03/20 | Volume 146 | Thannhausen, Germany, March 12, 2020

sofw journal

Home & Personal Care Ingredients & Formulations

powered by SOFW

home care

Why Proteins Improve Cleaning

Determination of CMCs

03
2020
english

skin & hair care

Swiss Glacier Bacterium to Vitalize Tired Skin

Shot of Well-being for Healthy Skin

Towards a Sustainable Solution for Skin Health and Well-being

Clay-like Ingredient for a Better Skin and Well-being

Anti-inflammatory and Antioxidant Cosmetic Ingredient for Holistic Skin and Hair Care

Micellar Formulations – Old Wine in New Skins?

antiper-spirants

New Technologies for Specific Antiperspirant Actions

BRAND NEW

Hello I'm the SOFW AWARD.

SCIENTIFIC HONOR FOR THE BEST ARTICLES OF THE PREVIOUS YEAR.

sofw journal
powered by SOFW

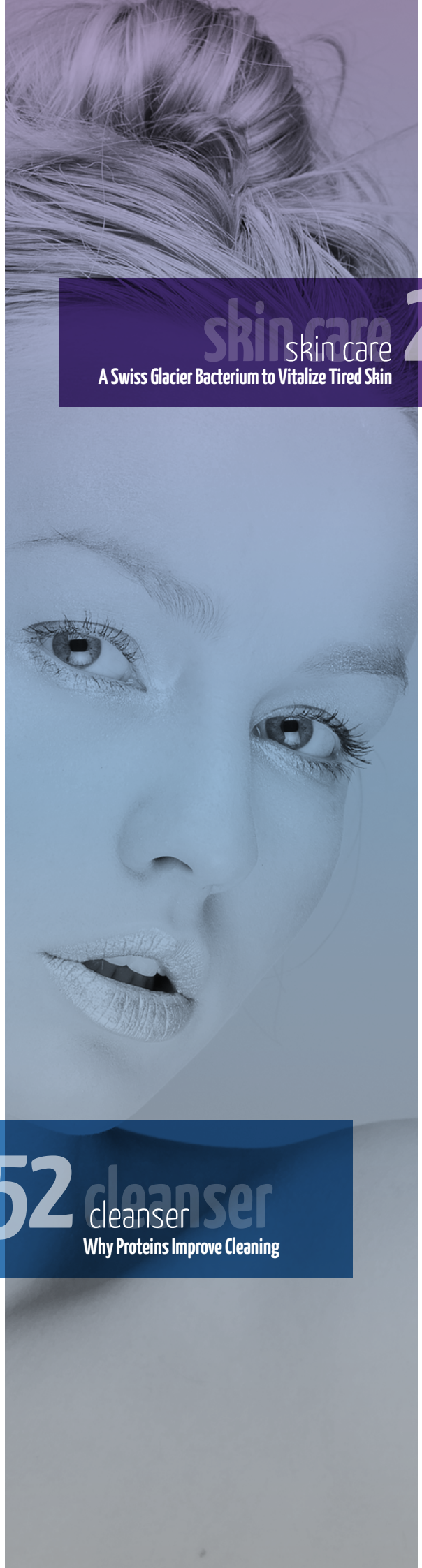
I honor the top three scientific papers of one year in the SOFW Journal.

I am awarded by an independent jury and **I AM BEAUTIFUL.**

More on **www.sofw.com/award**

SOFW

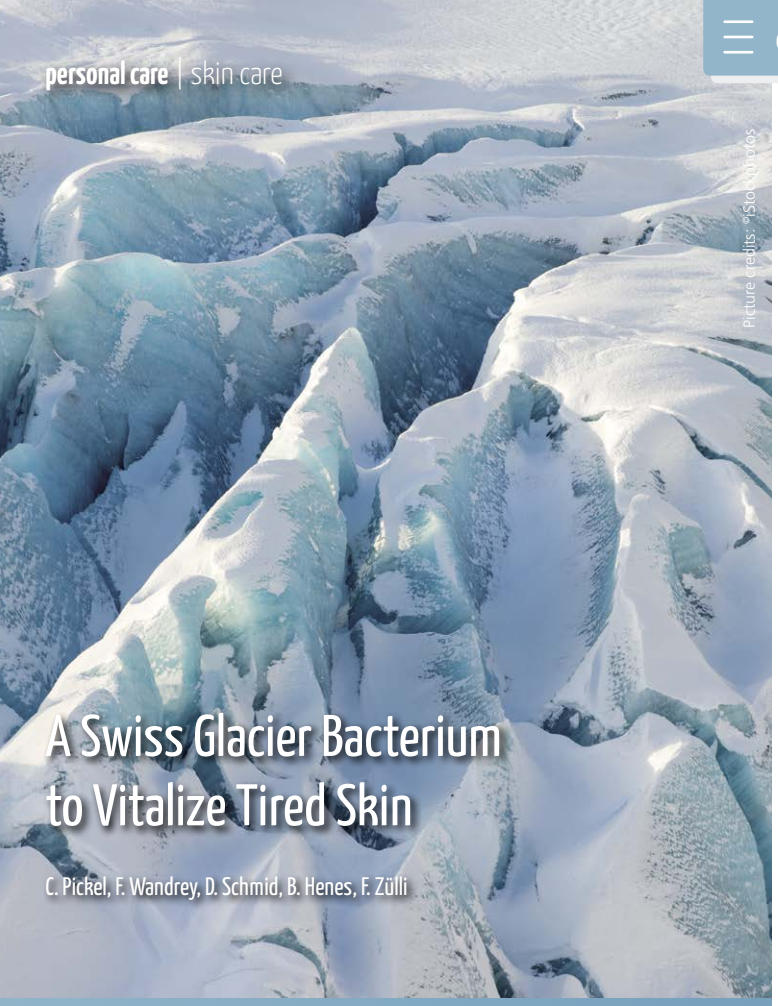
TAKE THE OPPORTUNITY TO WIN!



personal care

home care

content

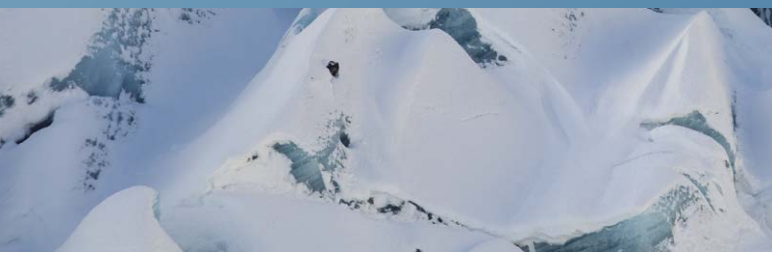


Picture credits: ©Stockphotos

A Swiss Glacier Bacterium to Vitalize Tired Skin

C. Pickel, F. Wandrey, D. Schmid, B. Henes, F. Zülli

Today's society is characterized by demanding jobs, long working hours and an increasingly hectic lifestyle, which frequently results in a lack of sleep. This is often markedly reflected on the skin by a tired appearance of the face. Recent research showed that also on the molecular level, lack of sleep causes stress and leads to premature aging. A major stress mechanism, the unfolded protein response (UPR) of the endoplasmic reticulum (ER), gets compromised by sleep deprivation, leading to the accumulation of misfolded proteins which damage the cell. To target this novel cellular aging mechanism, an extract of the psychrotolerant Swiss glacier bacterium *Iodobacter ssp*. was developed and analyzed for its efficacy to reduce ER stress and visible signs of tiredness. Treatment of aged fibroblasts with the extract led to an increase in the expression of ER chaperones which mediate the UPR and energized cells through an increase of ATP levels in a cellular model of sleep deprivation. Placebo-controlled randomized clinical studies conducted with sleep-deprived and overworked volunteers demonstrated that treatment with the Iodobacter-derived active ingredient IceAwake™ improved several skin parameters associated with skin aging, leading to a vitalized and rejuvenated appearance.



