10/20 I Volume 146 I Thannhausen, Germany, October 19, 2020

sofw journal

Home & Personal Care Ingredients & Formulations

powered by SOFW

10
2020
english

personal care

Essential Aspects of Filling Stations for Cosmetic Products in the Retail Trade

Effect Pigments with Customized Performance

HLB

Sense or Nonsense?

antiperspirants

Anti-Odor Properties of Sodium Bicarbonate

Microbiota-respectful Deodorant which Allows Axilla Perspiration

home care

Effective Soil Release & Bio-Based – First of Its Kind for Laundry Detergents

Enhancing Antimicrobial Efficacy of Sodium Benzoate

Process Technologies to Optimize Detergent Manufacturing

skin & hair care

Beauty and the Mask – Eyes Only

Novel Pentapeptide Enhances Skin's Natural, Healthy Glow

Functional Vegan Silk – Crafted by White Biotechnology to Utilise a New Way to Take Care of Damaged Hair

BASF
We create chemistry

Brighten up your day with our One-Fits-All Solution

Looking for a brilliant laundry solution? Our One-Fits-All Solution is sure to brighten up the day for your customers! By combining our new cellulase **Lavergy® C Bright 100 L** with BASF's best Home Care and I&I ingredients, we've made graying of garments a thing of the past. Whether white or colored clothes, cotton or synthetic fabrics – after multiple washes textiles still look like new. And the environment benefits too: Lavergy® C Bright 100 L meets the criteria for a variety of Ecolabels including European Flower, Blue Angel and Nordic Swan.

The BASF One-Fits-All Solution – makes laundry a pleasure!

https://www.homecare-and-i-and-i.basf.com

8 cleanser
Antimicrobial Efficacy of Sodium Benzoate

28 skin care
Beauty and the Mask – Eyes Only

home care

specialties

personal care

Effective Soil Release & Bio-Based – First of Its Kind for Laundry Detergents

K.J. Mutch, F. Mazzeo

abstract

As more and more consumers look for environmentally friendly laundry products but demands for effective washing performance remain high, detergent producers face the dilemma of how to increase the renewable carbon content without compromising on stain removal, extended clothing life and fabric preservation.

To provide support in tackling these aspects, Clariant has focused on fulfilling the gap in the Home Care market for a renewable-based soil release polymer that performs. It introduces the first bio-based, non-synthetically sourced soil release polymer for liquid laundry detergents (INCI: nonionic polyester). TexCare® SRN 260 Life is based on non-tropical raw materials, with a Renewable Carbon index (RCI) of 80 %, and is readily biodegradable.

Laboratory soil release and anti-deposition tests to evaluate performance and demonstrate effectiveness were conducted on synthetic-based fabrics. Those washed with liquid laundry detergent containing the novel soil release polymer came out up to six times cleaner than those washed with detergent only. It also prevented the redeposition of dirt during washing, which is the main culprit for graying, almost two times better than a standard detergent alone. The bio-based origin, and outstanding performance in soil release and in building a shield against stains provide unique support for more sustainable laundry products.

Introduction

Soil release polymers (SRP) are increasingly establishing their place in the toolbox of laundry detergent formulators. The worldwide market for Soil Release Polymers in Laundry is expected to grow at a CAGR of roughly 3.7% over the next five years, and will reach 689.2 million US$ in 2024, from 573.8 million US$ in 2019, according to a new study [1].

As synthetic fibers have gained in importance, with global polyester production now twice as high as cotton production [2], the recognized contribution of these polymer laundry additives to achieving the difficult removal of fatty stains from polyester-containing fabrics and also preventing stain redeposition during washing, will become more important than ever. However, the increasing consumer demand for more environmentally-friendly laundry detergents raises new challenges for detergent producers. This demand is predicted to drive growth in the 'green' segment of the Industrial and Home Care sector by 6.5% until at least 2024, according to a recent report by Research and Markets [3]. Hand in hand with this desire to use renewable ingredients with a low environmental impact, comes the expectation from consumers for no compromise on cleaning performance, while at the same time washing temperatures are steadily decreasing in order to consume less energy.

Detergent producers face the dilemma of how to increase the renewable carbon content of their products through weight-efficient ingredients and deliver sustainable washing without compromising on stain removal, extended clothing life and fabric preservation.

To provide support across these multiple considerations, Clariant has focused on fulfilling the gap in the Home Care sector for a renewable-based soil release polymer that performs. One of the leading supplier of soil release polymers has recently introduced the market's first bio-based, non-synthetically sourced soil release polyester for liquid laundry detergents (INCI: nonionic polyester).

Within this article we explore and evaluate the performance and effectiveness of the bio-based nonionic polyester in aiding a more sustainable laundering process with liquid detergents, which are now outstripping use of powder detergents the world over. The general adsorption mechanism and performance at low concentration of soil release polymers is initially assessed. Following this, laboratory tests demonstrating its effectiveness in terms of soil release, provision of fiber protection,

and whiteness preservation will be demonstrated. The subject of low washing temperatures is highlighted to emphasize and meet the sustainability criteria of the business segment.

First bio-based soil release polymer

The new bio poly ethyleneglycol propyleneglycol oligo ester carries the tradename TexCare® SRN 260 Life. It is the latest addition to Clariant's soil release polymer portfolio and identical in performance to its fossil raw material sourced counterpart TexCare® SRN 260. The nonionic polyester is based on non-tropical raw materials, with a Renewable Carbon Index (RCI) of 80 %. This makes it a unique offering within the industry. Due to its nonionic character, the detergent polymer exhibits a broad tolerance to the most common surfactant systems. It is readily biodegradable and requires no hazard labelling according to REGULATION (EC) No 1272/2008. The product is available as a clear liquid.

Soil release polymer dynamics on polyester

Soil release is understood to be the improvement in detergency that is brought about through the modification of a surface to be cleaned in a pre-treatment stage, i.e. before soiling occurs. In the case of polyethylene terephthalate-polyoxyethylene terephthalate (PET-POET) polymers, which are widely used in both liquid and powder detergents [4] the soil release properties have been attributed to a hydrophilization of the hydrophobic polyester surface [5]. As the removal of hydrophobic oily and fatty soils from polyester is impeded by the affinity of the soil for the hydrophobic surface, the change in the surface properties can lead to a large improvement in the soil removal.

Although the surface properties of soil release polymer films on polyester have been well studied [6], the dynamics are less well known. In order to study the deposition process, measurements were conducted on a quartz crystal microbalance with dissipation monitoring (QCM-D) [7]. This method uses the piezoelectric nature of quartz to its advantage, in that a polymer solution is injected into a flow cell over the crystal that is kept vibrating at its resonant frequency due to an applied current. Any increase of the mass of the crystal due to deposition from the solution is translated into a frequency change of the vibration and can be used to calculate a deposited mass. Measurements were conducted on a Q-Sense E4 (Biolin Scientific, Sweden) in simulated tap water of 3°dH at 25°C under a constant flow rate of 0.1 ml/min. Three solutions of increasing polymer concentration were sequentially pumped through the system before a final rinse with pure water.

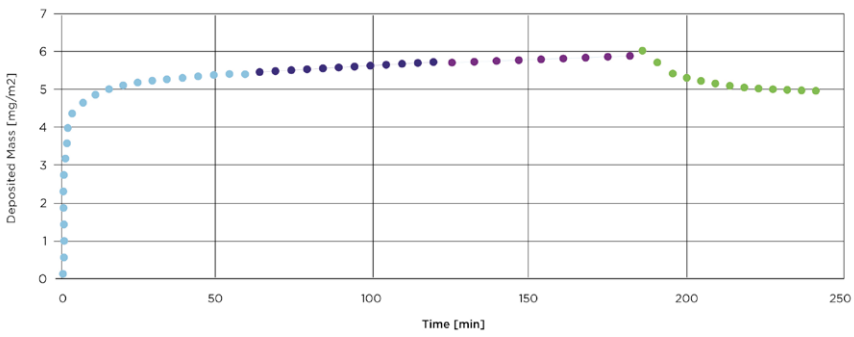


Fig. 1 QCM-D results of pure solutions of a SRP (TexCare® SRN 260 Life) flowing over a PET sensor. After rinsing, approximately 5 mg/m² of polymer remains on the sensor.

Fig. 1 shows the mass of polymer deposited onto a sensor coated with polyethylene terephthalate (PET), essentially mimicking a polyester fabric. It can be seen that the deposition from the polymer solution occurs very quickly, within the first 2-3 minutes of the experiment, but it is also evident that a significant amount of polymer remains on the sensor after rinsing with pure water, shown by the small mass decrease after around 180 minutes. It is also important to note that an increase in polymer concentration from 0.01 wt. % to 0.1 wt. % does not lead to an increase of polymer mass on the sensor. It can thus be concluded that the optimal polymer concentration in a wash liquor is reached at concentrations below 0.01 wt.-%, which corresponds to concentrations of 2 wt.-% or less in a detergent dosed at 4 to 5 g/l.

Performance of bio-based SRP at low concentrations

In view of the detergent industry's increasing preference for selecting weight-efficient ingredients, performance at low concentrations was analyzed. In doing so, it assesses the general suitability of soil release polymers to replace some of the less weight-effective surfactants that are traditionally used for soil removal.

The effectiveness of the new bio-based nonionic polyester can be verified by considering the performance data presented in **Fig. 2**. Here, polyester fabrics (WFK 30A) were pre-washed with a detergent containing varying amounts of the polymer, subsequently soiled with dirty motor oil, dried and finally washed with the same detergent used for pre-washing. The detergent composition is shown in **Tab. 1**.

Spectrophotometric measurements showed an increase in the amount of oil removed from the fabric even at very low concentrations. In this case, 0.25 wt.-% in the detergent. No significant benefit was found by going beyond the uplift achieved with polymer dosage concentrations of around 1 wt.-%.

From the results, it can be concluded that performance is achieved at low concentration and that the bio-based non-ionic soil release polyester is also effective in highly dilute conditions, such as those found in top-loading washing machines.

Materials & assessment methods

In the following sections, the specific performance offered by the bio-based soil release polymer is evaluated in a number of areas, considering the main benefit expectations from the laundry industry of such additives. The testing methods and results are included.

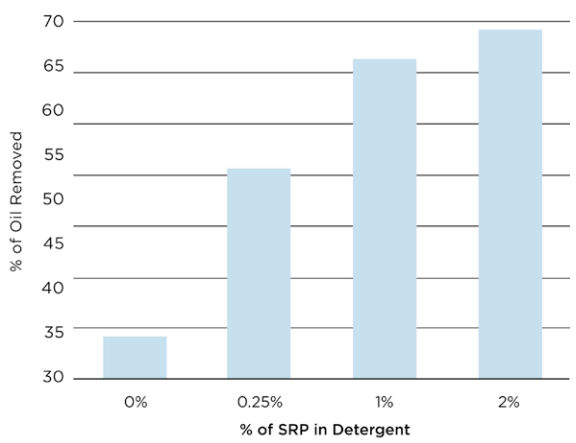


Fig. 2 Amount of dirty motor oily removed from polyester fabrics (WFK 30A) when washed with a liquid detergent containing increasing amounts of TexCare® SRN 260 Life dosed at 4.3 g/l at 40°C.

INGREDIENT	AMOUNT / WT.-%
Linear alkylbenzene sulfonate	7.0
Lauryl ether sulfate with 2 EO	9.0
Lauryl alcohol ethoxylate with 7 EO	7.0
Coconut oil fatty acids	3.7
Glycerine	5.7
Ethanol	2.0
Citric acid	3.2
Sequest DTMP	0.7
TexCare SRN 260	0.0 – 2.0 (see chart)
NaOH, 50%	Qs pH 7.6-7.8
Water	Qs 100

Tab. 1 Detergent composition.


INDUSTRY SOLUTIONS.
Consumer Care Solutions.
CHT
SMART CHEMISTRY WITH CHARACTER.
NO.
COMMODITIES.
YES.
HIGH-END SILICONES WITH EXPERTISE.
The CHT Group offers a wide range of speciality silicones for personal care products. With an international team of highly skilled experts, we are the first choice when it comes to expertise, performance, product range, and sustainability for hair care, skin care, and color cosmetics solutions. Our dedicated teams will personally take on every challenge when it comes to finding the solution that is best for you.
silicone-experts.cht.com

a) Improving detergency at low washing temperatures

The removal of solid fatty stains at ambient washing temperatures is one of the major challenges of polyester. Cold washing has always been the norm in many parts of the world, but the shift in environmental awareness of consumers in the USA and Europe means growing attention on energy conservation and reducing household energy consumption. Recent product innovations are supporting a transition to low temperature washing [8] and product claims regarding low-temperature washing performance are commonplace on liquid laundry detergent packaging. The use of synthetic-based soil release polymers has already been observed to play a significant role in alleviating this problem. Evaluation of the detergency performance of the new nonionic bio-based soil release polyester also confirms substantial soil removal even at 20°C.

A comparison was made of polyester items (mixed 50:50 polycotton fabrics) pre-washed and washed with a standard detergent and a standard detergent with the addition of 1% of the new bio-based soil release polyester. Soil removal was assessed with both dirty motor oil and carbon black/sebum. All fabrics were washed three times before soiling with the respective detergent, then soiled, then washed again with the respective detergent. Detergent concentration was 5g/L, with water hardness of 280ppm Calcium Carbonate. A front loader type washing machine was used.

The color difference indicates that the bio-based soil release polymer washes clothes up to six times cleaner than detergent only at 20°C, when soiled with carbon black/sebum (**Fig. 3**). They are twice as clean when compared to the use of detergent only at similar wash temperatures when soiled with dirty motor oil (**Fig. 4**).

In both cases, it can be concluded that an increase in washing temperature can only increase the removal of the soiling agents by a limited amount, whereas pre-washing and washing with a soil release polymer-containing detergent can lead to a large increase in soil removal at low temperature.

b) Fiber protection against dirt/soil anti-redeposition at low temperatures

One of the primary roles of traditional surfactants in a laundry detergent is the suspension of the soil in solution in order to prevent redeposition on the washed garments. The redeposition is one of the key causes of graying of fabric. Small amounts of soil release polymers are known to perform this task very effectively on all types of fabric, especially those containing polyester.

Evaluation of the potential of the new bio-based soil release polymer in terms of anti-redeposition and dirt-trapping benefit was performed under the following conditions: a detergent containing 1% of the new soil release polymer compared to a standard detergent when washed under stressed conditions of 1g/L detergent with 0.5g/L carbon black – olive oil mixed into wash water at 25°C. The modified jar test was used to replicate washing machine conditions.

Results showed that the addition of 1% of the new soil release polymer is almost twice as better than with a standard

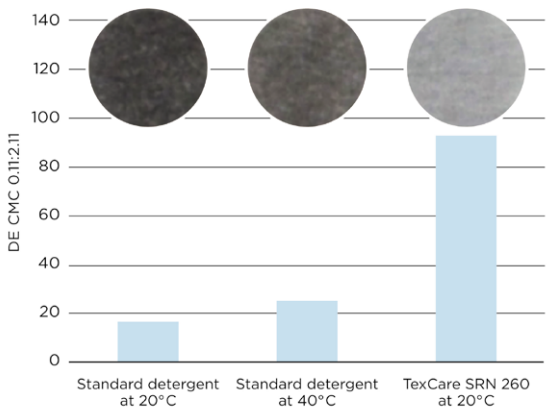


Fig. 3 Soil release effect on carbon black/sebum.

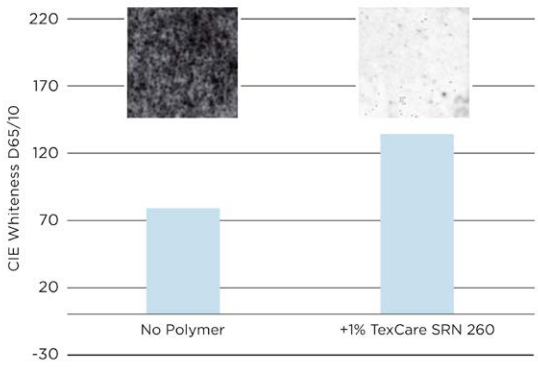


Fig. 4 Soil release effect on dirty motor oil: color difference comparison of standard detergent and standard detergent plus TexCare® SRN 260 Life at a washing temperature of 20°C.

detergent alone in terms of achieving whiteness protection (**Fig. 5**).

Conclusions & application discussion

From the performance demonstrated by the bio-based soil release polymer, it can be seen that the novel newcomer with its 80% RCI (Renewable Carbon Index) unites high renewable content with stain removal and fiber protection pre and during washing. Its soil release performance allows a reduction in washing temperature, detergent concentration and also mechanical action, which further helps to protect fabrics. Following the principles of Sinner's Circle and the fundamentals of soil suspension, by simply using a more gentle washing cycle it is possible to reach the same washing results if the product contains the bio-based soil release polymer.

TexCare® SRN 260 Life addresses the need for weight-efficient ingredients in detergents. Its addition to a formulation supports low temperature stain removal, even with cold washing, on polyester-containing fabrics at low concentration. The bio-based innovation also builds a shield against dirt and stains for superior whiteness maintenance of synthetic fabrics.

The availability of a bio-based soil release polymer provides value to liquid laundry detergent producers in general, but particularly "green-oriented" brands, looking to improve product cleaning performance and provide the sustainable washing experience consumers now demand. As its characteristics are like-for-like identical to the high level of performance achieved with the non bio-based counterpart, it enables a drop-in replacement for formulators wanting to raise the renewable content of detergent formulations.

References

[1] Industry Research - Global Soil Release Polymer in Laundry Market 2019 by Manufacturers, Regions, Type and Application, Forecast to 2024. Accessed via: https://www.industryresearch.co/global-soil-release-polymer-in-laundry-market-14357949

[2] Textile Clothing: Risk Analysis updated February 2020: https://www.coface.com/Economic-Studies-and-Country-Risks/Textile-Clothing

[3] Research and Markets: report on global household green cleaning products market 2017-2024 accessed via: https://www.hpci-events.com/household-green-cleaning-products-market-estimated-to-grow/

[4] *Gosselink E.P.*, Soil Release Agents in Powdered Detergents, Chapter 7, in Powdered Detergents, Edited by Showell M.S., Marcel Dekker, Inc. (1998)

[5] *O'Lenick Jr A.J.*, J. Surf. Det. 2, 553-557 (1999)

[6] *Calvimontes A., et. al.* Tenside Surf. Det. 42, 210-2016 (2005)

[7] *Rodahl M., et. al.*, Rev. Sci. Instrum. 66, 3924-3930 (1995)

[8] https://www.reviewed.com/laundry/features/why-cold-water-washing-is-the-future-of-laundry

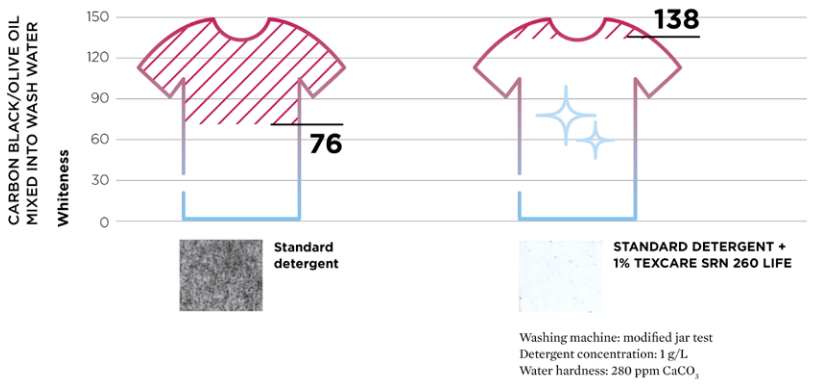


Fig. 5 Anti-redeposition benefit – delivering brighter whites.

contact

Dr. Kevin J. Mutch
Clariant Produkte (Deutschland) GmbH
Clariant Innovation Center
Industriepark Höchst
65926 Frankfurt am Main, Germany

Fabrizio Mazzeo
Clariant (Brazil) S.A.
Av. das Nações Unidas, 18.001
04795-900 Sao Paulo, Brazil

Corresponding Autor:
Fabienne Schindler
E-mail: fabienne.schindler@clariant.com

Enhancing Antimicrobial Efficacy of Sodium Benzoate

S. Foster, P. Rempala, D. Mueller, L. Dornan, A. Yarnell, J. Blankenship

abstract

In many cleaning product applications in the market place, the inclusion of sodium benzoate as a stand-alone preservative is providing sufficient microbial control. It was observed that inclusion levels are typically lower, resulting in economical use and, consequently, feasibility in a wider pH range of cleaning products compared to what was generally believed. For sodium benzoate to effectively preserve, formulations require sufficient concentrations of the undissociated organic acid, which is benzoic acid. The acid equilibrium constant (pK_a) of benzoic acid determines the degree of acid dissociation, and a larger value would result in increased benzoic acid concentrations. Titrations show that in the presence of home care surfactants, the apparent pK'_a increases as surfactant concentration increases, indicating a higher concentration of the active substance. This was confirmed with ^{13}CNMR. Microbial challenge testing was performed to verify that the increased apparent pK'_a yields increased antimicrobial efficacy. A surfactant was selected to increase the benzoic apparent pK'_a with substantial improvements in microbe reductions versus controls. Finally, a theoretical model based on surfactant properties was adopted to explain the observed pK'_a increases. This model provides a basis for detailed formulation advice and justification to the enhanced antimicrobial efficacy of Kalaguard® SB sodium benzoate.

Background

Organic acids have been used as preservatives for centuries, stretching back to ancient fermentation methods used to preserve foods after harvest to sustain peoples through the winter. In recent years, sodium benzoate has become increasingly valued outside of food and beverage applications, including cosmetics, pharmaceuticals, and home care. It offers a combination of effective antimicrobial action, good water solubility, and an eco- and consumer-friendly profile.

Consumer demand, retailers, and NGO's have driven significant shifts in approaches to home care preservation. Many traditional chemistries, such as isothiazolinones, have become subject to regulatory and labeling changes. At the same time, the preference for eco-friendly and skin-friendly products has escalated. Manufacturers are challenged to create formulations that are effectively preserved, contain ingredients that are gentler on the skin, and meet needs for green labeling.

Sodium benzoate has a beneficial profile that can meet the needs for green preservatives. It is biodegradable, nature identical, and considered non-irritating to the skin by a WHO assessment. Sodium benzoate is classified as a low risk substance by the European Commission, preferred over classical biocides to encourage the use of products with a more favorable environmental or human or animal health profile. Last year, Emerald was able to register sodium benzoate as an antimicrobial active (Kalaguard® SB) , extending the palette of preservatives for cleaning products and fulfilling the need for (cost) efficient, non-sensitizing, and eco-friendly preservation.

There are a number of factors that can make preservation more challenging in home care products. As formulations have become more eco-friendly, they are more likely to have increased concentrations of water, which leads to increased water activity, as well as biorenewable ingredients, which may have organic content that can serve as nutrients for microbial growth. In many product applications in the market place, the inclusion of sodium benzoate as stand-alone preservative is providing sufficient microbial control. It was observed that inclusion levels are typically lower, resulting in economical use in a wider pH range compared to what was generally believed to be effective. This article provides scientific justification of the observed enhanced antimicrobial activity.

Mechanisms for antimicrobial action and pK'_a shifts

The mechanism of preservative action for organic acids such as benzoic acid (and its respective salt, sodium benzoate) is well known. In the un-dissociated state, benzoic acid readily crosses cell membranes, decreasing internal pH and disrupting processes required for sustaining microbe life [1]. Sodium benzoate is the acid-salt of benzoic acid, and therefore, the concentration of unionized benzoic acid and benzoate can be estimated from the pH, the pK_a value of the acid, and total (free acid + anion) concentration using the *Henderson–Hasselbalch* equation [2]. It is important to note that the pK_a of an acid is tied to a solvent, with water being most common.

The concept of pK_a is straightforward in dilute solutions of a pure acid in a pure solvent, such as water. However, home-care formulations contain surfactants, solvents, solubilizers, and other ingredients. In these formulations, the undissociated benzoic acid concentration can be increased beyond what the pH and pK_a would suggest, especially when anionic surfactants are present [3, 4].

In solutions containing surfactants, the concentration of acid [HA] can be segregated between $[HA]_{solution}$ (=[HA]) and $[HA]_{micelle}$ (=[HA@M]), creating an additional quasi-chemical equilibrium with an apparent pK'_a greater than pK_a in solution alone [4].This results in larger concentrations of acid at higher pH levels. We denote this as apparent pK'_a rather than pK_a due to multiple equilibria. This apparent pK'_a effect has been observed for benzoic acid combined with anionic surfactants [3]. Pelezetti attributed this behavior to increased partitioning within the surfactant micelles [3]. In the presence of anionic detergents, the $[HA]_{micelle}$ may not be segregated permanently, as these detergent molecules rapidly exchange in solution. Thus, $[HA]_{micelle}$ may be bioavailable to prevent and eliminate microbial contamination in formulations. The overall impact of these effects is that the presence of anionic surfactants has the potential to enhance the preservation power of sodium benzoate by increasing the benzoic acid concentration, without the addition of more preservative in the formulation.

Experimental

Titrations, carbon-13 nuclear magnetic resonance spectroscopy (^{13}CNMR), and challenge testing were used to measure the increased apparent pK'_a in the presence of anionic surfactants and determine the resulting increase in microbial efficacy.

To study the impact of pK'_a on microbial control, efficacy testing was conducted through independent, third-party testing. Challenge inoculations of microbes *Pseudomonas aeruginosa* (ATCC 9027), *Escherichia coli* (ATCC 8739), *Staphylococcus aureus* (ATCC 6538), *Aspergillus brasiliensis* (ATCC 16404), and *Candida albicans* (ATCC 10231) were made at 10^5 CFU/mL. Log reductions of the microbes were measured at various time points using standard dilution and plating techniques.

A range of surfactants was selected due to their common use in home care formulations such as cleaners, detergents and laundry care. In addition, several surfactants were selected based on their potential to be used in green-focused products, such as whether they are considered to be more mild on the skin, biodegradable, or palm-oil-free. The surfactants used were sodium lauroyl methyl isethionate (SLMI), sodium lauryl sulfate (SLS), cocoamidopropyl betaine (CAPB), and linear alkylbenzene sulfonate (LAS).

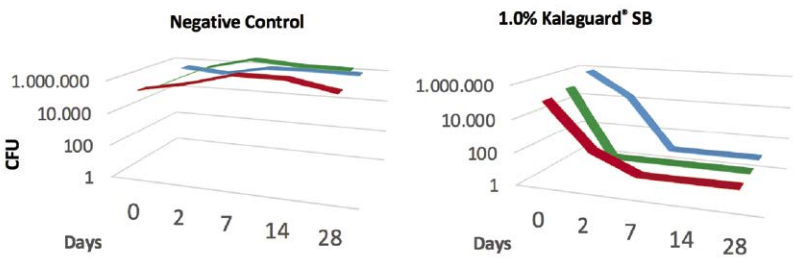


Fig. 1 Microbial counts in colony forming units (CFU) for ■ *Escherichia coli,* ■ *Pseudomonas aeruginosa,* and ■ *Aspergillus brasiliensis* of hand dishwash formulation pH 6.0.

Results and Discussion

Observing increased antimicrobial efficacy

Preservative testing of Kalaguard®* SB (sodium benzoate) in homecare formulations has resulted in surprising efficacy when compared to published minimum inhibitory concentration values. **Fig. 1** shows the antimicrobial performance of 1 wt% Kalaguard SB in challenge testing a hand dishwash formulation at pH 6.0. The formulations were self-efficacious versus *Candida albicans* and *Staphylococcus aureus*. These results meet the European Pharmacopeia criteria for log reduction requirements versus bacteria and fungi.

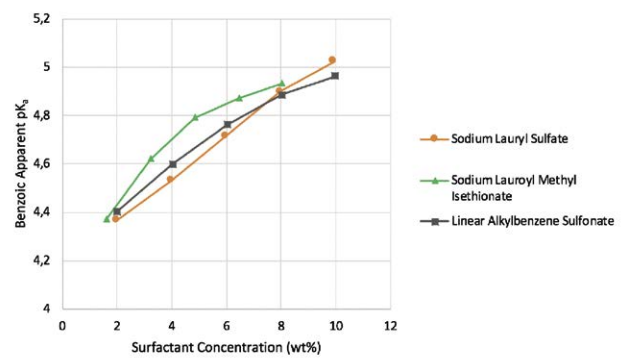


Fig. 2 The apparent pK'_a of benzoic acid increased with the concentration of various surfactants.

What is interesting about these results is that, according to the *Henderson-Hasselbalch* equation and benzoic acid pK_a in water of 4.19, the concentration of undissociated benzoic acid is 129 ppm at pH 6.0. In a previous study at pH 6.0 in nutrient agar, the minimum inhibitory concentration (MIC) of benzoic acid was measured to be 160 ppm for both *Escherichia coli* and *Pseudomonas aeruginosa* and 1000 ppm for *Aspergillus brasiliensis* [5]. Of course, there are differences in experimental methods and results between MIC determination and challenge testing, but the differences in values and outcomes was substantial, suggesting that an unexplained mechanism was increasing the antimicrobial efficacy.

The pK_a of benzoic acid in water is 4.19 [6]. However, the titrations of sodium benzoate combined with anionic surfactants indicated that the pK'_a of benzoic acid was significantly larger. As surfactant concentration increased, the apparent pK'_a increased, as shown in **Fig. 2**. The anionic surfactants SLMI and LAS shifted the pK'_a to approximately 4.9, while SLS shifted the pK'_a to an even higher value of approximately 5.02. These results demonstrate a significant shift in the concentration of undissociated benzoic acid, with an increase as much as six times higher at pH 6.0.

