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# Surface-Active Functionals for Wash Off Stable Hair Color Modifications

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Objective:
Purpose of the research conducted was to find a cosmetically elegant on-hair deposition approach for color-modifying actives. Treatments should not be damaging to the hair, avoid the use of strong oxidants such as hydrogen peroxide and provide uniform and wash-off-stable color effects.

The molecular combination of vitamin C with a color-modifying functionality-unit provides an option to evenly and permanently attach said functionality to the hair fibre. Thereby vitamin C can be used as antioxidative linker capable to physically and chemically attach the color-modifying functionality to the hair matrix. In this paper we investigate the color modifying effects of two different functionalities, one being a UV absorber ("UV vitamin C") the other being a colorant ("VIS vitamin C").

Methodology:
Various protocols were applied to test the products for their wash-off resistancy (A), their antioxidative power on hair (B), and their color modifying and UV absorbing properties (C). Test A included a 5-times incubation of moisty hair strands with product-containing hair fluids (45min at 40°C) followed by rinse-off and drying steps. Finally 10 shampoo-washings were applied. Remission spectra were recorded on the dried hair before and after the shampoo-washings. L*a*b* color values were calculated from these remission spectra. In test B product-treated hair strands were sprayed with DPPH radicals and the resulting color visually assessed. In test C color and UV absorbing properties were compared for strands that were either ammonia-pre-treated, then 1-time product-treated, or not pre-treated with ammonia but 1-time or 7-times product-treated. Remission spectra (250-2500nm) were recorded on the washed and dried hair strands with a Perkin Elmer lambda 900 with integrating sphere. Absorption was calculated according to the following equations: absorption = log (1/transmission); transmission = $\sqrt{}$ $remission_{verum}$ / $\sqrt{}$ $remission_{placebo}$ (standardized against $BaSO_4$)

Results and Discussion:
UV-absorbing properties were tested according to test C and are (partly) illustrated in Fig. 1: All hair absorbance profiles show strong UV absorbances with maxima in the UVA between 330-340nm. A substantial built-up effect in UV protection can be seen for the 7-times treated strand. Here the absorbance extends into the visible region which might be explained by a Maillard-type reaction between the product and the hair proteins. The two single-treated strands do not significantly differ in this case.

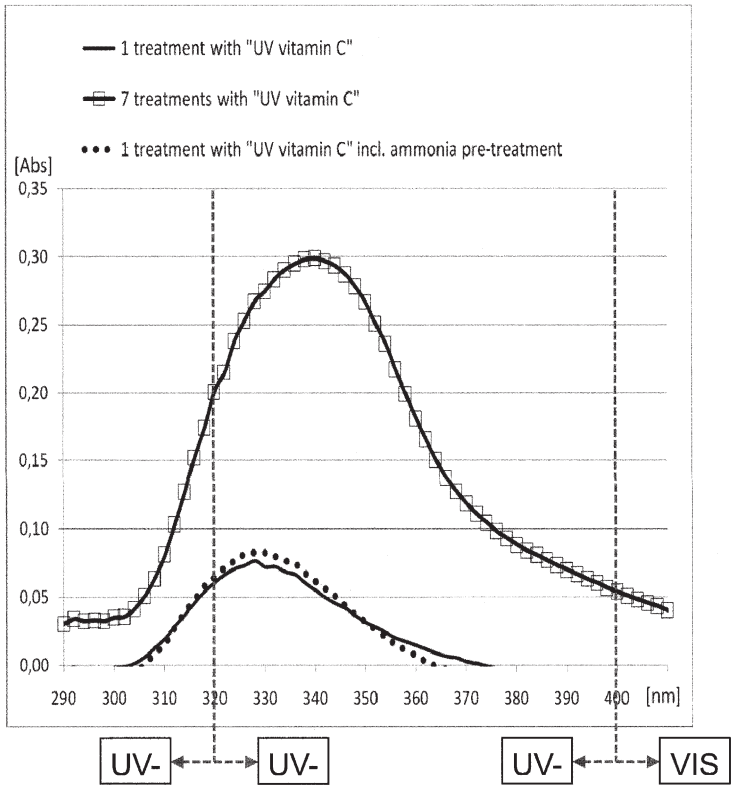



Fig. 1: White hair treated with "UV vitamin C"; absorption profiles calculated from remission spectra (versus placebo).

In a wash-off resistancy test according to test A, "UV vitamin C" showed almost unchanged colors on blond hair before and after 10-shampoo-washings (dL*da*db* [versus untreated] "before 10 shampoo-washings": dL* = -5.0; da* = 1.5; db* = -2.6); "after 10 shampoo-washings": dL* = -5.7; da* = 1.7; db* = -2.4).
The assessment of the antioxidative power according to test B resulted in homogeneously-yellow strands for hair treated with "UV vitamin C" and dark colored strands when treated with comparative antioxidants (vitamin C, bioflavanoid, beta-carotene).
The following color values were measured with test design C) and "VIS vitamin C" as test product: dL*da*db* [versus untreated] "ammonia pre-treatment + 1 product treatment": dL* = -10.4; da* = 5.8; db* = 11.2); "no ammonia pre-treatment + 1 product treatment": dL* = -5.9; da* = 1.3; db* = 2.6; "no ammonia pre-treatment + 7 product treatments": dL* = -8.9; da* = 5.5; db* = 9.2.

Conclusion:
Substances out of the described concept were found to be adhesive to the hair fibre. The delivered functionalities provide stable color or anti-UV-performances. Due to their built-in antioxidant function and due to the option of avoiding hydrogen peroxide, treatments are considered as "mild" to the hair. In addition they help to overcome galenic disadvantages such as unpleasant greasiness or smell.

# Make-Up and Biological Activity on the Skin

Karl Lintner, Ph.D.[1],
Philippe Mondon[2], Emmanuel Doridot[2], Nada André[2], Olga Gracioso[2]
[1]KAL'IDEES S.A.S., Paris, France, [2]SEDERMA, S.A.S., Le Perray en Yvelines, France

## INTRODUCTION:

Color cosmetics (including foundations, lip sticks, mascara and similar decorative formulas have until recently not been designed for inclusion of active ingredients which would, in addition to the immediate masking and decorative effect, achieve biological results in the long run. This situation is changing with the introduction of BB creams ("Blemish Balm"), also called "Fusion concepts", with "active" powders, with soothing lipsticks, moisturizing foundations and similar claims and concepts.

## OBJECTIVE:

The objective was to explore the possibility of combining decorative cosmetic formulations with biologically active ingredients in order to achieve visible short-term effects and beneficial long-term physiological changes. Among the difficulties encountered, one may note formulation, and efficacy measurement (claim substantiation) as instrumental methods from skin care are not always well adapted.

## METHODS:

Three examples are chosen: "active volumizing lipbalm", "eye lash lengthening mascara" and "active anti-wrinkle foundation". The active ingredients (peptides, ceramides) are examined in cell culture studies with normal human keratinocytes, fibroblasts and full thickness human skin biopsies; DNA chip analysis of whole human genome in response to stimulation; ELISA, immunoblotting, histological staining; clinical trials using instrumental analysis (aeroflexmeter™, ultrasound echography, moisturemeter™, FOITS, macrophotography, image analysis).

## RESULTS:

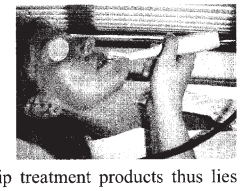
For inclusion in decorative **lip treatment** balms and sticks, a novel peptide (Pal-$KMO_2K$) was designed to stimulate Collagen I, III, IV, and Fibronectin synthesis in normal human fibroblasts. Collagens I and IV, however, produced by dermal cells and deposited in the extracellular space, need to undergo a maturation phase in order to ensure the elasticity of the skin, and to bind the dermis to the epidermis. One of the components necessary for the maturation of certain collagens is the chaperone protein HSP47 (heat-shock protein). Specific to collagen-producing cells, HSP47 binds to collagens I through V. This function enables the collagen to be reinforced and protected, particularly against proteases, before it exits the cell. The absence of HSP47 in mice yields animals whose skin is fragile; Fig. 1 shows the increase in HSP47 synthesis in presence of increased concentrations of the peptide.

*Increase (135% $p<0.01$) in **HSP47** with **Pal-$KMO_2K$** (Western blot):* →

Furthermore, increased synthesis of ECM molecules was observed on human dermal fibroblasts when incubated with the peptide: Collagen I: +133%, Collagen III: +223%, Fibronectin: +73%; EDJ related Collagen IV: +84% and Laminin 5: +62%; moisture promoting Hyaluronic acid: +101% and Aquaporin 3: +31%, all this at the level of 4-6 ppm, which is easily incorporated into lip treatment products (balm, stick, gloss). A clinical trial with a lip-balm containing 5ppm of the peptide evaluated lip tissue firmness, density, moisture, surface quality and curvature/volume with the mentioned techniques. Whereas the placebo balm showed no measurable effects, the "active" lip-balm increased lip moisture, tissue firmness and density, measurably improved softness and shape (volume) after one month ($p<0.01$ to $p<0.05$). One needs to mention the first measurement of lip tissue firmness, for which a novel instrumental technique (Aeroflexmeter™) was used: a non-contact pressurized air nozzle coupled to a laser detection device[1]. To quantify dermal tissue density, an ultrasound miniprobe designed for studying gum disease was used for the first time on lips. The difficulty in formulating "active" lip treatment products thus lies mostly in the technique of claim substantiation, given the special nature of lip tissue.