Introduction

Lack of sleep impacts on the cellular stress response

A long day at work, then you get stuck in commuter traffic, just quickly prepare dinner – time flies and suddenly it's past bedtime… most people can relate too well! In today's hectic society, lack of sleep is a common phenomenon which often results in a tired appearance marked by a loss of radiance, an increase in wrinkles such as crow's feet and dark circles around the eyes. But besides causing a tired appearance on the macroscopic level, sleep deprivation is also an aging factor on the molecular level.

It affects a cellular process termed the unfolded protein response (UPR), which is initiated upon accumulation of misfolded proteins and required for the clearance of these potentially toxic proteins [1]. The UPR takes place in the endoplasmic reticulum (ER), an intermediate station during the life cycle of the vast majority of cellular proteins. Following their synthesis from mRNA through ribosomes, proteins are transported into the ER, where a class of helper proteins, termed chaperones, assists in the correct folding and assembly of proteins and protein complexes. Only then, they can continue their journey in the correct shape and fulfill their intended functions in the cell. Cellular stress was shown to upregulate BiP (binding immunoglobulin protein), one of the major chaperones of the ER which is also involved in the UPR [2, 3]. Interestingly, recent research showed that expression of BiP is upregulated at night prior to increased expression of collagen, thereby supporting cellular regeneration processes [4]. It has further been shown that sleep deprivation affects protein folding and causes ER stress, leading to the activation of the UPR and an increase in the expression of BiP [5, 6]. This likely helps the cells to cope with the stress and aids regeneration and repair of the cell. However, it was shown that in aged cells, not only the basal expression of chaperones is decreased, but also their ability to activate the UPR upon sleep deprivation is reduced [3, 7]. The resulting accumulation of wrongly folded proteins causes further ER stress and leads to subsequent damage of the cell. Besides impairing the functions of the ER itself, ER stress also results in the formation of mitochondria-associated membrane (MAM) contact points, through which stress signals are transferred to the mitochondria [8]. In line with this, sleep deprivation also causes a reduction of mitochondrial activity, leading to reduced energy levels as reflected by a drop in the levels of ATP, the cellular energy currency [9]. This further prevents the activity of ATP-dependent chaperones upon lack of sleep, deteriorating cellular stress and causing cell damage.

Taken together, inadequate sleep, similar to UV irradiation or oxidative stress, is correlated with reduced skin health, as it weakens the skin's ability to repair itself at night and consequently accelerates skin aging. Therefore, active ingredients for cosmetic use which support the skin in coping with cellular stresses due to lack of sleep are desirable.

Harnessing the potential of extremophile organisms

Most organisms are viable only under (close to) optimal conditions, such as mild temperatures, atmospheric pressure, neutral pH and salt concentrations close to the organism's own. However, there are various groups of organisms termed "extremophiles" that have evolved to being able to survive and even thrive under extreme conditions. Among these, there are prokaryotes, e.g. archaea and bacteria, but also eukaryotes and even metazoans which grow despite very high or low temperatures, pH values, salt concentrations or pressures, and even under permanent ionizing or UV radiation. These organisms can be regarded as true masters of survival, and understanding their survival strategies has been the subject of numerous scientific studies. It has, for example, been shown that bacteria that are able to proliferate at temperatures as low as 5°C (so-called psychrotolerants) express enzymes which are active even at low temperatures and contain a large number of secondary metabolites with various functions [10, 11]. Transferring the capabilities of these extraordinary organisms into skin care by harnessing their biological potential and using them as a source for interesting and novel secondary metabolites for use in cosmetic application is of big interest for cosmetic scientists.

A freshness kick for tired skin with a Swiss glacier bacterium

In order to discover and harvest novel extremophile microorganisms for use in cosmetics, an expedition to a glacier in Valais, Switzerland, was undertaken. Due to the continuous shrinking of glaciers in the past decades, more and more microbes that have been hidden below permanent ice for centuries have become accessible. A sample of the soil exposed underneath the retreating glacier was taken and analyzed for its microbial content. This led to the identification of *Iodobacter ssp.*, a rod-shaped bacterium which belongs to the group of cold-tolerant organisms. After many years below the glacier ice layer, it has been reawakened and harnessed for the development of a novel active ingredient for skin care. Large-scale cultivation under optimized conditions and extraction of the *Iodobacter ssp.* strain followed by spray-granulation of the extract on a maltodextrin carrier yields the active ingredient IceAwake™ [INCI: Succinic Acid (and) Maltodextrin (and) Aqua/Water]. The efficacy of this novel active to reduce ER stress as a cause of prematurely aged and tired skin was investigated *in vitro* and *in vivo*.

Methods and Results

Iodobacter ssp. extract re-activates chaperone expression in aged fibroblasts

To assess the effect of *Iodobacter ssp.* extract on the process of protein folding, the expression of helper proteins required for flawless protein production was analyzed in aged cells. For this, normal human dermal fibroblasts were cultured for 17 passages prior to the experiment in order to mimic the aging process. Following treatment of these aged fibroblasts with 1 % *Iodobacter ssp.* extract for 24 h, cells were harvested, and total RNA was extracted. An untreated control was performed in parallel. Complementary DNA (cDNA) was synthesized from total RNA and the expression of target genes was assessed by RT-qPCR.

Among others, the expression of several chaperones involved in protein folding in the ER was analyzed in aged fibroblasts treated with an extract of *Iodobacter ssp.* and compared to an untreated control. The expression levels of key chaperones involved in the UPR, namely BiP, endoplasmin, calnexin and calreticulin, were increased by up to 100 % in aged fibroblasts when treated with 1 % *Iodobacter ssp.* extract (**Fig. 1**).

Previous research has shown that cellular stress, caused by factors such as sleep deprivation, leads to an upregulation of various ER chaperones [5, 6]. This is an important mechanism that helps the cells to prevent the accumulation of misfolded proteins. However, according to recent studies, aged cells lose the capacity to activate the UPR and therefore can no longer efficiently prevent protein misfolding and aggregation [3, 7]. Additionally, the expression of BiP, the main chaperone responsible for assisting correct protein folding and preventing of protein aggregation, declines in aged cells [7]. Together, this leads to an amplification of the ER stress and initiates a vicious cycle in which aged cells can only insufficiently recover from stress during the night and therefore get damaged even further. The results of this study show that treatment with an extract of *Iodobacter ssp.* reverses these aging effects by supporting protein folding in the ER via upregulation of chaperone expression.