The apparent pK'_a of benzoic acid was observed to increase in combination with anionic surfactants, as shown in **Fig. 2**. Will the increased pK'_a also yield increased antimicrobial efficacy? A microbial challenge experiment was designed to measure the efficacy of increased pK'_a through surfactant selection. SLMI, from **Fig. 2**, was sourced preservative-free from a raw material producer. Sodium benzoate and SLMI were formulated in tryptic soy broth at pH 6.5, which is an extremely favorable environment for microbe growth and, therefore, a very rigorous test for antimicrobial efficacy. Log reductions were measured in **Tab. 1** versus microbes *P. aeruginosa, E. coli, S. aureus, A. brasiliensis,* and *C. albicans*. The surfactant and sodium benzoate alone are not efficacious versus the microbes, with the exception of *S. aureus.* Combining the surfactant and sodium benzoate raises the pK'_a to 4.93 and enables efficacy, resulting in complete kills for *P. aeruginosa, C. albicans,* and *A. brasiliensis* and increasing efficacy versus *E. coli.*

wt% SLMI	wt% Sodium benzoate	Benzoic Apparent pK'_a	*E. coli*	*S. aureus*	*P. aeruginosa*	*C. albicans*	*A. brasiliensis*
0	0	–	TNTC	TNTC	TNTC	TNTC	1.00
0	1	4.19	0.67	0.02	TNTC	-1.05	0.42
8.1	0	–	TNTC	3.6	TNTC	TNTC	0.36
8.1	1	4.93	1.43	3.13	Complete Kill	Complete Kill	Complete Kill

*Too numerous to count (TNTC)

Tab. 1 Log reduction of microbes in soy broth at pH 6.5 at 14 days, demonstrating that sodium benzoate combined with SLMI surfactant resulted in greater antimicrobial efficacy.

*Registered trademark of Emerald Kalama Chemical, LLC

Measuring pK'$_a$ with ^{13}CNMR

NMR was utilized to further expand upon and measure the behavior of benzoic acid/sodium benzoate when combined with surfactants. ^{13}CNMR has been used in previous literature to study the pH behavior of carboxylic acids to detect the changes in chemical shift of the carboxyl carbon. This change in chemical shift of the carboxyl carbon can be shown in **Fig. 3** for sodium benzoate, with and without the addition of surfactants. Due to rapid proton exchange between undissociated and anion species, the carboxyl carbon resolves as a single peak between the extremes of pure undissociated acid and pure dissociated salt. These extremes can be shown at pH 1 (δ_{min}) versus pH 8 (δ_{max}), where virtually all of the benzoate species have been converted to benzoic acid or sodium benzoate, respectively. The NMR samples were dissolved in H_2O rather than D_2O to avoid correcting for the dissociation differences between H_2O and D_2O. A $CDCl_3$ capillary insert was used for an external lock signal and spectrum reference peak.

In **Fig. 3**, the chemical shift δ_{min} of benzoic acid was shifted upfield in the presence of surfactant, whereas the chemical shift δ_{max} of sodium benzoate remains unchanged. The chemical shift value of a carbon nucleus depends on the molecular magnetic environment, which depends upon the electronic structure and interaction with molecules of the environment. This upfield shift of δ_{min} of benzoic acid in the presence of surfactants represents a change in the local environment of the carboxyl carbons nucleus of pure aqueous versus surfactant-containing solutions. Hydrogen bonding withdraws electrons and shifts signals downfield (lack of hydrogen bonding, upfield). Based on literature precedents, the authors propose that this is a consequence of the surfactant micelles partially incorporating benzoic acid within the hydrophobic cores with reduced hydrogen bonding from reduction in water [3]. Conversely, the similar δ_{max} of sodium benzoate with and without surfactants suggests that sodium benzoate was not partitioned significantly into the hydrophobic core of the micelles, which was expected.

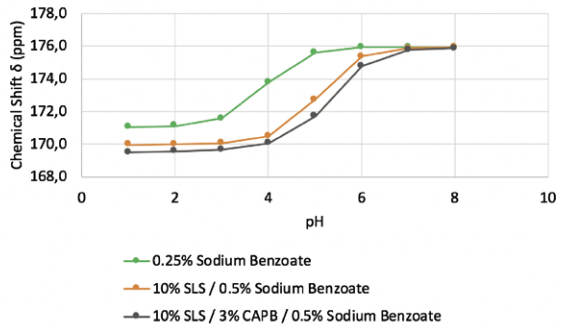


Fig. 3 ^{13}CNMR chemical shift of the carbon atom within the carboxyl functional group of sodium benzoate/benzoic acid at various pH levels.

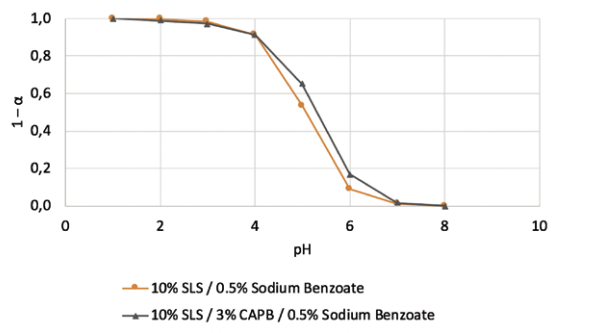


Fig. 4 Fraction of undissociated benzoic acid calculated from ^{13}CNMR chemical shift data at various pH.

Previous literature for ^{13}CNMR studies of carboxylic acids [7] has found that the chemical shift of the carboxyl carbon increases linearly with added base to the equivalence point, and thus, the fraction of dissociation can be determined through a linear equation.

Equation 1 Determination of benzoic acid dissociation (ionization) degree α from the carboxyl carbon chemical shift.

$$\alpha = \frac{[A^-]}{[A^-] + [AH]}$$

$$1 - \alpha = 1 - \left(\frac{\delta - \delta_{\min}}{\delta_{max} - \delta_{min}}\right) = fraction\ of\ undissociated\ benzoic\ acid$$

Using **Equation 1** and the chemical shift data of **Fig. 3**, curves can be calculated, as are presented in **Fig. 4**. The pK_a that results from interpolation of these curves is shown in **Tab. 2**.

Using **Equation 2**, the pK'_a can be interpolated at $\alpha = 0.5$, and these values are listed in **Tab. 2**. The presence of each of the surfactants shifts the pK'_a according to ^{13}CNMR measurements and was in reasonable agreement with the trends and values obtained through titrations. The 10% SLS/0.5% sodium benzoate sample pK'_a of 5.08 was very close to its titrated value of 5.02 in **Fig. 1**. Cocoamidopropyl betaine (CAPB) contains a carboxylic acid group and was not titrated to determine the apparent pK'_a, however, the NMR data shows a substantial shift when added to SLS. This increase in pK'_a is presumably due to the change in the hydrophobic structure of the micelles and results in an increase in partitioned benzoic acid.

Modelling Apparent pK'_a Values Based on Micelle Partitioning

The apparent pK'_a can be calculated based on assumptions governing the partitioning of benzoic acid between micellar aggregates and the rest of the solution. The partitioning relationship is illustrated in **Fig. 5**, showing the equilibrium between base and acid (K_a) in water and the partition equilibrium between acid in water and acid in the micelle hydrophobic core (k_{HA}).

Consider binding (partition between micelle and bulk of the solution) of the undissociated monoprotic acid, HA (like benzoic acid, A = C_6H_5COO) and its anion, A^- (conjugated base) to the micelles [4], treated as chemical equilibrium:

$$HA + M \rightleftharpoons HA@M \qquad k_{HA}$$
$$A^- + M \rightleftharpoons A^-@M \qquad k_{A^-}$$

$$k_{HA} = \frac{[HA@M]}{[HA][M]}$$

$$k_{A^-} = \frac{[A^-@M]}{[A^-][M]}$$

where [X] is molar concentration of species X (solution treated as a single phase). The anion, A^-, is more hydrophilic than the neutral acid, HA, therefore it is expected that $k_{A^-} < k_{HA}$. The dissociation constant, pK_a^w, refers to dissociation in water. We acknowledge that pK_a^w should be determined at equal ionic strengths for detergent and detergent-free solutions, if concentrations are used instead of activities, as *Debye-Hückel* theory expounds upon electrolyte impact on activity of ions [8]. We realize that further work might be required to include ionic strength effects in the surfactant/benzoic acid systems that the authors studied, but omission of ionic strength still seems to allow insight into experimental data and could be a starting point to more rigorous treatments.

The following can be shown:

$$pK'_a = pK_a^{app} = pK_a^w + log\frac{(1 + k_{HA}[M])}{(1 + k_{A^-}[M])}$$

The equation above is very closely related to **Equation 7** in review chapter on micelles by *McHedlov-Petrossyan et al* [4]. Based on observed lack of detergent influence on ^{13}CNMR chemical shift of the carboxylic group at high pH values (under practically quantitative ionization conditions), the authors postulate that k_{A^-} is negligible (near zero, no binding of A^- to the micelles).

Sample	Apparent pK'_a
10% SLS/0.5% Sodium benzoate	5.08
10% SLS/3% CAPB/0.5% Sodium benzoate	5.31

Tab. 2 pK'_a interpolated from benzoic acid 0.5 molar fraction using ^{13}CNMR data in **Fig. 4**.

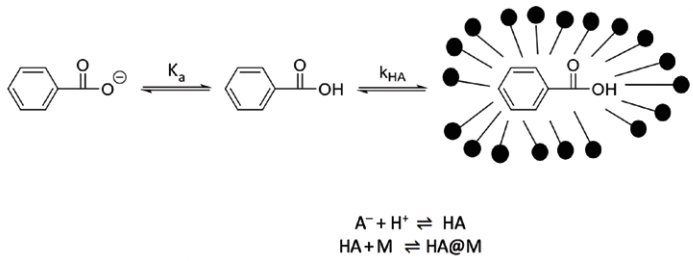


Fig. 5 Depiction of the equilibrium between base and acid dissolved in water and acid incorporated into the micelle core.

$$pK'_a = pK_a^{app} = pK_a^w + log(1 + k_{HA}[M])$$

Let $[S_m]$ represent concentration of the surfactant, S, bound in micelles with aggregation number n, then:

$$[M] = \frac{[S_m]}{n}$$

Equation 2 Apparent pK'_a of benzoic acid with surfactants. For full derivation details, please contact author.

$$pK'_a \approx pK_a^w + \log\left(1 + k_{HA}\frac{[S_m]}{n}\right)$$

The variable $[S_m]$ is the concentration of surfactant within micelles and is calculated from an equation that uses the critical micelle concentration (CMC) and two additional parameters (A, r) to describe detergent behavior ($[S_m]$ = fcmc, A, r([S]0), [S]0 – total detergent concentration) developed by *Al-Soufi et al* [9]. The parameters used to calculate $[S_m]$ were as follows: r describes the sharpness of the CMC transition and was set equal to 0.48 for LAS based on previous work by *Al-Soufi et al* [9]. Value of A is a function of r determined by the mass balance of the surfactant and was set to 1.0 [9]. The CMC for linear alkylbenzene sulfonate (sodium dodecylbenzene sulfonate) was reported to be 2.33×10^{-3} M [10]. The aggregation number (n) for the surfactant molecules within a micelle changes based on surfactant concentration. In absence of measured aggregation numbers at concentrations used in this study, the authors assume the aggregation number to equal 43, based on previous studies for LAS assuming 50/50 ratios of LAS6 and LAS4 isomers [11].

In **Equation 2**, the value of k_{HA} is found from the least-square fit of the model to the experimental measurements of pK'_a. **Fig. 6** depicts the experimental results graphed with the fitted (predicted, theoretical) values from **Equation 2**. The value for k_{HA} that from the least squares non-linear fit was 684 M^{-1} (r^2=0.99992). These parameters result in a theoretical pK'_a behavior that closely matches experimental results achieved through titrations. The data further supports partitioning as being one of the main factors enabling the increased pK'_a values of benzoic acid in the presence of anionic surfactants.

Conclusions

Overall, the use of anionic surfactants in combination with Kalaguard® SB sodium benzoate demonstrated antimicrobial synergy. This provides formulators with the possibility to economically apply sodium benzoate in a pH range up to 7. This method can also be used to predict the inclusion levels of sodium benzoate that are required to achieve sufficient antimicrobial efficacy within a given formulation. It ensures that the formulation meets consumer needs for products with skin-friendly, eco-friendly labeling, by reducing the level of preservative needed in the formulation and eliminating chemistries under regulatory scrutiny, such as isothiazolinones.

The method for enhanced preservation through pK'_a manipulation was validated through several methods. The pK_a of benzoic acid is 4.19 in water, yet titrations and NMR experiments with homecare surfactants such as sodium lauroyl methyl isethionate (SLMI), linear alkylbenzene sulfonate (LAS), cocamidopropyl betaine (CAPB), and sodium lauryl

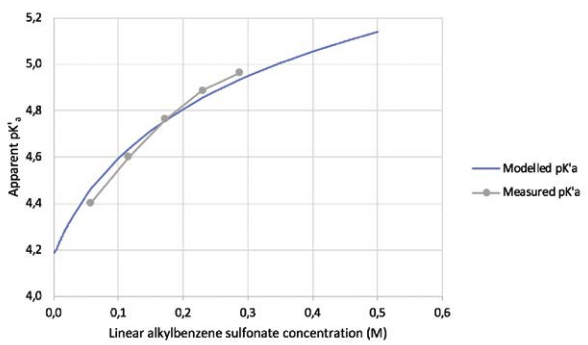


Fig. 6 Graph of apparent pK'_a versus LAS concentration from experimentally measured and theoretically derived values from **Equation 2**.

sulfate (SLS) increased pK'_a values. The ^{13}CNMR results confirmed the benzoic acid apparent pK'_a increases observed in the titration studies and demonstrated chemical shift evidence of partitioning of benzoic acid within micelle cores. The observed increases in pK'_a were expected to translate into enhanced antimicrobial efficacy, which was confirmed using microbial challenge testing. Anionic surfactant SLMI was used to increase the pK'_a of benzoic, and substantial improvements in antimicrobial action were observed as a result. A theoretical model based on surfactant partitioning was employed to explain the observed pK'_a increases. Theoretical values and experimental values closely matched for the LAS studies and indicated that partitioning is likely the main factor increasing the apparent pK'_a of benzoic acid. The model predicts that increasing the k_{AH} equilibrium between acid in water and acid within the micelle core would increase pK'_a, likely increasing antimicrobial efficacy further.

References

[1] *Chipley, J. R.*, Sodium Benzoate and Benzoic Acid. In Antimicrobials in food, Third ed.; Davidson, P. M.; Sofos, J. N.; Branen, A. L., Eds. CRC Press: 2005; pp 11-48.

[2] *Henderson, L. J.*, Concerning the relationship between the strength of acids and their capacity to preserve neutrality. Am. J. Physiol. 1908, 21, 173-9.

[3] *Pelizzetti, E.; Pramauro, E.*, Acid-base titrations of substituted benzoic acids in micellar systems. Analytica Chimica Acta 1980, 117, 403-406.

[4] *McHedlov-Petrossyan, N. O.; Vodolazkaya, N. A.; Kamneva, N. N.*, Acid-base equilibrium in aqueous micellar solutions of surfactants. In Micelles Structural Biochemistry, Formation and Functions and Usage, Bradburn, D.; Bittinger, T., Eds. Nova Science Publishers, Inc.: 2014; pp 1-71.

[5] *Paulus, W.*, Microbicides for the Protection of Materials: A Handbook. Springer Netherlands: 1993.

[6] *Serjeant, E. P.; Dempsey, B.*, IUPAC Chemical Data Series, No. 23: Ionization Constants of Organic Acids in Aqueous Solution. Pergamon: Oxford, 1979.

[7] *Cistola, D. P.; Small, D. M.; Hamilton, J. A.*, Ionization behavior of aqueous short-chain carboxylic acids: a carbon-13 NMR study. Journal of lipid research 1982, 23 (5), 795-9.

[8] *Daniels, F.; Alberty, R. A.*, Physical Chemistry. Second Edition ed.; John Wiley & Sons: New York, 1961; p 744.

[9] *Al-Soufi, W.; Piñeiro, L.; Novo, M.*, A model for monomer and micellar concentrations in surfactant solutions: Application to conductivity, NMR, diffusion, and surface tension data. Journal of Colloid and Interface Science 2012, 370 (1), 102-110.

[10] https://www.stepan.com/Products/Surfactants/BIO-SOFT%C2%AE/BIO-SOFT-%C2%AE-D-40.aspx.

[11] *Tucker, I.; Penfold, J.; Thomas, R. K.; Dong, C. C.; Golding, S.; Gibson, C.; Grillo, I.*, The Adsorption and Self-Assembly of Mixtures of Alkylbenzene Sulfonate Isomers and the Role of Divalent Electrolyte. Langmuir 2011, 27 (11), 6674-6682.

contact

Stephen Foster, Ph.D.
Pawel Rempala, Ph.D.
Daniel Mueller, Ph.D.
Lauren Dornan
Amber Yarnell, Ph.D.
Jenna Blankenship

Corresponding Author:
Stephen Foster
Emerald Kalama Chemical
1296 Third St., N.W.
Kalama, WA 98625

28 – 30 OCTOBER 2020

SEPAWA® CONGRESS

AND EUROPEAN DETERGENTS CONFERENCE
ECC ESTREL CONGRESS CENTER BERLIN

We are Fragrance

Scientific Conference

Consequences of Olfactory Loss
M. D. Thomas Hummel
Smell and Taste Clinic, Department of Otorhinolaryngology, TU Dresden

- Thursday, 29 October 2020, 09:00 – 09:45
  Room 2

We are Sustainability

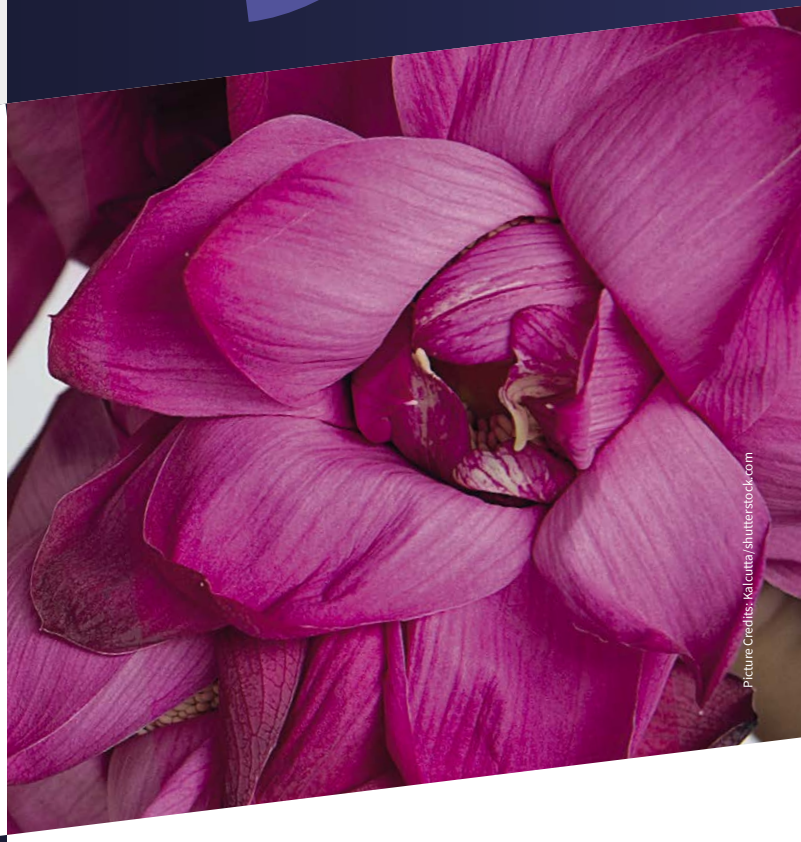
Scientific Conference – Sustainability LUV

Social and Biodiversity Standards in Carnauba Wax Production
Louisa Lösing, Deutsche Gesellschaft für Internationale Zusammenarbeit (GIZ) GmbH

- Thursday, 29 October 2020, 09:30–10:00
  Convention Hall I CD

Sustainability and Ecolabel Criteria: Re-thinking the Anaerobic Biodegradation Criterion for Linear Alkylbenzene Sulfonate (LAS)
Dr. John Heinze, Council for LAB/LAS Environmental Research (CLER)

- Thursday, 29 October 2020, 10:00–10:30
  Convention Hall I CD



Picture Credits: Kalcutta/shutterstock.com

#sepawacongress
#welovesepawa

www.sepawa-congress.com

Specials

Forum for Innovation Special – Home Care

Sustainable Innovation in Home Care During the Pandemic Era
Richard Hopping, Mintel Group Ltd.

- Thursday, 29 October 2020, 12:30–13:00
  Room 2

Scientific Conference Special – Home Care

Effective Lobbying Today – the Association TEGEWA and the European Chemicals Policy
Dr. Alex Föller, Verband TEGEWA e.V.

- Thursday, 29 October 2020, 09:00–10:00
  Room 3

Scientific Conference Special – Fundamental Research

What has COVID-19 Changed in People's Everyday Life and Conclusions with Regard to a 2nd Wave
Prof. Dr. med. Axel Kramer, Institute of Hygiene and Environmental Medicine, University Medicine Greifswald

- Thursday, 29 October 2020, 15:00–15:45
  Conventionhall I CD

How can cleaning agents components be produced in a more environmentally friendly and effective way? With fluid bed and spouted bed technologies, plant manufacturer and process expert, Glatt Ingenieurtechnik, offers modern, established solutions

Process Technologies to Optimize Detergent Manufacturing

G. Ding, A. Teiwes

As a manufacturer of consumer goods, the detergent industry must respond to megatrends like no other. One of these drivers is sustainability; eco-friendly biosurfactants have been available since 2009 and, now, alternative complexing agents such as α-ADA or MGDA-Na3 can be produced from renewable raw materials and used to replace phosphates. According to the German Zukunftsinstitut (Future Institute), the neo-ecology megatrend is causing a reorientation of the values of global society, culture and politics and will have a defining impact on the 2020s [1].
Consumers and business partners alike are asking about transparent value creation and whether social aspects are being considered. As an example, seamless traceability has long been standard practice. Yet, the ongoing microplastics debate calls for a rethink when it comes to encapsulated fragrances or when the fibres of functional clothing have to be fished out of wastewater. Solutions for all these requirements can only be realised if the chemical industry and original equipment manufacturers work closely together. Innovative raw materials is one side of the coin; technological advances in laundry washing is the other!

An interplay of forces

Saving resources such as water and energy raises fundamental questions in terms of cleaning, which can be divided into four key factors: temperature, time, mechanical force and chemical functionality. For overall effectiveness, if one of these components is reduced, the others must become correspondingly more efficient.

- For low-temperature detergents, for example, stain removal is relatively difficult and requires catalysts that activate bleaching agents in the formulation.
- Likewise, the thermal inactivation of bacteria at temperatures < 40 °C can only be achieved by biocides, but these are critical because they promote resistance.
- Energy savings can be compensated by higher mechanical forces in the washing machine; but, for synthetic fibres, this means more microplastics!

Thus, many attempts at optimisation induce conflicting side-effects, which must be carefully assessed. Ultimately, the easiest way to overcome these issues is to improve the washing activity itself and extend cleaning times. Two groups of ingredients that are particularly important when it comes to optimising the cleaning effect are surfactants and catalysts, especially enzymes. More compact detergents also reduce the consumption of raw materials and packaging materials.

Great success from small particles

Smaller particles offer a range of benefits, such as lower bulk densities, improved dosing and a consistent composition. There is also greater scope for specific shapes, sizes and the release of particular ingredients. An enabler of such solutions is fluidised bed processing, which facilitates the preparation of user-defined particle (**Fig. 1**). Chemical synthesis can also be augmented by simultaneously targeting different areas of the fluidised bed with separate spray liquids. The wide range of available options allows a diverse array of end product properties to be achieved, which are further influenced by the properties of the starting materials, the plant geometry and fine-tuned process parameters.

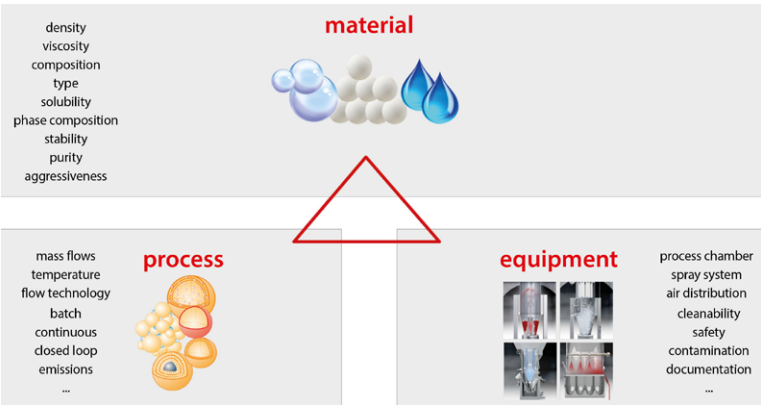


Fig. 1 Influencing factors of material, process and equipment properties on the particle design. (©Glatt)

Ingenious biocatalysts

Enzyme granulation provides a good illustration of the effect of the various variables. Enzymes play a key role as biocatalysts in detergents. They break down organic substances in the fabric, which can then be washed out in small, water-soluble fragments. Proteases are used to treat protein-containing dirt, lipases for fatty stains and cellulases help to smooth fibres.
As high molecular weight proteins, enzymes are temperature-sensitive and potential allergens that have a sensitising effect upon contact or inhalation. Strict guidelines are applied for work areas where enzyme dust occurs. In detergent production, concentrations of ≤15 ng/m³ in the ambient air are required to avoid sensitisation [2]. Granulated, compact enzyme particles show hardly any abrasion and are also easy to film coat (**Fig. 2**). This not only provides additional protection for the user, the enzyme itself demonstrates improved stability in the detergent during storage. To be able to produce a wide variety of applications – from powders to pellet-shaped compact detergents to dishwasher tabs – enzyme particles of different sizes and formulations with coordinated activities are required.

Fig. 2
Enzyme granulate

End product properties: abrasion stability and particle size

If the target is a dust-free enzyme particle with a defined particle size, which is also uniformly round and has an even surface, various processes are available. Enzymes are obtained by fermentation and accumulate as a solution with a very low dry weight. Traditionally, drying is done in a spray tower wherein the drying time is limited by the fall height. Owing to the plant geometry, the atomised liquid droplets must dry within a few seconds before they are discharged at the lower outlet. The result is a fine powder. Integrated agglomeration by recirculation optimises the product properties in terms of particle size, but not abrasion stability (**Fig. 3**) [3]. If the spraying direction changes, for example, the atomising and drying process delivers completely different particles (**Fig. 4**).

Spray granulation brings enzymes into shape

Although the dust particles combine with the spray solution in the tower dryer to form an agglomerate, during spray granulation in a spouted bed apparatus (a further development of the fluid bed, **Fig. 5**), they serve as starter cores for continuous layering. Owing to the reversal of the spray direction and the geometry of the spray zone in the process chamber, the finely atomised droplets of the enzyme solution are spread onto the surface of starter cores. They evaporate directly in the area of the spouted bed around the nozzle. The required energy is absorbed from the conditioned air stream in the immediate environment of the product,

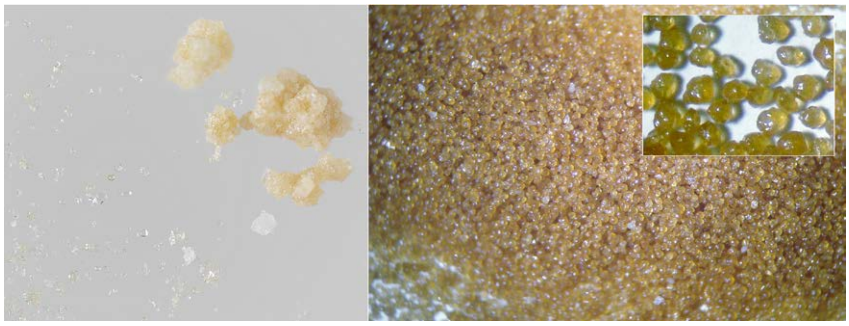


Fig. 3 & 4 Spray dried enzyme powder/agglomerate and spray granules from an enzyme solution. (©Glatt)

Fig. 5 ProCell apparatus for the spouted bed process patented by Glatt (EP 1 638678 B1). (©Glatt)

Fig. 6 Principle of product flow during spouted bed granulation in the Glatt ProCell. (©Glatt)

which reduces the thermal load on the sensitive protein molecules (**Fig. 6**).
The dried granules then fall back and are conveyed past the spray nozzle until they are finally discharged. The required size is determined by sieving out the desired fraction, which is then fed back into the process – as well as ground top grain – and forms new starter nuclei. These are required for stable process control and uniform particle size distribution. The surface quality depends on the solids content, viscosity and stickiness of the spray solution. An excessive solids content leads to a rougher surface and increases the potential for dust formation. The spouted bed process is particularly suitable for drying enzyme solutions to form uniform granules, which build up their very compact, smooth surface from low-concentration solutions.

Drying, agglomeration, synthesis

Most particle formulation processes work purely physically and are based on drying and agglomeration. The inclusion of chemical synthesis opens up further formulation possibilities for active ingredients and complete mixtures. The advantage here is that multi-step manufacturing processes such as wet chemical synthesis combined with filtration and drying can be done in a single step.

Reactive spray and binder granulation

The process of reactive spray granulation is gaining more and more importance in a wide range of applications (**Fig. 7**). As early as the 1990s, Glatt developed and patented technical solutions for the reactive granulation of sodium percarbonate as a bleaching or oxidising component in fluidised beds [EP 0787 682 B1]. The major challenge was to enable the chemical reaction of hydrogen peroxide and sodium carbonate to take place in the fluid bed. However, if the two reactant solutions of hydrogen peroxide and sodium carbonate are introduced into the fluid bed together through the same spray nozzle, the components usually crystallise very quickly. This clogs the spray nozzle and leads to operational downtime.
When using fluidised bed apparatus, a hydrogen peroxide and a soda solution are sprayed onto sodium percarbonate cores via two separate spray nozzles, with water being evaporated at the same time. The disadvantage of this process is that it's difficult to achieve homogenous mixing if the reactants are introduced into the granulation apparatus via two or more spatially separated spray nozzles.