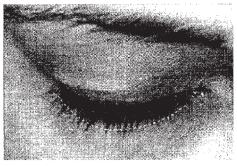
Table 1: ultrasound mesurment of lip tissue density

| | Superficial dermis density restoration | |
|---|---|---|
| | T0 | T 1 month |
| Mean | 113.51 ± 14.37 | 123.17 ± 15.63 |
| Improvement vs. T0 | +8.5% | |
| Significance vs. T0 | p<0.01 | |
| Maximum value | 38.1% | |

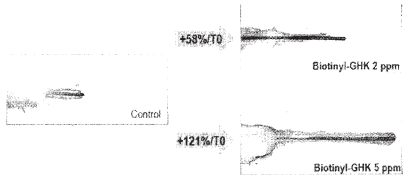


Similarly, to claim an **eye-lash elongation** and **strengthening** activity for a mascara type formulation poses methodological challenges.

[1] Mondon et al. IFSCC 2010, Buenos Aires



A different peptide, Biotinoyl-GHK[2] tested *in vitro* on isolated hair follicles showed significant hair growth properties, stimulation of collagen IV and laminine 5 synthesis, as well as increased cell proliferation[3].



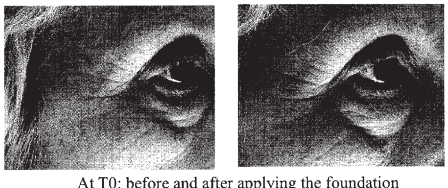

A placebo controlled study measuring the eye lash lengthening and thickening effect of a mascara containing the peptide, combined with panthenol, demonstrated highly significant ($p<0.001$) increase in length of eyelashes after one month of use, with also significant difference compared to the placebo preparation. Lashes (as determined by macrophotography and image analysis) were also thicker (increased volume by 67%, $p<0.001$ *vs.* placebo) and less likely to fall out (tendential observation).

Table 2: Change in eyelash lengths with time: peptide used at 5 ppm

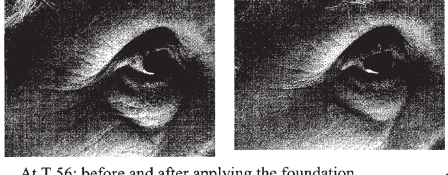
| Length (mm) | Mean variation | |
|---|---|---|
| | T 15 days | T 30 days |
| Versus T0 | +10.6% up to +32%, p<0.001 | +17.0% up to +43%, p<0.001 |
| Versus placebo | x 2.6, p<0.001 | x 2.7, p<0.001 |

Finally, a tinted fluid **foundation** was formulated to determine in a clinical trial over two months, if the combination of a chemotactic peptide implicated in wound healing and a barrier repair ceramide may together help reducing the deep wrinkles of the eye zone. Genomic (see table 3), proteomic (stimulation of elastin synthesis) and cell morphology studies showed that the peptide was likely to act on the ECM, presenting together with the barrier repair capacity of ceramide II the power to reduce the appearance of wrinkles in the face.

| Change in gene expression Pal-oligopeptide | % vs. control |
|---|---|
| Granulocyte chemotactic protein GCP2 | 227% |
| Ephrin receptor gene | 179% |
| Plasminogen activator inhibitor 2 | 166% |
| EGF response factor (or ERF1) | 154% |
| Calmodulin gene | 150% |



At T0: before and after applying the foundation



At T 56: before and after applying the foundation

The particular challenge lay again in showing the effects in a clinical trial, as it is well known that the application of a foundation often leads to an apparent aggravation of the visibility of the wrinkles. Without resorting to physical techniques ("soft focus" and the like), it was possible to demonstrate the restoring power of this "active foundation".

## CONCLUSION

The examples presented show, on the basis of the extensive *in vitro* and clinical data, that this approach to simultaneous immediate skin beautifying and long term physiological skin care is possible and justifiable. The measurable results on the three panels of volunteers are in good correlation with the proposed *in vitro* mechanisms of activity of the ingredients formulated in these make-up preparations.

[2] V. Arul V, Gopinath D, Gomathi, K and Jayakumar R. (2005) Biotinyl-GHK peptide incorporated collagenous matrix: A novel biomaterial for dermal wound healing in rats. Journal of Biomedical Materials Research: Applied Biomaterials 73: 2, 383-391

[3] C Mas-Chamberlin, P Mondon, F Lamy, O Peschard & K Lintner (2005). Reduction of Hair-loss: Matrikines and plant molecules to the rescue. Proc. ASCS Conference Bangkok, Thailand.

# High Performance Color Cosmetic Coating for Prevention of Skin Injury Due to Thermal Insult

D. Michelle McCluskey, Robert Lochhead, Ph.D., Paige Buchanan, Laura Anderson and Kelli Booth

*SciGenesis, LLC and University of Southern Mississippi*

**Introduction:**

Military personnel face a significant threat from IEDs, road side bombs and other ballistic heat events when deployed abroad, especially in combat/conflict zones such as Afghanistan and Iraq. Part of this is due to the exposure of the skin of the hands and face which is left unprotected by the traditional gear worn daily. Current military camouflage face paint does not provide any type of protection from these scenarios and in lab studies has proven to act as an accelerant when exposed to open flame. The current commercially available products are also aesthetically undesirable; their waxy base is occlusive to the skin and also presents difficulty in removal. As such, SciGenesis, LLC in collaboration with Dr. Robert Lochhead, Ph.D. and the University of Southern Mississippi have developed a unique color cosmetic coating which will protect the exposed skin of the soldier using a combination of mechanisms to increase the time required to cause a second degree burn from 2 seconds to 4 seconds, corresponding to a heat flux reduction of 40 kW/m2 down to 15 kW/m2 and thereby providing precious time for personnel to respond. These cosmetic camouflage face paints must meet the rigorous performance requirements set forth in MIL-DTL-32000 in addition to these added benefits.

**Materials and Methods:**

Proof of concept has been established from a leveraged multi-pronged approach to protect skin from intense thermal insult comprising of: reflection of the near infrared radiation via addition of NIR reflectance pigments and diversion of heat through the endothermic evaporation of water from hydrogels within the formulation. The hydrogels are designed to remain swollen by perspiration to ensure their efficacy for the duration of wear under extenuating conditions. Hydrocarbons were minimized in the formulation because they absorb in the flame region and literally 'add fuel to the fire'. Therefore, the formulation was based upon silicones. We selected performance pigments to provide the specific color(s) required by the MIL-DTL-32000 currently in use. The table below is representative of the formulation at the most recent stage of development.

| Ingredient Name | Wt% Final Formulation |
|---|---|
| **Pigment Phase Composition: Phase I** | |
| DI Water | 14-16% |
| Butylene Glycol | .09-.15% |
| Glycerin | .09-.15% |
| Propylene Glycol, Diazoldinyl Urea, Methyl Paraben and Propyl Paraben | .09-.15% |
| IR Reflecting pigment (iron oxides and chromium oxides) | 7-12% |
| Dimethicone | 1.5-2.5% |
| Cyclopentasiloxane | 7-9% |
| PVP/Hexadecene | 1.5-3% |
| Ammonium Nonylphenol ether sulfate | 7-12% |
| Dimethicone | .5-2% |
| Total Pigment Phase: | 48 |
| **Hydrogel Phase Composition: Phase II** | |
| Deionized water | 44-47% |
| Sodium Chloride | .2-.3% |
| Calcium Chloride | .1-.2% |
| Magnesium Sulfate | .005-.015% |
| Potassium Dihydrogen Phosphate | .005-.001% |
| Triethanolamine | 1-2% |
| Acrylates/C10-3- Alkyl Acrylate Crosspolymer | 1-4% |
| Total Hydrogel Phase: | 48 |
| Polyester-5 | 4 |
| | 100 |

Formulating of this product is conducted in a two stage process. The 'pigment phase' is an oil in water emulsion with no phase inversion occurring during preparation, resulting in the water phase as the external phase. The hydrogel phase is prepared separately and 'pre-swollen' in the presence of salt concentrations typically found in human sweat to guarantee the stability of the gel in the event the wearer begins to perspire. These two phases are combined using counter-rotation and film former is added before a final mixing event on the counter-rotating equipment. The resulting product is a smooth continuous cream that when applied, feels cool and breathable to the wearer. In order to match the shades required in MIL-DTL-32000 monochrome shades are prepared using the aforementioned techniques and then blended to achieve the appropriate color. Confirmation of the color-match is obtained using a BYK handheld colorimeter to take three consecutive CIE LAB readings which are then averaged. Sweat resistance is characterized by spreading the formulation onto clean untreated fine woven polyester having a pore size of approximately 100um, this sheet is then exposed to simulated sweat from both above and below. Transfer resistance is characterized by spreading the formulation onto the same type cloth and allowed to rest for ten minutes, 100% cotton is then placed over the top of the cloth with a 2lb. weight for ten seconds. The cotton sheet is lifted and inspected for evidence of transfer.