ER stress is reduced by *Iodobacter ssp.* extract in a cellular model of sleep deprivation

Cellular stress caused by sleep deprivation not only impairs ER function and protein folding, but also affects mitochondria,

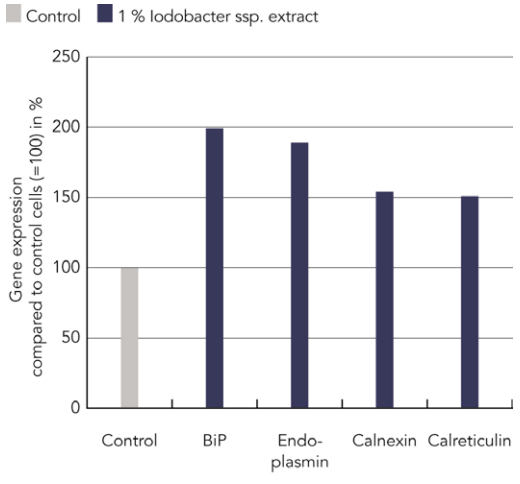


Fig. 1 Gene expression of chaperone genes in aged fibroblasts treated with *Iodobacter ssp.* extract relative to untreated cells.

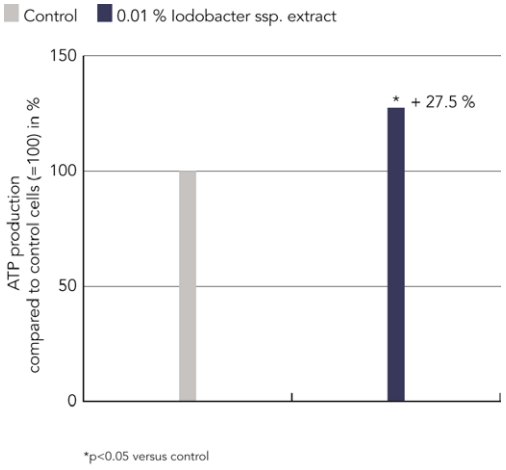


Fig. 2 ATP levels in AD fibroblasts upon treatment with *Iodobacter ssp.* extract relative to untreated cells.

the cells' powerhouses required for production of the energy equivalent ATP. A decrease in ATP levels is therefore a sign of impaired mitochondrial function [9]. Moreover, the formation of so-called mitochondria-associated membrane (MAM) contact points between mitochondria and the ER was shown to be an indicator of these impairments [8]. The efficacy of *Iodobacter ssp.* extract to alleviate these signs of cellular stress was tested in fibroblasts isolated from a patient with Alzheimer's disease (AD), which combine impairment of mitochondrial function and increased ER-stress and can thus be used as a cellular model for sleep deprivation.

In a first experiment, AD fibroblasts were treated with 0.01 % *Iodobacter ssp.* extract for 120 minutes or left untreated prior to measurement of ATP levels by chemiluminescence. Compared to the untreated control, *Iodobacter ssp.* extract increased ATP production by 27.5 % after 120 minutes of treatment (**Fig. 2**).

In a second experiment, the formation of MAM contact points resulting from interactions between the mitochondrial protein GPR75 and the ER protein SERCA2 was assessed in AD fibroblasts. As before, these cells were treated with 0.01 % *Iodobacter ssp.* extract for 120 minutes or left untreated, and healthy cells were cultured in parallel. Subsequently, MAM contact points were stained and the signal as well as nuclear counterstaining was visualized by fluorescent microscopy. Quantification showed that the number of MAM contact points was increased in the sleep deprived cell model, while treatment with the *Iodobacter ssp.* extract led to a significant and visible reduction of MAM contact points in these cells (**Fig. 3** and **4**).

The results of these experiments confirm that an extract of *Iodobacter ssp.* supports cells in dealing with mitochondrial and ER stress. The extract alleviates known signs of cellular stress and supports the function of ER as well as mitochondria. Moreover, an increase in ATP levels boosts chaperone function, as the activity of these helper proteins is energy dependent. Therefore, treatment with *Iodobacter ssp.* extract facilitates correct protein folding through an additional mechanism and despite aging and sleep deprivation.

IceAwake™ ameliorates visible signs of tiredness in a mixed study population

The efficacy of the active ingredient IceAwake™ to reduce visible signs of tiredness was further evaluated in two independent randomized, double-blind, placebo-controlled clinical studies. For this, twenty-one Caucasian men and women (44-66 years, mean age: 53.7 years) and twenty-three Asian women (41-57 years, mean age: 50.7 years) with chronic lack of sleep and/or a tired appearance with dark circles around the eyes and medium-deep crow's feet wrinkles were enrolled. Having completed a washout phase of three to seven days, the volunteers were asked to apply a cream containing 2 % IceAwake™ on one half of the face while treating the other half of the face with a placebo cream without an active ingredient. At the beginning of the study and after two weeks of twice daily treatment, the wrinkle depth of crow's feet was determined using a PRIMOS Premium (GF Messtechnik, Germany) or a PRIMOSlite (Canfield, Germany). Moreover, the skin radiance and visible tiredness of the volunteers was evaluated by clinical grading performed by experts.

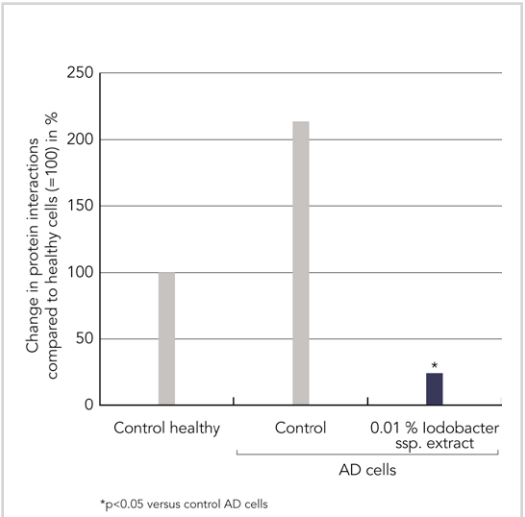


Fig. 3 Quantification of MAM contact points in healthy cells or AD fibroblasts treated with the Iodobacter active or left untreated.

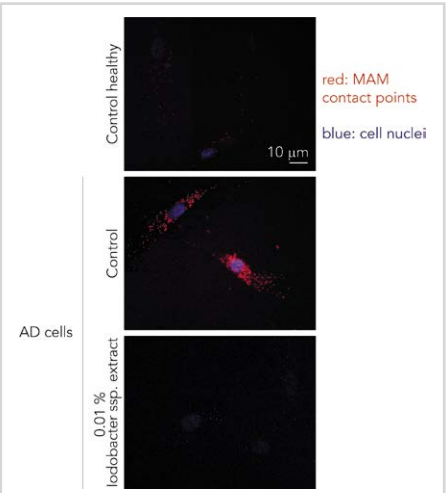


Fig. 4 Immunofluorescence staining of MAM contact points in healthy cells or AD fibroblasts treated with the Iodobacter active or left untreated.

mibelle GROUP biochemistry

Approved by ECOCERT RAW MATERIAL
COSMOS APPROVED

IceAwake™
Fighting aging in sleep-deprived skin

Today's hectic lifestyle inevitably affects both the quality and quantity of sleep, resulting in a tired appearance in the short term, and it is also an important aging factor in the long term. On a cellular level, this is caused by inefficient protein folding in combination with a lack of ATP.
IceAwake™ helps to reduce this stress in the skin cells. It is based on a biotechnologically produced bacterium which was reawakened after being discovered by Mibelle Biochemistry under a Swiss glacier.