Playing with atomisation technology

Yet, German patent [DE 20 60 971] elucidates a way to integrate a special type of spray nozzle for use in reactive spray granulation processes. The intensive mixing of the educts should lead to a better product quality and yield, which is achieved immediately after they're injected into the spray mist. Special spray nozzles of this type are designed as three- or four-component units in such a way that, in this example, two spray solutions are conveyed separately to the nozzle outlet and are pneumatically atomised with the aid of one or two atomising agents (**Fig. 8**).
The various liquid and atomising gas lines are arranged concentrically: liquid one is delivered in the centre of the arrange-

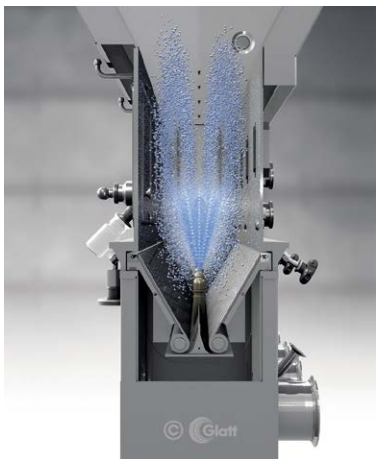


Fig. 7 Reactive spray granulation and reactive binder granulation. (©Glatt)

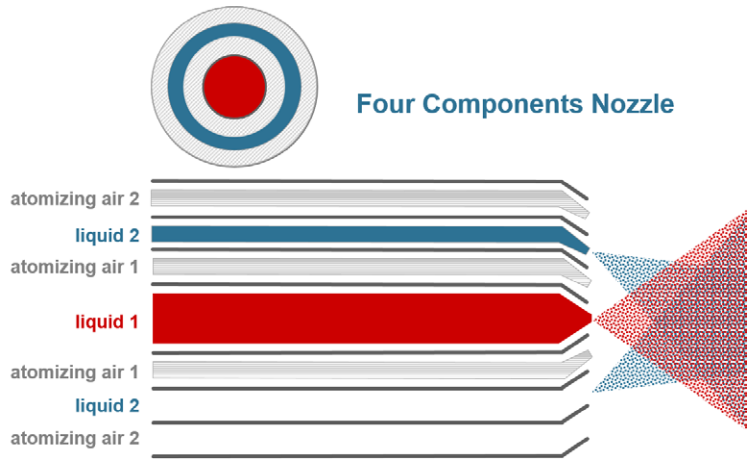


Fig. 8 A nozzle system with annular gas lines ensures that highly reactive solutions do not mix intensively until they reach the spray cone. (©Glatt)

ment (up to the top of the nozzle) where an annular gas line provides the atomisation. The unit is in turn surrounded in a ring by a second liquid line, which is encased by an external second atomising gas line. This design ensures that when different liquids are supplied separately, the two spray liquids first mix outside the nozzle in the spray cone and then react with each other in the spray jet. The particle formation or granulation takes place simultaneously. This basic principle can also be applied to other reactive fluid bed formulation processes such as spray granulation, agglomeration or coating.

Why anionic surfactants are treated in compartments

Another example from the field of reactive detergent granulation is the formulation of anionic surfactants in fluid beds. For example, sodium alkylbenzene sulfonate (LAS-Na), one of the world's most widely used anionic surfactants for detergent powder formulations, can be formulated by injecting alkylbenzene sulfonic acid (HLAS) onto a fluidised bed of sodium carbonate particles. The neutralisation process forms a dry product that can be mixed with other detergent components and/or used for compression [4].

In some cases, structuring agents are also integrated into the reactive formulation process of LAS-Na. The same obstacles occur as in the reactive spray granulation of sodium percarbonate. Mixing the components before the spray process would lead to solidification of the liquids and thus disrupt the injection process. Multi-component nozzles or compartmentalisation – reaction zones – help to eliminate these material-related challenges.

In general, the main hurdles in reactive spray granulation are the interaction of reaction and particle growth kinetics as well as the wetting and drying behaviour of the fluidised particles. This results in a need to optimise the residence time distribution and adjust both the fluid injections and the thermodynamic conditions. For the transition from batch to continuous processing, this interplay must be considered and further investigated to ensure successful process development.

If horizontal fluidised beds are divided into several zones, this reduces the residence time distribution, equalises the flow and thus allows several (different) steps to take place in a continuously operated apparatus. This not only enables new product shapes and manufacturing processes to be developed, it also has a direct effect on product homogeneity. For example, reactive spray granulation can be done in the first zone and subsequent drying, cooling or other types of thermal treatment can be done in a downstream part of the apparatus. Compartmentalisation also helps to separate incompatible substances into different processing zones.

Conclusion: size is important!

Highly reactive detergent components are safer and easier to handle if the particles have a compact structure, a specific size and a dense surface structure. Fluid and spouted bed processing offer two state-of-the-art technologies that enable tailor-made particle design for safe application and perfect cleaning performance.

References

[1] zukunftsInstitut. https://www.zukunftsinstitut.de/dossier/megatrend-neo-oekologie/. Access 31 July 2020.

[2] *Cyril Martela, Gunnar D. Nielsenb, Adriano Maric, Tine Rask Lichta, Lars K. Poulsena* (2010). SCIENTIFIC / TECHNICAL REPORT submitted to EFSA. Bibliographic review on the potential of microorganisms, microbial products and enzymes to induce respiratory sensitization1, CFP/EFSA/FEEDAP/2009/02: page 12 ff.

[3] *Kessler, H.G* (2011). Food and Bioprocess Engineering - Dairy Technology. 2. Reprint. Publisher A Kessler: Page 270 ff.

[4] *Sarah Germaná, Stefaan Simons, JudithBonsall, BrendanCarroll.* LAS acid reactive binder: Wettability and adhesion behaviour in detergent granulation. Powder Technology. Vol. 189, Issue 2, Jan. 2009. https://doi.org/10.1016/j.powtec.2008.04.012

contact

Gudrun Ding | Head of Business Development Process Technology
Arne Teiwes | Process Technology, Deputy Head of Technology Center and R&D

Glatt Ingenieurtechnik GmbH
Nordstraße 12
99427 Weimar | Germany

www.glatt.com/ptf

Forum Cosmeticum 2020

28 – 29 October
Estrel, Berlin

www.forum-cosmeticum.org

Uncover the Invisible

Enjoy excellent scientific presentations at the Forum Cosmeticum 2020, and visit over 300 suppliers at the simultaneous SEPAWA® CONGRESS.

Selected overarching topics:

- Microbiome: „I spy with my little eye"
- Microbiological Quality Management: „Safety first"
- Light, UV and the Environment: „New waves for future"
- New Detection Methods: „Seeing is believing"

Conference Program now online

IN CONJUNCTION WITH
28 – 30 OCTOBER 2020
SEPAWA® CONGRESS
AND EUROPEAN DETERGENTS CONFERENCE
ECC ESTREL CONGRESS CENTER BERLIN

Direct contact:
info@dgk-event.de

Forum Cosmeticum is an event together with

Organized by

PART 1

» Part 2 will be published in SOFW Journal 12-2020

HLB – Sense or Nonsense?

J. Venzmer

abstract

The Hydrophilic/Lipophilic Balance (HLB) concept as introduced by *Griffin* in 1949 means assigning a single number between 0 and 20 to fatty alcohol ethoxylates in order to describe their hydrophilicity/lipophilicity and assuming that this number is useful to predict the applicability as emulsifier. This approach has severe limitations as it ignores crucial parameters such as temperature, presence of the oil phase, pH, salt, or processing during emulsion manufacturing. However, despite these deficiencies, HLB values are still given today by the surfactant manufacturers in their brochures and product documentations. The only meaningful purpose of the HLB value is that it provides (especially in case it has been determined experimentally) a rough guesstimate of the water solubility or dispersability of an emulsifier. All other properties, often a consequence of the molecular architecture especially of polymeric emulsifiers, cannot be predicted by a single HLB value. This paper critically discusses the limitations of the HLB concept in order to avoid misconceptions, especially when dealing with non-fatty-alcohol-ethoxylate amphiphiles.

Introduction

For many years, the quest for HLB values of non-fatty-alcohol-ethoxylate surfactants is a nuisance to physical chemists, as long as they are well aware of the inherent deficiencies of the HLB concept. The original approach by *Griffin* [1] to assign a single number to a fatty alcohol ethoxylate in order to describe its hydrophilicity/lipophilicity and to assume that this number is useful to predict the applicability as emulsifier has severe limitations. It ignores not only crucial parameters such as temperature, presence of the oil phase and the type of oil, pH, salt, or processing during emulsion manufacturing, but also the molecular architecture of (polymeric) emulsifiers. Although these limitations are well-known to surfactant experts, why are HLB values still used today? The main reason seems to be a vicious circle: The customers of the surfactant suppliers are asking for HLB values because the surfactant manufacturers are providing them – most probably only because the customers are asking for them! Even the extensions of the HLB concept which were proposed in the last several decades, often in terms of other or more refined methods to generate HLB values, cannot solve the inherent problems. This is the reason why the CESIO Working Group "Test Methods of Surfactants" and the TEGEWA Working Group "Surface Active Substances" have decided to deal with the topic of HLB, since A) spreading the word about the deficiencies of the HLB concept and B) discussing alternatives (see part 2 in sofw journal 12-2020 [2]) should be of interest to both the manufacturers and/or developers as well as the customers and/or formulators of surfactants.

History – *Griffin* and *Davies*

Based on theoretical considerations suitable emulsifiers should fulfill two requirements. First, they should have a proper balance between attraction for the water phase and attraction for the oil phase. This first requirement could be met by small molecules like simple alcohols (methanol, ethanol), but these do not meet the second requirement, which is the enrichment at the interface, leading to a reduction in interfacial tension. While the interfacial activity can be measured by determination of interfacial tension, the balance between polar and non-polar portions of a surface-active molecule is much less straight-forward to quantify. According to a rule of thumb in emulsion technology, known as *Bancroft's* rule [3], water-soluble emulsifiers tend to give o/w-emulsions and oil-soluble emulsifiers w/o-emulsions. The first attempt to extend this entirely qualitative rule into some kind of quantitative relationship has been undertaken by *Griffin* [1] at the Atlas Powder

Company. They have published a systematic ranking of emulsifiers in 1948, based on evaluations of the type of emulsion (o/w vs. w/o) and their stabilities prepared from a series of oils and fatty alcohol ethoxylates as surface-active agents [4]. This experimental approach to generate these HLB numbers was pretty time-consuming – for each value as many as 75 emulsions had to be prepared by the following procedure: Shaking 95 ml of oil and 95 ml of water in the presence of 10 g of emulsifier and observing the result of this procedure after storage for 24 hrs. **Tab. 1** shows *Griffin*'s HLB scale and its correlation with water dispersability and potential use of the amphiphiles. Later on, the HLB numbers for fatty alcohol ethoxylates were calculated as weight-% ethylene oxide in the molecule divided by 5 [5]. Using those calculated HLB numbers and performing emulsification tests, the required HLB for a specific oil phase was determined as guidance. Therefore, the HLB concept has helped to reduce random trial-and-error by a more systematic approach, eliminating a large number of otherwise failed emulsification attempts.

Since the calculation of HLB numbers by *Griffin* (HLB = wt%EO/5) was basically only suitable for fatty alcohol ethoxylates, *Davies* [6] suggested to calculate HLB values by adding so-called group numbers assigned to the different hydrophilic (even ionic!) and lipophilic groups of the amphiphiles. It is hardly ever reported that the *Davies* scale differs substantially from the *Griffin* scale in the entire range of practical applications; unfortunately, for reported HLB values, it is typically not specified how the values have been derived and to which scale they refer to.

Alternative methods to generate HLB values

Experimental determination of HLB values has a chance to capture the influence of molecular architecture, which is not accounted for in case of the calculation methods by *Griffin* or *Davies*. Several approaches have been reported over the years. *Greenwald* [7] could find a reasonable correlation of the HLB numbers with solubility characteristics by titration of

HLB	Dispersability in water	Suitable as
1-4	nil	defoamer
3-6	poor	w/o emulsifier
6-8	milky dispersion upon agitation	wetting agent
8-10	stable milky dispersion	wetting agent o/w emulsifier
10-13	translucent	o/w emulsifier
13-20	clear solution	o/w emulsifier solubilizer detergent

Tab. 1 HLB values, water solubility and potential uses of surfactants.

the surfactant dissolved in e.g. 4% benzene in dioxane and titration with water, the end point being the appearance of a second phase. Other experimental methods to derive HLB values are based on interfacial tension between water and toluene [8], gas chromatography [9, 10, 11], dielectric constant [12], NMR [13], Relative Solubility Number (RSN; by titration) [14], or – as a rough guesstimate of HLB – simply the water solubility/dispersability, using the original classification by *Griffin* (**Tab. 1**).

Benefits of the HLB concept – Examples of success

In the typical B2B relation between the surfactant manufacturers and surfactant formulators, it is rarely the physicochemical specialists which are talking to each other. Therefore, it is obvious that a deep dive into different surfactant theories and concepts is rarely part of the negotiations between the salesperson of the surfactant supplier and the purchasing person of the customer. Intentionally developed as a guidance for emulsification, the HLB concept, however, can help even non-experts to categorize surfactants into defoamers, wetting agents, emulsifiers, detergents and solubilizers as depicted in **Tab. 1**. Although the HLB scale is only a rough guesstimate, it is due to its extreme simplicity and ubiquitous use in surfactant product brochures a highly appreciated tool that helps to preselect substitutes from different supplier portfolios. One successful example is the replacement of nonylphenol ethoxylates by environmentally more benign surfactants during the past decade. The next best alternative to a nonylphenol ethoxylate turned out to be in most cases an alcohol ethoxylate with a comparable molecular weight, cloud point and HLB value.

Limitations of the HLB concept – Examples of failure

Even in the preface of a monograph on HLB [15], it is explicitly stated that A) the applicability of the HLB system is often overestimated, and B) *Griffin's* and *Davies'* HLB numbers represent different hydrophilicity scales – but this aspect often seems to get ignored. In the scientific literature, there are several examples demonstrating the deficiencies of the HLB system, e.g. *Balson* gives a number of surfactants with either the same HLB numbers but different phys.-chem. properties or vice versa [16]. He concludes that "HLB remains a scientific curiosity rather than a useful concept". *Griffin* himself has given oleic acid (used as oil phase!) an HLB value of 1, whereas to sodium oleate he assigned a value of 18. This shows that a single number characterizing a molecule without specifying the conditions such as pH is just not helpful to predict interfacial behavior. Often, the type of emulsion formed is significantly influenced by the process of emulsification; therefore, not even the unambiguous prediction whether an emulsifier is suitable for o/w- or for w/o-emulsions is possible. Moreover, in case of polymeric emulsifiers, molecular architecture rather than overall chemical composition determines the interfacial properties, which makes the assignment of a calculated HLB number – independent of the molecule's amphiphilicity – nothing but useless. For example: A random EO/PO-copolymer is not amphiphilic, whereas a corresponding block copolymer, consisting of the very same number of EO and PO units, is known to have emulsifying properties.

Especially for polymeric amphiphiles, the calculated HLB value neglects the effect of molecular size and surfactant architecture. Many technical processes have characteristic times in the order of (milli-)seconds and are dominated by diffusion and/or adsorption kinetics. Diffusion of surfactants towards a surface or interface is directly affected by the size (i.e. hydrodynamic radius) and consequently molecular weight of the surfactants. Differences in diffusion/adsorption kinetics are directly reflected in the application performance of the surfactant. In the following, three examples are shown in which surfactants of identical calculated HLB value, but different molecular structures behave quite differently.

Surfactants with an HLB value between 6 and 10 are categorized as wetting agents according to Griffin's concept. The wetting behavior on Parafilm of solutions of a castor oil ethoxylate with 20 moles of EO and a C12-14 alcohol with 4 moles of EO has been compared (**Fig. 1**). Both surfactants possess a calculated HLB value of 10, but differ significantly in their molecular weight (MW). The much higher MW castor oil ethoxylate performs significantly poorer as compared to the lower MW fatty alcohol ethoxylate. This is not too surprising, since "speed" is known to be crucial for wetting processes.

The second example concerns the cloud point of nonionic surfactants [17]: Clear surfactant solutions turn turbid above

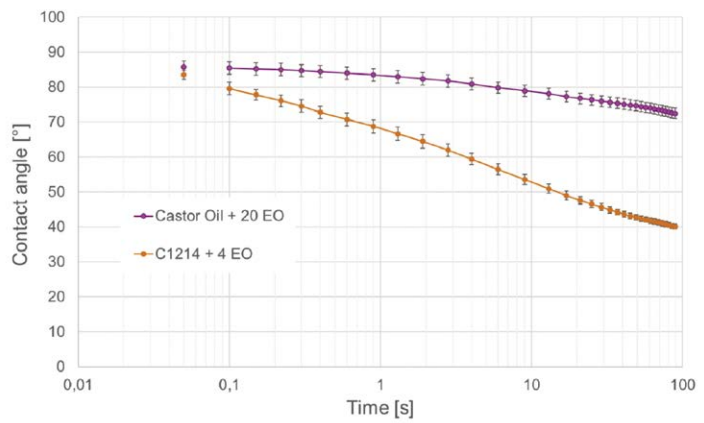


Fig. 1 Contact angle of a 0.1% aqueous solutions of castor oil ethoxylate and a fatty alcohol ethoxylate having the same HLB value of 10 (according to Griffin) on Parafilm (DataPhysics OCA50, 25°C, 50% rel. humidity, droplet volume 3.7 µL; average of at least 8 droplets).

the cloud point, because the system then forms a surfactant-rich phase dispersed in a surfactant-poor phase. The reason is that the H-bonds, which keep the ethoxylates in solution, break up at elevated temperatures. Above the cloud point, nonionic surfactants cannot really be used any more for solubilizing or emulsifying purposes, but they can act now as antifoaming agents [18]. Therefore, the cloud point is a pretty good measure of the suitability of a fatty alcohol alkoxylate for certain applications, and it takes into account one of the most important features of nonionic surfactants, their temperature dependent solubility. Generally speaking, there is some correlation between cloud point and HLB [2]: Hydrophilic, highly ethoxylated, water-soluble ethoxylates have an HLB >10 (*Griffin*) and a high cloud point, whereas more hydrophobic ethoxylates with an HLB <10 (*Griffin*) have a cloud point below room temperature. However, there is no strong correlation between calculated HLB and cloud point, as shown in the following example C12-14EO9 vs. i-C13EO9. These two alcohol ethoxylates have identical hydrophilic headgroups (9 EO); there are only differences in the structure of their hydrophobic tail: either linear C12/C14 with on average of 12.7 methylene groups, or branched i-C13. Both of these nonionic surfactants have the same HLB value, whether calculated according to *Griffin* (13.7) or to *Davies* (5.7). However, their cloud points are quite different (**Fig. 2**). Therefore, HLB can hardly be used to predict the application performance of these products.

The third example shows the effect of order of addition of EO and PO onto a C18 alcohol; in a simplified oil/water system, based on a semisynthetic metalworking formulation, the emulsifier efficiency (i.e. the minimum mass fraction $\tilde{\gamma}$ of emulsifier needed to form a one-phase microemulsion, for details see [2]) of C18+6PO+6EO is considerably higher as compared to C18+6EO+6PO, although both emulsifiers have the identical calculated HLB values.

Conclusion

The original approach by *Griffin* of introducing the HLB concept to reduce the number of emulsification trials did make sense – but it seems like over the decades the original idea

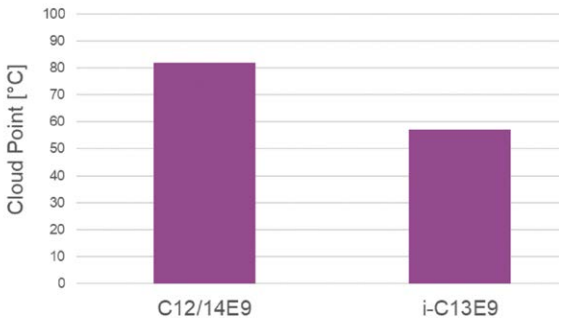


Fig. 2 Cloud points of two alcohol ethoxylates of identical calculated HLB.

Component	Formulation I [g]	Formulation II [g]	$\tilde{\gamma}$
Oil phase	33	33	
Water phase	55	55	
Emulsifier C18+6PO+6EO	3		0.03
Emulsifier C18+6EO+6PO		7.8	0.08

Tab. 2 Comparison of emulsifier efficiency $\tilde{\gamma}$ in an oil/water system with emulsifiers of same calculated HLB, but different molecular architecture (C18PO6EO6 vs. C18EO6PO6). $\tilde{\gamma}$ is defined as the minimal mass fraction of surfactant $\tilde{\gamma} = m_{surfactant} / (m_{oil} + m_{water} + m_{surfactant})$ which is necessary to form a one-phase microemulsion (see [2]).

got lost and the HLB number has been taken more seriously than intended originally. The HLB values reflect the result obtained when shaking – at room temperature – equal amounts of water and oil, using a fixed amount of emulsifier. All other extensions to derive either "more precise" HLB values or develop other – less laborious – methods to generate HLB values still suffer from this limitation. Moreover: Calculating HLB values for some modern PEG-free emulsifiers makes even less sense than for the ethoxylates it has been developed for. However, if the formulators would like to get an idea about the dispersability of an emulsifier in water, the HLB value can be used as an indication – especially if it has been determined experimentally. *Steven Abbott* states in his textbook [20] that HLB should be banned, because the concept was not only useless, but has caused huge damage because it has stood in the way of a much better systems. It should be clear that such alternative, better methods should consider parameters such as the type of oil, salt and pH; therefore, these alternatives have to be more complex than a single universal number assigned to an emulsifier alone. An introduction into two of these alternative concepts will be the topic in sofw journal 12-2020 [2].

Remark

This paper is a joint effort of the members of the CESIO Working Group "Test Methods of Surfactants" and the TEGEWA Working Group "Surface Active Substances": *Roland Borner* (Chemische Fabrik Schärer&Schläpfer AG), *Wolfgang Brennich* (Zschimmer&Schwarz GmbH&Co KG), *Karsten Holtin* (Kolb Distribution Ltd.), *Carmen Pey* (Kao Corporation S.A.), *Arjan Gelissen* and *Renke Rommerskirchen* (Sasol Germany GmbH), *Natascha Schelero* (Clariant Produkte (Deutschland) GmbH), *Kati Schmidt* (BASF SE), *Louis Schwarzmayr* (Nouryon Surface Chemistry AB), *Michael Stapels* (Kao Chemicals GmbH) and *Joachim Venzmer* (Evonik Operations GmbH). We would like to thank *Brigitte Bartsch* and *Sabine Diesveld-Koller* for experimental support.

References

[1] *W.C. Griffin*, Classification of surface-active agents by "HLB", J.Soc.Cosmet. Chem. 1 (1949) 311-326.

[2] *A. Gelissen, R. Rommerskirchen* and *J. Venzmer,* Alternative concepts to HLB, SOFW Journal, 146, 12/2020.

[3] *W.D. Bancroft,* The Theory of Emulsification V, J.Phys.Chem. 17 (1913) 501-519; The Theory of Emulsification VI, J.Phys.Chem. 19 (1915) 275-309.

[4] Atlas Powder Company, „Surface Active Agents", Wilmington, Del., 1948.

[5] *W.C. Griffin*, Calculation of HLB values of non-ionic surfactants, J.Soc.Cosmet. Chem. 5 (1954) 249-256.

[6] *J.T. Davies,* A quantitative kinetic theory of emulsion type, I. Physical chemistry of emulsifying agent, in: Gas/Liquid and Liquid/Liquid Interface, Proceedings of the Second International Congress of Surface Activity, London, April 1957, J.H. Schulman (ed.), Butterworths Scientific Publications, London, 1957, 426-438.

[7] *H.L. Greenwald, G.L. Brown* and *M.N. Finman,* Determination of the Hydrophile-Lipophile Character of Surface Active Agents and Oils by a Water Titration, Anal.Chem. 28 (1956) 1693-1697.

[8] *A.H.C. Chun* and *A.N. Martin,* Measurement of hydrophile-lipophile balance of surface-active agents, J.Pharm.Sci 50 (1961) 732-736.

[9] *V.H. Huebner*, Determination of the Relative Polarity of Surface Active Agents by Gas Liquid Chromatography, Anal.Chem. 34 (1962) 488-491

[10] I.*G.A. Fineman*, Polarity index of surface-active ethylene oxide adducts, J.Am. Oil Chem. 46 (1969) 296-298.

[11] *P. Becher* and *R.L. Birkmeier,* The determination of hydrophile-lipophile balance by gas-liquid chromatography, J.Am.Oil Chem. 41 (1964) 169-172.

[12] *W.G. Gorman* and *G.D. Hall,* Use of dielectric constants in the classification of surfactants, J.Pharm.Sci. 52 (1963) 442-446.

[13] *G. Ben-Et* and *D. Tatarsky,* Application of NMR for the determination of HLB values of nonionic surfactants, J.Am.Oil Chem. 49 (1972) 499-500.

[14] *J. Wu, Y. Xu, T. Dabros* and *H. Hamza,* Development of a method for measurement of relative solubility of nonionic surfactants, Coll.Surf.A: Physicochem. Eng.Aspects 232 (2004) 229-237.

[15] *P.M. Kruglyakov,* Hydrophile-Lipophile Balance of Surfactants and Solid Particles – Physicochemical Aspects and Applications, Studies in Interface Science Vol.9 (*D. Möbius, R. Miller*, eds.), Elsevier 2000.

[16] *T.G. Balson,* HLB: Is It a Valuable Concept or a Curiosity, in: Industrial Applications of Surfactants IV, D.K. Karsa (ed.), Royal Society of Chemistry, 1999, 175-192.

[17] Surface active agents – Determination of cloud point of non-ionic surface active agents obtained by condensation of ethylene oxide, EN 1890:2006

[18] *R. Chaisalee, S. Soontravanich, N. Yanumet* and *J.F. Scamehorn,* Mechanism of antifoam behavior of solutions of nonionic surfactants above the cloud point, J. Surf. Det. 6 (2003) 345-351.

[19] *H. Schott,* Hydrophile-Lipophile Balance and Cloud Points of Nonionic Surfactants, J. Pharm. Sci. 58 (1969) 1443-1449.

[20] *S. Abbott,* Surfactant Science – Principles & Practice, free download at www.stevenabbott.co.uk

contact

Joachim Venzmer

Evonik Operations GmbH
Goldschmidtstr. 100
45127 Essen | Germany
E-mail: joachim.venzmer@evonik.com

BYK Additives
Diversity for cleaning and care

Whether it's surface cleaners, care products, or detergents for the household, industrial and institutional sector, the optimization of a diverse range of product characteristics is BYK's clear commitment. With its innovative additives, BYK provides effective one-stop product solutions.

www.byk.com

A member of ALTANA

BYK

content

Picture Credits: dragana991/www.istockphoto.c

Beauty and the Mask – eyes only

How wearing a face mask affects the choice of the right skin care products

S. Hettwer, E. Besic Gyenge, S. Breitenbach, B. Suter, B. Obermayer

Behavioural changes

Within a very short period of time, wearing a face mask has led women to focus more on the eye area when applying make-up. A survey by the French IFOP showed that 44% of women who regularly used decorative cosmetics no longer put make-up on their lips under the mask. The proportion of women who used make-up daily decreased by half. The trend is now strongly moving towards skin care and wellness at the expense of color cosmetics (Premium Beauty News, July 12, 2020). Surveys in France, Germany and U.K. confirm this. Of those over 55, 46% say they have more often taken up beauty and body care activities to promote their health. Of the 16-34 year olds, 45% say they do this to reduce stress and anxiety. Overall, 59% said that beauty and body care activities were used to have a good time (Mintel survey, 2020).

In uncertain times, people want to feel safe and secure. This leads to a strong demand for cosmetics that care for consumers and make them feel good. In other words, products with fragrances and active ingredients that help people to relax and cope better with stress. The personal care industry can respond with more positive and emotional marketing. However, the products must also be effective enough to generate long-term trust and interest, which is very important for emotional products.

What happens above the mask?

The mask covers a large part of the face. Therefore it is not always easy to correctly assess the emotions and state of mind of the person opposite. To emphasize the beauty of the face, a large part of the "surface" is also hidden. As the surveys show, it makes little sense to wear lipstick under the mask. It remains to accentuate the eye area and obviously, there are no limits in the color cosmetics field.

The situation is different with rejuvenating cosmetics. Here too, the eye area offers great opportunities for application, because the wrinkles around the eyes make a big difference. And just as in the color cosmetics area, the immediate effect is desired. For this purpose, we offer LIFTONIN®-XPRESS, the "Instant Wrinkle Minimizer". Whether on the train, while shopping or in a zoom meeting. With LIFTONIN®-XPRESS you can even out wrinkles around the eye area or on the forehead in seconds, for a fresh and young look above the mask (**Fig. 1**).

What happens under the mask?