**Thermal Challenges**

Certainly the crux of this research rests on the formulation's ability to protect a substrate from thermal insult. There are currently few ASTM standard methods available capable of simulating real world fire and blast scenarios. As such, SciGenesis, LLC and collaborators at the University of Southern Mississippi have developed two in-house challenges in addition to utilizing Ballistic TGA and Cone Calorimetry.

The 'flame-test' comprises of a inverted MAP torch mounted six inches above the surface of an aluminum coupon. A thermocouple is mounted to the reverse side and five grams of product is spread evenly over the surface of the coupon. The tip of the flame when the torch is lit touches the surface of the formulation and the temperature detected by the thermocouple is reported as a function of time. Figure 1a below illustrates the flame-test set up. Current product performance in the flame test shows a 22 second average time to 60°C. Testing to determine protection again irradiative heat is carried out using a custom designed quartz lamp emitter from ProTherm Inc. Sensors typically imbedded in 'fire mannequins' used to mimic the thermal conductivity of human skin to evaluate textiles are purchased from Pyrocal. The formulation is applied to the surface of the sensor at approximately 4mil thickness and the sensor is then exposed to the lamps which produce temps of approximately 700°C. Again, the temperature as a function of elapsed time is recorded and the time in seconds required for the sensor to detect 60°C is marked. Figure 1b is data representative of the product performance for the quartz lamp challenge.

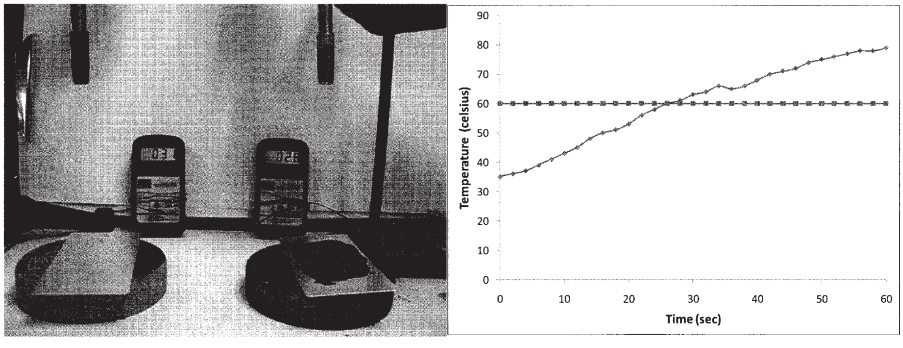



**Figure 1 (a) Flame-test set up** **(b) Quartz Lamp data for current prototype**

**Conclusion**

In summary, SciGenesis, LLC has successfully demonstrated the ability to formulate a high performance color skin cream coating which protects the skin from thermal insult during blast scenarios while also providing to the wearer the aesthetics and comfort of conventional cosmetic products. We anticipate this product will successfully enter the military and first responder market and provide much needed protection to our military and first responder personnel both at home and abroad.

# FRONTIERS OF SCIENCE AWARD LECTURE

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## COSMETICS AND TOILETRIES®

# Biomaterials and Biotechnology: From Discovery of the First Angiogenesis Inhibitors to the Development of Controlled Drug Delivery Systems and the Foundation of Tissue Engineering

Robert Langer, Sc.D.

*Massachusetts Institute of Technology*

In this presentation, I would like to talk about how I became involved in the field of biomaterials and about some of the research my group and I have conducted to understand and create biomaterials that could be useful in various areas of medicine and possibly cosmetics.

During the past 30 years, a whole new field of materials-based drug delivery systems has emerged. These polymer-based systems have had a dramatic impact on safe and effective drug delivery. In my postdoctoral work, I trained with Dr. Judah Folkman to figure out how blood vessels grow in the body. He wanted to see if there was some way to isolate a substance that could stop this blood vessel growth, something he called an "angiogenesis inhibitor." When I started working with him, angiogenesis inhibitors were only theoretical, and many people did not agree with Dr. Folkman's concept that if you could stop blood vessels from growing to tumors, the tumors would stop growing. Moreover, this area of blood vessel growth was difficult to study. We realized that in order to solve this problem, we would not only need to isolate an angiogenesis inhibitor, which is often in the form of a large molecule, but we would also need to find an assay. We chose the eye of a rabbit, because there are normally no blood vessels in the eye. We put a tumor in the eye that would mimic what happens in the human body. Over time, blood vessels grew from the edge of the cornea to the tumor. At that point, we wanted to stop those blood vessels, but to do that we needed a controlled-release polymer that could deliver the different molecules I was isolating. Since there were no such systems at that time, I decided to try to develop one. I can trace my interest in the drug delivery field back to this project.

In 1974, one of my goals was to see if we could release different molecules that I might isolate from cartilage, the tissue I was then studying for mining angiogenesis inhibitors. A lot of the molecules were large. At that time, there were no systems for delivering these kinds of molecules for long time periods and in a way that would be safe in the human body. So the work I was doing was somewhat basic—to help in angiogenesis research. However, from the standpoint of potential practical impact, something happened that I could not have anticipated. That was the advent of biotechnology and genetic engineering where, for the first time, it became possible to create large molecules such as peptides and proteins in a commercial way. But these molecules faced serious delivery challenges. Swallowing them did not work because they were too large and would be destroyed by enzymes or acid in the stomach or intestines. They were also too large to use in a patch. If you tried injecting them, they were quickly destroyed by enzymes. Delivering any of these molecules on a chronic basis would require a way to deliver them in an unaltered form, and yet protect them from harm.

When we started this work, the conventional wisdom in the field was that it could not be done. Scientists felt it was not possible to slowly release large molecules from biocompatible polymers. Against this background, I began working in the laboratory to see if I could come up with a way to make tiny systems that could deliver molecules for long times. After two years of experimentation, I had found many different unsuccessful methods. Finally, I discovered one way to make it work. My students and I took hydrophobic polymers like ethylene vinyl acetate copolymer or lactic glycolic acid copolymer and dissolved them in organic solvents, usually at low temperatures like -80°C. We added the proteins to them and slowly dried off the solvent. This is how we created small microspheres or even nanospheres.

We published in *Nature* that you could use this approach to release molecules of almost any size, from 14,000 MW to a quarter of a million MW. These molecules could be released for more than 100 days *in vitro.* Although the release rates were not constant in those initial studies, we later developed ways to ensure constant rates. For example, we were able to achieve a constant release of albumin for over 50 days. At first, our concepts were not well accepted, but today these approaches are now widely used.

Furthermore, most drug delivery systems are built around the idea of engineering materials to do things they have never done before. That has been one of the primary pursuits of my research. Another facet of our work has been investigating how materials find their way into medicine, and creating newer, possibly better, materials. Being a chemical engineer, I had once thought that experienced chemical engineers or chemists were the driving force for bringing materials into medicine. But the closer I looked into that theory, the less I found it to be true. Rather, medical doctors were the ones who identified problems in their field and, urgently wanting to fix them, came up with materials solutions. They would search their surroundings, for example, their homes or local stores to find objects that closely resembled the organ or tissue they wanted to fix. They would then adapt that material for use in the human body. While that practice has resulted in some solutions, it has also created problems.

For example, in 1967 at the National Institutes of Health, some clinicians and engineers wanted to make an artificial heart. They started by asking, what object has a good flex life, like a heart? The answer they came up with was a ladies' girdle. They then determined that since the girdle was made of a polyether urethane, they would make an artificial heart from that material. Today, many years later, we find that the artificial heart is still made of that same material—polyether urethane. Yet, when blood hits the surface of the artificial heart (the ladies' girdle material), it can form a clot. That clot can then go to the patient's brain and cause a stroke, which could result in death.

Similarly, dialysis tubing was originally made of sausage casing. The vascular graft, which is an artificial blood vessel, was developed by a Texas surgeon who searched for possible materials in a clothing store, based on what fabric would be easiest to sew. He chose Dacron. Of the two materials chosen for breast implants, one is a lubricant (silicone) and the other is a material used for stuffing mattresses (polyurethane).

Against this background, we and others began thinking that we needed to find a model for solving medical problems other than to search for materials in everyday settings. As a chemical engineer, I believed that researchers could take an engineering design approach, asking the question, what do we really want in a biomaterial from an engineering standpoint, from a chemistry standpoint, and from a biology standpoint? If we could answer those, we could then synthesize the materials from first principles.

We started by developing a particular class of materials—synthetic degradable materials. Using an engineering design approach we designed a new family of polymers that led to a new FDA approved treatment for brain cancer. We have also used similar approaches to create new hair care products. All of this will be examined in my lecture.

# A New Hair Straightening System Showing High Performance and Low Damage

Timothy Gao, Ph.D., Charles Moses, Zhi Li, Jung-Mei Tien, Ying Xia He and Peter Landa

*Croda Inc., 300-A, Columbus Circle, Edison, NJ, 08837, USA*

## INTRODUCTION

Hair straightening is one of the most popular hair treatment procedures in salons across the world. Currently, there are mainly three different types of hair straightening systems based on chemicals and mechanisms: Hair relaxing – using hair straightening solutions containing NaOH; Japanese straightening – using hair straightening solutions containing thioglycolate/dithiodiglycolate and followed by a hot flat iron (~180°C) treatment; Brazilian hair straightening – using straightening solutions containing formaldehyde and followed by high temperature (~220°C) flat iron treatment. While all these methods achieve straight hair, but all have drawbacks - making hair dry and damaged. Recently, Occupational Safety and Health Administration (OSHA) issued a hazard alert, warning that formaldehyde-containing hair-straightening products could cause serious health problems, including increased risk of cancer. Several countries have recalled the use of the formaldehyde based hair straighteners (1).