- **Energizes tired skin**
- **Reduces wrinkles after only two weeks**
- **Increases radiance despite a hectic lifestyle**

In clinical studies conducted on volunteers who experience poor sleep quality and a hectic lifestyle, IceAwake™ visibly rejuvenated the skin appearance by decreasing wrinkle depth and increasing skin radiance.

SWISS QUALITY PRODUCT

www.mibellebiochemistry.com

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

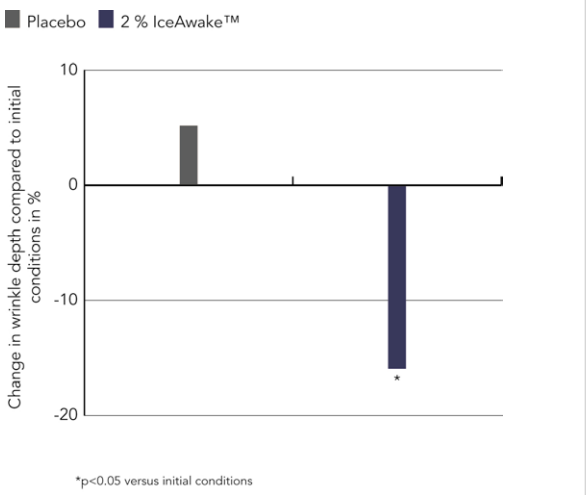


Fig. 5 Decrease of wrinkle depth upon treatment with IceAwake™.

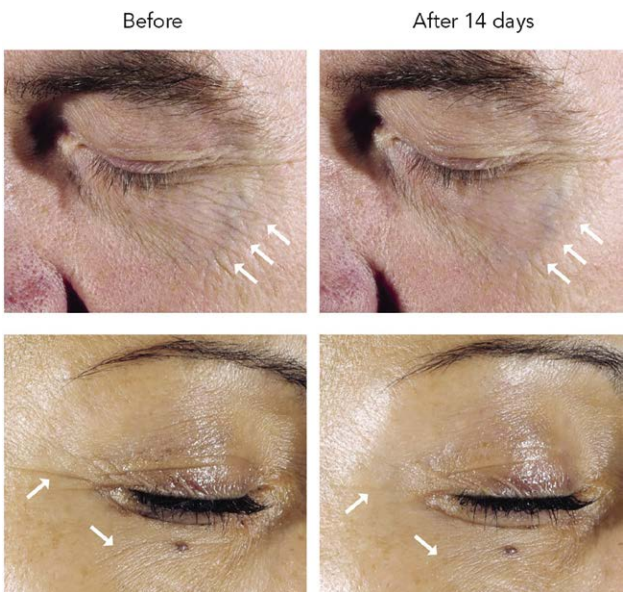


Fig. 7 Visible improvement of wrinkle depth and dark circles with IceAwake™.

After only 14 days of application of a cream containing 2 % IceAwake™ on Caucasian skin, the wrinkle depth of crow's feet was significantly reduced by 15.9 % compared to initial conditions (**Fig. 5**). Moreover, clinical-grade evaluation confirmed a significant reduction of visible facial tiredness compared to initial conditions, which was observed in 71 % of the volunteers (**Fig. 6**). The reduction in wrinkle depth as well as the improvement of tiredness were also visible in photographs of male and female volunteers (**Fig. 7**).

In a questionnaire about their lifestyle, the Asian volunteers enrolled in the second clinical study indicated to have late bedtime (56.5 % go to bed after midnight), sleep only very little hours (34.8 % sleep less than five hours per night) and with bad to very bad quality (95.6 %), while being faced with night overtimes or heavy workload at least once per week (60.8%). In this study population, the significant reduction of wrinkle depth following 14 days of treatment with a cream containing 2 % IceAwake™ was confirmed (data not shown). In addition, skin radiance significantly increased by 9.2 % compared to initial conditions and the placebo treatment (**Fig. 8**). Overall, these data demonstrate that after just 14 days of application, IceAwake™ rejuvenates tired skin by reducing wrinkle depth and visible signs of tiredness as well as increasing skin radiance.

Conclusion

The active ingredient IceAwake™ was developed using an extract of *Iodobacter ssp.*, a microorganism discovered in the soil under a retreating Swiss glacier. This extremophile bacterium has likely survived under permanent ice for centuries and is therefore an interesting source for an active ingredient to vitalize end energize tired skin. Mechanistic studies showed

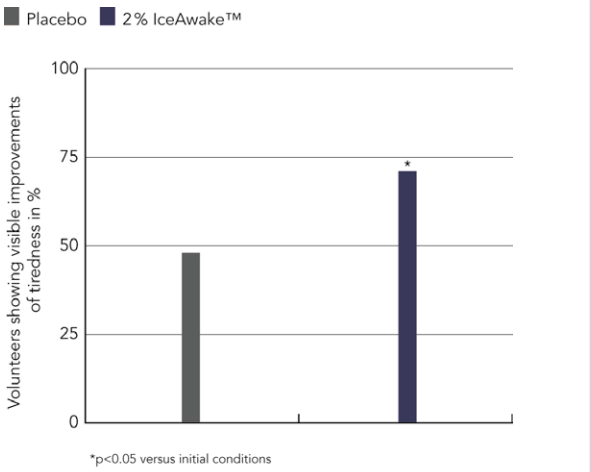


Fig. 6 Reduction of tired appearance upon treatment with IceAwake™.

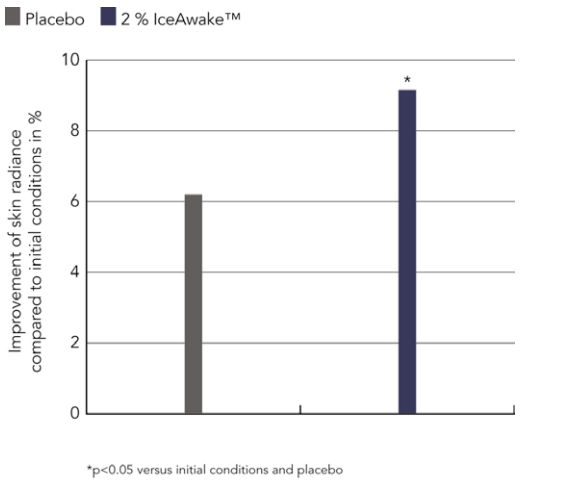


Fig. 8 Increase in radiance with IceAwake™.

that treatment of cells with *Iodobacter ssp.* extract leads to an upregulation of key chaperones involved in protein folding and thereby supports the removal of accumulated, misfolded proteins. The increased ATP levels following treatment with the *Iodobacter ssp.* extract additionally ensure the availability of the energy required for chaperone activity. Both mitochondrial and ER function are known to be impaired upon aging and sleep deprivation [3, 7, 8]. The results show that these impairments were improved in stressed cells upon treatment with *Iodobacter ssp.* extract. These findings translate to clinical studies, where it was found that application of a cream containing IceAwake™ leads to a significant and visible improvement of the wrinkle depth of crow's feet compared to a placebo cream. Moreover, the positive effect on tired and stressed skin was confirmed through clinical grading by experts who attested a significant improvement of skin tiredness and radiance after just two weeks of treatment with the cream containing IceAwake™. Regular application therefore leads to a visible skin rejuvenation and an increase in radiance despite a hectic lifestyle.