In many countries in Asia for a long time, an expression of mutual respect to protect their fellow human beings from infectious respiratory diseases, is wearing a face mask. This is a rather unusual sight in the rest of the world, up to now. However, the COVID-19 pandemic has also led to the wearing of masks there, which can cause cosmetic problems for the skin. We see three main causes that can affect the skin when wearing masks:

- Mechanical irritation of the skin
- Increase in humidity
- Increase of the air temperature

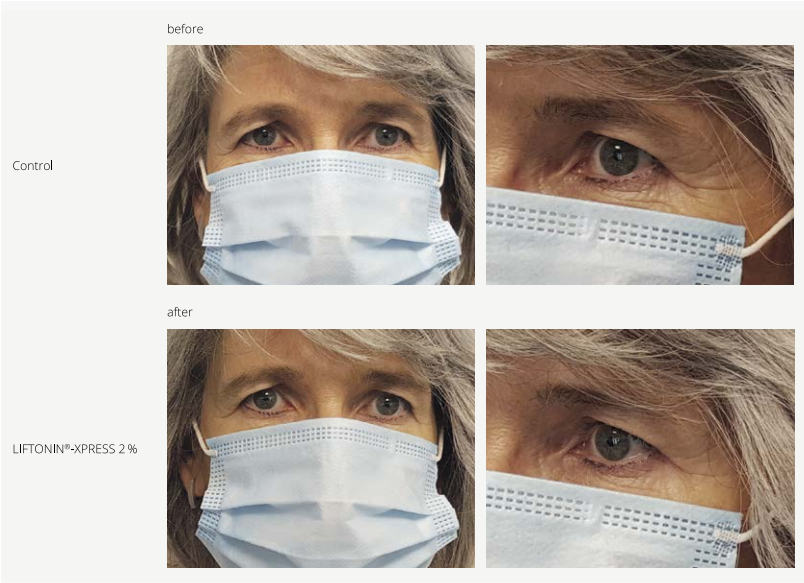


Fig. 1 The application of 2 % LIFTONIN®-XPRESS leads to an immediate reduction of wrinkles. In less than a minute.

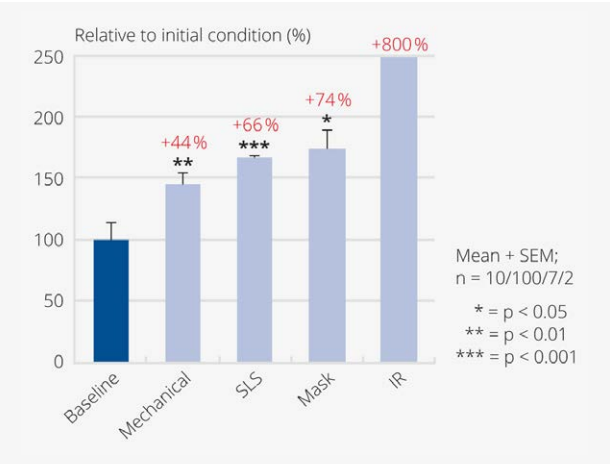


Fig. 2 After 2 hours of wearing a simple protective mask, the TEWL increases by almost 75%.

Mechanical irritation in the areas where the mask touches the face, such as the chin, cheeks and the bridge of the nose, can damage the skin barrier and cause an inflammatory reaction that leads to reddening of the skin. Skin irritation and discomfort can be the result. Permanent wearing of masks may intensify these skin reactions by mechanical activation of the TRPV1 receptor. It is therefore important to suppress corresponding inflammatory reactions from the beginning. Not least to avoid an acne outbreak (maskne).

But it is not only the mechanical irritation of the skin that causes problems. Internal studies show that the air conditions under the mask also lead to an undesired change of the skin barrier. The transepidermal water loss (TEWL) under the mask was on average almost 75 % higher than without wearing a mask (**Fig. 2**). Indeed, *Van Rensburg et al.*, Skin Res Technol. 2019, describe that TEWL correlates positively with an increase of skin temperature. The TEWL is strongly influenced by the functioning skin barrier and any damage, be it chemical, mechanical or physical, directly expresses itself in an increased TEWL. The measurement of the TEWL is therefore the most important means to detect a damage of the skin barrier. In own studies it could be determined that the skin barrier is strongly disturbed by mechanical irritation (tape stripping), washing with SLS and massive IR irradiation (heat). Wearing a mask for only 2 hours already had a strong effect here. Increasing the temperature under a mask has the effect of a permanent irradiation with low doses of infrared light, which demonstrably disturbs the skin barrier. In addition, there is also mechanical irritation at the points where the mask lies on the face.

These skin changes can therefore occur as a result of wearing a mask:

- Disruption of the skin barrier
- Skin redness
- Skin irritations and sensitive skin
- Acne outbreak

Since the trigger – wearing the mask – cannot be prevented, cosmetic solutions must be based on the expected disorders. This is where cosmetic active ingredients having the following effects can be of help:

- Reduction of the transepidermal water loss to strengthen the skin barrier
- Reduction of the microcirculation to counteract reddening of the skin
- Blocking of the TRPV1 receptor to counteract inflammation and skin irritation
- Skin microflora and acne control

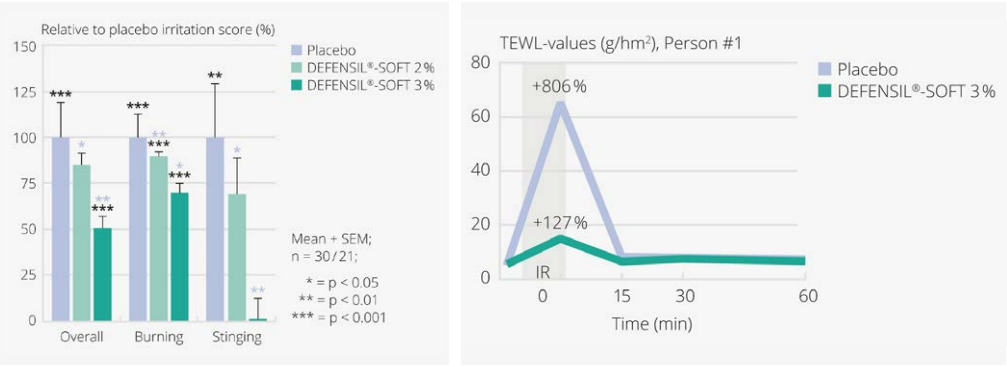


Fig. 4 DEFENSIL®-SOFT can reduce skin irritation and discomfort within 3–5 minutes. It prevents heat-related transepidermal water loss, reduces microcirculation and skin redness.

RAHN offers various cosmetic actives as a solution

DEFENSIL® protects against increasing transepidermal water loss due to skin irritation and thus strengthens the skin barrier. While cortisone and panthenol were unable to stop an increase in transepidermal water loss after irritation with SLS, the skin barrier remained virtually intact with 2% DEFENSIL® and was able to regenerate very quickly (**Fig. 3**).

Thanks to its anti-inflammatory properties, DEFENSIL® reduces the microcirculation triggered by an increase in temperature within minutes and can thus reduce skin redness (see DEFENSIL® documentation).

We pursue a different approach with DEFENSIL®-SOFT. It reduces heat-related transepidermal water loss and redness. DEFENSIL®-SOFT acts as a neuro-soother, which can bring the hypersensitivity of the skin due to the constant irritation by the mask back into the normal range. An increase in microcirculation is effectively reduced (see DEFENSIL®-SOFT documentation). A few minutes are sufficient to reduce sensations (**Fig. 4**). With 2 % you already get remarkable results, which are considerably intensified with 3 %.

We recommend REFORCYL® for more mature skin or in cases of mechanical damage to the skin barrier due to chafing of the mask on the skin. At an application level of 3 % it strengthens the skin barrier after mechanical stress as impressively determined in a tape stripping assay. The moderate mechanical removal of corneocytes already showed a 10 % increase of the transepidermal water loss, which could be completely compensated after 14 days. After 28 days, it was even 10 % lower than baseline level. Placebo, however, did not lead to any improvement (**Fig. 5**). The reason for the fast regeneration is the increased lipid synthesis and refilling of fatty acids which are important for the skin barrier. REFORCYL® is therefore very well suited for mature, dry skin whereas young skin needs different care, as oily skin tends to acne.

SEBOCLEAR™-MP takes care of the causes of maskne

Maskne (acne caused by wearing a mask) has been a rapidly developing skin impurity since the COVID-19 pandemic. In addition to adequate cleansing and moderate exfoliation, sebum

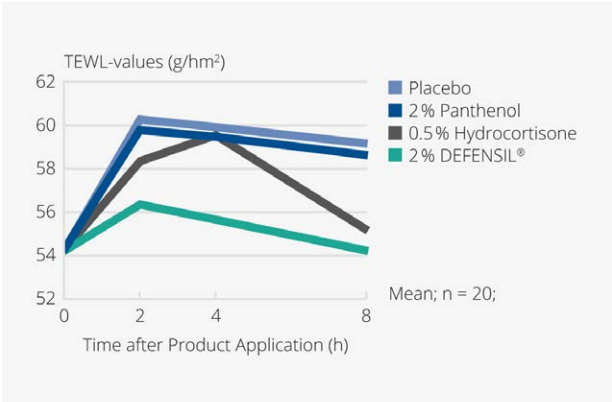


Fig. 3 DEFENSIL® maintains control of the skin barrier after irritation with SLS.

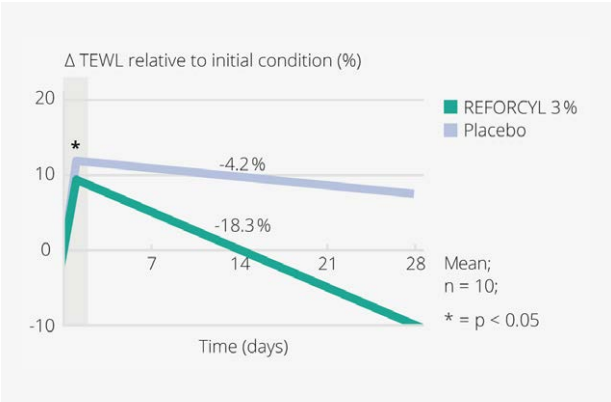


Fig. 5 Single tape stripping simulates a damage of the skin barrier, as one can imagine a mechanical abrasion of corneocytes under a mask.

mibelle GROUP biochemistry

BSB Innovation Award® NATURAL Products 2020
2. Prize Category Most Innovative Raw Material

Approved by ECOCERT RAW MATERIAL

COSMOS APPROVED

Alpine Rose Active
Clearing age-promoting cells

Eliminating senescent cells has emerged over the past few years as a promising anti-aging therapy in the medical field and with Alpine Rose Active this novel "senolytics" concept has now been adapted for cosmetics for the first time. Alpine Rose Active was shown to specifically clean-up misdirected, age-promoting senescent cells, and in clinical studies it significantly reduced skin redness, increased skin elasticity, and protected the skin from UVA induced photo-aging.

- **Eliminates senescent skin cells**
- **Reduces redness and increases skin elasticity**
- **Rejuvenates the deep layers of the skin**

Alpine Rose Active is a purified extract from the leaves of the organic alpine rose, which is one of the most typical and iconic Swiss alpine plants. This robust and resilient plant grows in the high Alpine regions of Switzerland and it is carefully harvested by sustainable wildcrafting. COSMOS approved.

SWISS QUALITY PRODUCT

Mibelle AG Biochemistry, 5033 Buchs/Switzerland, Phone +41 62 836 17 31

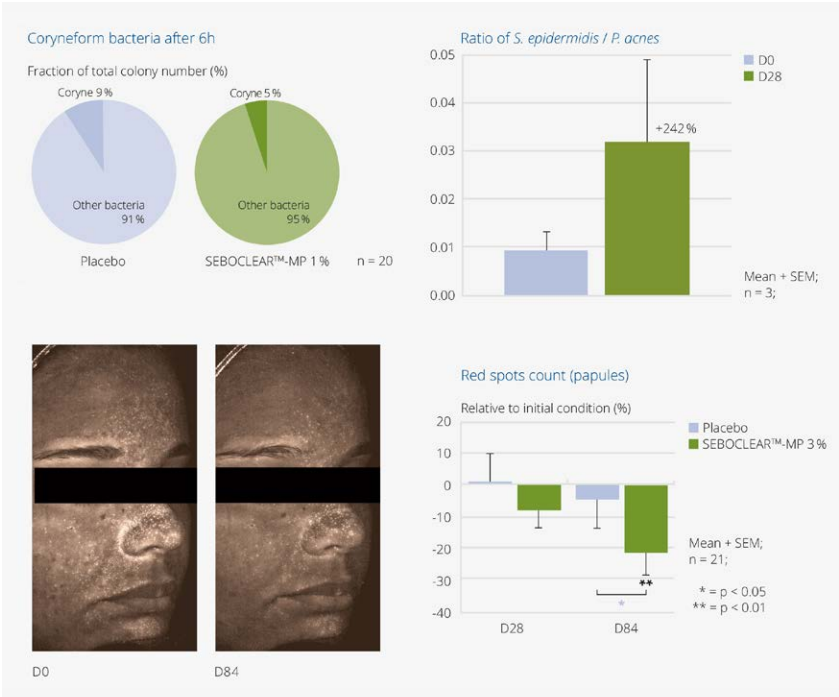


Fig. 6 SEBOCLEAR™-MP reduces corynebacteria and propionibacteria and promotes healthy skin microflora, recognizable by the reduction of porphyrin-positive pores. It reduces the number of inflamed papules, which can develop into acne pimples when irritated.

Skin Balancing SOS Treatment

(700.482.0001)

St	Substance	INCI name USA	% [w/w]	Manufacturer
1	Water demin.	Water	60.50	several
2	TEGO Feel C 10	Cellulose	1.00	Evonik, DE
	Keltrol CG-SFT	Xanthan Gum	0.50	CP Kelco, US
3	Tego Alkanol 1618	Cetearyl Alcohol	3.50	Evonik, DE
	Dermofeel GSC	Glyceryl Stearate Citrate	3.00	Evonik, DE
	Amisoft HS-11P(F)	Sodium Stearoyl Glutamate	0.20	Ajinomoto, JP
	Tegosoft CI	Cetearyl Isononanoate	7.00	Evonik, DE
	Verstatil PC	Phenoxyethanol, Caprylyl Glycol	1.00	Evonik, DE
	Myritol 318 MB	Caprylic / Capric Triglyceride	18.00	BASF, DE
	Dermofeel Toco 70 non-GMO	Tocopherol, Helianthus Annuus (Sunflower) Seed Oil	0.10	Evonik, DE
4	DEFENSIL®-PLUS	Octyldodecanol, Ribes Nigrum (Black Currant) Seed Oil, Helianthus Annuus (Sunflower) Seed Oil Unsaponifiables, Cardiospermum Halicacabum Flower/Leaf/Vine Extract, Tocopherol, Helianthus Annuus (Sunflower) Seed Oil, Rosmarinus Officinalis (Rosemary) Leaf Extract	3.00	RAHN AG, CH
5	SEBOCLEAR™-MP	Propanediol, Maclura Cochinchinensis Leaf Prenylflavonoids	2.00	RAHN AG, CH
6	Perfume NihilO001	Perfume	0.20	Huber the Nose, CH

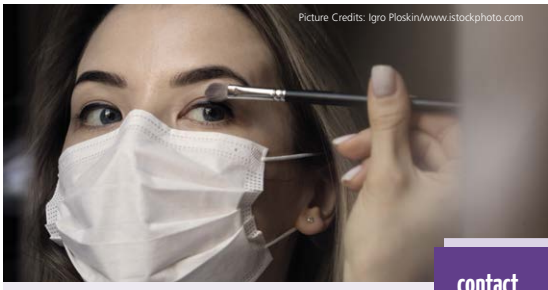
Production: Mix 2 and add to 1 while stirring, the phase must be homogenous, heat to 75°C while stirring / Mix 3 and heat to 75°C while stirring / Add 1&2 to 3 while stirring strongly, homogenise and cool to 40°C while stirring / Add the remaining ingredients separately, homogenise strongly, cool to 25°C while stirring.

Formulation Example of a formulation perfectly designed to strengthen the skin barrier and prevent maskne when wearing face masks.

control and inhibition of acne-causing bacteria is essential. In the humid climate under the mask, the growth conditions of corynebacteria and propionibacteria are optimized. It is therefore important to keep them in check and reduce inflammatory skin reactions. This is where SEBOCLEAR™-MP can help, as it quickly reduces the number of coryneform bacteria and balances the ratio of the good skin microbiota (*S. epidermides*) to the acne germ *Propionibacterium acnes*. SEBOCLEAR™-MP markedly reduces the number of pores populated with *P. acnes* and suppresses acne formation, especially in the chin/nasolabial folds/bridge of the nose (**Fig. 6**).

The important thing with this face care is to take intensive care of the skin barrier. For this purpose, we have developed the "Skin Balancing SOS-Treatment" with 2 % SEBOCLEAR™-MP and 3 % DEFENSIL®-PLUS. DEFENSIL®-PLUS, as an intensified version of DEFENSIL®, is RAHN's flagship product for the protection and repair of the skin barrier. In addition to supporting the skin's own barrier active ingredients, it has a strong anti-inflammatory effect, which, in synergy with SEBOCLEAR™-MP, ensures optimum protection when wearing a mask.

With the selected RAHN-Cosmetic Actives you can get the most important skin irritations under control when wearing a mask.



contact

Stefan Hettwer | stefan.hettwer@rahn-group.com
Emina Besic Gyenge
Sandra Breitenbach
Brigit Suter
Barbara Obermayer

RAHN AG
Dörflistrasse 120
8050 Zürich | Switzerland

NEW

Immedeline® PG

5mins wrinkles invisible, tightening effect for **8h**.

Source from precious peach resin in Jade Dragon Snow Mountain, with 4:1 golden ratio technique, Immdeline® PG brings you tightening and anti-wrinkle effects in just 5 minutes and it effect lasts for 8 hours.

Clinical test (VISIA) 5% Immedeline® PG

+ 86-21-34556071 export@greaf.com.cn www.greaf.com

7th Floor, Building No. 1, Yuanmei Road No. 58, Minhang District, Shanghai 200241, China

GREAF®
Active Ingredients

Functional Vegan Silk – Crafted by White Biotechnology to Utilise a New Way to Take Care of Damaged Hair

J. Müller, N. Blosl, M. de Tollenaere, A. Scandolera, K. Nessbach

abstract

Human hair can be damaged in many different ways. Frequently in everyday life, the natural fibres come into close contact with heat or chemical agents, which attack and damage their structure and can impair their visual appearance. In recent decades, various substances have been identified and developed by the cosmetics industry to minimize such influences. Previously, it has been reported that Silkgel, a breakthrough nature-inspired biopolymer from Givaudan Active Beauty, offers many unique benefits particularly for the use in hair care applications such as anti-pollution, anti-colour-fading and sensorial features.

A completely new application for this highly innovative, vegan and biomimetic raw material presents its use as conditioning agent to protect and repair the hair structure from damages. Due to its defined composition and its ability to self-assembly, the biotechnologically derived vegan silk polypeptides inside the Silkgel are able to form a protective and conditioning film onto the surface of the hair. Therefore, vegan silk molecules can represent a valid alternative to traditional and chemical protective conditioning agents based on hydrolysates such as keratin, wheat protein and wheat starch. According to our latest findings, it has been demonstrated that vegan silk polypeptides used as protective or repair conditioning agents were able to increase the breaking force as well as to decrease the 3D volume fraction of the damage (x-ray nanotomography) of thermally treated and dyed hair. Thus, in contrast to conventional raw materials based on e.g. silicones, which are commonly used for hair conditioning and protection, vegan silk represents a true ecological, natural-inspired and vegan alternative with an excellent protective and repairing performance for both thermal treatment and colouration of hair, thereby meeting the expectations of the highly innovative clean beauty market.

The hair styling market will continue to grow with the rising consumer demand of innovative active ingredients

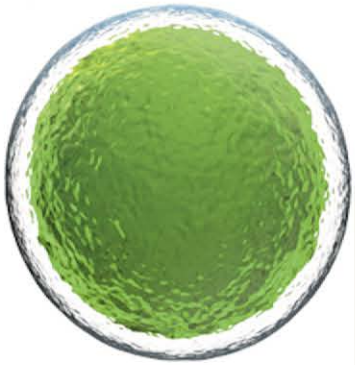
The global hair styling market is expected to exceed the $28 billion by 2027, growing at an average annual growth rate (CAGR) of 5.1% [1]. Growth of the hair care segment is driven by a rising customer tendency to experiment with hair styles and colours. Social media e.g. Instagram and beauty blogs have become even more important channels for advertising and promoting of hair styling products in the last few years [2]. Seven out of ten consumers have already purchased beauty products based on an influencer´s recommendation. Generation Z and Millennials (now aged 22-37), more conscious about their looks and appearance, are the strongest drivers behind the hair styling category (**Fig. 1**) [1, 2].

It is well known, that the repeated and excessive use of hair styling tools such as curling wand, straightener or blow dryer can damage the hair and cause moisture loss, frizz, dryness, split end and hair breakage. Chemical treatments (e.g. hair dyeing and bleaching) can also negatively impact the hair texture and quality. In fact, these chemical treatments are the leading cause of changes in hair structure and ultimately hair damage. However, in general a thermal induced damage has higher degree of damage. Damaged hair has less shine and smoothness, thus directly indicating poor hair health [3]. In addition damaged hair has reduced elasticity and breaks easily under tension. The tensile strength of the hair shaft is related to its elasticity and is defined as stretching without breaking [4]. Healthy hair fibres have high resistance to stretching with a high tensile strength. Environmental conditions such as UV radiation and air pollution can also have an impact on hair quality [5, 6].

To prevent the hair effectively from taking damage or to repair it after heat or chemical treatment, a variety of hair care products are available on the market. Typical examples for these hair styling products are leave-in spray conditioners. They protect the hair from damage providing heat protection combined with a conditioning effect. Alternatives are intensive treatment rinse-off repair conditioners or masks that protect the hair from damages caused by hair dyes. Commonly used ingredients of these products are silicones, which can effectively form a protective layer on the hair fibres. However, as it can be difficult to wash silicones out of the hair entirely they can cause a "build-up effect" on hair [7]. Other protective and widely applied con-

Fig. 1 Social media presents an attractive opportunity for advertising and promoting hair styling products.

Greenaltech
Microalgae Technologies



ALGAKTIV® BodySKN
Upcycling skincare, lifting sustainability

ALGAKTIV® BioSKN
The ultimate skin super food

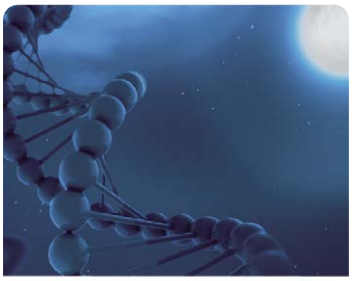
ALGAKTIV® RestoreSKN
Dermal renewal for rapid lifting

ALGAKTIV® GenoFix Day
Light powered skin care

ALGAKTIV

World of Microalgae

ALGAKTIV® LightSKN
Correct age spots for an even tone



ALGAKTIV® GenoFix Nite
Deep night repair

VEGAN

CHINA LISTED

SUSTAINABLE

Approved by ECOCERT RAW MATERIAL
COSMOS APPROVED

ALGAKTIV® UrbanSKN
Shield the skin from urban life

ALGAKTIV® Zen
Prevent lifestyle skin aging

ALGAKTIV® Optimmunist
Protect and care for sensitive skin

GloryActives

Distribution in Germany:

Your partner for cosmetic active ingredients

GloryActives GmbH
Am Pruessee 46
21514 Guester (Germany)

+49 4158 209469
info@gloryactives.de
www.gloryactives.de

ditioning agents are proteins and protein hydrolysates. Particularly frequent, protein hydrolysates for hair care are obtained from keratin and wheat. Since these hydrolysates have no defined composition and molecular structure, the components of the hydrolysed proteins could lose their functionality of the full length protein [8]. Givaudan´s innovative vegan silk protein inside of Silkgel is non-hydrolysed and is therefore fully functional in its performance. In particular, vegan silk proteins are able to form a protective film on the surface of the hair.

Since the consumers are becoming more and more concerned about their impact on the environment, the demand for innovative, environmentally friendly active ingredients for hair care is constantly growing. Givaudan Active Beauty offers Silkgel, a hydrogel containing a vegan silk protein with a high potential for this rising market. Recently published studies have demonstrated the high potential of Silkgel for the hair care market. Major claims represent anti-pollution, anti-colour fading and sensorial improvements [9].

For the evaluation of further beneficial qualities of Silkgel for the hair care segment, the present study focuses on its protective properties and recovery effects on thermally or chemically damaged hair using highly innovative research methods such as tensile testing or x-ray nanotomography.

Materials and Methods

Evaluation of Silkgel as protection and hair repair agent

The efficacy of Silkgel as hair repair and protection agent has been assessed on natural European blond hair (Kerling International Haarfabrik GmbH). The hair protection studies were performed applying Silkgel prior to the damage induction by a thermal treatment (**Fig. 2A**). In order to demonstrate the hair repair effect of silk, Silkgel was applied after the colouration process of natural hair wefts and also after thermal treatment of these samples (**Fig. 2B**).

For both, the hair protection and the hair repair approach tensile testing was the method of choice to determine the quality of the hair based on the strength of its breaking force.

Ex vivo measurements of hair breakage using tensile testing

To analyse the effect of functional vegan silk polypeptides used as protective and repair conditioning agents to improve the hair quality an *ex vivo* tensile testing method was established using human hair wefts. Based on the determination of the breaking force of single hair fibres, this procedure is in particular suitable for generating comparable values to evaluate the degree of damage of the hair structure. A hair fibre of known length and diameter is stretched at a certain rate until it finally breaks. The measured breaking force is proportional to the integrity of the intermediate filaments and the surrounding matrix of the cortex and therefore it represents a characteristic for the degree of hair damage [10].

In a first step, the hair diameter of the corresponding sample was determined, before 10 hairs per sample were consecutively clamped into the tensile tester (Zwick BT1-FK0.5N. D14 from Zwick / Roell GmbH & Co. KG) to determine the breaking force. The thickness was measured using a precision micrometer (ID-H0530 from Mitutoyo GmbH). As each hair becomes thinner from the shaft to the tip, an average of five diameter measurements was taken.

The samples were tested following DIN EN ISO 2062 standard originating from the textile industry with a pulling speed of 250 mm/min and an effective clamping length of 80 mm. The tensile strength was calculated according to the following formula:

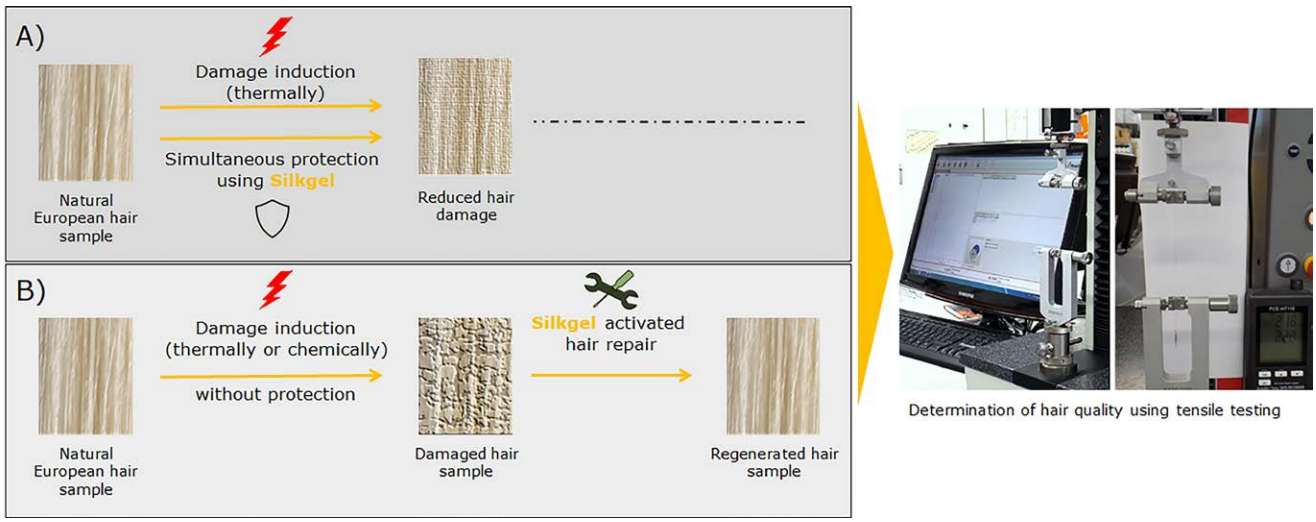


Fig. 2 Procedure of hair repair and hair protection analysis using tensile testing.
A) To investigate the protective effects of Silkgel an aqueous solution containing 3% of Silkgel was sprayed on the hair prior to the treatment of the hair wefts with a hair straightener. The film-forming properties of the silk proteins prevent the hair fibres to take serious damage from the heat treatment by a physical protection barrier.
B) Hair repair of chemically treated (coloured) hair wefts was induced using an aqueous solution of 2% Silkgel or a benchmark hair conditioner after the dyeing procedure. The regeneration of thermally damaged hair was performed by the application of Silkgel in an aqueous solution of 3%.

$$\% \text{ tensile strength increase} = \frac{F_D - F_T}{F_D}$$

F_T: break force of silk treated hair
F_D: break force of non-silk-treated hair (blank)

In order to minimize diameter dependent fluctuations the measured breaking force values are given standardized to the diameter of the respectively measured hair. Following formula was used:

$$F_S = \frac{F_M}{2 \times \pi \times r^2}$$

F_S: **standardized** maximum breaking force
F_M: **measured** maximum breaking force
r: radius of the hair sample

Afterwards the data were analysed using 1-way Analysis of Variance (ANOVA), followed by a post-hoc analysis Tukey Honest Significance Difference (THSD) test, using the R programming language. Probability values $p < 0.05$ were considered statistically significant. For probability values $p < 0.01$ the term highly significant was true and probability values $p < 0.001$ were considered the most significant.