A new Cystine Hair Smoothing System (CHSS) has been developed by Croda Inc. to reduce hair damages and eliminate the health risk from using formaldehyde. This paper presents our recent studies on this new system

## EXPERIMENTAL

### *Materials*

- Natural curly hair was purchased from International Hair Importers, Inc., New York and used for straightening.
- Four different hair straightening systems were evaluated (CHSS, a Lye-Relaxer, a Thio-perm formula, and a commercial market product).

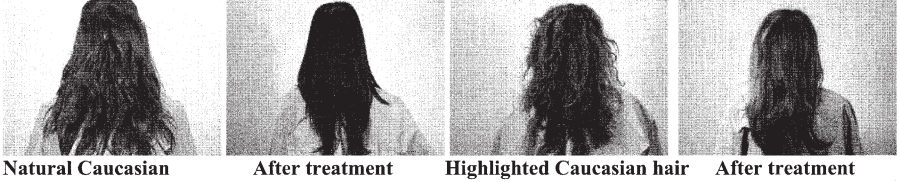
### *Instruments and Test Methods*

- An Atomic Force Microscope (AFM), Dimension Icon-System from Bruker Nano Surfaces, USA was used to generate 3D images of hair surfaces and quantitatively determine average changes in cuticle heights and angles before and after respective straightening processes
- A Miniature Tensile Tester (MTT-670) from Dia-Stron, UK was used to determine tensile strength of hair fibers at constant 22°C and 65%RH
- A K100 Tensiometer from KRUSS, Germany was used to measure dynamic advancing contact angle of single hair fibers at room temperature (~22°C)

## RESULTS AND DISCUSSION

### 1. Evaluation of Hair Straightening Performance

Full head images of panellists with Caucasian thick hair before and after CHSS treatments are shown in Figure 1. It can be seen that after 5 weeks of treatment, the hair still remained straight.

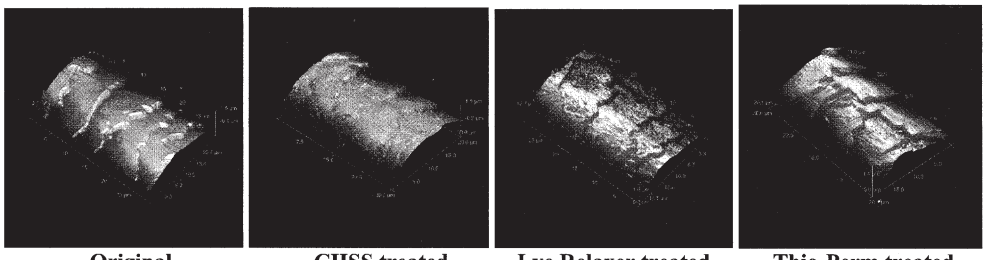


**Natural Caucasian** **After treatment** **Highlighted Caucasian hair** **After treatment**

**Figure 1 Images of Caucasian hair before and 5 weeks after treatments**

### 2. Determination of Hair Damage by AFM

Typical AFM 3-D images of hair surfaces treated with different hair-straightening systems are presented in Figure 2. Experimental results obtained from image "Section Analysis" (2) are listed in Table 1. It is observed that CHSS remained the surface cuticle integrity during the straightening process and showed much better protection from damages compared to relaxers, commercial hair straighteners, and Thio-perm formulations.



**Original** **CHSS treated** **Lye Relaxer treated** **Thio-Perm treated**

**Figure 2 AFM 3D images of hair surface with different hair-straightening treatments**

**Table 1 "Section Image Analysis" Results and Contact Angle Measurements**

| Treatment | Cuticle height (nm) | Cuticle angle (°) | Cuticle layers | Contact angle (°) | Change, % |
|---|---|---|---|---|---|
| Original | 673 | 5.6 | 5 | 90.3 | 0 |
| CHSS | 667 | 5.6 | 5 | 87.8 | -2.8 |
| Thio-Perm | 555 | 23.0 | 4 | 86.0 | -4.8 |
| Commercial | 544 | 3.3 | 3.5 | 86.0 | -4.8 |
| Lye Relaxer | 413 | 33.1 | 3.5 | 81.2 | -10.2 |

### 3. Changes in Tensile Strength of Hair Fibers

Figure 3 explains average relative changes in tensile strength of hair fibers treated with different hair straightening systems. It is found that hair fibers treated with CHSS remained the hair strength and showed the least increase in strain-to-break which is an indication of hair plasticization.

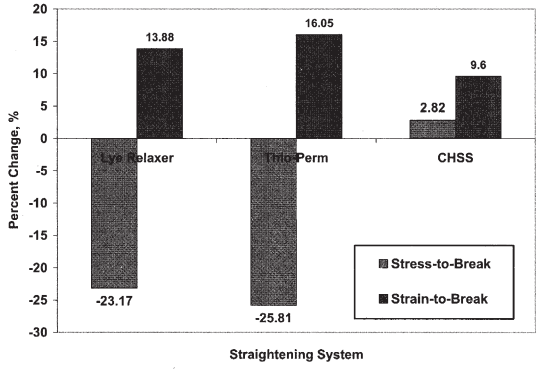



**Figure 3 Relative changes in Tensile strength of hair fibers after different treatments**

### 4. Changes in Average Dynamic Advancing Contact Angles of Hair Fibers

Data on average dynamic advancing contact angles and their relative changes are also listed in Table 1. It is clear that hair treated with CHSS showed the least changes in average dynamic advancing contact angle compared to other systems.

## CONCLUSIONS

- CHSS is a complete, safe and significantly milder system with functional proteins and actives for healthier hair and enhanced effects
- Regime usage remains hair strength and reduces damage from repeated straightening treatments
- AFM is a very useful and powerful tool to study modifications on hair surface in a 3-D mode.

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# Models for Evaluation of the Enviromental Stress on Hair Fibers

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**Introduction:**
Environmental pollution is and remains an actual topic. The skin and the hair are in direct contact with the environment and they consequently remain the main targets for the effects of environmental stress.
Skin has intrinsic mechanisms for protection against external aggressions such as pollution, whereas hair lacks such intrinsic protection and regeneration systems. Therefore it is of great importance to design experimental models to mimic the effects of pollution on hair fibers and to develop the topical treatments that would help to counteract these aggressions and repair related induced damages.

**Objective:**
The objective of the study is to present some models to demonstrate and to quantify the effects of pollution and environmental stress on hair fibers' properties.

**Methods:**
In a "city pollution model" the negative effects on hair caused by consecutive exposure to exhaust gas and UV radiation are evaluated. Hair tresses are put in an environmental chamber and stressed by exposure to exhaust gas (6 hours) /UVB irradiation (16 hours; UVB 2.5 mW/cm²) cycles. The combability measurement serves as the end point of quantification of hair surface damages.
In a dust particle test, the adhesion of carbon particles on hair, similar to those prevalent in urban pollution, is visualized and quantified. Small hair tresses are brought in contact with a defined quantity of carbon particles. The excess of carbon particles is removed by standardized mechanical shaking. After the observation by scanning electron microscopy, the adhered particles are quantified by image analysis.
In addition to these two original models, other approaches for the demonstration of the deleterious effect of UVB effects on hair fibers are presented: determination of level of the aromatic amino acid tryptophan and modifications of biomechanical parameters such as the post-yield slope.
The overall protein modifications are evaluated and quantified after the reaction with dansyl chloride.
The oxidative damage of UVA is measured by the formation of free radicals in hair fibers.

**Results:**
***Environmental pollution***
The alternate exposure of human hair to exhaust gas and UVB irradiation has caused the modification of hair surface properties that were quantified by an increase of combing parameters, combing work and maximal combing force. The increase was proportional to the extent of exposure to pollutant (Figure 1).

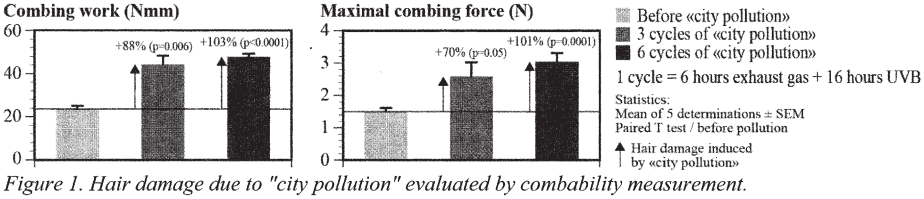
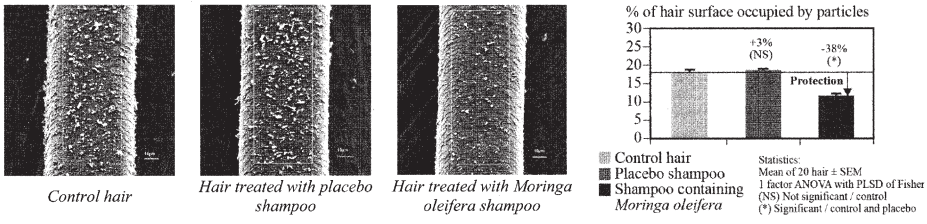


*Figure 1. Hair damage due to "city pollution" evaluated by combability measurement.*

The adhesion of particles was determined as a percentage of surface occupied by the particles to the total surface of the measuring window. In control non treated hair this surface represents about 20%. This percentage was not modified when hair was pre-treated with a simple shampoo formulation and was dramatically reduced after pre-treatment with a shampoo containing *Moringa oleifera* proteins (Figure 2).