Formulations

Energizing Serum with IceAwake™ (see **page 66**).

References

[1] *P Walter and D Ron*, The Unfolded Protein Response: From Stress Pathway to Homeostatic Regulation. Science. 2011; 334(6059):1081-1086

[2] *MJ Gething,* Role and regulation of the ER chaperone BiP. Semin Cell Dev Biol. 1999; 10(5): 465-472

[3] *N Naidoo et al.*, Aging Impairs the Unfolded Protein Response to Sleep Deprivation and Leads to Proapoptotic Signaling. J Neurosci. 2008; 28(26):6539-6548

[4] *A Pickard et al.*, Preservation of circadian rhythms by the protein folding chaperone, BiP. FASEB J. 2019; 33(6): 7479-7489

[5] *N Naidoo et al.,* Sleep deprivation induces the unfolded protein response in mouse cerebral cortex. J Neurochem. 2005; 92(5):1150-1157

[6] *Naidoo et al.,* Aging and sleep deprivation induce the unfolded protein response in the pancreas: implications for metabolism. Aging Cell. 2014; 13(1): 131-141

[7] *RC Taylor,* Aging and the UPR(ER). Brain Res., 2016; 1648(Pt B): 588-593

[8] *AR van Vliet and P Agostinis*, Mitochondria-Associated Membranes and ER Stress. Curr Top Microbiol Immunol. 2018; 414: 73-102

[9] *S Chikahisa and H Séi,* The role of ATP in sleep regulation. Front Neurol., 2011; 2: 87

[10] *NJ Russell,* Psychrophily and resistance to low temperature. In: C Gerday and N Glansdorff (eds) Extremophiles (Life under extreme environmental Condition), Encyclopedia of Life Support Systems (EOLSS) Developed under the Auspices of the UNESCO. Eolss Publishers, Oxford, UK.

[11] *F Zakhia et al.,* Cyanobacteria in Cold Ecosystems. In: R Margesin et al., Psychrophiles: from Biodiversity to Biotechnology. Springer-Verlag Berlin Heidelberg, 2008; Chapter 8, 121-135

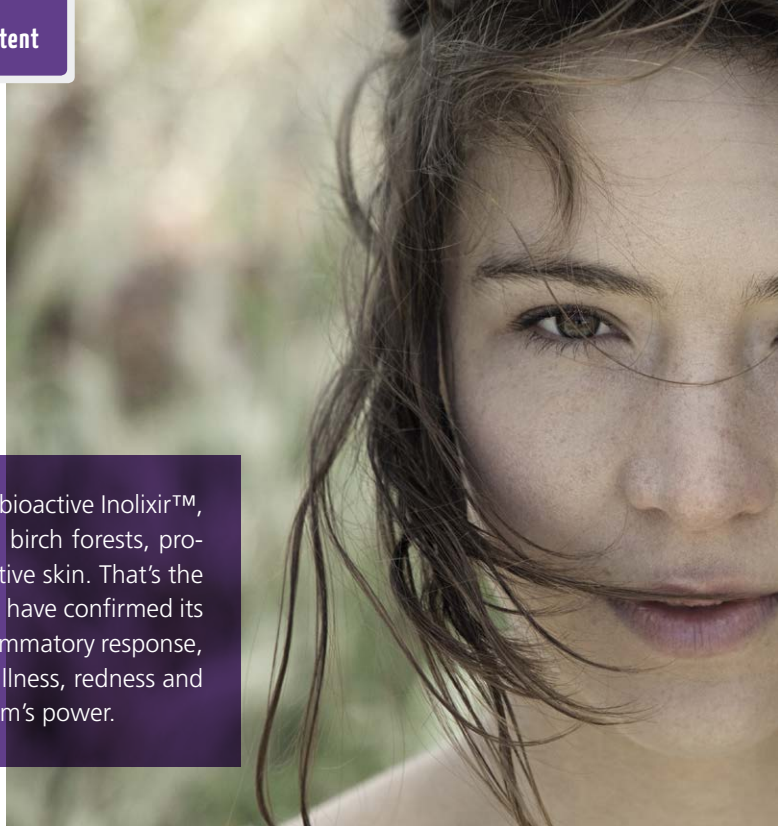
[12] *N Naidoo,* Cellular stress/the unfolded protein response: Relevance to sleep and sleep disorders.

contact

Dr. Christina Pickel
Dr. Franziska Wandrey
Dr. Daniel Schmid
Bernhard Henes
Dr. Fred Zülli

Mibelle Biochemie, Schweiz
Mibelle Biochemistry, Mibelle AG
Bolimattstrasse 1
5033 Buchs | Switzerland

Corresponding author:
Dr. Christina Pickel
Mibelle Biochemistry, Mibelle AG
Bolimattstrasse 1
5033 Buchs | Switzerland
E-mail: christina.pickel@mibellegroup.com



A Shot of Well-being for Healthy Skin

BASF's Inolixir™, a chaga mushroom extract, boosts tired and sensitive skin

P. Moussou, S. Leoty-Okombi, C. Boury, F. Trombini, V. Andre-Frei

From a centuries-old superfood to a "magic" ingredient: BASF's latest bioactive Inolixir™, a 100% natural extract of the chaga mushroom native to Canadian birch forests, promotes skin health with fast and long-lasting efficacy for tired and sensitive skin. That's the feedback from trial participants – and various *in vitro* and *in vivo* studies have confirmed its efficacy. By fortifying the skin's protection system and reducing pro-inflammatory response, it delivers visible results on fine lines and dark circles, as well as skin dullness, redness and comfort. A special extraction method is key to harnessing the mushroom's power.

Introduction

For at least half a millennium, people in Northern Eurasia have valued the chaga mushroom (*Inonotus obliquus*) for its various health benefits. Its nutritious conk – a dense black mass formed on the surface of birches and other host trees deep in Nordic forests [1, 2] – is traditionally consumed as a hot infusion in Eastern European countries, the Baltics and Russia to boost immunity and overall health [3, 4]. Thanks to the mushroom's unique biological properties, including antioxidant and anti-inflammatory characteristics, it can be used to treat stomach upsets and intestinal pain, satisfy hunger, and decrease tiredness. In North and Central Russian folk medicine, tinctures were also used for prophylaxis and for treating gastric disorders, and even cancer [5]. Based on its traditional uses in folk medicine and several scientific studies looking at its biological properties, the chaga mushroom has become a popular superfood in recent years. Online retailers stock powders and pills, tinctures and tea bags, capsules and even raw conks from the mushroom.

