, the New Generation Relaxer System outperforms the benchmark in terms of hair strengthening. This is reflected in the data for the complete system; the New Generation Relaxer System gives significantly better strengthening performance than the benchmark system.

KERAVIS: Consumer-perceivable strengthening

A Consumer preference sensory analysis was led on a panel of 34 people. Six light blond bleached tresses were washed twice with 10% SLES solution. Then, three tresses were washed with a basic shampoo and three tresses were washed with the basic shampoo containing 0.25% active Keravis PE. After detangling, tresses were combed 10000 times on a Repetitive Combing Wheel. All experiments were conducted at 50%RH.

One tress with Keravis PE and one tress without Keravis PE were randomly selected and evaluated visually in a Forced Preference Test. Assessors were asked to choose which tress looked strongest and healthiest.

31 out of the 34 respondents (91%) selected the tress treated with the Keravis PE shampoo as looking strongest and healthiest.

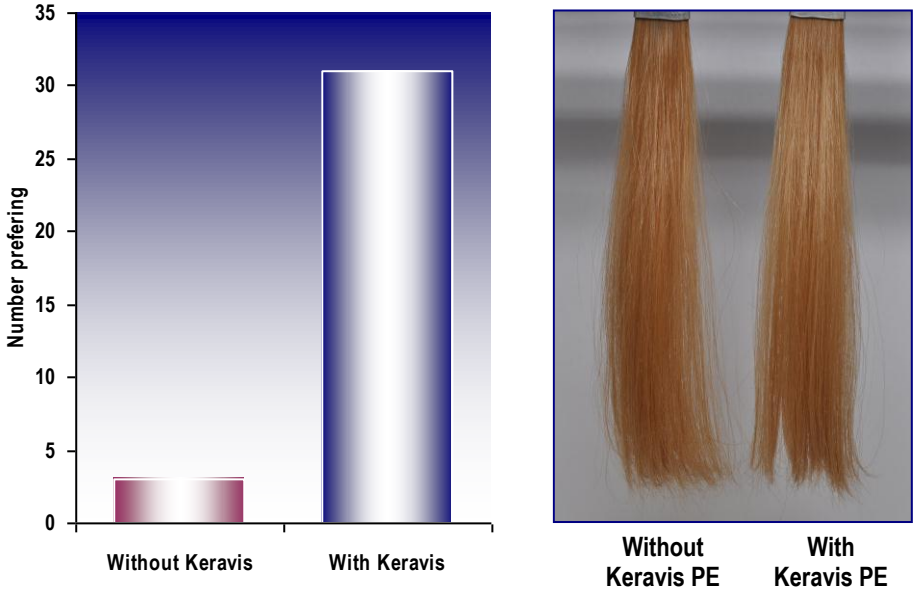


Figure 17: Consumer preference test (result and evaluated tresses photograph)

Keravis gives a clear visually-perceivable improvement in the appearance of the hair after repeated combing: less damaged, more shine, more aligned fibers and less fuzzy ends.

Substantivity

The substantivity of Keravis to hair was measured using radiolabelling techniques. By radioactive tagging of the protein it is possible to measure the radioactivity of the hair after treatment and relate this to protein uptake. In these studies ^{125}I was used for labelling purposes. Keravis contains tyrosine residues and these are radiolabelled by iodination with ^{125}I as illustrated in Figure 18.

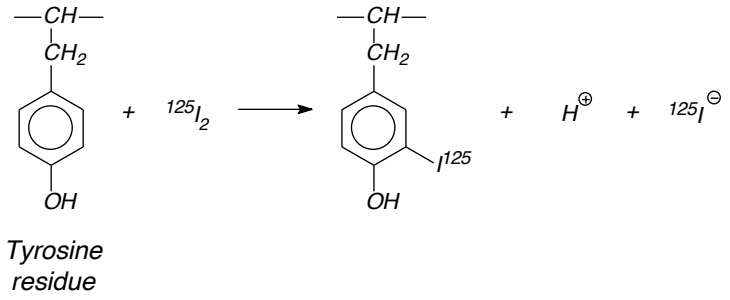


Figure 18: Radiolabelling of proteins using ^{125}I

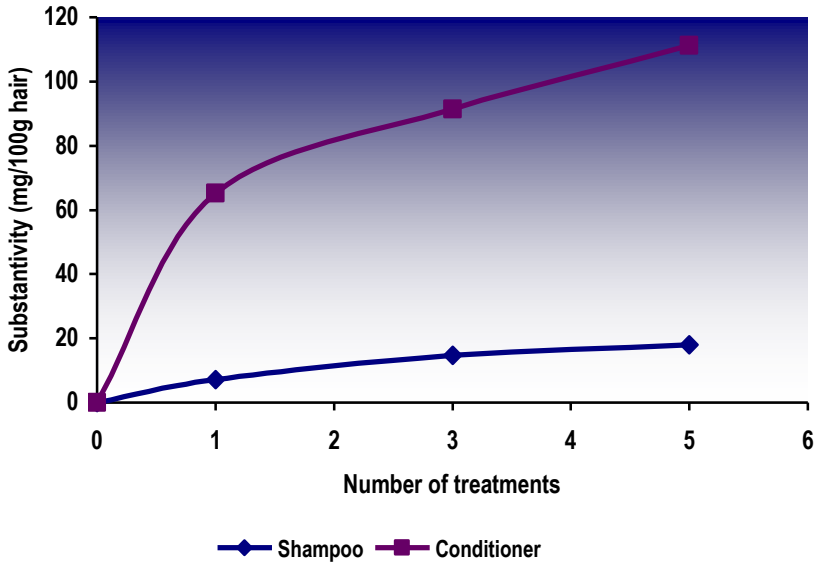


Figure 19: Substantivity of Keravis

The data show that Keravis is substantive to normal European brown hair when applied from a basic shampoo or a conditioner base. As expected, the substantivity is greater when applied from a conditioner base than a shampoo base.

Formulation, Regulatory Matters and Health and Safety

Keravis PE is recommended for all hair care products including shampoos, conditioners, hair repair and treatment products and styling aids.

Keravis PE is a water-soluble, clear yellow liquid. It can be incorporated into cold mix systems, and can also be used at elevated temperatures up to about 60°C. Typical use levels are 1 - 5 % as supplied.

Keravis PE is considered to be an acceptable cosmetic raw material. A separate material safety data sheet is available on request.

PERSONAL CARE

References:

1. JA Swift and AC Brown, "Hair breakage: the scanning electron microscope as a diagnostic tool", J Soc Cosmet Chem 26 289 (1975).

2. JA. Swift, S. Chahal, D. Coulson, and N. Challoner; “Flexabrasion – A Method for Evaluating Hair Strength”, Cosmetics and Toiletries, 116, 12, 53 – 60, December 2001.

3. D. Jones, S. Chahal, E. Smart; “Flexabrasion hair strength – a comparison of two commercial actives:, *Cosmetic & Toiletries Worldwide*, 2005.