The lower particles adhesion on the surface after the treatment with *Moringa oleifera* proteins is linked to the film forming properties and also to the positive charge of these proteins (1, 2).




***UVB photodegradation of hair***

The reduction of the tryptophan content in human hair after UVB irradiation may be considered as a measure of photo-degradation of hair proteins.

Both spectrocolorimetric determination (3) and the measurement of fluorescence spectrum of tryptophan on previously irradiated hair (4) are useful tools for the evaluation of UV induced hair damage (Figure 3).

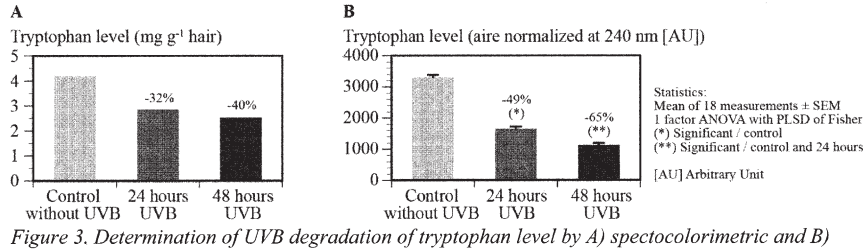



*Figure 3. Determination of UVB degradation of tryptophan level by A) spectocolorimetric and B) spectrofluorimetric methods.*

Another approach to the evaluation of hair protein modifications due to the UVB irradiation is the measurement of hair fluorescence after the reaction with dansyl chloride (5).

The measurement of tensile properties is one of the most widely used hair evaluation techniques (6). From the analysis of load-extension curves, we have selected the post-yield slope as the most representative parameter of hair damage due to UVB. The decrease of post-yield slope may be interpreted as a mechanical weakening of the hair and a deterioration of hair fibres. A dose dependent decrease in post yield slope with an increasing UVB dose has been demonstrated.

***UVA hair oxidative damage***

Oxygen radicals are believed to be involved in the photodegradation of hair fibers. The UVA irradiation causes oxygen radical formation.

The technique of evaluating free radical formation by fluorescence is based on the measurement of the oxidation of terephtalic acid dianion by hydroxyl radicals formed during UVA exposure of hair (7).

Carefully prepared blank experiments allow distinguishing between the fluorescence intensity caused by the release of fluorescent material from the hair fibers without UVA and the fluorescence due to the oxidation of terephtalic acid into 2-hydroxyterephtalic acid by UVA induced free (hydroxyl) radical formation.

**Conclusion:**

Hair is exposed to environmental stress every day and throughout the year. Even if the concept of protecting hair against environmental stress is not entirely new, the formulation of hair treatment products able to attenuate its harmful effects and the development of new models capable to support the protective claims for hair care are necessary.

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# A Global Approach to the Differentiation of Styling Polymer Performance on Asian, Caucasian, Mulatto and African American Hair

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**Introduction**

A key driver in the hair care market is fulfilling the needs of consumers of various ethnic backgrounds. Hair types differentiated by racial background have different degrees of stylability or the ease to which the hair can be put into a particular shape. Also, it has to be held in this shape despite environmental or mechanical stress. Although there is no difference chemically between the major ethnic hair groups[1], each hair type, whether it is of Asian, Caucasian, African, or mixed racial descent, has a distinct morphological structure. These structural differences affect how hair responds to mechanical stress. This, taken together with the degree of damage of the hair and its consequent response to humidity, are factors in how styling resins perform in the styling process. It is essential, then, in the differentiation of hair styling resins to study not only the chemistry and features of the polymers themselves, but also the nature of the hair upon which the resin is attached.

**Objectives**

This paper will explore the important physical and chemical features of styling resins to properly differentiate their use for hair types of different racial origin that have their own characteristic propensity to be placed and held into a particular style due to differences in morphological structures.

**Methods**

Tests were conducted using hair tresses based on different degrees of curl. Hair types ranged from Asian straight hair to moderately curly hair such as is characteristic of hair from people of African-American decent[2]. To show structure-property relationships on performance of fixative resins, a cross section of polymers from simple hydrophilic homopolymers to more advanced functionalized copolymers were selected for testing. In order to understand the behavior of the different hair types, both with and without polymer pretreatments, a collection of appropriate test methods were employed. The mechanical property of stiffness of polymer treated hair was measured at two humidities using a Texture Analyzer. For this, hair was tested in the configuration of omega loops and planar configurations applicable for three point bending.[3] With the support of an equation that approximates the deformation of hair tresses formed in an omega loop, as well as the parallel axis theorem, the composite nature of the polymer-fiber assembly interaction is described, both on a theoretical basis as well as through supporting data.[4] High humidity curl retention of untreated and polymer pretreated hair of various types were measured using the industry accepted test method, except for using the natural length instead of the extended length of the hair tress in the standard equation.

**Results**

Texture analysis on hair tresses shaped into omega loops showed that there is an exponential increase in stiffness when treated with a styling polymer. Also, Caucasian hair consistently has a higher stiffness compared to Asian and Mulatto hair types. This is attributed to a greater number of inter-fiber connections for polymer treated Caucasian hair, since more fibers of this hair type can be packed into a fixed unit volume due to its smaller diameter. High humidity curl retention also shows differences in performance among the different hair types. Untreated tresses show that Asian hair has a lower curl retention compared to hair that has a more distinct curl such as Mulatto and African American. These trends are consistent when hair is pretreated with hydrophilic polymers. Polymers used as a pretreatment that form increased hydrophobic and mechanically durable films show high humidity curl retention despite hair type.

**Conclusions**

Both the mechanical properties of hair, which are determined by hair morphological features, as well as the performance properties of the polymeric film, need to be considered to properly recommend a fixative polymer for a particular racial hair type. These morphological features affect the ease or difficulty of putting hair into a particular style. Results from this study demonstrate that the factors to consider in hair stylability that are dependent on hair morphological structures consist of:

1. The composite nature of the hair fiber assembly interaction which is based on the packing density of fibers of different diameters where the more fibers per unit volume results in more inter-fiber connections. The consequence of this is higher stiffness. Also, the weave of the hair based on its degree of curliness also affects this packing density.
2. Hair assemblies of different ethnic origin have different mechanical properties. Here the polymeric resin has to counteract the hair's natural tendency to revert to its natural configuration.

From this it is concluded that polymers that create a resinous film with a greater mechanical durability, especially under the stress of high humidity, can be recommended for hair that is hard to style. An example is Polyquaternium-69 which contains in its chemical makeup monomers with specific functional groups that create an elastic, durable, and hydrophobic film that allows it to be resistant to the stress of mechanical fatigue and humidity. From this study an example of an application of this polymer is its use for Asian type hair which is hard to style. In this case a more durable polymer is needed, due to less inter-fiber polymeric connections and a high mechanical strength to counteract the force for the hair reverting to its straight configuration.

Overall findings demonstrate that there is a complex interaction and inter-dependant relationship between the multiple factors of fixative polymer chemistry, morphology and mechanical properties of hair, and hair fiber assembly configuration.

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# An Alternative Method for Reshaping Hair

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## Introduction

Personal care and the methodologies used to affect one's appearance very likely dates back to antiquity. An important component of personal care is the practice of reshaping or resetting the hair on the scalp. Known variously as perming, relaxing, water setting and heat setting, for example, it involves the use of a physical (heat, water) or chemical (thioglycolate, alkali) agency to change the shape of the hair assembly on the scalp. In brief, in the temporary setting of hair (vulnerable to moisture and physical pressure), either water or heat is used to endow hair with a desired shape. Styling formulations are constitute another component of temporary setting of hair tresses. Perming of hair, which results in a more permanent shape, reducing chemicals, such as thioglycolic acid solution, are used to break the structurally important disulfide bonds of the hair protein and the reshaped hair is treated with hydrogen peroxide to reform the broken disulfide bonds in the new set.

Further breakthroughs in this area call for investigating potentially new methods. In this context, if one were to look, *schematically*, upon the scission of the disulfide bond as a reduction process involving first the breakage of this bond and subsequent combination of the sulfur with H atoms, cathodic reduction that has been extensively used in other areas appears to be a good candidate to break the disulfide bond. The present work is a communication of some preliminary results wherein cathodic reduction of hair has been used to reshape hair successfully.

## Experimental

In this preliminary feasibility study, no specific attempt has been made to optimize all the parameters such as the electrolyte composition and physical state (gel, viscous fluid), electrode potentials, cell design, etc.