Hair protection study – thermal protection

For the determination of the protective impact of functional vegan silk on damages caused by thermal straightening, a solution was prepared containing 3% of Silkgel in water. The formulation was used like a leave-in conditioning spray and applied to natural European hair wefts (remis, color 9/0) prior to the thermal straightening procedure (220°C for 1 minute). In addition, a commercially available benchmark product (Express-repair conditioner) was used as control. The test setup simulates a typical use of a heat protection spray (**Fig. 2A** and **Fig. 3**).
The hair tresses were first washed with shampoo and then dried with a towel. Afterwards, the corresponding protective formulation (Silkgel or benchmark) was sprayed on damp hair (3x spraying corresponds to approx. 300 µl) like a leave-in

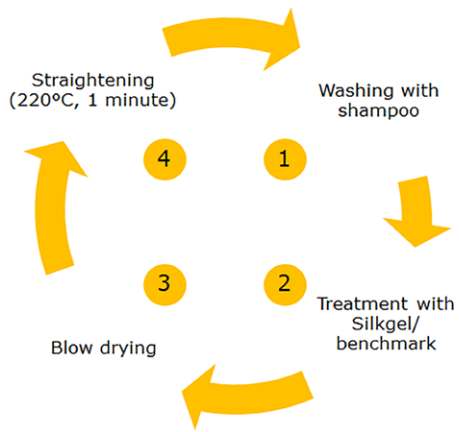


Fig. 3 Treatment cycle for analysis of the protective effect of functional vegan silk against thermally induced hair damage.

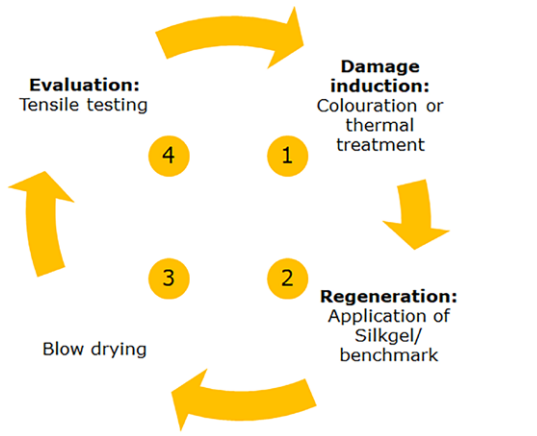


Fig. 4 Treatment cycle for analysis of the repair effect of functional vegan silk against thermally and chemically induced hair damages.

product. Subsequently, the threads were gently blow-dried (50°C). This was followed by heat treatment with the hair straightener at 220°C for 1 min. This treatment was performed over 20 times for several weeks (**Fig. 3**) and afterwards the tensile strength of the samples could be evaluated.

Hair repair study – regeneration of thermally and chemically induced damages

The efficacy of Silkgel as hair repair agent after the dyeing process has been assessed on natural European blond hair after thermally and chemically induced damages. To analyse the effect of Silkgel on chemically damaged hair wefts, the hair tresses were coloured red using an ammonia based colouration agent and treated either with an aqueous solution of Silkgel (2%) or a benchmark hair conditioner (containing hydrolysed wheat and rice starch) after the dyeing procedure (**Fig. 2B** and **Fig.4**).

Thermally induced hair quality loss was induced by the treatment with a hair straightener over 20 times at 220°C for 1 minute (as previously described in **Fig. 3**). Afterwards hair repair was induced by the application of Silkgel in an aqueous solution (3%) as well as with Silkgel in a leave-in formulation (composition: 10.0% oil phase, 5.0% glycerol, 3.0% Silkgel, aqua ad to 100%). The application was repeated over 14 times (**Fig. 4**). In addition, a typical benchmark product claiming hair repair was tested.

Another way to determine the efficacy of Silkgel as hair repair agent after the bleaching process has been assessed on light blond hair after chemically and thermally induced damages. To analyse the repair effect of Silkgel, the hair tresses were bleached using 9% hydrogen peroxide and 3% ammonium persulfate solution for 1 hour at 40°C. The hair tresses were rinsed 3 times with water and blow dried for 1 hour. Afterwards, the hair tresses were straightened using a hair straightener at 220°C for 1 minute to increase the breakage of disulfide bonds and increase porosity (3 passages). In a final step, 0.5 mL of Silkgel in an aqueous solution (2%) was applied and incubated overnight with gentle massages after the bleaching procedure. Untreated and non-exposed (bleaching solution and straightening) hair tresses were used as negative control representing native hair quality (**Fig. 5**).

The hair porosity analysis was performed using X-ray tomography. A resolution of 800 nm was applied to generate 3D images of all samples. Quantification of the damage caused by the treatments was performed using image analysis techniques. Two parameters were analysed: the 3D volume fraction of the damage out of the total volume of the sample and the increase of 3D area created by pores/openings due to damage. Four samples per condition were analysed.

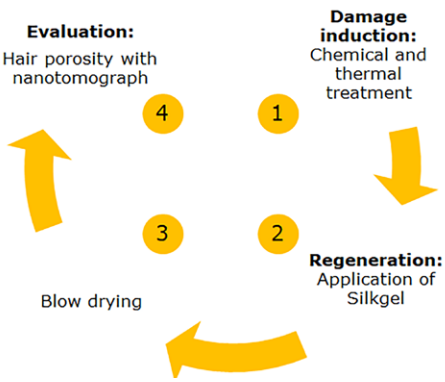


Fig. 5 Treatment cycle for the nanotomography analysis of functional vegan silk repair effect against chemically and thermally induced hair damages.

Results

Vegan Silk – An excellent protection agent for hair care

As protection claims belong to the top 5 beauty functional claims of the hair styling market, and as vegan silk has already been proven to provide anti-colour fading and anti-pollution effects in the past, Silkgel has been screened for its potential as a heat protection agent [9, 11].

Silkgel protects the hair from thermal damages

The analysis of the tensile strength of thermally damaged hair was carried out in order to identify the tensile strength reduction for unprotected thermally treated hair tresses as well as to evaluate a potential protective effect by the use of vegan silk. The detected breaking force for the unprotected tresses was set 100% and used for the calculation of the tensile strength increase of the protected tresses (**Fig. 6**).

The tensile test showed that the maximum breaking force of unprotected, thermally treated hair was reduced by around 30% compared to non-thermally treated hair. That corresponds to the data given in relevant literature [10]. By using the corresponding protective formulations (benchmark and silk solution), a highly significant increase in the maximum breaking strength of the thermally treated tresses by 56% and 47% was recorded compared to unprotected thermally treated hair ($p < 0.001$). *Nicholson et al.* (2016) observed for the use of protective collagen, keratin und wheat hydrolysates tensile strength increase percentages between 18 to 29%. Thus, with the results not only the protective effect of the silk proteins against thermal damage in leave-in products

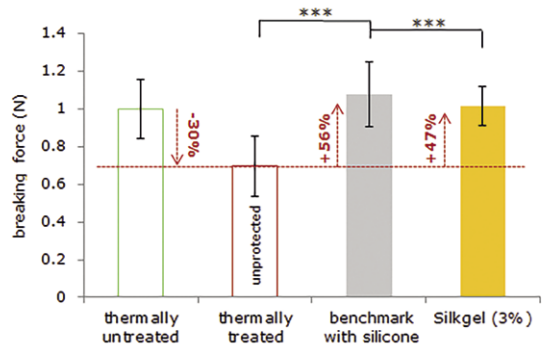


Fig. 6 Mean breaking force (N) with standard deviation and tensile strength percentage increase for thermally treated tresses. The samples were damaged for 20 times and treated with the respective formulations (unprotected, benchmark or aqueous silk solution at 3%) prior to the straightening procedure. As benchmark product a commercially available, silicone-based heat protection spray was used. Using the THSD test statistically most significant differences could be detected. (***) $p < 0.001$.

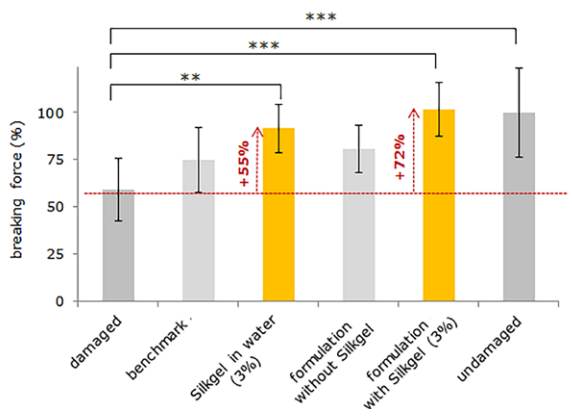


Fig. 7 Standardized mean breaking force (%) with standard deviation considering the hair diameter of each sample and tensile strength percentage referred to the damaged/unprotected sample. As benchmark a commercially available express-repair conditioner containing hydrolysed silk was analysed. Using THSD testing, compared to the unprotected hair sample, statistically highly and most significant differences could be detected for the samples treated with Silkgel. (**) $p < 0.01$; (***) $p < 0.001$.

in general, but also their increased effectiveness over conventional conditioning agents (collagen, keratin and wheat hydrolysates) could be proven.

Real added value – Silk treatments for hair repair

Silkgel was already proven to provide numerous benefits for hair care applications such as anti-pollution effectivity, sensorial improvement of hair fibres, anti-colour fading and brand-new heat protection properties. In addition the ability of silk to regenerate and repair already damaged hair was analysed.

Regeneration of thermally damaged hair up to healthy state

To evaluate the influence of thermal hair treatment to the tensile strength and to evaluate the potential of vegan silk as repair agent six hair samples have been examined using the tensile tester. The determination and comparison of the standardized maximum breaking force (FS) of the hair samples are shown in **Fig. 7**. As previously described the thermal treatment of the hair damages its structure and reduces its resistance by around 30%. The subsequent application of formulations containing silk polypeptides over 14 times improves the hairs resistance significantly. Using Silkgel at 3% in an aqueous solution increased the hairs breaking force by up to 55%. Using a newly developed conditioner formulation this value increased even more by up to 72%. Thus the hair quality is again in the range of completely intact hair. Even in comparison to the express-conditioner benchmark containing animal-derived silk, a significantly enhanced repair effect was demonstrated for Active Beauties vegan silk.

Total repair – Silkgel treatment after colouration

For the evaluation of the tensile strength after dyeing of hair and the potential of vegan silk as repair agent four hair samples have been examined using the tensile tester. The determination and comparison of the standardized maximum breaking force (FS) of the dyed hair samples are shown in **Fig. 8**.

It could be shown, that hair dyeing itself damages the fibrous hair structure revealing in a reduced maximum breaking force (-25% compared to uncoloured hair). In addition, the treatment of the coloured samples with the Silkgel solution (2%) decisively increased the hairs maximum breaking force by 35%, thus resulting in a hair quality level of undamaged hair and performing even better than the wheat and rice starch containing benchmark. Taking into account the natural character of vegan silk, using this raw material for hair conditioning and protection represents an excellent ecological and biodegradable alternative to conventional ingredients such as silicones but also other natural ingredients such as animal derived silk, collagen, keratin and wheat hydrolysates.

Silk induced repair visualized using x-ray nanotomography

To visualize and measure the efficacy of Silkgel as repair agent x-ray nanotomography was conducted after the chemical and thermal treatment of hair wefts and the 3D volume fraction and area increase were determined. The hair wefts were either untreated, chemically and thermally damaged (bleached control) or chemically and thermally damaged with subsequent application of Silkgel (**Fig. 9**).

It could be demonstrated that the bleaching procedure induced a lightening of the hair tresses and a rough-touch effect.

In **Fig. 9**, we can observe the good condition of the cuticle in the untreated group. Indeed, the green colour represents a healthy hair and in contrary, the blue colour reflects damages. The hair fibres of the bleached control are reduced in diameter and more damaged areas are observed in comparison to samples treated with 2% of Silkgel.

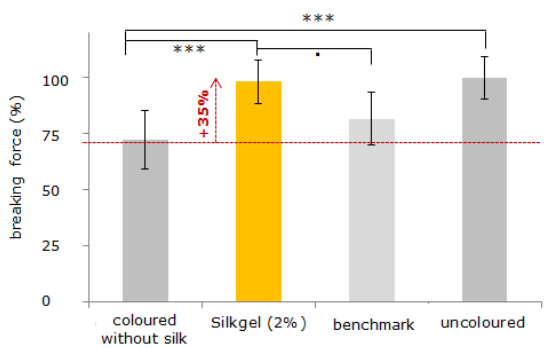


Fig. 8 Standardized mean breaking force (%) with standard deviation considering the hair diameter of each sample and tensile strength percentage referred to the treated/coloured sample. As benchmark a commercially available conditioner containing hydrolysed wheat and rice starch was analysed. Using THSD testing, compared to the coloured/damaged hair sample, statistically highly significant differences could be detected for the samples treated with Silkgel. (***) $p < 0.001$; (·) $p < 0.05$.

In **Fig. 10**, the bleached control showed an increase of 3D volume fraction of the damage and 3D area.
The quantification reflects the increase of the blue colour presence in **Fig. 9** and the macroscopic observations. The quantification of the damages in Silkgel condition demonstrated a lower degree of damages in comparison with the bleached control. Indeed, in presence of Silkgel the 3D volume fraction of the damage is reduced by 56% and the 3D area by 63% ($p < 0.1$). These results demonstrated that a single application of Silkgel at 2% is able to restore hair damages and especially strongly reduced the hair porosity mediated by bleaching.

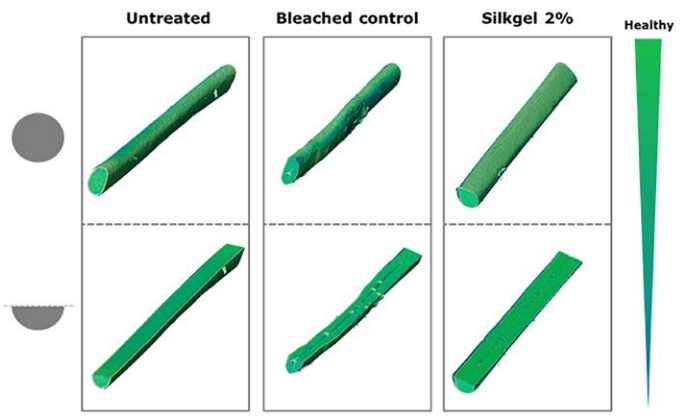


Fig. 9 3D representative images of the samples. Blue areas represent strongly damaged hair sectors, whereas lighter green areas stand for healthy hair quality.

Conclusion

Functional vegan silk – Meeting the performance and clean beauty market expectations

The hair styling market and its continuously growing demand for real innovations is mainly driven by the young generation (aged between 22 and 37 years) that are very conscious about their looks and appearance [1, 2]. Besides the performance of their styling products, "Millennials" are increasingly focusing on a healthy lifestyle that is compatible with their environment. Most recently, especially on social media

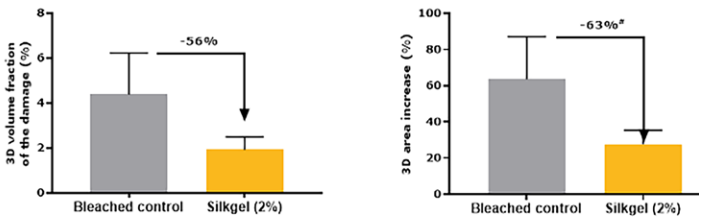


Fig. 10 Hair porosity quantification after bleaching with or without Silkgel application for 3D volume fraction and area increase. The results are expressed in percentage of the untreated control (0%).

frequently. Clean beauty products include ingredients ethically sourced and are made with the health of our bodies and the environment in mind [12]. The investigation of functional vegan silk polypeptides used as protective and regenerating conditioning agent against thermally and chemically induced hair damages for such a clean beauty market has become a great success. In particular, it has been demonstrated that functional vegan silk protects human hair from damage when thermally treated using a hair straightener. The tensile strength of silk protected hair could be increased by almost 50%, which corresponds to the hair quality level of undamaged hair and is in the same range as when using a commercial heat-protection conditioner containing silicones.

Furthermore, a second approach was chosen to evaluate the potential of vegan silk as repair agent for damages caused by hair-dyeing or hair straightening procedures. For both concepts a considerable increase in hair quality could be observed through the use of silk. Even heavily thermally damaged hair could be strengthened by up to 72% by the application of vegan silk as nourishing repair treatment. This means that the hair quality is again in the range of undamaged hair. Likewise, in the case of pre-damaged hair caused by the dyeing process, it has been shown that even a single after-treatment of the hair with silk increased its strength by 35% to the same level as uncoloured hair. Compared to other commonly used more or less "ecological" considerable raw materials for hair repair claims such as collagen, starch, animal-derived silk, keratin or wheat hydrolysates vegan silk was certified a substantially better performance [10]. The enormous hair repair potential of Silkgel could also be visualised using x-ray nanotomography. The method evidenced that a single application of Silkgel at 2% is able to restore hair damages and especially strongly reduces the hair porosity mediated by bleaching. The 3D volume fraction of the damage was reduced by 56% and the 3D area by 63% ($p<0.1$).

Typically vegan silk is used in leave-in products, but also for rinse off products the usage of this outstanding material can result in an enhanced protection and repair performance from concentrations of 2% upwards.

It is well known that consumer awareness for clean beauty is increasing and more and more products are being advertised with the vegan label or free from silicone claims [11]. In addition, protection and repair properties are one of the five key claims for styling products. In this context, vegan silk represents a true innovative solution for the hair styling market, which on one hand has an excellent performance as a protective and repair agent and on the other hand is vegan, environmentally friendly and biodegradable and therefore meets the expectations of the clean beauty market. All in all, one can really call vegan silk a multifunctional hair care champion for the clean beauty market.

References

[1] https://www.futuremarketinsights.com/reports/hair-styling-products-market

[2] https://www.mintel.com/blog/beauty-market-news/what-to-expect-from-beauty-influencer-marketing-in-2020 .

[3] *Barel A. O., Paye M., Maibach H. I.*, Handbook of Cosmetic Science and Technology. Fourth Edition, CRC Press: Taylor & Francis Group, 2014, pp. 327.

[4] *Kleiner. A.*, Anti-Pollution trend for hair care. COSSMA, 2015, pp. 16-18

[5] *Marsh J. M., Gray J., Tosti A.*, Healthy Hair. Springer International Publishing Switzerland, 2015, pp. 63-65.

[6] *Baran R., Maibach H.*, Textbook of Cosmetic Dermatology. Informa Healthcare. London, 2010, pp. 389.

[7] *Baki G., Alexander K. S.*, Introduction to Cosmetic Formulation and Technology. Wiley, New Jersey, 2015, pp. 493-500.

[8] *Simon T., Römer L., Slotta U.*: The Reinvention of Silk – Fully Functional Silk Proteins for Innovative Cosmetic Products. SOFW Journal 9-2014, pp. 40-46.

[9] *Blosl N., R. Ott, Dr. Schacht K.*: Efficient Hair Protection – the Multi-functional Silk Polypeptide as New and Promising Strategy against Pollution. SOF Journal 7/8-2017, pp. 42-45.

[10] *Nicholson, S., Daniels, G., Grant-Ross, P. & Tamburic, S.*, 2016. An *ex vivo* comparison of the tensile strengthening properties of protein derivates on damaged hair. IFSCC, pp. 1-6.

[11] *Di Gesu, R.*, 2019. A year of innovation in hair styling products, 2019, MINTEL.

[12] https://cleanbeautybox.com/pages/definition-of-clean-beauty

contact

Joachim Müller
Natalie Blosl
Kristin Nessbach

Givaudan Deutschland GmbH (AMSilk office)
Am Klopferspitz 19 im IZB
82152 Planegg | Munich | Germany

Morgane de Tollenaere
Amandine Scandolera

Givaudan France SAS
Route de Bazancourt
51110 Pomacle | France

Corresponding author:
Joachim Müller
E-mail: joachim.mueller@givaudan.com

Picture Credits: aslysun/Shutterstock.com

Anti-odor Properties of Sodium Bicarbonate Assessed by Sensory Evaluation

S. Mouzon, J. Perez

Nowadays, more and more people are concerned about the safety and carcinogenicity of some components used as antiperspirants inside deodorant formulas, for instance aluminum salts [1]. The consumers are searching for a smooth alternative to this kind of ingredients that would not impede perspiration. SEQENS Mineral Specialties carried out a sensory analysis to compare the anti-odor efficacy of three different formulas of roll-on deodorants: one with sodium bicarbonate, one with anti-odor active and one with placebo. 24 subjects have participated in the study and a trained jury of five people has assessed the perspiration smell intensity, and the hedonic value of the smell 8 and 12 hours after the standardized application. Furthermore, the formulas were hidden (masked review). This study allows to prove scientifically that sodium bicarbonate has efficient anti-odor properties and can be an alternative to antiperspirants for the formulation of deodorants.

Introduction

Perspiration is a physical excretion that is normally odorless. But bacteria, which expand because of excretions, generate the strong odors people can smell. In fact, perspiration is made up of minerals, but also toxins that provide good conditions for bacteria to grow and produce odors. In recent decades, antiperspirants have dominated the deodorant market. But with rising concerns about the safety and carcinogenicity of some of them [1], the cosmetics industry has decided to enter the natural deodorant market, producing natural odor-fighting compounds.

Sodium bicarbonate (INCI name: Sodium bicarbonate), also known as baking soda, is a mineral compound which is used since Egyptians. It is registered under CAS number 144-55-8. The most common form of sodium bicarbonate is a fine white powder, produced by crystallization from synthetic or natural soda ash. The manufacturing process allows to produce high-quality sodium bicarbonate, aluminum and heavy-metal free.

Its many properties make sodium bicarbonate an exceptional and versatile compound. Over the years, it has become a key ingredient with hundreds of applications, from housework to personal care and healthcare. Sodium bicarbonate is not classified as hazardous according to CLP regulation [2], it does not contain any allergen and it is not listed as ingredient with restricted level of use according to cosmetic regulation [3]. Furthermore, sodium bicarbonate can be used for natural formulas as it has a natural origin index of 1 according to NF ISO 16128 standards and SEQENS Mineral Specialties product range is COSMOS approved.

Thus sodium bicarbonate could be an anti-odor alternative to other antiperspirants. It could prevent odor from acidifying, by regulating pH levels and neutralize these odors, without impeding perspiration.

To prove scientifically this property, SEQENS Mineral Specialties carried out a sensory analysis or sniff-test (monitored by DERMSCAN laboratory) to evaluate and compare the deodorant efficacy of three roll-on deodorants 8 and 12 hours after a single standardized application to the armpits [4].

Materials and Methods

Each deodorant formula contains various ingredients which have specific functionalities (non-exhaustive list for a liquid formula, for instance roll-on deodorant oil-in-water emulsion):

- Emollient: to moisturize the skin, to enhance the skin feel and to help the application of the formula, for instance PPG-15 stearyl ether
- Surfactant: to produce stable emulsions, for instance the family of Steareth components (polyoxyethylene fatty ethers derived from stearyl alcohols)
- Solvent: for instance water
- Thickener: to increase the viscosity of the formula and enhance its application, for instance Hydroxyethylcellulose
- Absorbent agent (optional): to create a soft feeling on the skin (by absorbing moisture from perspiration), for instance Tapioca starch
- Deodorant agent: to prevent the unpleasant smell from perspiration. There are two categories of components with two different mechanisms:
  - Anti-odor agent: it neutralizes the unpleasant odors, without impeding perspiration, for instance Farnesol (acrylic sesquiterpene) which has specific antimicrobial activity against bacteria responsible for body odor. However, this component must be mentioned on ingredients list of the formula if its concentration exceeds 0.001% for leave-on products like deodorant formulas to be compliant with cosmetic regulation [3] because it is present on the list of 26 allergens mentioned by European Directive 2003/15/CE.

· Antiperspirant agent: it neutralizes the unpleasant odors by impeding perspiration, for instance aluminum salts. However, some of these components must not exceed a specific concentration to be compliant with cosmetic regulation [3].

- Preservative: to guarantee the stability of the formula and to prevent from microbial growth over time, for instance DMDM Hydantoin.

The three formulas used for the sensory analysis are:

- Roll-on deodorant with sodium bicarbonate (at 20%) – formula 1
- Roll-on deodorant with anti-odor active (Farnesol, at 1%) – formula 2
- Roll-on deodorant without anti-odor active and without sodium bicarbonate (placebo) – formula 3

24 subjects have participated in the study. The kinetics of the study is visible on **Tab. 1**.

On D-7, the 24 subjects come to the laboratory without having applied any product to the armpits since the previous evening, are informed about the trial objectives, the procedures and the risks of the study and sign two copies of the Consent Form. The technician verifies inclusion and non-inclusion criteria, explains to the subjects the product application conditions and frequency and gives to the subjects:

- the neutral shower gel to be used at home in replacement of the usual hygienic product on the week before D0, this neutral shower gel allows to avoid that fragrances distort the results of the test;
- the daily file to write down their possible unpleasant sensations or medications.

On D0t0, the 24 subjects come to the laboratory one hour at maximum after taking a shower with the neutral shower gel and without having applied any perfume. They bring back their daily file and the neutral shower gel.

The technician determines the products to be used on the armpits according to the randomization list and wipe the two armpits with a single-use tissue. The technician applies 0.40g ± 0,05g of the formula 1, 2 or 3 on right or left armpit according to the randomized list and the formulas are hidden (masked review). Each formula is directly applied with a pipette on the armpit, then it is spread with light circular massage with the index. The single standardized application is exactly the same for the three formulas. The subjects leave the lab and commit to not take a shower or bath, go to the swimming pool until the end of the study.

On D0t8h and D0t12h, the subjects return to the laboratory, having followed the previous instructions and without having taken a shower and without wearing perfume.

A trained jury of five people assesses the perspiration smell intensity (**Tab. 2**), and the hedonic value of the smell (**Tab. 3**) 8 and 12 hours after the standardized application.

Perspiration smell intensity	
0	none
1	just perceptible smell (perception threshold)
2	very little intense
3	little intense
4	mildly intense
5	noticeable intense
6	rather intense
7	obviously intense
8	notably intense
9	extremely strong
10	excessively strong

Tab. 2 Structured scale for **perspiration smell intensity.**

Hedonic value of the smell	
1	extremely unpleasant
2	very unpleasant
3	unpleasant
4	slightly unpleasant
5	neither pleasant nor unpleasant
6	slightly pleasant
7	pleasant
8	very pleasant
9	extremely pleasant

Tab. 3 Structured scale for **hedonic value of the smell.**

	Measurement zone	D-7	From D-7 to D0t0	D0t0	D0t8h D0t12h
Information of the subject about study conditions and collection of his/her informed consent		•			
Verification of inclusion and non-inclusion criteria		•			
Distribution of the neutral shower gel		•			
Use of neutral shower gel at home, in normal conditions of use, during the study until D0t0	Body		•		•
Wiping of the two armpits and products application according to the randomization list	Armpits			•	
Sensory evaluation (sniff-test)					•

Tab. 1 Kinetics of the study.

The efficacy of the product is pointed up by comparing the scores obtained for each formula. If we take into account these two scales, the formula which will be the most efficient will have the lowest score for the perspiration smell intensity and the biggest score for the hedonic value of the smell. The sensory evaluation of deodorants is the only method allowing to estimate directly the efficacy of products on the perspiration smell. The training of an experimented jury allows a fast, precise and reproducible evaluation.

	Parameters	Kinetics	Mean ± SEM
Perspiration smell intensity	Formula 1	t8h	5.1 ± 0.4
		t12h	4.2 ± 0.5
	Formula 2	t8h	6.6 ± 0.4
		t12h	5.5 ± 0.6
	Formula 3	t8h	6.8 ± 0.5
		t12h	5.9 ± 0.5

Tab. 4 Scores for **perspiration smell intensity** 8 and 12 hours after application of the formula.

Results and interpretation

Syntheses of the results, given in mean (n=16) and standard deviation of the scores of the jury (n=5) obtained for each subject under both armpits, are presented in the **Tab. 4** and **5**.

- If the mean obtained for one formula is between [X.0; X.3], the interpretation corresponds to the value rounded off to X for the attribution of the term. For example, the perspiration smell intensity for formula 1 after 8 hours since the application is 5.1; the interpretation is 5 which means "noticeable intense".

	Parameters	Kinetics	Mean ± SEM
Hedonic value	Formula 1	t8h	3.1 ± 0.2
		t12h	3.8 ± 0.3
	Formula 2	t8h	2.3 ± 0.2
		t12h	3.1 ± 0.3
	Formula 3	t8h	2.3 ± 0.3
		t12h	2.8 ± 0.3

Tab. 5 Scores for **hedonic value of the smell** 8 and 12 hours after application of the formula.

- If the mean obtained for one formula is between [X.4; X.7], the interpretation corresponds to the values X and (X+1) for the attribution of the term. For example, the perspiration smell intensity for formula 2 after 12 hours since the application is 5.5; the interpretation is between 5 and 6 which means "noticeable intense to rather intense".
- If the mean obtained for one formula is between [X.8; (X+1).0], the interpretation corresponds to the value rounded up to (X+1) for the attribution of the term. For example, the hedonic value of the smell for formula 3 after 12 hours since the application is 2.8; the interpretation is 3 which means "unpleasant".

Mixed ANOVA model is used to compare the three formulas together for each kinetic point (t8h and t12h) and to check if the formulas are significantly different or not. Syntheses of the results are presented on the **Tab. 6** for perspiration smell intensity and on the **Tab. 7** for hedonic value of smell.
The data analysis shows a statistically significant difference in the perspiration smell intensity in favor of the armpit treated with the formula 1 compared to the armpits treated with the formulas 2 and 3, 8 and 12 hours after the single standardized application.
However, no significant difference is noticed between the formula 2 and formula 3.
The data analysis shows a statistically significant difference in the hedonic value of the smell in favor of the armpit treated with the formula 1 compared to the armpits treated with the formulas 2 and 3, 8 and 12 hours after the single standardized application.
However, no significant difference is noticed between the formula 2 and formula 3.