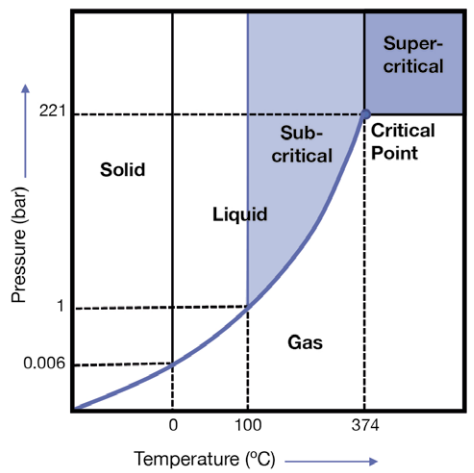


Fig. 1 Phase diagram of water as a function of temperature and pressure.

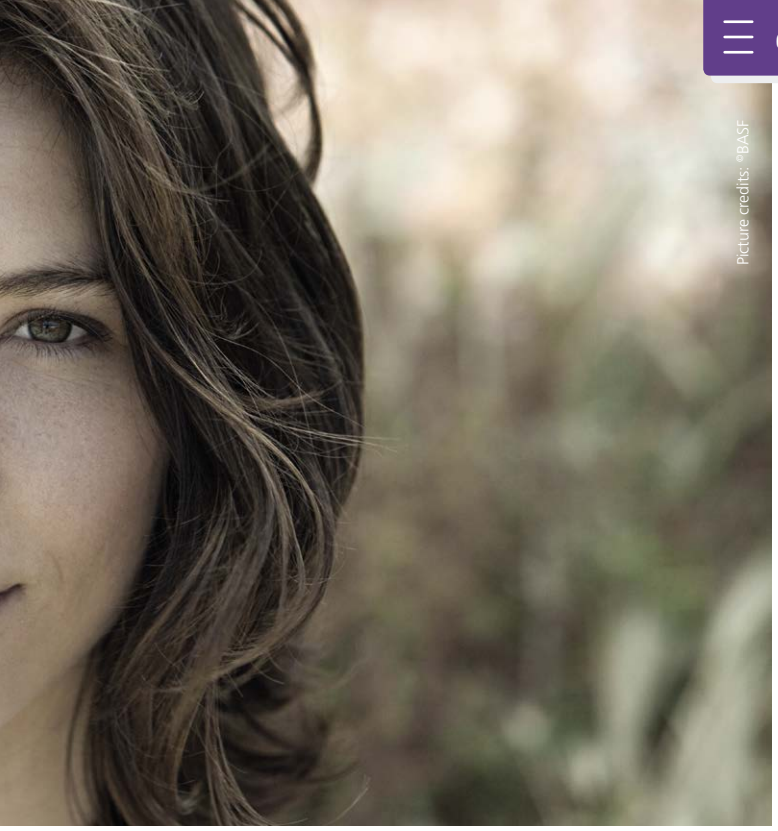
Now, BASF scientists have developed a novel bioactive ingredient from this remarkable mushroom: Inolixir™ (INCI: Glycerin (and) Water (and) Inonotus Obliquus (Mushroom) Extract) fortifies the skin's natural protection system by strengthening barrier function and the microvascular network – working on all fronts to return skin to a healthier condition. What's more, the active reduces the pro-inflammatory response thanks to its antioxidant and anti-inflammatory properties, with visible results on fine lines, dark circles, and skin dullness, redness and comfort.

Subcritical water extraction

A novel extraction process was used to harness the chaga mushroom's biological activity in skin care: Subcritical water extraction (SWE) uses superheated, pressurized water as an extraction solvent, instead of organic solvents. Water is in a subcritical state when it is above boiling temperature (100°C) but below critical temperature (374°C), and at a high enough pressure to keep it in liquid state (see **Fig. 1**). Subcritical water differs from cooler water in polarity, surface tension, and viscosity, making it suitable for the efficient extraction of polar and less-polar phytochemicals (see **Tab. 1**) [6].

When the water temperature ↑	
Dielectric constant ↓	Polarity ↓, similar to some organic solvents (e.g. MeOH at 200°C)
Surface tension ↓	Wetting of the raw material ↑
Viscosity ↓	Penetration inside the raw material and diffusion ↑

Tab. 1 Changes in extraction potential with the SWE process.


Picture credits: ©BASF

(involucrin x 4.9-fold), and epidermal cell cohesion as proven by an increased expression of the transmembrane proteins integrin β1 (+89%), claudin 1 (+20%) and occludin (+62%) (data not shown).

To measure the active ingredient's microvascular protection potential, BASF researchers proved that it stimulates collagen IV and VE-cadherin synthesis in endothelial cells. They also fed the proinflammatory cytokine TNFα into specially developed 3D reconstructed dermal sheets containing endothelial blood and lymphatic cells. The TNFα presence mimicked inflammatory stress, leading to an impairment of the microvascular network (measured by immunostaining of the microvascular wall structural protein CD31, see **Fig. 3**), whereas Inolixir protected the capillaries against stress-induced collapsing.

Finally, two hemi-face, double-blind, randomized, placebo-controlled clinical studies were performed to evaluate the benefits of the chaga extract for healthy skin.

An *in vitro* test proved the superiority of SWE compared to other common extraction methods for chaga. SWE was able to increase the polyphenols content by a factor of at least 1.6 compared to conventional water or hydroethanolic extraction (see **Fig. 2A**). At the same time, SWE demonstrates a better anti-inflammatory performance compared to hydroethanolic extract: SWE extract reduced the secretion of the pro-inflammatory signal protein IL-8 by human keratinocytes stimulated by a pro-inflammatory cocktail (see **Fig. 2B**).

Inolixir's efficacy on the skin has been proven in various *in vitro* and *in vivo* studies.

A strengthened skin barrier with well-protected vessels

In vitro, the chaga extract showed skin-barrier strengthening properties: It stimulated keratinocyte differentiation

A spa experience for tired skin

Thirty-two female volunteers aged 35 to 51 years took part in the first study. All of them were working, declared to be tired and stressed, and had dark circles under their eyes. They applied a facial cream containing Inolixir at 1% or a facial cream placebo twice daily for 28 days. The results of the study were compared

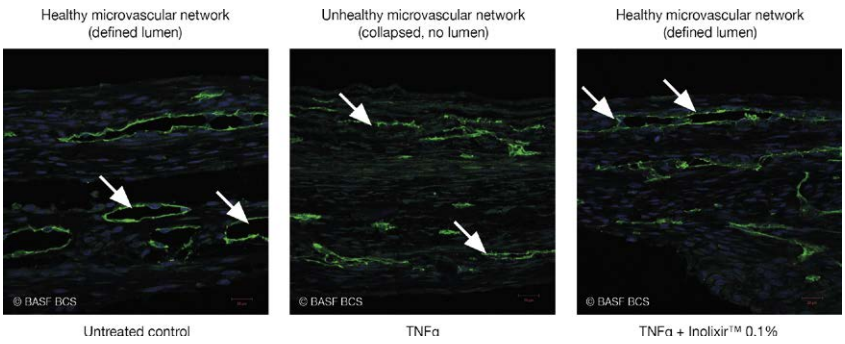


Fig. 3 Protection of the microvascular network in a 3D vascularized dermis model.