***Electrolyte and the electrochemical cell:*** The electrolytic reduction has been attempted in the pH range of 4 to 10. The electrolyte, for each pH used, was a commercially available buffer solution. The electrode arrangement is shown in Fig. 1. It consists of three rows of metallic pegs (stainless steel) embedded in an insulator back plate. The middle row of pegs is connected in parallel and form the anode part of the unit. The outer rows are again connected in parallel and form the intra-connected cathode array. A small swatch of medium brown Caucasian hair was wound around the cathode pegs, contacting them, to give the hair fibers a wavy shape. The electrode assembly with hair in place was dipped in the respective electrolyte solution and electrical power turned on. The voltage has been varied in the range of 2 to 5 volts, as long as it is above that needed for the electrolysis of water. It is recalled that depending on the cell conditions, overvoltage figures in this minimum voltage needed to observe gas evolution. The current through the cell varies with the applied voltage and the impedance of a particular run. In this exploratory work, currents from a few amperes to tens of amperes have been used.

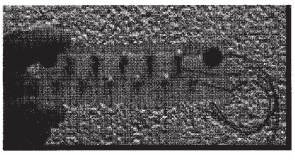

**Fig. 1 The electrode array**

***Post-electrolysis treatment of the tress:*** Assuming that the process here is the disulfide scission to give –SH ends and that the reformation, like in the chemical route, needs a peroxide oxidation step, the tress, after electrochemical reduction was dipped in hydrogen peroxide solution (6%, pH 5) for 10 min and rinsed in water.

**Results and Discussion**

Images of the resultant tresses obtained, respectively, at pH's 4, 7, 8 and 10 are shown in Fig. 2 (1 h electrolysis). Medium brown hair as-is, before electrochemical reduction, is also shown for comparison. In order to demonstrate that what was observed and shown in Fig. 2 was indeed the result of an electrochemical cathodic reduction of hair, and not from any extraneous factor like ohmic heating, the experiment was repeated at one of the pHs, namely 10, but with the polarities changed. That is, the hair contacting the pegs was now part of the anode assembly. No wavy hair was produced as shown in Fig. 3. It is also seen from Fig. 2 that pH 7 and above give better defined waves.

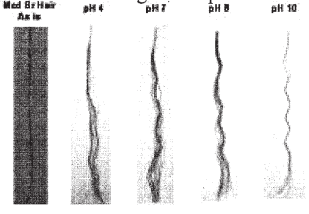


**Fig. 2 Electrochemically reshaped tresses**

**Fig. 3 Hair tress contacting the anode after electrochemical treatment**

The ability to produce waves in hair over this wide range of pH suggests that what is happening is the electrolysis of water and that the hair, being in the vicinity of the cathode, is being subjected to the reductive process.

What is hypothesized here, based on these results, is that, during the hydrolysis of an aqueous system, H• atom free radicals are produced which would normally combine to produce hydrogen gas at the cathode. However, before these short lived H• atoms recombine, if a susceptible substrate such as the disulfide bonds in the keratin structure were to be in the vicinity, some of these hydrogen atoms would react with the disulfide bond to yield the –SH moieties. That this could be a hydrogen atom initiated free radical reduction process was tested by carrying out an identical electrochemical cathodic reduction run, but with the electrolyte (buffer at pH 10) also containing 0.4% benzophenone – 4, a known free radical scavenger. The absence of any well defined wave after the cathodic reduction is shown in Fig. 4. This supports the view that this is a free radical (H•) reaction with the disulfide bond.

It is further suggested that the process described here is the generally practiced "perming" process, except that the agent that causes the scission of the disulfide bond is different. That the cathodically reduced hair needs the subsequent peroxide oxidation step is demonstrated when one examines some preliminary dry tensile mechanical data for the hair fibers encountered here (Table 1). It is noticed that the tensile mechanical properties are, on the whole better for the peroxide treated post-electrolysis fibers compared to those that were not peroxide "neutralized". Hence, it is reasonable to suggest that the chemistry at the disulfide bond is the scission of the bond during cathodic reduction and the regeneration of some of them during the peroxide treatment phase.

**Fig. 4 Hair tress after cathodic reduction in electrolyte containing benzophenone - 4**

**Table 1 Comparison of tensile properties****

| Hair sample | Elastic Modulus (GPa) | Work to extend 15% per unit vol ($MJ/m^3$) | Post-yield modulus (GPa) | Break stress (MPa) | Work to break per unit volume ($MJ/m^3$) |
|---|---|---|---|---|---|
| Control | 2.73 ± 0.06 | 13.63 ± 0.12 | 0.33 ± 0.01 | 193.25 ± 4.28 | 63.46 ± 2.60 |
| ER at pH 10 | 2.92 ± 0.06 | 11.74 ± 0.22 | 0.29 ± 0.02 | 161.27 ± 9.89 | 47.46 ± 4.67 |
| ER @ pH 10 No peroxide oxidation | 2.95 ± 0.04 | 11.54 ± 0.13 | 0.22 ± 0.03 | 130.24 ± 8.72 | 34.09 ± 3.90 |

**Uncertainties are the standard errors; ER=Electrolytically reduced

In summary, electrochemistry of keratins in the hair fibers opens up new avenues for exploring one of the most interesting fibers in nature. It is conceivable that this electrolytic route might also offer another alternative to the glycolate or other methods currently in use for "perming" hair.

# Polarization Spectroscopy Imaging for Assessment of Erythema and Blanching in Skin Care Product Testing

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OBJECTIVE:

There is a need for quantitative and versatile methods for laboratory and work-site assessment of skin erythema and blanching in the development and testing of skin care products. The objective of this presentation is to explain the operating principle of polarization spectroscopy imaging for investigation of the skin microcirculation and other skin parameters and to give some examples of how this emerging technology - introduced under the name Tissue Viability Imaging (TiVi) in the scientific literature - is used in practice.

METHODOLOGY:

Polarization spectroscopy imaging is a method used to map local concentrations of red blood cells (RBC) within the skin microcirculation (1). A digital camera fitted with cross-polarizing filters blocks out direct skin surface reflections producing photos that are analyzed using only the components of the red and green color planes (Fig. 1). Since RBCs mainly absorb light in the green wavelength region, areas in the green plane of the photo corresponding to high RBC concentrations display attenuated values, while corresponding areas in the red plane are virtually unaffected. By the use of an algorithm that calculates the relative difference between each element in the red and green planes, an RBC concentration map is constructed in which the values of the individual elements scale linearly with the local RBC concentration in the skin.

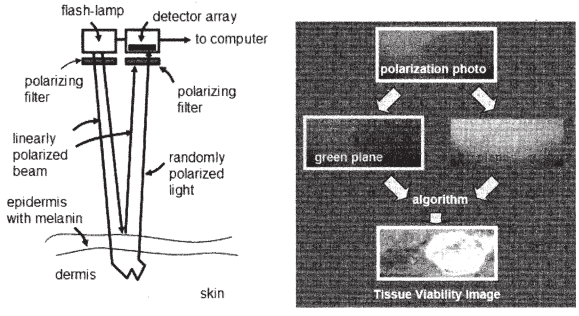


Fig. 1. Operating principle of TiVi (left). Construction of a TiVi-image from the red and green plane (right).

RESULTS:

By use of mathematical modelling of light scattering in tissue and an in-vitro model comprising a transparent tubing system filled with blood of known RBC concentration, the linear relationship between measured and actual RBC concentration was established (cc = 0.997). The influence of a varying degree of melanin in the epidermal layer on the estimated local RBC concentration can be eliminated to ensure the integrity of the RBC maps.

APPLICATIONS:

Typical applications of TiVi include visualization of skin erythema following the topical application of a vasodilating agent such as methyl nicotinate. Since erythema accumulates over a time period of ten minutes, about 100 images may be captured in sequence at a rate of 6 images per minute to visualize the process. Following the capture of all images, an integrated software "wizard" calculates and graphs both the average erythema intensity and the erythema area increases over time within the designated test areas (Fig. 2), crerated by user defined regions of interest (ROI). Using the TiVi technology, Wiren et al (2) investigated changes in erythema following application of 0.5% benzyl nicotinate (BN) in formulations with different lipid content. They observed that BN formulations containing 10% fat induced erythema more rapidly and with higher intensity than formulations with higher fat content and concluded that the rate of penetration of the active ingredients was inversely related to the lipid content.

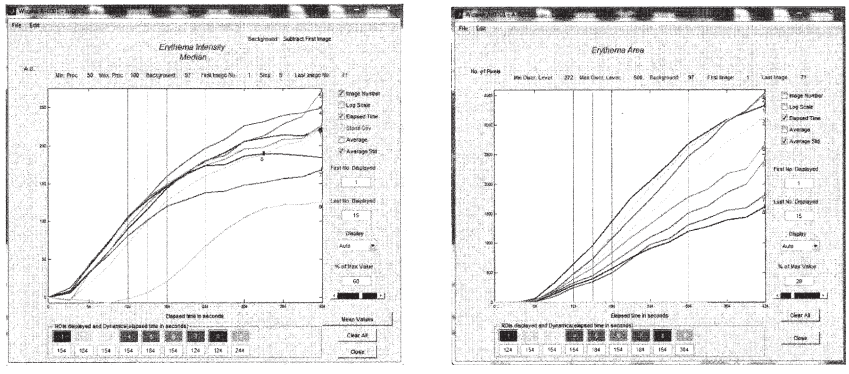


Fig 2. Time course of erythema intensity (left) and erythema expansion (right) following topical application of methyl nicotinate (10 mmol). Note the delayed effect inside a region of interest drawn in between the test areas, indicating lateral diffusion of the applied vaso-active substance (lowest curve in left diagram).