			Formula 2	Formula 3
t8h	Formula 1	mean* ± SEM	- 1.3 ± 0.3	- 1.3 ± 0.3
		p	<.0001	<.0001
		sign.	Yes	Yes
	Formula 2	mean* ± SEM		0 ± 0.3
		p		0.9932
		sign.		No
t12h	Formula 1	mean* ± SEM	- 1.3 ± 0.4	- 1.1 ± 0.4
		p	0.0018	0.0052
		sign.	Yes	Yes
	Formula 2	mean* ± SEM		0.2 ± 0.4
		p		0.6589
		sign.		No

* adjusted mean

Tab. 6 Scores for **perspiration smell intensity** 8 and 12 hours after application of the formula according to ANOVA model.

			Formula 2	Formula 3
t8h	Formula 1	mean* ± SEM	0.6 ± 0.1	0.5 ± 0.1
		p	<.0001	<.0001
		sign.	Yes	Yes
	Formula 2	mean* ± SEM		- 0.1 ± 0.1
		p		0.3598
		sign.		No
t12h	Formula 1	mean* ± SEM	0.6 ± 0.2	0.6 ± 0.2
		p	0.0023	0.0048
		sign.	Yes	Yes
	Formula 2	mean* ± SEM		- 0.1 ± 0.2
		p		0.7642
		sign.		No

* adjusted mean

Tab. 7 Scores for **hedonic value of the smell** 8 and 12 hours after application of the formula according to ANOVA model.

Conclusions

Under these study conditions, 8 and 12 hours after a single standardized application, the data collected highlight a significant deodorant efficacy with the formula 1 compared to the formulas 2 and 3. This efficacy is characterized by a statistically significant difference in the perspiration smell intensity and in the hedonic value of the smell in favor of the armpit treated with formula 1 compared to the armpits treated with formulas 2 and 3. Furthermore, no significant difference is shown between the formula 2 and formula 3.
This study proves that sodium bicarbonate can be used as anti-odor agent for deodorant formulas. When used in a roll-on deodorant, it can be an effective alternative to aluminium salts and other antiperspirants. Sodium bicarbonate can also be used for deodorant formulas with other galenic forms, for instance deodorant cream (example of formula on request).

Find information about our Sodium Bicarbonate product range and formulas on our website https://www.seqens.com/en/business-lines/specialties/mineral-specialties/bicarbonates/

References

[1] *Welzel, J.* (2020). *Grüdl, S., Banowski, B., Sättler, A., Förster, T.* and *Welss, T.* New Technologies for Specific Antiperspirant Actions. SOFW, 146 (03/20), pp. 46-51.

[2] Classification, Labelling, Packaging (CLP) regulation, 1272/2008

[3] Regulation (EC) n° 1223/2009 of the European Parliament and of the Council of 30 November 2009 on cosmetic products, Annex III

[4] Standard guide for sensory evaluation of axillary deodorancy – E1207-09. ASTM International – 2012

contact

Dr. Sophie Mouzon | sophie.mouzon@seqens.com
Jeanne Perez | jeanne.perez@seqens.com

Seqens Mineral Specialties
Usine de La Madeleine, rue Gilbert Bize
54410 Laneuveville-Devant-Nancy | France

Get a healthy, youthful, fresh-from-holiday look... all year round

SYN-GLOW™ is the unique pentapeptide that partners with skin to reveal its natural sun-kissed tone. It is clinically proven that SYN-GLOW™ promotes a **visible healthy glow in just 5 days**, with **long-lasting results** and a **natural personalized effect**.

Thanks to its unique mode of action, SYN-GLOW™ also strengthens skin defenses from within.

100% of consumers noticed their skin looked fresher and more revitalized.

For more information, scan the QR-Code to visit our website ***www.dsm.com/personalcare*** and search **SYN-GLOW™**

NUTRITION · HEALTH · SUSTAINABLE LIVING

Novel Pentapeptide Enhances Skin's Natural, Healthy Glow by Boosting MC1R

D. Imfeld, F. Pascucci

abstract

To address an identified consumer demand for skin care products that can create a natural-looking, lightly suntanned complexion all year round, DSM has been investigating safe means of stimulating skin pigmentation. Its novel pentapeptide Benzoyl Dipeptide-18 D-Phenylalanyl Arginyl D-Tryptophan Diproylamide Mesylate (trading under the name SYN-GLOW™) has been designed and synthesized to act as a melanocortin 1 receptor (MC1R) agonist. Its efficacy has been tested in *ex-vivo* and *in-vivo* studies which have demonstrated its potential to initiate and enhance skin pigmentation, with long-lasting results. The findings are presented here.

A lightly tanned complexion is desirable to many consumers

Consumer research from recent years suggests that when it comes to the facial complexion, beauty standards are evolving away from a deep tan to a more natural-looking "glow" [1]. This is true across all age groups and especially so among the under 55s, where more than 60% of consumers say that they like to have a "light tan to look good and healthy" [1]. Many people make a strong association between words such as "some tan, a good color, or not pale" and the idea of "glowing" skin, and this trend is starting to be reflected in the market. In 2018 for example, product launches for facial and neck care products that featured the word "glow" doubled in percentage in the US and Europe [2], and in 2019, the social media hashtag for "glowing skin" was used more than 3.5 million times [2].

To understand more about the motivations behind this trend DSM has been engaging with consumers via its dedicated Consumer Insights Hub and has also conducted a survey of more than 6000 respondents across the globe. This research has found that the "natural glow" that comes with a suntan, and the appearance of freshness and well-being people feel just after a holiday in the sun are indeed highly sought after. Consumers strongly associate this look with a healthy, revitalized and youthful appearance and report that it boosts their confidence, even to the point of feeling able to go make-up free.

Consumers also say that they would like to have this "sun-kissed" look all year round. However, although there are currently a range of self-tanning and make-up products on the market, many people are unhappy with the results. The main complaints given about such products concern superficial, uneven, and artificial-looking results that do not match an individual's specific skin tone. There is therefore a clear gap in the market for a product that safely gives consumers a bespoke, natural-looking, lightly tanned complexion all year round.

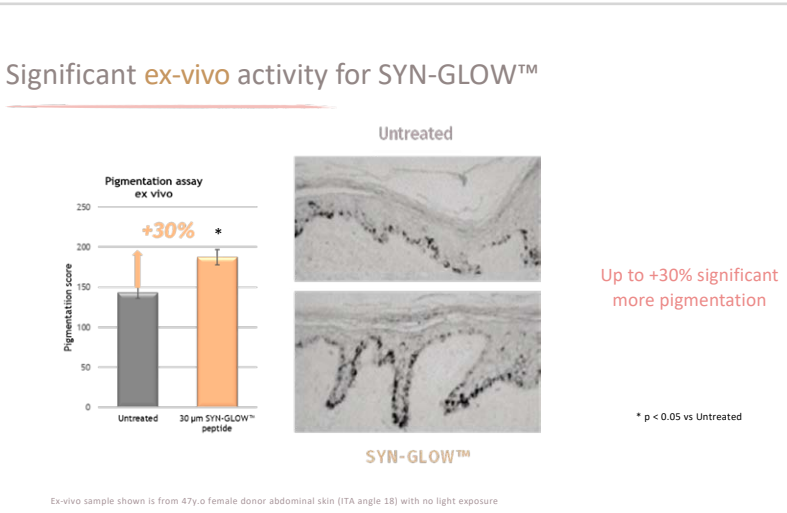


Fig. 1 a) Pigmentation assay *ex-vivo* on human skin. Pigmentation increased 30% in presence of 30 µM SYN-GLOW™ peptide. Vertical axis represents pigmentation score. b) example for histologic staining of melanin *ex-vivo* on human skin (untreated vs. pentapeptide treated).

Could activating MC1R with a synthetic peptide offer a safe, sustainable answer?

To address this need, DSM has turned to synthetic, small molecular weight peptides, tailor-made to deliver specific biological effects. With their known mode of action, these defined active compounds are already proven to be particularly effective at addressing common signs of skin ageing. They are also becoming a highly relevant solution at a time when many consumers require products that deliver visible results but are also consistent with broader environmental, health, and societal concerns such as sustainability and veganism.

DSM was one of the first companies to develop peptides for cosmetic applications and, drawing on more than 60 years' experience of peptides technology, it is still committed to investigating their potential to deliver new skin care benefits. To identify possible structural features that could be responsible for a molecule's biological activity, the company's scientists begin by modelling molecules virtually to identify the most promising lead compounds. Based on these *in silico* results, they then synthesize and perform a biological screening test to narrow the selection to those which are most effective. This technique, which is called rational design, facilitates understanding about how compounds or peptides perform at molecular level and how skin actives bind to their biological target within the skin.

Through this rational design approach, DSM has developed and tested a brand-new pentapeptide that works selectively with the skin's natural processes to provide consumers with the summer glow they desire – all year round; safely and naturally.

ex-vivo investigation and findings[1]

Melanocortin-1 receptor (MC1R) is localized in the cellular membrane. In this role, it acts as the unique gateway to control of the melanogenesis pathway which is responsible for the production of natural skin pigmentation. Activation of MC1R on epidermal melanocytes therefore represents a key step in initiating skin pigmentation and additionally, it is known to regulate cellular defense mechanisms, to cope with oxidative stress, and to enhance DNA repair mechanisms [3].

D-Phenylalanyl Arginyl D-Tryptophan Diproylamide Mesylate was identified, *in-vitro*, from a library of more than 100 peptides as the best candidate for activating MCR1 in order to stimulate skin pigmentation[1]. To investigate its melanin synthesis activity in a more complex system, it was tested *ex-vivo* on a human skin model. Human skin tissue was topically treated with the peptide for six days and under these conditions the pentapeptide was found to stimulate pigmentation by 30% ($p < 0.05$ vs vehicle) [4] compared to vehicle treated control tissue. (**Fig. 1**)

In view of these findings, DSM's scientists also investigated whether the peptide was able to induce three key melanogenesis markers *ex-vivo*: melanocyte-inducing transcription factor (MITF), tyrosinase (TYR) and tyrosinase-related protein-1 (TYRP-1). By immunohistochemistry, using specific antibodies against each of the three proteins mentioned above, it was shown that the pentapeptide was able to significantly induce protein expression of all three markers [4], thus confirming activation of the MC1R-dependent natural melanin synthesis pathway[1].

In addition to the peptide's pro-pigmentation effects, a further *ex-vivo* study found that it was able to induce activation of Nrf2 upon UVA irradiation [4]. Nrf2 is a transcription factor that stimulates intrinsic antioxidant reactions in the cell and therefore helps to protect skin against UV-induced oxidative stress. (**Fig. 2**)

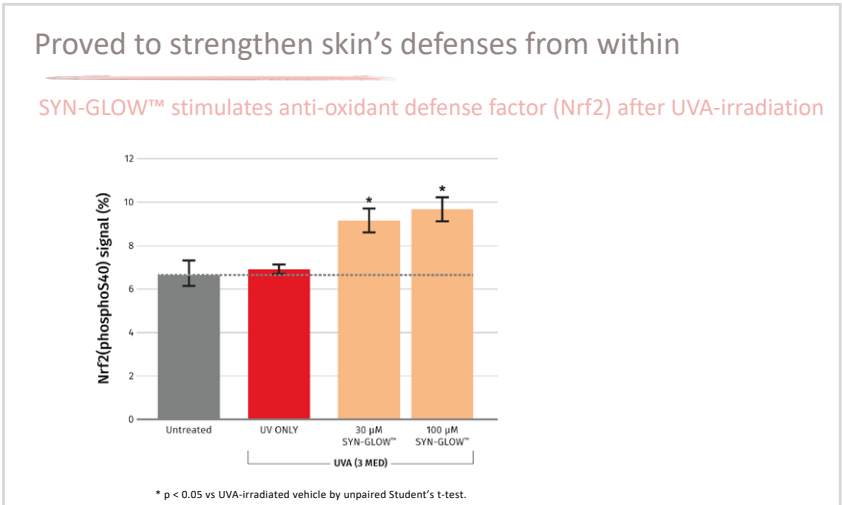


Fig. 2 a) SYN-GLOW™ peptide activates Nrf2 by serine 40 phosphorylation of after UVA-irradiation *ex-vivo*. *p<0.05 vs UVA-irradiated vehicle by unpaired Student's t-test.

[1] NB – Scientific evidence based on *ex-vivo* investigation, not intended to be a final product claim.

in-vivo study

Study design

Having confirmed the pentapeptide's potential for stimulating skin pigmentation *ex-vivo*, scientists conducted an *in-vivo* study. This involved 29 volunteers and was carried out over a period of 29 days which included a 7-day pre-conditioning phase, a 15-day application phase and a 7-day post application phase. All volunteers applied a product containing 3% commercial formulation of D-Phenylalanyl Arginyl D-Tryptophan Diproylamide Mesylate, to one section of their inner forearms and a product containing 5% of a commercially available competing peptide to another section.

During the pre-conditioning and application phases, subjects applied both products twice daily. Then, on days 1 to 4, predefined spots of their inner forearms were exposed to UV light in the form of a sunlight simulator (type Single Port XPS400 from Solar Light Co.), 40% below the minimal erythemal dose (MED). This level was used to simulate normal life conditions as it corresponds to approximately 15 to 30 minutes of outdoor exposure on an average spring day in a mid-European country. To maintain highly standardized conditions, subjects kept their arms covered with long sleeve dress at all other times during the study.

To quantify change in skin color, ITA° measurements were taken on the first day of the pre-conditioning phase and on days 0, 5, 10, 15 and 22. Photographs of treated skin were taken on day 15. For control purposes, measurements were also taken for a section of each subject's forearm which had been exposed to UV light on days 1 to 4 but had not been treated with any product (= control section). (**Tab. 1**)

In-vivo study

- 29 Volunteers
- application at inner forearm of 3% SYN-GLOW™ and 5% of competitive Peptide*

	Product application phase (2 times per day)																	Post application phase						
Day		0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22
ITA Measure	Pre-conditioning phase	ITA	«Real life» simulation**				ITA					ITA					ITA & Photo							ITA

*Benchmark = Hexapeptide-1
**We exposed skin to some UV light, in order to simulate "real life". UV exposure was done with a sunlight simulator, 40% below the minimal erythemal dose (MED): this roughly corresponds to 15-30min outdoor, during avg Spring day in mid-European Country

Tab. 1 *In-vivo* study.

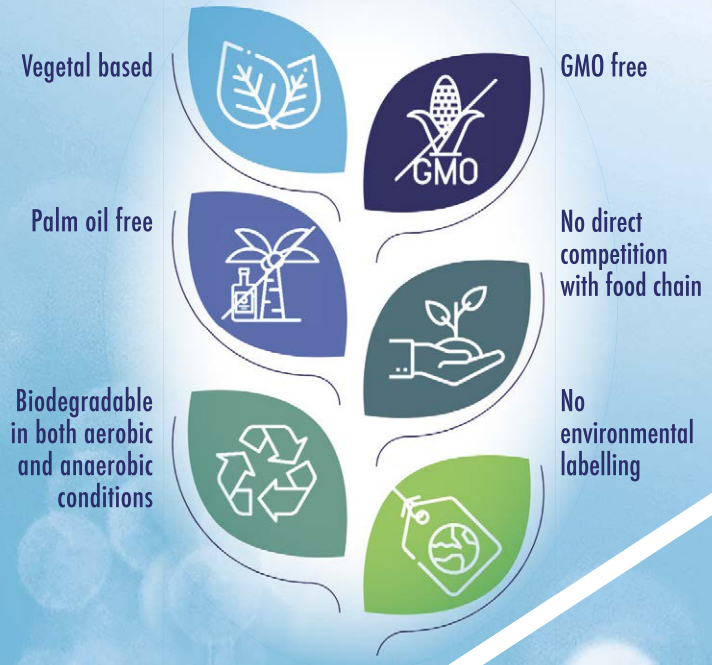
Study findings confirm enhanced and long-lasting pigmentation *in-vivo*

As the graph in **Fig. 3** illustrates, on sections of the forearm treated with 3% D-Phenylalanyl Arginyl D-Tryptophan Diproylamide Mesylate, the visible pigmentation effects, measured and expressed as a negative delta ITA°, were significantly higher after 5 days than they were in either the control section of the forearm or the section treated with a

Fig. 3 Graph representing the visible pigmentation effects of DSM's novel pentapeptide compared to a competing peptide and an untreated, control section. The control group was only gently irradiated with UV, as described in the text. Vertical axis = negative delta ITA° of visible effects. More negative delta ITA values represent more intense and darker skin color. Statistics: *p<0.05, **p<0.01 against control group

INNOVATIVE BIO-BASED SURFACTANTS FOR CLEANING PRODUCT APPLICATIONS

Unique sustainable profile



SENSIO®

A real sense to cleaning

Approved by ECOCERT RAW MATERIAL

DETERGENTS

Can be used in EU ECOLABEL detergents

Contact us: hpc@arkema.com

arkema.com

ARKEMA

INNOVATIVE CHEMISTRY

Fig. 4 Comparative images of the test area in five volunteers at day 15. Skin sections treated with one of the products are shown and the darker spots in the middle of the sections were both treated with one of the products and UV-irradiated.

5% competing peptide. These effects were also sustained at a higher level and for a considerably longer period both post exposure to UV (days 5-15) and in the post application phase (days 15-22).

More pronounced and visible effects of the pentapeptide compared to a benchmark peptide can be seen in **Fig. 4** which contrasts the individual pigmentation effect photographed at day 15 in five volunteers.

Additionally, to illustrate the pentapetide's potential pigmentation effect facially, a virtual reality method was used to digitally create an artificial face, based on the mathematical average of facial images taken from nine volunteers. The delta ITA values obtained on forearms were then transferred to the artificial face using the results for two subjects at days -4, 10 and 22. This is shown in **Fig. 5**.

Consumer perceptions from facial application of pentapeptide

Another *in-vivo* study involving direct facial product application and a qualitative questionnaire was also conducted. This study was based on 16 female volunteers aged 20-55 (skin phototypes II-IV) carrying out planned outdoor activities and who applied a formulation containing Benzoyl Dipeptide-18 D-Phenylalanyl Arginyl D-Tryptophan Diproylamide Mesylate to the face twice daily for 10 days. The feedback from this study proved to be particularly positive with **100% of respondents reporting that they had noticed their skin looked "fresher" and "revitalized", 90% that their skin looked less "dull" and "tired", 80% reporting a "healthy facial glow" and 70% stating that they had a more even skin tone**.

In Summary

With facial beauty trends now favoring a lightly tanned appearance, there is a gap in the market for skin care products that can give consumers a natural-looking, glowing complexion that compliments individual skin tones.

DSM has used its 60 years of expertise in peptides technology to identify and test a novel, synthetic pentapeptide, with a unique amino acid sequence. Benzoyl Dipeptide-18 D-Phenylalanyl Arginyl D-Tryptophan Diproylamide Mesylate (trading under the name SYN-Glow™) activates the Melanocortin-1 receptor (MC1R) which is responsible for enhancing skin complexion. (NB – scientific evidence not intended to be a final product claim).

In *ex-vivo* tests, the peptide was found to enhance skin pigmentation. Additionally, it may offer a defense against solar induced oxidative stress through activation of the cellular antioxidant system (via Nrf2). (NB – scientific evidence not in-

Fig. 5 Augmented reality representation of a typical face representing skin color at day 0 (baseline before UV irradiation, and then, in split-face view (left side UV only and right side UV plus SYN-GLOW™ treated, at day 10 of the study and day 22 (7 days after last product application). Two individual representative datasets from subject 3 (upper faces) and subject 6 (lower faces) are shown

Supported claims

- Natural healthy glow in just 5 days
- Prolonged summer bronze
- Personalized result, bespoke complexion
- Strengthened skin natural defenses from within

tended to be a final product claim).

In-vivo studies show that the pentapeptide provides a natural, healthy glow, visible in just five days (+90% vs control, high significance) and that these results are long-lasting (up to two and a half weeks proven visibility, high significance better than control).

The results measured were backed up with very positive, subjective feedback from volunteers.

Synthetic peptides are a clean and sustainable answer to growing demand for high-efficacy solutions that are backed by scientific evidence. They also meet requirements for consumers looking for vegan and environmentally friendly cosmetic alternatives. SYN-GLOW™, the peptide featured in these studies, has been assessed internally according to four pillars of sustainability and is 98% biodegradable and has a low carbon footprint.

Formulations including SYN-GLOW™ (at a recommended dosage of 1-3%) could therefore help meet the needs of consumers looking for sustainable products that work with their own skin to deliver a natural looking glow or suntanned effect all year round.

Consumers report that having a sun-kissed, "back from holidays", glow on the outside helps them feel happier and more confident on the inside. In the current, uncertain climate, when so many people have put their travel plans on hold, DSM'S range of ready-to-use formulations containing Benzoyl Dipeptide-18 D-Phenylalanyl Arginyl D-Tryptophan Diproylamide Mesylate could help people achieve that back-from-holidays look while staying at home.

References

[1] DSM Sun Protection and Skin Cancer Survey, 2017/2018. (Global Data excluding Asian Countries)

[2] Mintel GNPD Data

[3] *Kadakaro Chen et al,* Alpha Melanocyte Stimulating hormone suppresses oxidative stress through a p53 mediated signalling pathway in human melanocytes, Molecluar Cancer Research, 10 June 2012

[4] *Jackson E et al,* Discovery of a Highly Selective MC1R Agonists Pentapeptide to Be Used as a Skin Pigmentation Enhancer and with Potential Anti-Aging Properties, International Journal of Moleclu-ar Sciences 2019, 20, 6143

contact

Dr. Dominik Imfeld | Senior Lead Scientist
Francesca Pascucci | Global Marketing Manager

DSM Nutritional Products Ltd
P.O. Box 2676 | 4002 Basel | Switzerland

Essential Aspects of Filling Stations for Cosmetic Products in the Retail Trade

B. Hirschmann, B. Huber, M. Ibel, E. Kratz, B. Pelzmann, C. Marx

abstract

In the wake of a discussion about a possible saving of packaging materials, systems for the filling of cosmetic products in the retail trade are partly available. Of course all conditions imposed by the EC Cosmetics Regulation as well as the supplementary national regulations in Germany and Austria apply also to the products sold in this form. For these filling stations it is, however, necessary to attach special attention to compliance with the Cosmetics GMP conditions. In these cases the filling, which is subject to high hygienic requirements in industrial production, is outsourced to the retail trade. This is why comprehensive, robust hygiene concepts are necessary in particular for these systems.

The authors have put together some essential aspects which need to be complied with when using such filling stations. In this connection it was taken into account that the organisation of the filling process in the retail trade can be designed quite differently in detail. It is necessary to consider both filling stations which are offered to customers for self-service as well as stations which are operated by retailer staff.

What needs to be taken into account from the point of view of the manufacturer, who acts as responsible person:

Determination of the procedure for the filling. Here, in particular the following scenarios are possible:

1. The customer receives in the store a receptacle intended for (re-)filling and brings the latter along with him for the next filling. He cleans the receptacle before the refilling.
2. The customer brings along another receptacle to fill the product into the latter.

Particularities of the safety assessment:

- Taking into account of the respective scenario – use, (multiple) refilling, (multiple) re-use –, depending on the type of product (rinse-off or leave-on); more particularly, with a view to:
  - · The safety of the filling process
  - · Possible product residues in the container
  - · As well as the risk of microbial contamination.
- A specific hygiene concept should be prepared for the respective filling system. In this connection the product withdrawal by the consumer, the cleaning of the filling nozzle and a possibly insufficient preservation are seen as particularly critical points.
- Additional challenge tests may be necessary for the simulation of the entire refilling cycle (more particularly, for scenario 1). The calling in of a microbiologist with experience in the field of cosmetics makes sense.
- If necessary, it should be checked whether the addition of water and/or detergents (residues of the cleaning process) influences the integrity and safety of the product, which is to be filled into the receptacle.
- Definition of a microbial quality management to comply with the Cosmetics GMP at the refilling.
- When mentioning the period after opening or a date of minimum durability it must be ensured that the information applies during the entire duration of the filling from the large container for each refill receptacle. If necessary, this duration must be restricted accordingly and the date of opening of the large container must be documented.
- If necessary, a number of the maximum possible safe refills of the original receptacle should be defined. Organisational measures for the establishment that this level has been reached, should then be fixed. (scenario 1)
- In the event of receptacles brought along by customers (scenario 2), this should be taken into account at the safety assessment. If necessary, the eligible receptacles must be defined ("only receptacle with certain properties/suitable materials, eg made of glass") and, if applicable, the use of receptacles which could be confused with foodstuff packaging should be excluded taking into account Art. 3 a) EC Cosmetics Regulation in conjunction with Directive 87/357/EEC.

Securing of training of the staff of the retailer, more particularly concerning the proper implementation of the hygiene concept taking into account Cosmetics GMP (e.g. cleaning of hoses, replacement of the large container, if necessary, visual inspection of the receptacles of the customer to be filled, etc.) and, if necessary, concerning the perishability/limited durability of the products offered.

Information for the customer "on site"

- In the event of self-filling by the customer, the latter should receive clear instructions about the refilling procedure and

the cleaning and/or drying of the refill receptacle. This can be done eg through a public notice at the filling station and, if necessary, be illustrated by easily understandable pictograms. General information of the customer about a possible microbiological contamination of the products at the filling in soiled and/or insufficiently cleaned and/or not dried receptacles might also make sense.

- The labelling of cosmetic products which are packed in the retail trade at the request of the buyer, is governed by the national provisions issued by the respective member state in accordance with Art. 19.4 EC Cosmetics Regulation.
  - In Germany, § 5 of the German Cosmetics Ordinance "for the labelling of not prepacked cosmetic products" must be complied with, according to which mandatory information within the meaning of Article 19.1 EC Cosmetics Regulation must be "be mentioned on an enclosed or attached label, tape, tag or card".
  - In Austria § 3 of the Cosmetics Implementation Ordinance "Labelling of unpacked cosmetic products" must be complied with, according to which labelling must be carried out in accordance with Art. 19 of the EC Cosmetics Regulation and must be attached on the goods by means of tag, label or in a similar form.
  - According to these provisions the customer must be provided for each refill product with all mandatory information under cosmetics law at least in the form of an "enclosed" paper; this should be made available by the responsible person for the respective large container.
  - Particularly for scenario 1, it must be ensured that the INCI list, the batch identification and the shelf life, which can deviate from the labelling on an original receptacle, are correct.
- In Germany it is recommended to use as a large container a receptacle with a **volume not exceeding 20 litres** taking into account the special provisions on "drum and container warehouses" according to § 31 AwSV (Ordinance on facilities for handling substances that are hazardous to water).
- Clear **contractual agreements of the responsible person with the retailer**, where the cosmetic product is to be filled, are urgently recommended to avoid liability risks.

What needs to be taken into account from the point of view of a retailer?

For refill systems of a manufacturer who acts as responsible person

- Strict compliance with all the conditions imposed by the responsible person. In the event of deviations an own liability of the retailer is possible.
- Implementation and observance of the hygiene concept of the responsible person, more particularly, compliance with the hygiene criteria for staff and for filling of the products (Cosmetics GMP) incl. documentation.
- Training of the employees concerned.
- In the event of a corresponding condition imposed, possibly inspection of the receptacles brought along for identifiable contaminations and/or suitability of the receptacles for the filling. If the receptacles are not clean or appear to be unsuitable for refilling, the consumer must be informed accordingly and the filling must possibly be refused. In hygienically objectionable receptacles the stated shelf life of the product cannot be guaranteed.
- If necessary, verification whether the receptacles brought along cannot be confused with foodstuff packaging.
- For each filled product handing over (at least as an "enclosure") of the information made available by the responsible person for the respective product (in Germany in accordance with § 5 of the Cosmetics Ordinance, in Austria in accordance with § 3 of the Cosmetics Implementation Ordinance), taking into account, more particularly, the correct batch identification.
- If necessary, documentation of the date on which the large container was opened.
- Compliance with the specifications of the calibration regulations concerning the labelling of the filling quantity.

For own refill systems (without the support of the supplier of a large container)

- The retailer is the responsible person for the cosmetic products refilled by him, since the filling of a product, which is already on the market, into smaller receptacles represents a modification in such a way that compliance with the applicable requirements may be affected (Art. 4.6 EC Cosmetics Regulation).
- In this case the retailer has all the obligations of a responsible person (see above and under https://www.ikw.org/fileadmin/ikw/downloads/Schoenheitspflege/Leitfaden_Verantwortlichkeiten_02_2013.pdf)

Literature

EC Cosmetics Regulation 1223/2009 https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=CELEX:02009R1223-20200501

DIN EN ISO 22716: https://www.ikw.org/schoenheitspflege/themen/detail/gute-herstellungspraxis-kosmetik-gmp-heute-eine-selbstverstaendlichkeit-280/
· Leitlinien für Kosmetik-GMP (Guidelines for Cosmetics GMP)
· Checkliste für Kosmetik-GMP (Check list for Cosmetics GMP)

Germany: Kosmetik-Verordnung https://www.gesetze-im-internet.de/kosmetikv_2014/KosmetikV_2014.pdf

Austria: Lebensmittelsicherheits- und Verbraucherschutzgesetz (LMSVG) BGBl. I Nr. 13/2006 idgF

Kosmetik-Durchführungsverordnung (BGBl. II Nr. 330/2013 idgF))

authors

Brigitta Hirschmann | CVUA Rheinland (Chemical and Veterinary Investigation Office Rheinland)
Winterstraße 19, 50354 Hürth

Birgit Huber | IKW (German Cosmetic, Toiletry, Perfumery and Detergent Association)
Mainzer Landstraße 55, 60329 Frankfurt am Main

Matthias Ibel | IKW (German Cosmetic, Toiletry, Perfumery and Detergent Association)
Mainzer Landstraße 55, 60329 Frankfurt am Main

Evamaria Kratz | CVUA Karlsruhe (Chemical and Veterinary Investigation Office Karlsruhe)
Weißenburger Straße 3, 76187 Karlsruhe

Birgit Pelzmann | AGES Wien (Austrian Agency for Health and Food Safety)
Spargelfeldstraße 191, 1220 Wien, Austria

Claudia Marx | LAVES Niedersachsen (Agency for Consumer Protection and Food Safety of Lower Saxony)
Am alten Eisenwerk 2 A, 21339 Lüneburg

A New Microbiota-respectful Deodorant which Allows Axilla Perspiration

Ò. Expósito, A. Guirado, D. Robustillo, A. Gallego, M. Mas, P. Riera, S. Ruiz, D. Luna, S. Laplana, T. Ruiz, K. Lingen

abstract

The body odour modulation has historically been a concern for the human being and studying the microbiota involved represents an approach which this topic may naturally be faced from. The new active Deobiome Noni (INCI: Morinda Citrifolia Callus Culture Lysate) is an active ingredient made from *Morinda citrifolia* plant stem cells that introduces the concept of biological deodorant. By combining an innovative prebiotic technology and the latest advances in plant stem cell technology in the skin microbiota treatment, it has been demonstrated how to modulate the body malodour while being respectful with the skin microbiota and its ecosystem. The mechanism of action of the active has been evaluated by performing several *in vitro* and *in vivo* tests to demonstrate the effective body odour reduction while allowing the natural axillary perspiration.