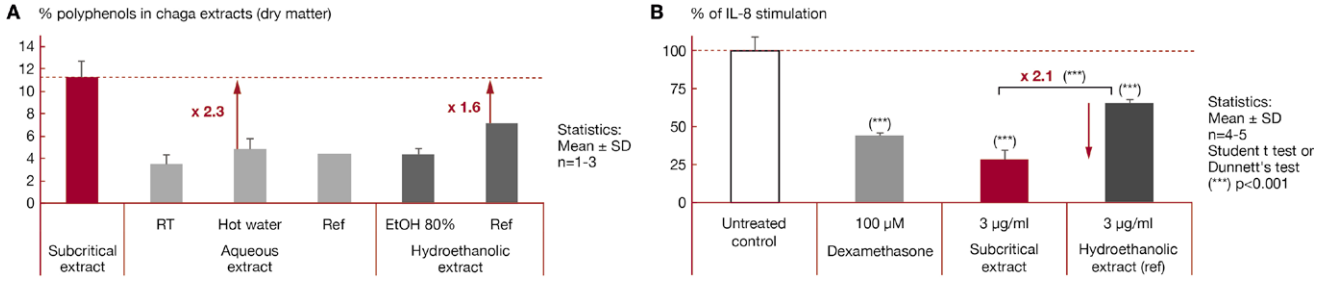


Fig. 2 A. Dosage of polyphenol content according to extraction methods. Ref are commercial extracts. RT: room temperature. B. Anti-inflammatory activity of extracts. Dosage of IL-8 (ELISA) on keratinocytes stimulated by a pro-inflammatory cocktail (TNFα, IFNγ and PolyI:C)

Fig. 4 Illustrative picture showing improvement in signs of fatigue (fine lines) with Inolixir™.

with those obtained after a relaxing five-day spa cure including facial sauna and massages. A majority of the volunteers said their dark circles appeared less dark (78%) and less large (75%) after 28 days, that their skin was more beautiful (63%) after 28 days, and that their facial features were less tense after 7 and 28 days of application (72% and 75% respectively). All these self-assessments significantly outperformed the placebo and were globally similar to the relaxation spa experience (see **Fig. 4**).

A wave of radiance and comfort for sensitive skin prone to redness

The second clinical trial involved 33 female volunteers aged 35 to 58 years with intolerant, reactive skin and facial redness. The efficacy of Inolixir at 1%, applied twice a day for 28 days, was evaluated by Chromameter measurement and self-assessment.

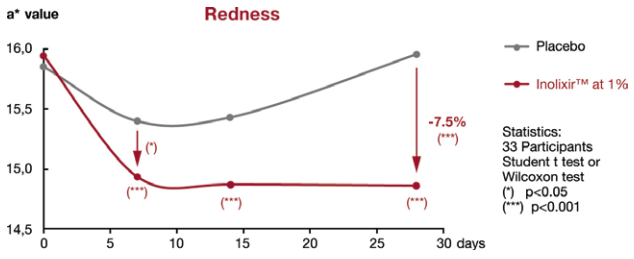


Fig. 5 Chromameter measurement of skin redness: a* value. Inolixir at 1% versus placebo.

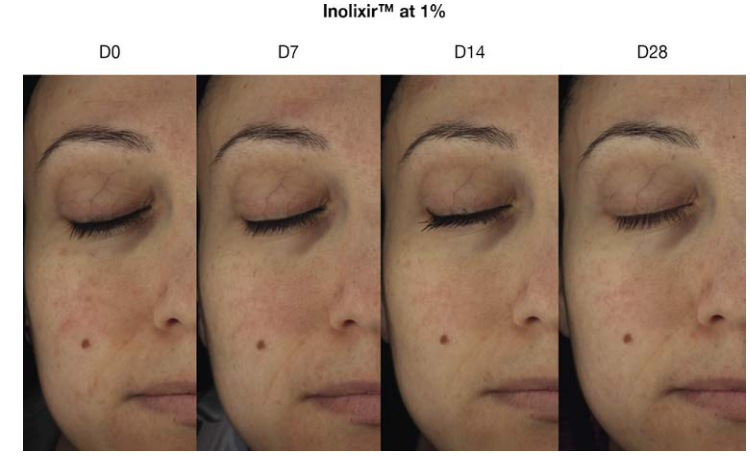


Fig. 6 Illustrative picture showing decrease in facial redness with Inolixir.

The active ingredient demonstrated a significant reduction in cheek redness (chromameter, a* value) after one week compared to baseline (D0) and the placebo (**Fig. 5** and **Fig. 6**). The redness did not appear again during the 28-day treatment, whereas no improvement was observed with the placebo over time. A similar significant improvement in skin brightness (chromameter, L* value) was also observed. The volunteers themselves also noticed this improvement after 28 days: a significant majority agreed that their skin redness was reduced (73%), their skin felt soothed (88%), looked and felt healthier (85%) and was more comfortable (91%).

Conclusion

The chaga mushroom's biological activity can be harnessed efficiently for skin care using eco-friendly subcritical water extraction technology. The new highly efficient extract Inolixir responds to the needs of personal care manufacturers to offer customers green, 100% natural solutions.
It offers genuine, visible benefits, promoting skin health with a dual action approach that delivers quick and long-lasting results for tired and sensitive skin.

Formulations

Relaxing face cream (SC-FR-19-BC-50810) (see **page 67**)

References

[1] *Lee MW, Hur H, Chang KC, Lee TS, Ka KH, Jankovsky L,* Introduction to Distribution and Ecology of Sterile Conks of Inonotus obliquus, Mycobiology 36(4), (2008), 199-202.
[2] *Balandaykin ME, Zmittrovich IV.* Review on chaga medicinal mushroom, Inonotus obliquus (higher Basidiomycetes): realm of medicinal applications and approaches on estimating its resources potential, Int J Med Mushrooms, 17(1), (2015), 95-04.
[3] *Shikov AN, Pozharitskaya ON, Makarov VG, Wagner H, Verpoorte R, Heinrich M,* Medicinal Plants of the Russian Pharmacopoeia; their history and applications. Journal of Ethnopharmacology, 154, (2014), 481-536.
[4] *Glamoclija, J, Ciric A, Nikolic M, Fernandes A, Barros L, Calhelha RC, Ferreira ICFR, Sokovic M, Griensven LJLD,* Chemical characterization and biological activity of Chaga (Inonotus obliquus), a medicinal "mushroom", Journal of Ethnopharmacology 162, (2015), 323–332.
[5] *Shashkina MY, Shashkin PN, Sergeev AV,* Chemical and medicobiological properties of Chaga (Review), Pharmaceutical Chemistry Journal, 40(10), (2006), 37-44.
[6] *Cvjetko Bubalo M, Vidovic S, Radojcic Redovnikovic I, Jokic S,* New perspectives in plant extraction of biologically active compounds by green solvents, Food and Bioproducts Processing, 109, (2018), 52-73.

contact

Philippe Moussou | philippe.moussou@basf.com
Sabrina Leoty-Okombi | sabrina.leoty-okombi@basf.com
Carole Boury | carole.boury@basf.com
Florence Trombini | florence.trombini@basf.com
Valérie Andre-Frei | valerie.andre-frei@basf.com

BASF Personal Care and Nutrition GmbH
Rheinpromenade 1 | 40789 Monheim | Germany
www.carecreations.basf.com

Visit us at **in-cosmetics 2020** in Barcelona
March 31 to April 2, **Booth K50**

For **Anna, Naomi,** and **Carl**, living in the city and being connected to nature are not mutually exclusive. They live and love a green life.