Following topical application of desoximetasone gel 5% (Topicort®, TaroPharma, Taro Pharmaceuticals USA Inc., Hawthorne, NY, USA) on the volar side of the forearm under occlusion for 6 hours to produce skin blanching, readings with TiVi and Minolta Chromameter CR 200 (Minolta, Tokyo, Japan) were compared (3). The TiVi system delivered values similar to those recorded with the CR 200 device; however, the remote recording capability and investigator independence afforded by the TiVi system yield critical advantages. One unique function of TiVi is that the entire progression of the blanching process from start to finish can be observed and related to normal skin RBC concentration by capturing photos through a transparent occlusion cover. This is only possible due to the effect of the integrated TiVi cross-polarization filters which sharply reduce specular reflections from the cover material.

CONCLUSION:

The emerging technology of polarization spectroscopy imaging has been integrated into a portable device (Tissue Viability Imager) that allows for investigation of skin microcirculation and other skin parameters in the laboratory as well as at the work-site. Early applications demonstrate how this emerging technology can be used in the assessment of skin erythema and blanching in skin product development and testing.

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# Factors Influencing Hand Skin Health in Health Care Workers: Practices, Environment, Genetics and Treatment Response

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A national patient safety goal is to reduce health care associated infections (HAIs) as they affect 1.7 million people per year and cause 99,000 deaths. Routine hand hygiene prevents health care associated infections yet compliance is only 30-57%, primarily due to irritant contact dermatitis (ICD) including painful fissures or bleeding cracks. Chronic ICD may *negatively* impact infection control, since bacteria counts are higher with skin compromise, resulting in lost work time and increased costs. Repetitive hand hygiene leads to chronic irritant contact dermatitis in nearly all HCWs regardless of season (1, 2). Inflammation is particularly high in winter. Importantly, the skin does not recover during time off and HCWs return to work with significant skin compromise. Erythema and scaling from chemical irritants are associated with a polymorphism at position −308 on the TNFα gene, a portion of the promotor region that regulates TNFα production. Individuals with a G to A transition (AA/GA genotypes) have a lower threshold for irritation (3). The severity of skin irritation and the AA genotype are related in wet-work occupations (4). Subjects AA have a more intense neurosensory response to lactic acid and water than GG (5). Uninvolved skin in people with an atopic diathesis has subclinical inflammation and greater irritancy to sodium lauryl sulfate (SLS). Patients with atopic dermatitis have increased SC permeability, reduced ceramides and filaggrin gene mutations.

We hypothesized that the responses to chronic exposure, i.e., repetitive hand hygiene, would differ for TNFα AA versus GG genotypes and that irritation from sodium lauryl sulfate (SLS) will be more rapid and more severe in AA versus GG. The effects of TNFα -308 and atopy were examined under: (a) exposure and regression from hand hygiene, (b) intensive lotion treatment (10x daily) and (c) repetitive exposure and regression from SLS under patch on previously undamaged skin.

The key findings were as follows:

- Excess erythema decreased for AA and increased for GG during exposure while it increased for AA and decreased for GG during regression (6).
- Following four weeks of intensive lotion treatment, AA had a greater reduction in excess erythema (7) than GG while GG had a greater reduction in skin dryness (Fig 1) (6). Atopy and heightened neurosensory irritation from water and lactic acid significantly influenced the skin responses.
- In the patch test, mimicking the work cycle and time off, image a* and visual erythema were higher in AA for water, 0.05% SLS and 0.1% SLS versus no treatment (Fig 2) (6).
- More protein was removed from AA with repeated tape stripping indicating a less cohesive SC.

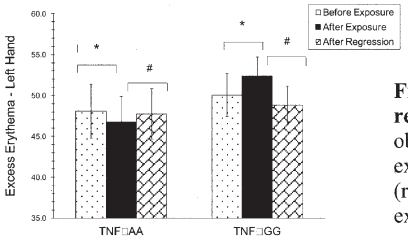


**Fig. 1. Response to hand hygiene exposure and regression**. Significant group differences were observed. For AA, excess erythema decreased during exposure and increased during time away from work (regression) ($p < 0.05$). For GG, it increased during exposure and decreased during time off ($p < 0.05$).

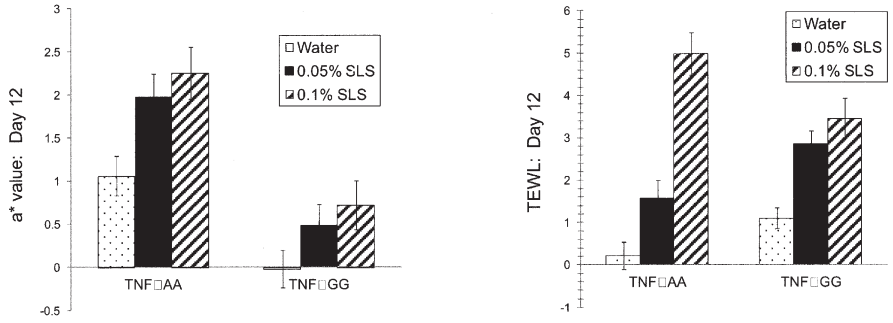


**Fig. 2. Repetitive SLS Exposure**. The AA genotypes had higher erythema (a* value from image analysis) for water, 0.05% SLS and 0.1% SLS following repetitive patch exposure (day 12) than GG ($p < 0.05$). TEWL was higher for GG than AA for water and 0.05% SLS and higher for AA for 0.1% SLS ($p < 0.05$). Dryness (not shown) was higher for AA than GG with atopic class as a covariate ($p < 0.05$).

**Discussion and Implications**

Intensive application of lotions may be required to mitigate the skin damage and counteract the impact on infection control. Currently, the choice of lotions for the health care setting is limited as they must be free of petrolatum and/or mineral oil to maintain glove integrity. Subjects with the TNFα -308 polymorphism and/or those with atopy represent excellent populations for the evaluation of milder hand hygiene regimens. Use and development of effective treatments for barrier compromise in HCWs is a clear need for the safety of patients and care providers alike. Historically, hand rubs have been associated with increased in hand hygiene compliance, though levels are only around 60%. Intensive lotion treatment as part of normal HCW hand skin care and/or innovation of non-damaging skin sanitizing systems may be required to achieve "clean hands without compromise".

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# Development of Cationic Bodywash Formulation Containing Novel Opacifier Utilizing Ultra Small Angle Neutron Scattering (USANS)

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**OBJECTIVE** - Opacifier technology has been applied in bodywash and shampoo formulations to provide an opaque and creamy appearance, which are often perceived by consumers as luxury products. However, most existing opacifier technologies are anionically stabilized, and have compatibility issues with cationic ingredients, such as conditioning agents in the formulation. A novel cationic opacifier was developed to offer more formulation flexibility.

The opaque formulations of personal care products present difficult structural characterization challenges. Typical optical characterization methods such as microscopy or optical scattering methods are limited for revealing structural properties of opaque samples. We will discuss the utilization of ultra small angle neutron scattering (USANS) to characterize the wet state structure of opaque body wash formulations containing cationically stabilized opacifier particles.

**METHODOLOGY** - Due to the highly penetrating nature of neutron radiation, neutron scattering methods are ideally suited for structural characterization of optically opaque samples. The National Institute of Standards and Technology Center for Neutron Research (NCNR) has a Perfect Crystal Small Angle Neutron Scattering (SANS) instrument capable of performing ultra small angle neutron experiments which probe size scales from 0.1 μm to 10 μm. This size range is typical of what is expected for dynamic clusters of colloidal particles making USANS an ideal tool for determining the extent of clustering in colloidal systems. All measurements were conducted at the NCNR on the BT5 Perfect Crystal Diffractometer described by Barker et al.[1] Samples were injected into demountable quartz window cells with 2 mm path lengths. The approximate counting time for each sample is 3.5 h. Data reduction was performed using the USANS Reduction package provided by NIST, using the Igor Pro® software package from WaveMetrics™ Inc. The scattering, $I(q)$, from fractal clusters has been given by Teixeira.[2] There are seven fit parameters in the Teixeira function: the volume fraction of particles; the particle radius (Å); the fractal dimension of the clusters; the correlation length of the clusters (Å); the scattering length density (SLD) of the particles ($Å^{-2}$); the scattering length density of the solvent ($Å^{-2}$);and an isotropic background scattering term ($cm^{-1}$). The volume fraction of particles, particle radius, scattering length density of the particle, and background parameters are fixed based on previous measurements. The SLD of the solvent is used as a fitting parameter because of the unknown scattering length density contributions of the surfactant, NaCl, and polyquat.