The challenge of fighting body odour

The flourishing clean movement is among the new trends of consumers who search and choose products based on natural and sustainable ingredients. This movement is also visible in the cosmetic sector.

In the deodorant market, there is a need for alternatives to the conventional products. New products that could be respectful with the skin and its important physiological functions such as the sweat production, as well as with the skin microbiota.

The human being has been creating habits of personal hygiene that sweep the skin ecosystem and obstruct the natural function of the armpit.

The daily battle against body odour is still dominated by two classical strategies: deodorants and antiperspirants. The so-called deodorants often focus on eliminating bacteria by applying alcohol bases, bactericidal actives, or cover malodour by using perfume. On the other hand, traditional antiperspirants' strategies are based on clogging the pores with derivates of aluminium salts (Aluminium Chlorohydrate), thereby depriving the bacteria in the axilla of malodour precursors and water. In addition, antiperspirants are often antimicrobials which can cause armpit irritation and create a feeling of dryness in the skin.

Vytrus Biotech has broadened the scope of axillary care by designing a biological deodorant ingredient that effectively eliminates the bad odour. The strategy is an innovative combination that addresses the care of the microbiota through two key channels: using a new prebiotic technology, and the microbiota re-balancing through the interaction with bacterial communication (Quorum Quenching).

Deobiome Noni: The First Biological Deodorant

The new active ingredient discussed in the article (INCI NAME: Morinda Citrifolia Callus Culture Lysate) combines an innovative use of prebiotics and the latest in plant stem cell technology in the field of microbiota: Quorum Quenching (**Fig. 1**).

The active ingredient follows two strategies which act synergistically against the formation of malodorous molecules derived from the armpit commensal microbiota:

Biological strategy

Morinda citrifolia, known as Noni plant, is a native plant from Southeast Asia (Indonesia) and Australia. It is used worldwide for its properties: antitumor, anthelminthic, analgesic, anti-inflammatory, immuno-stimulant, several skin diseases, urinary tract disorders, fever, diabetes, etc.

The concentrated metabolome of the plant stem cells from *Morinda citrifolia* is rich in specific fractions of terpenes. This

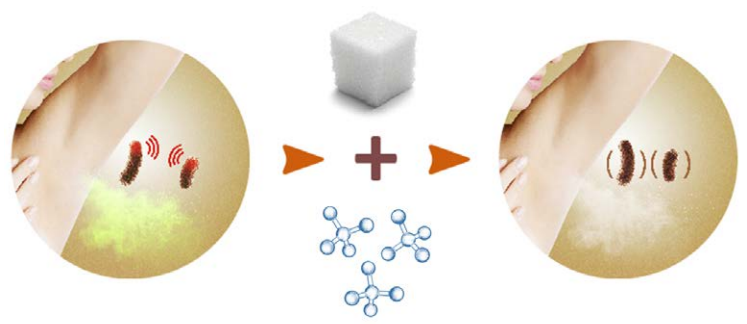


Fig. 1 Mechanism of action of Deobiome Noni.

molecular cocktail is designed to efficiently interfere with different bacterial species and prevent the exponential growth of certain species that would cause an imbalance in the bacterial ecosystem. The active ingredient is rich in anti-quormones (anti-Quorum Sensing molecules) and has been shown to be able to prevent the formation of bacterial communication or coordination factors (Niche Factors) and unwanted bacterial strategies involved in generating the bad smell. This ingredient represents a new activity profile for this species claiming the Plasma Rich in Cell Factors as a new combination of bioactives (metabolome) from the plant Morinda citrifolia.

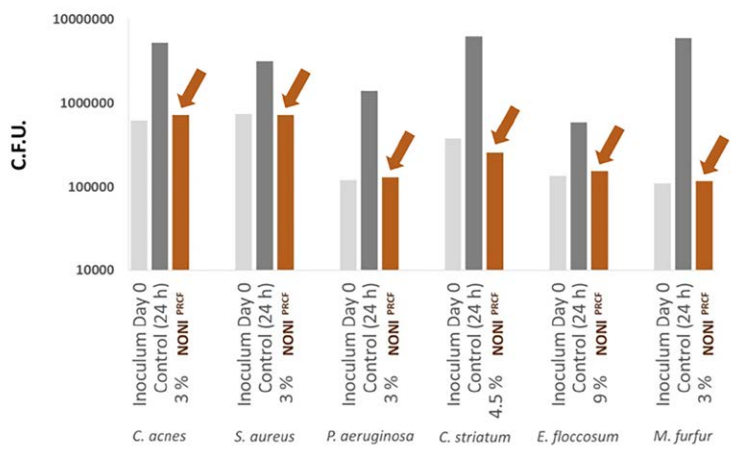


Fig. 2 Analysis of the broad spectrum bacteriostatic and fungistatic effect.

Prebiotic strategy

A specific and innovative prebiotic cocktail based on sugars that shifts the metabolism of commensal microbiota from lipids to polysaccharides. Microorganisms detect these polysaccharides as an easier alternative to metabolize the nutritional supply and change their metabolism from lipids to sugars resulting in less odorous by-products.

In vitro efficacy

The first strategy of the active (biological activity) is based on its anti-Quorum Sensing (Quorum Quenching) characteristics. A series of *in vitro* tests were carried out to evaluate the properties of the active that demonstrate the interaction with the microbiota responsible for body odour.

In vitro 1: Analysis of the broad spectrum bacteriostatic and fungistatic effect

Firstly, the broad spectrum bacteriostatic and fungistatic effect was demonstrated by quantifying the microbial population in the absence and presence of the active ingredient. It was found that there was significant variation between the untreated cultures and the cultures treated with the active after 24 hours in various species: *C. acnes, C. aureus, P. aeruginosa, C. striatum, E. floccosum and M. furfur.* The active showed a bacteriostatic effect stopping the growth of the indicated species and maintaining the initial levels but without bactericidal effect (**Fig. 2**).

In vitro 2: Anti-Quorum Sensing effect

Secondly, the anti-biofilm effect (the formation of biofilm is one of the first consequences of Quorum Sensing activation) in *Corynebacterium striatum*, *Staphylococcus aureus, Cutibacterium acnes* and *Malassezia furfur* was evaluated by fluorescence staining and Laser Scanning Confocal Microscopy. The active was shown to inhibit the biofilm formation up to 99% while keeping bacterial cells in suspension live (**Fig. 3**).

In vivo Efficacy

In order to demonstrate the efficacy of the active, several *in vivo* tests were performed.

In vivo 1: Evaluation of armpit odour modulation

A first group of 19 volunteers between 20 and 56 years old, applied a placebo cream to one of their armpits and a cream containing 1% of the active to the other armpit to evaluate the armpit odour modulation.

Armpit molecular analysis

First, more than 100 molecules responsible for body odour were analysed by gas chromatography, being demonstrated

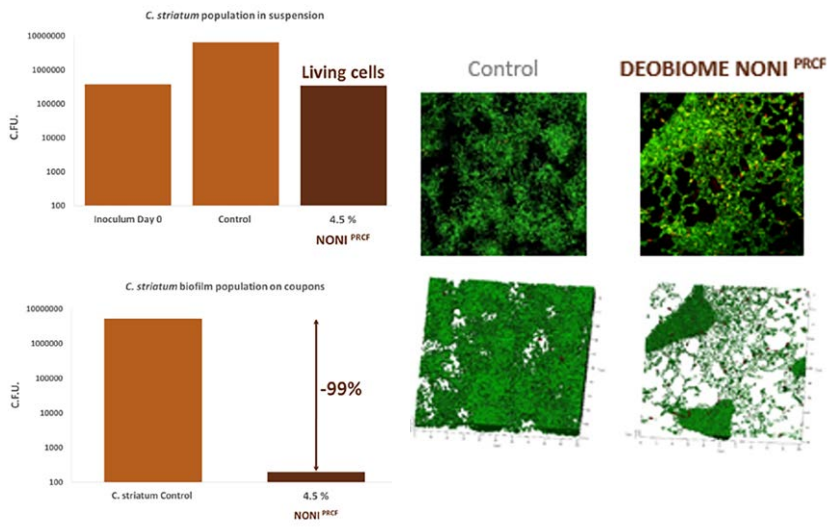


Fig. 3 Analysis of anti-biofilm effect on *Corynebacterium striatum*.

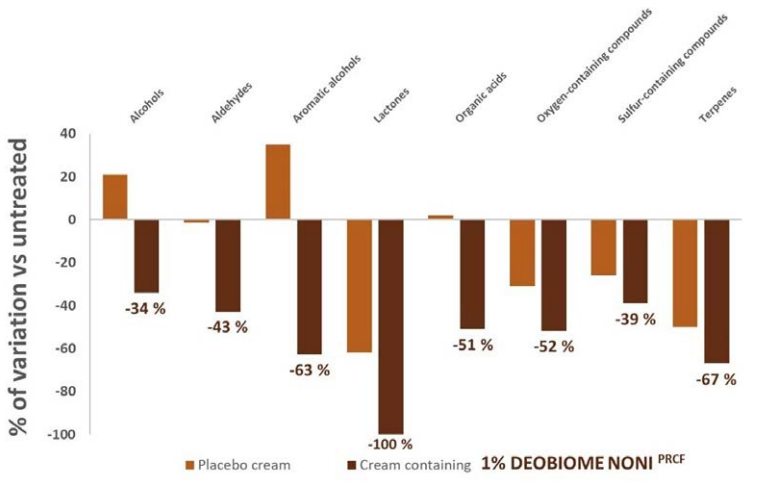


Fig. 4 Clinical evaluation of odorous volatile molecules reduction.

the reduction of these odorous molecules by 39%. The following graph clearly shows a decrease in these compounds after the application of the active vs placebo (**Fig. 4**).

Armpit odour intensity

On the other hand, the average intensity of underarm odour was studied in the 19 volunteers after having applied the active. The study showed a 30% reduction versus initial treatment and down to 82% (**Fig. 5**).

In vivo 2: Evaluation of axillary perspiration rate

In a second *in vivo* assay, the rate of axillary perspiration was evaluated in a panel of 8 volunteers, women and men, between 28 and 62 years old, who applied a placebo cream and another cream with a dose of 1% of the active ingredient.

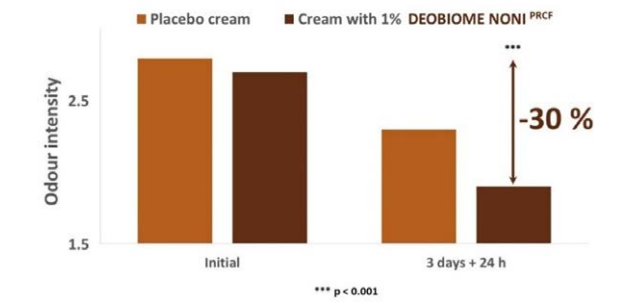


Fig. 5 Clinical evaluation of odour intensity reduction.

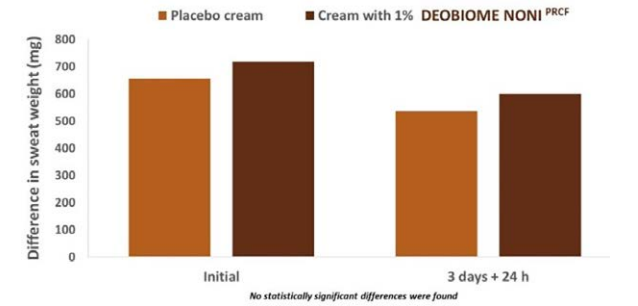


Fig. 6 Clinical evaluation of axillary perspiration rate.

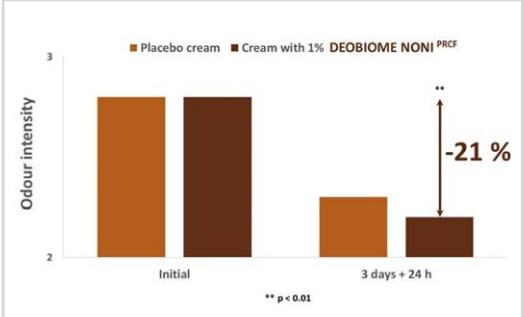


Fig. 7 Clinical evaluation of feet odour reduction.

The active was shown not to significantly alter natural armpit perspiration (**Fig. 6**).

In vivo 3: Evaluation of feet odour intensity modulation

And finally, in a third *in vivo* test made up of a group of 10 volunteers (23-57 years old), the average intensity of feet odour was evaluated. It was shown that with a cream that contained 1% of the active, a 21% average reduction of feet odour intensity versus initial measurement and down to 47% was possible (**Fig. 7**).

Conclusion

The active ingredient Deobiome Noni is an innovative deodorant treatment that respects the skin and its microbiota.
The treatment allows the armpit to breathe while avoiding the generation of bad odour, allowing the skin to breathe, and respecting the biological ecosystem.
Regarding the different applications in the cosmetic sector, the active ingredient has a wide range of applications in the field of reducing body, axillary, foot, and scalp odour, and rebalancing the microbiota.
Made from stem cells of the plant *Morinda citrifolia,* Deobiome Noni represents a scientific advance for the treatment of body odour, respecting the skin microbiota, thanks to the innovative mechanism of action of the active ingredient, being the first biological deodorant in the market.

contact

Ò. Expósito, A. Guirado, D. Robustillo, A. Gallego, M. Mas, P. Riera, S. Ruiz, D. Luna, S. Laplana, T. Ruiz

Vytrus Biotech S.L.
C/ Sant Gaietà 121, 2nd floor | 08221 Terrassa | Barcelona | Spain
E-mail: info@vytrus.com | www.vytrus.com

K. Lingen

DACH: novoclon GmbH
Nörderlinger Str. 25 | 40597 Dusseldorf | Germany
E-mail: vytrus@novoclon.com | www.novoclon.com

Effect Pigments with Customized Performance for Cosmetics

K. Mogare, T. Navale, F. Mazzella, M. Lergenmüller

abstract

In Effect pigments based on natural mica as well as on synthetically prepared substrates such as Synthetic mica, or Silica, Alumina or Borosilicates (Glass flakes), the surface area/particle size plays a crucial role in tailoring effect pigments with desired properties in cosmetic applications. With two pigment series, Sumicos™ HG ("high-gloss") Line and Prestige® Glow and Glam Line, we have recently build-up a technology to create new effect pigments that provide better chroma, color purity and sparkle in comparison to the conventional effect pigments. The article describes the science behind creating these effect pigments with improved reflectance.

Introduction

Pigment being a key ingredient that imparts color to any cosmetic product, cosmetics and color share a close-relationship. Color is result of how the light rays having different wavelengths interact with objects. These are further sensed by the receptors in the eye and the stimulation caused by each wavelength expresses the color. Effect pigments (special class of pigments) have distinct properties (Shimmer, Lustre, and Sparkle) that provide unique effects in end applications [1]. These substrate based pigments allow producing intriguing effects by modifying the refractive indexes of the coating layers on the substrate by using different metal oxides. In general, the intense color effect pigments that are available in the market are mostly based on synthetically prepared substrates such as Synthetic mica, Alumina, Silica or Borosilicates (Glass flakes) [2]. These substrates provide certain unique advantages such as smoother surface of the substrate. The uniform particle shape then allows for enhanced constructive interference whilst leading to increased chroma and lustre. The reduced thickness of these synthetically prepared substrates also allows depositing higher amounts of the metal oxides without losing the spectral interference. This helps in developing color travel pigments with increased flip-flop colors (angle dependent colors) [3]. However, these synthetically derived substrates are rather expensive. Based on our approach of creating intense colors on natural mica itself, we have developed a cost-effective technology that is dependent on surface area and particle size distribution. This has allowed us to generate new intense color effect pigments providing increased chroma, color purity and sparkle for cosmetic formulations.

Results & Discussion

Substrate and Particle size distribution plays a key role to develop any effect pigment. It is well known in effect pigments that a particle size distribution towards larger particles (e.g. 15-150µm) is resulting in better lustre and less coverage, whereas; in case of a lower particle size distribution (e.g. 5-25µm), the coverage is improved, whilst providing less lustre. Building further on this fact, we tweaked the particle size distribution within the same particle size range in order to enhance the color intensity and sparkle of effect pigments.

We selected the intermediate size range (10-100µm) to have a balance on coverage as well as sparkle at the same time. A few of the recent examples from this technology are as discussed below.

Fig. 1 provides the actual reflectance data for silver pearl pigment synthesized using different method and substrate. It can be observed that the reflectance of natural mica based Silver (Conventional Silver) is around 15% more than glass flake-based Silver. The intense Silver that we have created using a new technology has almost 25% more reflectance than the glass flake Silver. It is important to note that the particle size distribution of these 3 Silver pearls is almost in a similar range. The brightness and whiteness of Intense Silver is also superior and distinct than the conventional and glass flake based Silver (**Fig. 2**). The advantage of this new intense silver is that it provides exceptional sparkle and magnified

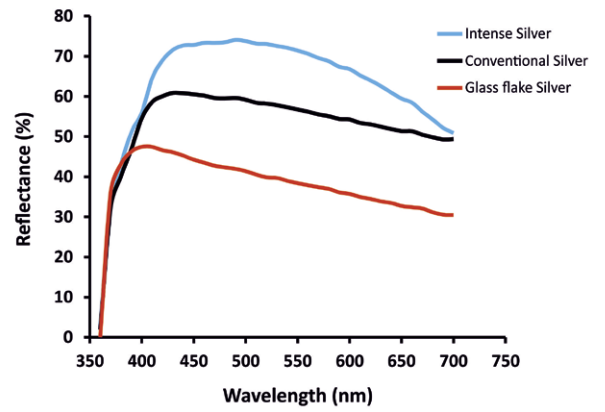


Fig. 1 Reflectance versus wavelength of Silver pearls; conventional natural mica-based Silver, Glass flake-based Silver, natural mica-based intense Silver.

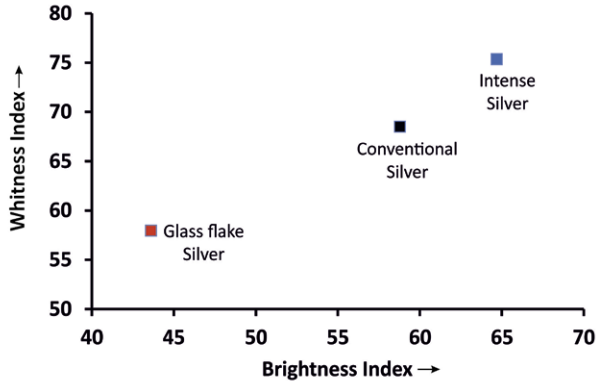


Fig. 2 Whiteness Index versus Brightness Index of Silver pearls in 10-100µ range; Conventional Silver (natural mica-based), Glass flake Silver, Intense Silver (natural mica-based).

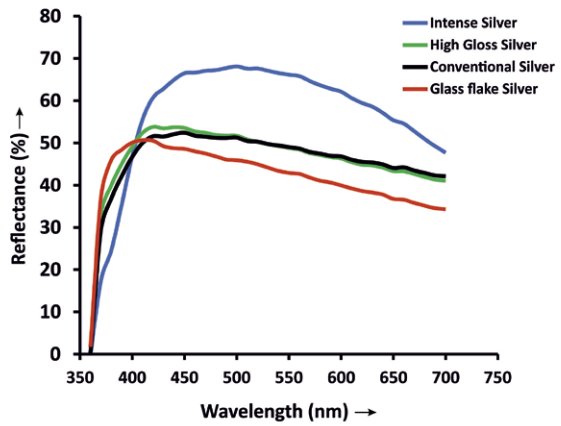


Fig. 3 Reflectance versus wavelength of Silver pearls in 20-150µ range; Conventional Silver (natural mica-based), Glass flake-based Silver, High Gloss Silver (natural mica-based) and Intense Silver (natural mica-based).

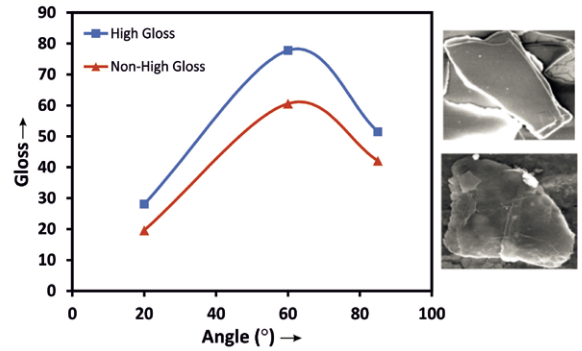


Fig. 5 Comparison of High Gloss product vs Non High Gloss products; Gloss at 20°, 60° and 85° angles with corresponding SEM images of raw mica that is used to produce the effect pigments (top: High Gloss Technology, bottom: Conventional Technology).

reflectance and in the cosmetic end applications, the particle size appears higher than actual. Similar effect is created for higher particle size Intense Silver (20-150µm), please refer **Fig. 3** and **4**.

Advantages

These new intense color effect pigments possess increased chroma at the reflection angle, display higher color purity and simultaneously an increased sparkle than traditional effect pigments. The enhanced reflectance and chroma of these pigments also provide an option to use lesser levels of these effect pigments in standard formulations. The customized particle size distribution allows creating unique visual effects in broad range of cosmetic formulations, primarily color cosmetic formulation such as creamy eye shadows, lip gloss or other lip products but also in Personal Care formulations, such as shower gels or hair shampoos.

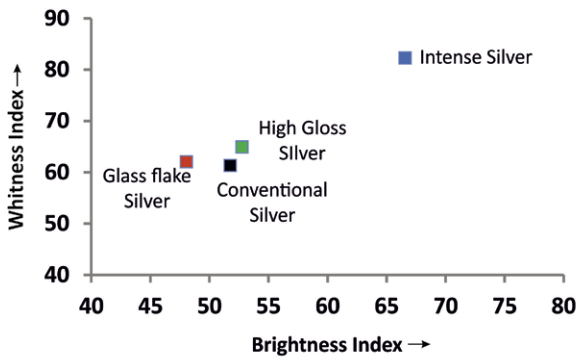


Fig. 4 Whiteness Index versus Brightness Index of Silver pearls in 20-150µ range; Conventional Silver (natural mica-based), Glass flake Silver, Intense Silver (natural mica-based).

High Gloss (HG) Technology

This technology, as the name indicates, was developed to produce high gloss (HG) effect pigments. This was achieved by using a special processing technique while preparing the raw mica, before it can be coated with metal oxides to produce an effect pigment. Wet mica grinding techniques are known since long time [4]. Creation of core material that acts as a substrate is an art of grinding. This technique allows better separation and finish of the mica layers while grinding and provides smoother and thinner raw mica substrate flakes for coating with metal oxides. This preparation allows enhancing the gloss by around 15 to 20% as compared to the non-high gloss effect pigments, please refer **Fig. 5**. Products developed using High Gloss technology provides brilliant and clean sparkle effects. These products are suitable for large particle sparkle trends.

References

[1] *Atwood, F. C.* (Atlantic Research Associates, Inc.) U.S. Patent 2,278,970, 1942.

[2] *Pfaff, G.; Franz, K.-D.; Emmert, R.; Nitta, K.; Besold, R.* In Ullmann's Encyclopedia of Industrial Chemistry: Pigments,Inorganic, Section 4.3, 6th ed. (electronic release); VCH Verlagsgesellschaft: Weinheim, Germany, 1998.

[3] *Pfaff, G.;* Reynders, Angle-Dependent Optical Deriving from Submicron Structures of Films and Pigments, Chem. Rev. 99, 1963-1981, 1999.

[4] *Bose M. J. Du,* Complete continuous wet mica grinding process, US Patent 3327951, 1967.

contact

Dr. Kailash M. Mogare | Deputy General Manager Research and Development
Dr. Tushar Navale | Manager Application Development
Mr. Frank Mazzella | Global Head of Research and Development
Dr. Matthias Lergenmüller | Vice President Cosmetics

Sudarshan Chemicals Ind. Ltd.
Pune | INDIA

Dewy Glow Iceberg Balm with CRYSTALIDE™ (*) and MATRIXYL® MORPHOMICS™ (**) | SE0092H

CRODA

sederma

This iceberg balm features an amazing shape-memory texture that breaks into pieces when taking it into your fingers. This unique texture is provided by the special combination of Volarest™ FL, Arlacel™ LC and Tween™ 60. Crodamol™ AB imparts a light skin feel without any drying effect. The bio-harmonical peptide Crystalide™ promotes a crystal skin and a glassy complexion, while bio- mimetic peptide Matrixyl® Morphomics™ fights against wrinkle appearance. A touch of cocooning that comforts the senses is added with Crodarom™ Delice Extreme, a natural extract of pecan nut and maple syrup. Add fun to your morning routine with this incredible iceberg balm that will erase the signs of ageing and leaves your skin dewy and luminous, from the inside.

Part	Ingredient	INCI Name	Functionality	% w/w
A	Water Deionised	Aqua	-	To 100
	Volarest™ FL[1]	Acrylates/Beheneth-25 Methacrylate Copolymer	Rheology modifier	2.50
	Potassium Sorbate		Preservative	0.10
	Tween™ 60[1]	Polysorbate 60	Surfactant	2.00
B	**Arlacel™ LC[1]**	Sorbitan Stearate (and) Sorbityl Laurate	Surfactant	1.70
	Pentylene Glycol		Humectant	3.00
	Phenoxyethanol		Preservative	0.80
C	**Crodamol™ AB[1]**	C12-15 Alkyl Benzoate	Emollient	3.00
D	Water Deionised	Aqua	-	2.00
	Sodium Hydroxide 30%		pH Adjuster	0.20
E	**Crodarom™ Delice Extreme[3]**	Water (and) Glycerin (and) Carya Illinoinensis (Pecan) Shell Extract (and) Acer Saccharum (Sugar Maple) Extract	Botanical Extract	1.00
F	**Crystalide™ Active ingredient[2]**		Skin glowing active ingredient	3.00
G	**Matrixyl® Morphomics™ Active ingredient[2]**		Anti-ageing active	2.00
H	Keshi 264285 D4	Perfume	Fragrance	0.10

(*) Patent pending
(**) Patent N°FR 3 029 782; WO 2016/097965; US 2018000717; EP 3256102; JP 2018500330; CN 106999401; KR 20170086680; BR 112017012474; MX 2017007819

Suppliers:
1: Croda 2: Sederma 3: Crodarom 4: Firmenich

Procedure:
Weigh Part A, mix well. Heat Part A to 75°C in bain-marie. Weigh Part B, mix well. Add Part B to Part A and stir well for 30 min at 75°C with normal helix stirring. Weigh Part C and heat to 75°C in bain-marie. Add Part C to previous part with strong rotor stator stirring. Weigh Part D, mix well. Adjust the pH with Part D, stir well. Add Part E to the previous part, mix well. Add Part F to the previous part, mix well. Add Part G to the previous part, mix well. Add Part H to the previous part, mix well.

Appearance: White opaque viscous cream; pH: 6.10± 0.5; Viscosity: 200.000 ± 10% Sp.93, 2.5rpm, 1 min, 25°C, Brookfield DV-I Prime

Stability: 3 months at 4°C, 25°C, 40°C and 1 month at 50°C

Centrifuge 10min @ 3000rpm and 1 x -80°C/+25°C 24 hours freeze-thaw cycle and autoclave 20min @120°C

This formulation was developed in France. Contact your local sales representative with enquiries as ingredient availability can vary by region.

Sun kissed Golden Body Oil | SU-BO-04

SUDARSHAN

Phase	Trade Name	INCI Name	Supplier	% (w/w)
A	Octyl Stearate	Octyl Stearate	Local Supplier	32.0
	Crodamol IPM	Isopropyl myristate	Croda India	32.5
	Crodamol IPIS	Isopropyl Isostearate	Croda India	11.0
	Argan Oil	Argania Spinosa (Argan) Kernel Oil	Katyani Exports	0.50
	Jojoba Oil	Simmondsia Chinensis (jojoba) Seed Oil	Katyani Exports	0.50
	Grapeseed Oil	Vitus Vinifera(Grape)Seed Oil	Katyani Exports	0.50
B	Bentone Gel ISD-V	Isododecane (and) Disteardimonium Hectorite (and) Propylene Carbonate	Elementis	15.0
C	**Sumicos Bronze Flash 43757**	Mica (and) Iron Oxide	Sudarshan Chemical Industries Limited	3.60
	Sumicos Flash Bronze HG755	Mica (and) Iron Oxide	Sudarshan Chemical Industries Limited	1.80
	Prestige Bright Gold 35164	Mica (and) Titanium Dioxide	Sudarshan Chemical Industries Limited	0.60
D	Euxyl PE9010	Phenoxyethanol (and) Ethylhexylglycerin	Schülke & Mayr GmbH	1.00
E	Sportsman 95199	Perfume	Oriental Aromatics Limited	1.00
Total				**100**

Procedure:
1. Add Phase A ingredients in a Main Vessel (MV).
2. Add Phase B to MV under continuous stirring.
3. Add phase C to MV, mix thoroughly.
4. Add phase D & E to MV, mix thoroughly.

Properties:
pH: Not applicable Appearance: Body oil with intense golden reflection

DISCLAIMER: This formulation is presented in a good faith and meant to demonstrate how our pearl pigments can be used in various formulations. No warranty of the accuracy or completeness of results, or fitness for a particular use is given; nor is freedom from the infringement of any intellectual/industrial property rights to be inferred. It is provided "as is" aimed at supporting customer development and requires further appropriate evaluations by customers. To the extent permissible by applicable law, Sudarshan Chemical Industries Limited shall in no way be liable for any reliance upon information or materials provided.

Isobionics® Santalol – a Close Alternative to Sandalwood Oil

Interview with Toine Janssen, founder of Isobionics®

Toine Janssen

What are the advantages of Isobionics® Santalol?

Thanks to its woody note, Isobionics® Santalol is a real alternative to sandalwood oil. It represents the heart note of sandalwood oil. It offers consistently high quality and is available regardless of weather or harvesting conditions.

Isobionics® Santalol is 100% free of endangered sandalwood trees. It is produced by fermentation from renewable raw materials: The starting material for the fermentation process is corn starch obtained from corn grown in Europe. Fermentation is an ancient cultural technique known from processes such as brewing beer and baking bread. In case of brewing beer, the glucose is transformed into alcohol.

Isobionics® uses a proprietary platform based on Rhodobacter sphaeroides. It uses the combination of two metabolic pathways to very efficiently produce terpenes. Products do not have yeasty off-notes and are thus ideally suited for flavor and fragrance applications.

Why does the F&F market need an alternative to sandalwood oil?

Sandalwood oil is extracted from the white sandalwood tree *Santalum album*. Plantations of sandalwood trees exist mainly in India. Sandalwood trees can be harvested after 15 years the earliest but high yields and good quality of oil is achieved from trees aged 30 to 40 years and above. This led to harvesting especially of mature trees which are most relevant for propagation. Due to depleting cutting of the white sandalwood tree over decades, the species is listed on the IUCN Red List. Sandalwood and its oil are deeply connected to India's culture and to save it from extinction, the Indian government strictly regulates plantations, harvest and trading of the wood and its products.

Bad weather conditions also influence the harvest. Therefore, original sandalwood oil is not sufficiently available for the F&F Industry in constant quality and price. With our new product Isobionics® Santalol, we respond to the unchecked market need. Isobionics® Santalol represents the heart note of sandalwood oil and its excellent cost positioning makes it an attractive alternative.

For which applications is Isobionics® Santalol suitable?

Thanks to its woody note, it is a real alternative to sandalwood oil, especially for use in fine fragrances and high-quality personal care products. In fragrance compositions, Isobionics® Santalol brings more volume and warmth. Floral-oriental fine fragrances are enhanced by the creamy sandalwood note.

Isobionics® portfolio is mainly based on fermentation processes. What contribution can fermentation make to mastering the industry's challenges?

I'll give you four good reasons: Consistently high quality, availability, effective production and scarcity of natural resources. Our fermentation technology lets us strictly control all conditions so that the quality of our ingredients is consistently high. This technology also makes it possible to produce year-round without weather-related restrictions. The result is the most important advantage for the F&F industry: The reliable availability of fermentation-based ingredients reassures our customers and simplifies delivery processes.

Fermentation becomes particularly interesting when plant products reach their limits. Innovations in fermentation can reconstruct highly complex molecules, providing easy access to natural aroma ingredients with a convincing purity and efficient production processes. And the final reason: Fermentation-based processes conserve already scarce natural resources, like sandalwood.

www.isobionics.com

Picture Credits: Sophie James/shutterstock.com

Clean Face, Clean Teeth, Clean Home: the All-in-one Natural Mineral Solution

Ostringen/Switzerland. September 10, 2020. At this year's SEPAWA, Omya is excited to update visitors on its personal and home care product portfolios: Within its cleansing agents range, the ingredients Omyacare® S 75-KP and Omyacare® S 95-KP have recently been approved by ECOCERT as COSMOS raw materials and gained NATURE certification. And for toothpaste applications, Omyadent®200 is a novel solution to address dentin hypersensitivity (DH). Attendees will also learn about the multiple ways in which mineral ingredients can create a new generation of functional, yet eco-friendly, end products - for example, surface cleaners based on marble, dolomite or limestone.

Variety of applications: The Omyacare® brand comprises bright-white particles sourced from natural minerals that work as cleansing agents with different grades of abrasiveness, and are thus adjustable according to the desired color, viscosity and texture of toothpaste and rinse-off applications. Thanks to the diversity of particle sizes within the Omyacare® product range, a huge variety of different applications are possible, such as bar soap, toothpaste, tooth tabs or handwash tabs. Underlining the company's commitment to maximum sustainability, the Omyacare® varieties S 75-KP and S 95-KP are now approved by the non-profit ECOCERT organization for raw materials to be natural, environmentally friendly and sustainable.

Tackling teeth sensitivity: Drawing on its vast expertise in developing advanced mineral ingredients, Omya has added Omyadent®200 to its dental ingredients line. This new grade of particle is highly efficient for desensitizing toothpaste applications. The co-processed mineral has an outer shell of hydroxyapatite, the main constituent of enamel and dentin, Dentin hypersensitivity can be treated by blocking dentin tubules, thus preventing irritations and dentinal fluid movement. Thanks to its tailored small particles, Omyadent® 200 penetrates the tubules and effectively occludes them, while the hydroxyapatite shell makes it resistant to acid attack.

Particles for perfect polish: When it comes to the home care segment, Omya's spotlight at SEPAWA is on surface cleaners. Depending on the individual product purpose, there are different mineral bases available, such as marble, dolomite or limestone. Ingredients of the brands Omyadol® and Omyacarb® deliver outstanding absorption properties, adjustable abrasivity and good polishing ability – all without harming environmental ecosystems. The cost-efficient, brilliant white and natural particles not only meet the highest expectations in terms of efficiency and sustainability, but also enhance the look and feel of finished products.

Distribution network expansion: All minerals can be combined with ingredients from the company's huge distribution portfolio. Its long-standing expertise and collaboration with certified producers mean that the company is able to provide a guaranteed sustainable supply for its customers. Omya is therefore happy to announce its collaboration with Aspanger, a producer of functional mineral substances and grinder of special mineral products. For its distribution portfolio, Omya focuses on Aspanger's natural MICA products, namely high quality and pure minerals which act as functional solutions in personal care products, such as decorative cosmetics and those with pearlescent pigments, and oral care.

Spotlight on science: During the SEPAWA Congress, Omya will participate in a Scientific Poster Session with the topic "Disruptive carrier acts as sequestrant by enhancing performance and sustainability profile in automatic dishwashers". (Exposition Poster 21).

www.omya.com

New Features of the Colors & Effects®

Online Service Platform Add Value, Convenience and Simplicity to the User Experience

- Product Comparison allows customers to compare products side-by-side to select the pigments that best fit their needs
- MySheets gives users a personalized library of product documents for their "favorited" pigments
- Sample Request makes it easy for customers to request a sample directly from the platform

Ludwigshafen/Germany, September 16, 2020. Colors & Effects® continues to improve the customer experience for its Online Service Platform by offering three new user-friendly functionalities in the Products area of the site: Product Comparison, MySheets and Sample Request. By registering for a personal account, users reap additional benefits to make the most of their online experience.

The Product Comparison feature allows users to evaluate multiple products side-by-side to help them find the pigments that best suit their business needs. Compare between two and four products within the same industry and assess pigments based on key attributes including product group, pigment effect and application. Users with a personal account can save their comparisons to the user dashboard for easy reference.
With MySheets, customers can "favorite" products to create a personalized library of product documentation that can be accessed through their user dashboard. Users have a single location for storing and referencing these documents, and anytime a document in the MySheets library is updated by Colors & Effects, users will receive a notification of this change. This ensures customers always have the latest information available at their fingertips.
Sample Requests for pigments of interest can now be completed conveniently through the platform. As customers browse pigment pages, they can submit a sample request by filling out a short form. Furthermore, customers can review their sample request history directly from their user dashboard.
MySheets and Sample Requests are available to customers with a registered and validated account.
The Online Service Platform, which launched in February 2020, encompasses the brand's corporate website and Pigment Finder, a product catalogue tool, which is now located under the Products area of the platform.

Visit the Online Service Platform
www.colors-effects.eu/products

For a Climate-friendly Industry: Using Carbon Dioxide and Hydrogen as Raw Materials for Sustainable Chemicals

Commissioning of the Rheticus system, from left to right: Stefan Kaufmann, Innovation Officer "Green Hydrogen" of the Federal Ministry of Education and Research; Anja Karliczek, Federal Minister for Research; Dr. Harald Schwager, Deputy Chairman of the Board of Management evonik Industries AG; Prof. Dr. Armin Schnettler, Executive Vice President New Energy Business, Siemens Energy

Marl/Germany, September 21, 2020. Evonik and Siemens Energy today commissioned a pilot plant sponsored by the German Federal Ministry of Education and Research (BMBF) that uses carbon dioxide and water to produce chemicals. The necessary energy is supplied by electricity from renewable sources. The pilot plant is located in Marl, in the northern Ruhr area, and its innovative technology of artificial photosynthesis should contribute to the success of the energy revolution. It is an essential part of the Rheticus I and II research projects, which are sponsored by the Federal Ministry of Education and Research (BMBF) with a total of 6.3 million euros.

German Federal Minister of Education and Research *Anja Karliczek* said on the opening ceremony in Marl, "I am delighted that we have today given the go-ahead for a new test facility of the very highest standard in Marl.With Rheticus, we are showing how we can set up climate-friendly production processes in the chemical industry and at the same time manufacture new innovative products. And this works not only here in Germany, but potentially all over the world. This opens promising opportunities for technology exports. We want to promote effective climate protection and continue to have a strong industrial base in Germany. I am firmly convinced that we can succeed in both. I am pleased that my ministry is now investing a total of 6.3 million euros in the new pilot plant in Marl with this goal in mind and wish all those involved every success."

Stefan Kaufmann, Member of the German Bundestag and Federal Commissioner for Green Hydrogen, states, "Today's start of the Rheticus pilot plant for the production of specialty chemicals is a real pioneering achievement. After all, a green hydrogen economy can only succeed in Germany as a country of innovation, if innovative technologies are used. This requires courage and a spirit of research. The project partners at Rheticus are exemplary in demonstrating this."

Harald Schwager, Deputy Chairman of the Executive Board of Evonik and responsible for innovations, said, "Climate protection is not possible without chemistry, because our industry supplies and develops solutions for the energy turnaround. Research projects such as Rheticus are a motivation and innovation driver for a sustainable society". At the same time, he warned against speed when phasing out fossil fuels. "Security of supply and reliability in political decisions set the framework in which new things are created."

Christian Bruch, CEO of Siemens Energy: "Our goal is to use innovative technologies to enable new, more sustainable solutions. With our hydrogen and CO electrolysis, we are building a bridge from green electricity to sustainable material applications. The close cooperation between politics, science and business partners, like Evonik, is an important step in this direction." The Rheticus research project is a spin-off of the Copernicus projects, one of the largest research initiatives of the German Federal Government on energy system transformation. Rheticus demonstrates how the Power-to-X idea can be successfully put into practice.

For the idea of artificial photosynthesis, which is behind the Rheticus experimental facility, the researchers took nature as a model. Just as plants use solar energy to produce sugar, for example, from carbon dioxide (CO_2) and water in several steps, artificial photosynthesis uses renewable energies to produce valuable chemicals from CO_2 and water through electrolysis with the help of bacteria. This type of artificial photosynthesis can serve as an energy store and thus help to close the carbon cycle and reduce carbon dioxide pollution in the atmosphere.

The pilot plant has started up in Marl, the largest Evonik site. It consists of a CO electrolyzer, developed by Siemens Energy, a water electrolyzer and the bioreactor with Evonik's know-how. In the electrolyzers, carbon dioxide and water are converted into carbon monoxide (CO) and hydrogen (H_2) with electricity in a first step. This synthesis gas is used by special microorganisms to produce specialty chemicals, initially for research purposes. These are starting materials for special plastics or food supplements, for example.

In the coming weeks, the composition of the synthesis gas and the interaction between electrolysis and fermentation will be optimized. In addition, a unit for processing the liquid from the bioreactor will be set up to obtain the pure chemicals.

After successful completion of the current Rheticus project phase (Rheticus II), Evonik and Siemens Energy will have a unique platform technology at their disposal that can produce energy-rich and valuable substances such as specialty chemicals or artificial fuels from CO_2 - in a modular and flexible manner.

www.evonik.com

New in our portfolio

High performance soil release polymers

Soil release polymers are specialized polymers used in laundry detergents to enable easier and more effective removal of oily stains from synthetic fibers like polyester and mixed polyester/cotton fabrics. Clothes that are treated with these soil release polymers will be protected against dirt and greying, and additionally will feel more comfortable to wear.

Soil release polymers are designed to fit perfectly with the structure of synthetic fibers, especially polyester. By washing a fabric with these soil release polymers they will adsorb on the fibers and show the following benefits:

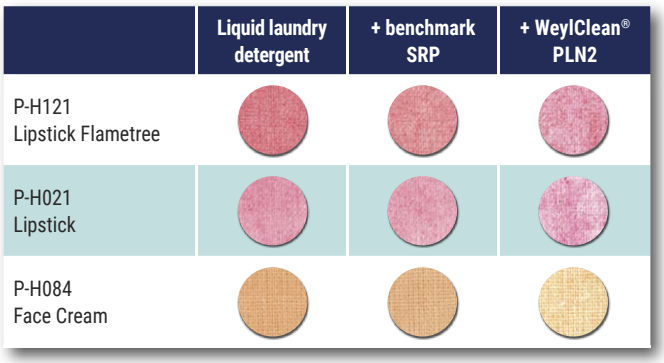
- Facilitating the dirt removal of hydrophobic stains in every wash cycle as the hydrophilic soil release polymer layer prevents the dirt sticking and penetrating into the fabrics
- Maintaining the whiteness and brightness of clothes as soil release polymers prevent greying of the synthetic fibers by an anti-redeposition effect and dispersion of soil
- Enhancing the breathability and comfort of clothes made out of synthetic fibers as soil release polymers increase the hydrophilicity of such fabrics

WeylChem is offering a range of soil release polymers for liquid and powder detergents that are compatible with most ingredients of standard formulations when formulated at an appropriate pH.

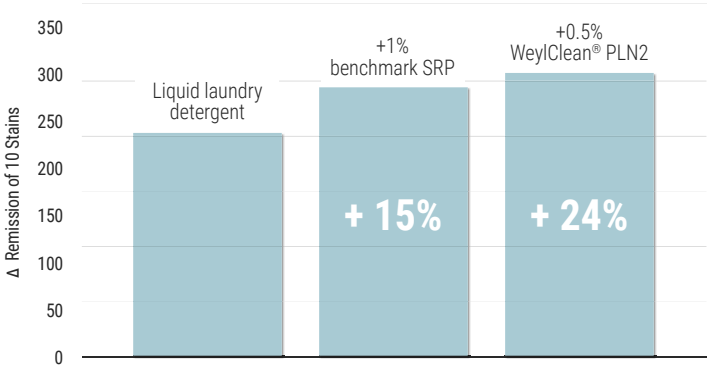
For powder detergents WeylClean® PSA1 is the optimal choice as an anionic and solid material with 100% activity. For liquid detergents there are two liquid products available, WeylClean® PLN1 and WeylClean® PLN2.

Next level premium grade WeylClean® PLN2 – high performance at low temperature

WeylClean® PLN2 is a new, premium grade with superior soil removal at 20°C that will be easily recognized by consumers (see picture 1). WeylClean® PLN2 can also reduce formulation costs, as a lower dosage level is required to achieve the same or even improved cleaning performance (see graph 1). Finally, WeylClean® PLN2 shows a lower viscosity reduction in your formulation compared to standard polymers. WeylClean® PLN2 is easy to formulate and can be used at a level of 0.2 – 1%.



Picture 1: WeylClean® PLN2 – Excellent soil removal at 20°C



Graph 1: Potential for cost reduction

If you are interested in WeylChem's range of soil release polymers WeylClean® PSA1, WeylClean® PLN1 or WeylClean® PLN2 and would like more information or samples please contact us at consumer.care@weylchem.com.

NORDMANN

Elementis and Nordmann Expand Cooperation in the Field of Personal Care

Hamburg/Germany, 15 September 2020. Nordmann has further expanded its long-standing cooperation with Elementis in the field of personal care. As of August 15, the international chemical distributor assumed control over the distribution of Elementis' Gilugel® products and active ingredients for antiperspirants in Austria, Bosnia-Herzegovina, Bulgaria, Croatia, the Czech Republic, Denmark, Finland, Germany, Hungary, the Netherlands, Norway, Poland, Serbia, Slovakia, Slovenia, Sweden and Switzerland.

The high-performance antiperspirant actives from Elementis offer the best moisture protection on the market and can be utilized in all standard product formats, e.g. aerosols, roll-ons and deodorant sticks.
Gilugel® products can be used in a variety of cosmetic formulations to serve several purposes, like rheological properties of emulsions.
Elementis is the world's leading manufacturer of rheological additives, natural oils, active ingredients and other specialties for a number of different industries.
Nordmann has been distributing Elementis products for personal care and HI&I for over 25 years. Expanding the already long-standing cooperation between the two companies further underscores Elementis' confidence in the comprehensive industry expertise and customer service excellence that Nordmann offers.
Mihaja Randriamahazomanana, Sales Director EMEA, Personal Care at Elementis, sees great potential in the newly intensified cooperation: "With the help of Nordmann's great understanding of the market, we can now extend our reach in Europe and further intensify the exchange of experience and knowledge between our two teams. We are looking very positively toward what lies ahead."
Petra Fisch, Sales Director Life Sciences Germany at Nordmann, adds: "We appreciate the outstanding quality of the products that Elementis provides, as well as their discerning know-how. Together, our companies have already launched many successful projects. We look forward to continuing this work on a larger scale and to pursuing the specific goals of our customers through integrated, comprehensive end-to-end consulting."

www.nordmann.global

ARKEMA
INNOVATIVE CHEMISTRY

Arkema Wins the 'Environment and Natural Resources' Prize at the Sustainable Industry Awards

Colombes/France, 15. September 2020. On 15 September at the ceremony of the Sustainable Industry Award organized by L'Usine Nouvelle, Arkema wins the "Environment and natural resources" prize for its new SENSIO™ product range.
With its new SENSIO™ biobased surfactant range, Arkema is the winner of the 'Environment and Natural Resources' category of L'Usine Nouvelle magazine's Sustainable Industry Awards. This prize awards to the best project in the category and reflects Arkema's ability for innovation and value creation towards sustainable development.

The bio-based SENSIO™ product range

The surfactant range SENSIO™, derived from castor oil, is designed for all formulation types in the detergent market. Thanks to its cleaning and foaming characteristics, it offers many advantages leading to more efficient cleaning products. Due to its origin and biodegradability, the range offers a unique sustainable alternative to traditional products.

Innovation for sustainable development

Arkema places innovation at the heart of its strategy in order to address the societal challenges and contribute to the positive evolution of an ever-changing world.
The Group offers usable, innovative and environmentally friendly solutions in such areas of biobased products, new energy sources, water management, electronics solutions, lightweight materials and design, and home efficiency and insulation.

www.arkema.com

PRODUCT LAUNCH

New BASF Fragrance Ingredient Velberry: A Fruity-Sweet Innovation

Ludwigshafen/Germany, September 1, 2020. BASF Nutrition & Health has brought Velberry™, a new, fresh and fruity-sweet fragrance, on the market. Velberry is the first substance developed jointly by BASF and its innovation ecosystem partners for the broad market.

BASF has formed the „Virtual Innovation Ecosystem", combining its own expertise in chemistry, regulatory matters and research & development with the strengths of its perfumery and formulation partners. This collaborative approach accelerates the development of new substances for the fragrance market. There is a high demand among perfumers for new fragrances in order to continuously provide consumers with new fragrance profiles for example in personal care products such as shampoos and body lotions. These new products have to give perfumers more freedom in terms of fragrance creations while meeting growing demands for sustainability, product safety and supply reliability at the same time.

Bringing innovative products to market is time consuming, cost-intensive and involves complicated regulatory efforts and financial risks. Only a few companies in the market have the resources needed for the tedious research and development work involved, which is why BASF founded the innovation ecosystem.

"With Velberry, we have succeeded not only in meeting our customers' desire for new substances to differentiate their fragrances, but also in meeting the growing demand for sustainable alternatives - all at an attractive price," says *Steffen Götz*, head of the BASF Aroma Ingredients division.

The experts search through the entire palette of BASF chemicals for molecules suitable for use as fragrances. The large number of potential candidates calls for a strict selection and prioritization process.

„We ask for feedback from perfumers, jointly evaluate the substances and then develop the most promising candidates. Our pipeline is filled and additional innovations are already on the way," says *Ralf Pelzer*, head of the New Business Development unit at BASF Aroma Ingredients.

www.basf.com

Breathe. Escape. Enjoy.

Bell EMEA Presents Air Care Trends 2021/22

Leipzig/Germany, September 17, 2020. The upcoming candle season is just around the corner. Bell Flavors & Fragrances EMEA introduces its latest fragrance collection for scented candles and reed diffusers coming with the slogan „Breathe. Escape. Enjoy." The compositions present themselves as different in character as the facets of life and cover elegant and earthy notes as well as intense fruity profiles. Manufacturers also benefit from Scenti Master Batch, a carrier material that Bell developed specifically for scented candles.

Fragrances to feel alive

The more turbulent and uncertain the times are, the more important the private sphere becomes. People focus on the essentials: family, home, community. They BREATHE and develop a peaceful environment at home. Natural materials express this feeling as much as soft, neutral colours. From an olfactive perspective, it is especially elegant fragrances that suit this feeling. Compositions of lily of the valley and fig, but also jasmine and sandal interpret the imperfect beauty that the theme implies.

On the other hand, there is a desire to ESCAPE the daily grind. The longing towards mysterious, distant lands, and also the origins of one's own homeland respond to the deep inner thirst for fulfilment. From an olfactive point of view, this is reflected by the warm notes of earthy spices. Thus, woody cinnamon notes meet extraordinary lavender mint compositions in this collection.

Ultimately, everybody wants to ENJOY life in all its facets and focus on the positive moments. The intense fragrances of ripened peaches, red berries or even orange, prickly pear, and lychee represent this hunger for life on an olfactive level.

Scented Candles without an oily film: Scenti Master Batch

Scenti Master Batch represents a fragrance delivery system that had been specifically developed for candles. It enhances the intensity of scented candles due to the possibility of incorporating dosages over five percent and the characteristic of not losing any oil. This is beneficial for the lifetime of the product and consumers benefit from a candle that releases its fragrances only after being lit. Last but not least, scented candles manufactured with Scenti Master Batch stand out due to their efficient and even burning.

Scenti Master Batch is available either in the form of a block or as pastilles. The latter is even easier to process as they melt more quickly, and as the material is not flammable, storage is convenient as well.

www.bell-europe.com

novozymes

Beyond "99.9%" Claims: Novozymes Launches Probiotic Solution in Cleaning

RALEIGH/United States, September 9, 2020. Novozymes, the world leader in biological solutions, today announced the introduction of Microvia probiotics for cleaning products. The microbial technology provides superior and continuous deep cleaning on hard surfaces over conventional chemistry alone – and enables consumers to avoid overuse of harsh cleaning and disinfection agents in their own homes.

"Novozymes scientists are leading the way to a future of cleaning with probiotics, rather than harsh chemicals, as the hero. You want a cleaner home? Add more bacteria!" says *Rene Garza*, Novozymes' Vice President for Household Care Americas. "People traditionally clean using chemical products that aim to remove all microbes, but there are beneficial bacteria within our home microbiome that can break down soils to natural harmless substances. Using probiotics in cleaners mean that we can boost the proportion of these helpful microbes, so they can continue the cleaning themselves."

As many begin to question the longer-term health effects of chemical cleaning and disinfection products, not least during the current COVID-19 pandemic, consumer interest in safer, more effective, and greener probiotic cleaner products is growing.

Cleaner and greener

Novozymes has been researching the use of probiotics in household cleaning for many years. As well as scientific studies into the effectiveness and safety of probiotic cleaning solutions, it has carried out extensive research into consumer perceptions of innovative, but naturally derived formulas that leverage probiotic technology.

"From our research and that of industry experts, it would appear that consumers are ready for natural products that clean by themselves1. Many surface cleaner users have expressed a positive interest in probiotic cleaning products, with almost as many concerned that disinfectants destroy good bacteria," remarks Rene Garza. "Novozymes Microvia probiotics add another dimension to the term 'living space'. The probiotic bacteria help create a thriving microbiome, so that dirty surfaces get a longer-lasting clean, even while the consumer sleeps!"

www.novozymes.com

Givaudan Active Beauty Launches Neosalyl™

100% Natural and Pure Salicylic Acid for Skin and Scalp that is Organic Compared to its Synthetic Alternative

Argenteuil/France, 15. September 2020. Givaudan Active Beauty launches Neosalyl™, a 100% natural and pure salicylic acid for skin and scalp that is organic compared to its synthetic alternative. Obtained by Green Fractionation from the natural essential oil of the Wintergreen plant, Neosalyl™ brings all its benefits in dermo-purification for skin, and shininess and anti-dandruff for hair care.

Schema process Neosalyl

Beauty consumers are looking for more natural products, massively rejecting chemicals, as they see natural ingredients as safe and trustworthy. According to Mintel, 72% of consumers would be most interested in using beauty, grooming and personal care products that are made using natural alternatives to synthetic ingredient or natural ingredients adapted in a lab. This trend keeps growing in any culture and challenges the composition and origin of personal care ingredients, as of today, 3 out of 4 women are scanning product composition and looking for hero

Givaudan

ingredients. In fact, salicylic acid is, with Hyaluronic acid and Retinol, one of the most known ingredients by consumers, always looking for performance to take care of their skin and hair.
Yohan Rolland, Global Category Manager said: "Due to its exfoliating properties, Neosalyl™ is a safe and effective peeling agent for a large number of cosmetic purposes for skin care, such as exfoliation, anti-inflammatory and anti-microbial benefits, raising it as the perfect active for prone-to-acne skin conditions. As a strong keratolytic agent, Neosalyl™ is also a very good synergistic active for the treatment of dandruff."
Neosalyl™ is extracted from Wintergreen leaves, sourced in Southwest of China in the Yunnan province by small farmers. Rich in essential oil composed of methyl salicylate, they immediately produce the crude essential oil of fresh raw material in a completely traceable supply chain. Then, it is in our Centre of Excellence in Avignon (France), that our phyto-experts begin a green fractionation process to transform the methyl salicylate and obtain the ingredient.
To warrant the authentication of the raw material and the natural origin of salicylic acid from wintergreen, our botanical experts apply an exhaustive set of analysis named IDPack. It includes botanical observation, DNA analysis, GC/FID and isotopic 14C measurement to clearly identify each botanical ingredient.

S3D powderful fond transparent

To inspire the beauty industry, Active Beauty formulation team crafted S3D® Powderful. Th is 11-Ingredient clean beauty cocktail combines Neosalyl™ with powdered hyaluronic acid (PrimalHyal™ 300) for skin immune defense reinforcement and wound healing, as well as biodegradable exfoliating particles (Biogomm'age W180) designed for sensitive skin. This water-free formula is highly concentrated. By adding few drops of water, it turns into a foaming milk to apply to the face. After a gently massaging into the skin and then rinsing, it reveals a cleaner skin and brighter complexion.

www.givaudan.com/activebeauty

index of advertisers

Publisher´s Ads

imprint

Organ

SEPAWA (Vereinigung der Seifen-, Parfüm- und Waschmittelfachleute e.V.)

Editor in Chief
Robert Fischer | robert.fischer@sofw.com

Editorial Content
editorial@sofw.com

Advertising
advertising@sofw.com

Subscriptions
subscription@sofw.com

Publisher
Verlag für chemische Industrie H. Ziolkowsky GmbH

Print

Holzmann Druck GmbH & Co. KG
Gewerbestraße 2 | 86825 Bad Wörishofen
Germany

Issues
10 issues per year + scheduled special issues

Address

Verlag für chemische Industrie H. Ziolkowsky GmbH
Dorfstr. 40 | 86470 Thannhausen
Germany

Phone
+49 8281 79940-0

Fax
+49 8281 79940-50

Email
vci@sofw.com

Internet
www.sofw.com

Advertisement Rates, See Media Info
www.sofw.com > SOFW Journal > Mediadata

Subscription, Prices
Print & Online Subscription
> EUR 235.00 (+ postage, + VAT where applicable)

Online Subscription
> EUR 195.00 (+ VAT where applicable)

Price/Issue
Germany: EUR 23.36 (+ postage, + VAT where applicable)
Other Countries: EUR 23.36 (+ postage, + VAT where applicable)

Picture Credits
Prostock-studio/stock.adobe.com

146th Volume: SOFW Journal Soaps, Oils, Fats, Waxes
93rd Volume: Containing Sections for Cosmetics, Aerosols and Perfumery
116th Volume: International Journal for Applied Science in the Chemical Industry

Jungbunzlauer

From nature to ingredients®

Clean up your cleaners

Sustainable home and laundry care
with ingredients of natural origin

Lactic Acid	Safe in-can preservative
Xanthan Gum	Biodegradable thickener
Sodium Citrate	Powerful builder in laundry detergents
Sodium Gluconate	Excellent chelator in alkaline solutions
CITROCOAT® N	Coated citric acid for stable powder formulations

Approved by ECOCERT RAW MATERIAL DETERGENTS

www.jungbunzlauer.com