**RESULTS** - Bodywash formulations containing different levels of amphoteric surfactant (cocamidopropyl betaine, CAPB), salt, and hydroxypropyl guar hydroxypropyltrimonium chloride as the conditioning agents, which has presented more formulation stability challenges comparing to Polyquaternium-10. Formulations were studied before and after heat-aging. Table 1 shows the optical evaluation of the formulations as well as the determined optical backscattering values. In spite of the variation in optical appearance, the backscattering values do not distinguish between the samples. The size of the free opacifier particles (150-220 nm) is too small to scatter light efficiently to produce an opaque formulation. Therefore, opacity of the formulation is presumed to be the result of clusters of individual particles. These clusters are dynamic in nature and settling of the clusters as large scale aggregates does not occur in cationic opacifier based formulations. The USANS data is shown in Figure 2. The data was fit to the fractal scattering model, where the primary building block of the fractal is the opacifier particle. The vertical line in Figure 2 shows the estimated q value at which the optical backscattering measurement is taken. The USANS data shows the reason for the insensitivity of the backscattering to the optical appearance. The two structural parameters obtained from the scattering are the fractal dimension ($D_f$) of the clusters and correlation length, $\xi$, of the clusters. The samples described in this preprint were part of a broader study. The results from the entire study are shown in Figure 2, where $D_f$ and $\xi$ of the formulations are plotted against each other. In general, clusters with $D_f$ of 3 give a bluish appearance, while formulations which have a $D_f$ less than 3 give an opaque appearance. A $D_f$ of 3 means the clusters of opacifier particles are densely packed, whereas lower $D_f$ clusters are less dense, hence more efficient scattering bodies. Very large clusters with $D_f$ of 3 also give an opaque appearance. The very high $\xi$ clusters are large enough to scatter light efficiently and give an opaque appearance. Therefore, an optimum combination of cluster $\xi$ and $D_f$ are desirable (crosshatched area in Figure 2) to obtain opaque bodywash formulations.

**Table 1. – Formulation Compositions and Optical Appearance Before (RT) and After (HA) Heat Aging**

| Bodywash Formulation | Cationic Guar | CAPB | NaCl | RT-appearance | RT-%BS | HA-appearance | HA-%BS |
|---|---|---|---|---|---|---|---|
| Sample A | high | high | low | Blue | 12.1 | Opaque | 28.3 |
| Sample B | low | low | high | Opaque | 29.5 | Opaque | 29.6 |
| Sample C | high | high | high | Opaque | 27.1 | Blue | 24.6 |

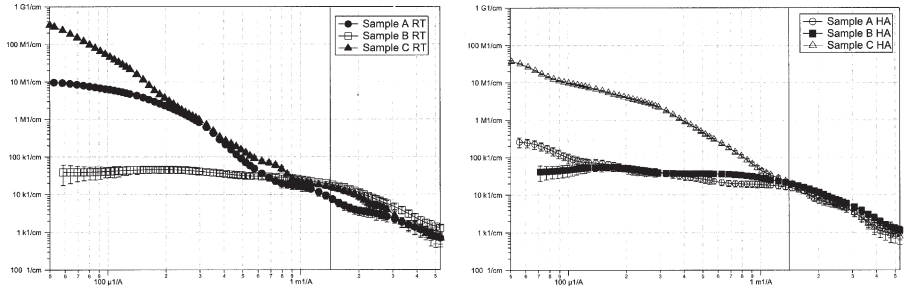


**Figure 1. –USANS data showing scattered intensity as a function of scattering wavevector, q, for non-heat aged samples (left) and heat aged samples (right). Intensity scale the same for both plots.**

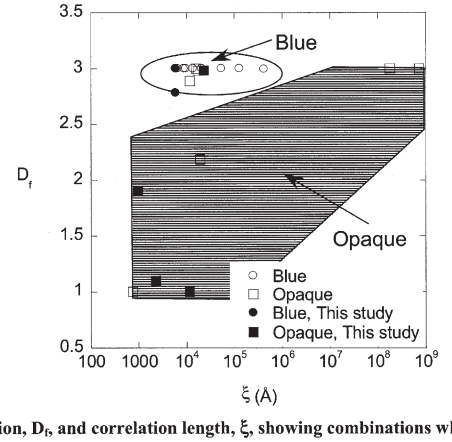


**Figure 2. – Map of fractal dimension, $D_f$, and correlation length, ξ, showing combinations which produce opaque and bluish bodywash formulations.**

**CONCLUSIONS** - Opaque body wash formulations were successfully characterized by the USANS technique. USANS provides insight on the stable wet stage structure formed by cationically stabilized opacifier particles in conditioning body wash formulations. The better understanding of opacifier particle interactions with surfactants, salt, and cationic polymer provided improved guidance on cationic opacifier polymer design to achieve stable and opaque formulations in a broad range of formulation conditions.

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# Formulating Hand Hygiene Products for the Healthcare Professional

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Studies have shown that a proper hand hygiene regimen can have a significant impact on decreasing the rate of healthcare-acquired infections (HAIs) [1-3]. Hand hygiene practices include handwashing with plain or antiseptic soaps, often in combination with alcohol handrubs [4]. Compliance with recommended practices results in an increase in the frequency in use of these products, which in turn can have a negative impact on skin condition, including dryness, redness (erythema), cracking and scaling [5]. Thus, increased hand hygiene compliance can result in poor skin health for the healthcare worker, an increase in both resident microflora and skin shedding, and ultimately also can result in decreased hand hygiene compliance.

In order to combat this unfortunate cycle, a careful approach to formulating products used in the healthcare market must be taken. Due to the significant differences in use-frequency in the healthcare environment, this formulation approach is often different than that taken to formulating similar products for the general consumer market.

## FORMULATION STRATEGIES

Though product type (lotion, handwash, alcohol handrub) will understandably affect ingredient selection during development, the general approach to formulating these products for the healthcare user is similar [6]. The choice of surfactants must keep in mind effectiveness; however, the surfactants should also be mild. High-frequency handwashing using a 'traditional' hand cleanser may lead to stripping of the lipids in the epidermis, and ultimately lead to increased dryness and irritation, so surfactants chosen should minimize de-fatting. Emollients used need to be balanced for both moisturization and aesthetic feel upon multiple applications. For example, a lotion that exhibits high tack may make it difficult for a healthcare provider to put on gloves prior to patient interaction.

There has been a significant increase in the U.S. in the usage of alcohol-based antiseptic handrubs (gel, rinse or foam forms) in the healthcare environment. Ideally, as part of an overall healthcare hand hygiene regimen, alcohol handrubs can be used when no visible soil is present. However, alcohol products can be very drying. Effective alcohol products are formulated to both enhance the efficacy of alcohol active and to maintain moisture levels on hands over multiple uses. Proper balance of moisturizers and skin conditioners can provide alcohol products that continue to maintain skin health on multiple uses.

A unique consideration when developing products for the healthcare market is the interaction with other products used by the healthcare staff. Specifically, chlorhexidine gluconate (CHG) is used in many products (e.g., healthcare personnel handwashes and surgical scrubs) as an active ingredient. Due to the cationic nature of the CHG molecule, it is both substantive to the skin and can be deactivated when it comes in contact with certain products that contain anionic ingredients. In addition, CHG-containing products are often used in high-risk areas, such as intensive care units, and therefore, products that can inactive the chemical reservoir on the skin of the cationic moiety of the CHG must be avoided.

Other attributes to be aware of include fragrance and color. For the consumer market, much care is taken to find a fragrance that covers up the other ingredients in the formula that will linger long after it has been applied. However, this approach may have a negative consequence if used for products used in the health-care setting. A large number of patients at any one time in a hospital may have allergies, be having respiratory difficulties, or may be much more sensitive to strong odors due to underlying chronic illnesses. If their healthcare provider is wearing perfume or is using a product with a strong fragrance, it may set off a negative respiratory response or nausea in the patient, causing further issues. Therefore, it is important to get input from the healthcare staff when selecting a fragrance.

## EVALUATION TECHNIQUES

Two additional considerations when developing a product for the healthcare setting involve testing to substantiate claims of mildness and moisturization. Patch testing is one common method to evaluate irritation of a product. Different patch test methods are utilized throughout the industry, ranging from 21-day cumulative irritation test [7] to 2-day occlusive testing with Finn chamber [8]. These occlusion techniques, however, may miss any impact the product has on solubilizing lipids in the skin that are then washed off during use causing dry skin to occur over time. Results from occlusion tests may conclude that a product is mild, but when said product is evaluated during actual use, it may be found to be less than mild due to dry skin-induced irritation.

Moisturization testing of products often utilizes different epidermal capacitance or impedence techniques to measure the water content in the epidermis. Three instruments commonly used include the Skicon®, NOVA DPM®, and Corneometer CM® instrument lines. Each instrument has unique strengths and weaknesses (e.g., sensitivity, accuracy, ease-of-use), so evaluating products using at least a couple of these instruments may provide a more complete understanding. It is important to evaluate the moisturization characteristics of a product as it will be used in a healthcare setting. Measuring the moisturization characteristics of a product at different time-points after use (e.g., 10-minutes, 2-hours, 4-hours) provides information on the how the product will affect the user long after it is used. Other approaches to consider include multiple use testing and alternating product testing (i.e., use of a handwash followed by a lotion).

## SUMMARY

Due to the complexities involved in skin condition of the health care worker, care must be taken in formulating products for these unique consumers. Developing a product that is effective, moisturizing and mild, even when it is used up to a hundred times a day, will result in better skin condition and lower irritancy for these professionals, ultimately leading to better hand hygiene and lower hospital acquired infection rates.

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