BASF
We create chemistry

Care Creations®

Their stories. Your products. Our commitment.

Life-loving consumers want to trust in a bright and life-friendly future. To satisfy these inner needs we are deeply committed to **achieving greater sustainability.** BASF's Care Creations® will support your sustainability strategy with ingredients that are ecologically sourced, conserve resources or help avoid negative environmental or social impact. Promise.

Every story matters.
carecreations.basf.com/sustainability

Towards a Sustainable Solution for Skin Health and Well-being

H. van der Hoeven, H. Prade

abstract

A novel cosmetic active ingredient, based on the fruit of *Annona cherimola*, here called Cherimoya (trade name: AnnonaSense CLR™), has shown to potently activate the skin's endocannabinoid system. It improves skin health and well-being and potently reduces skin sensitivity.

From CBD, via the endocannabinoid system to *Annona cherimola* fruit extract

CBD (Cannabidiol) is enjoying lots of attention in the cosmetic industry, currently. Is CBD worth the hype? There is still a lack of relevant and valid efficacy studies for its application for the cosmetic industry. Another potential concern, as was expressed by the American FDA, for instance, is the lack of safety data for CBD. CBD is truly a hype and, as is often the case with hypes, this looks to be largely irrational. Despite the fact that consumers are increasingly demanding and realistic, they still largely act irrationally. In the search of the next 'hope in a jar', CBD is appealing to them.

Our body's endocannabinoid system (ECS) is a biological system which safeguards our bodily homeostasis and health [1]. The ECS is strongly represented in our skin and supports the skin in dealing with daily stress [2]. Being on the periphery of our body, the skin is pre-eminently THE part of our body which is constantly in contact with outside stresses and is continuously trying to stay in homeostasis and biological balance to be able to perform its job as a physical and immunological barrier. The ECS is of eminent importance for our skin to be successful in doing so. Positively influencing and potentiating the skin's ECS creates a large opportunity for the cosmetic industry, therefore.

CBD is said to interact with our body's ECS, but is CBD the way going forward, though? Should our skin's ECS not be more in the focus than CBD? CBD is just a molecule, the ECS is one of the most important biological systems in the human body.

The Cannabinoid Type-2 (CB2) receptor plays an essential role in the ECS [3]. Virtually all cells in our skin show, on their outer membrane, CB2-receptors. These receptors interact with endocannabinoids, peptides which are produced in our body, to act on the ECS in the skin and ensure a healthy homeostasis. It is opportune to find natural molecules, which are proven to be safe and, from a regulatory point-of-view, can be used without hesitation, which have a potent agonistic effect on CB2 and, through this action, potentiate and support the ECS. With that, skin becomes more resilient, healthier and overall well-being of the consumer will be ensured.

The extract of the fruit of Annona cherimola, which is clearly distinct from Cannabis sativa and cannabidiol, shows potent agonistic effect on the CB2 receptor. CLR Berlin GmbH has developed AnnonaSense CLR™ (INCI: Annona Cherimola Fruit Extract, "Cherimoya") where its agonistic action on CB2 was proven to be extremely potent. *In vitro* and *in vivo* studies are shown below.

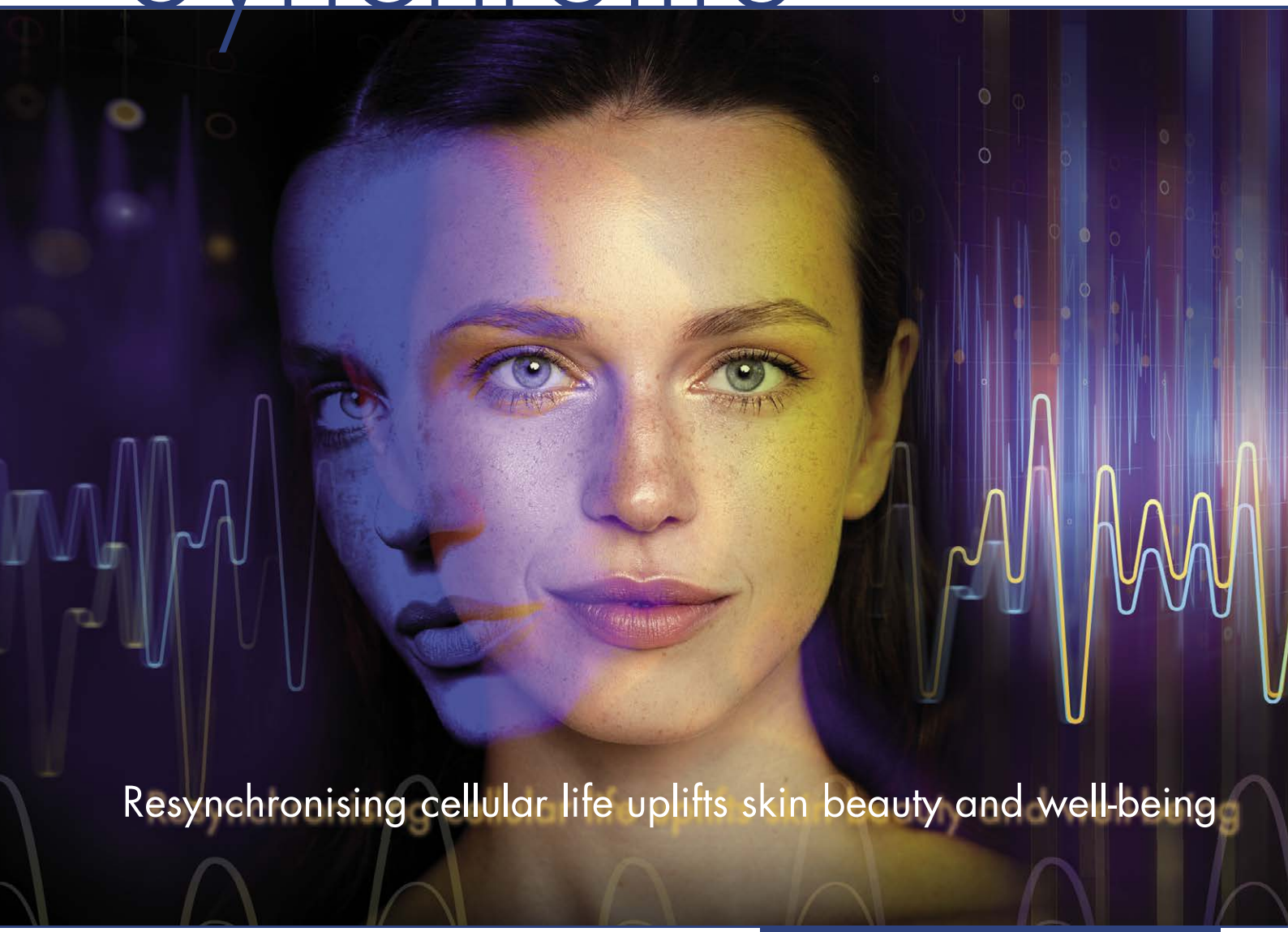
In search of homeostasis, health and well-being

In order to appreciate the importance, but also the complexity of our body's ability to manage 'health' the ECS needs to be put into the context of our body's EVS (Endovanilloid system). The activation of the EVS is largely detrimental [4], inducing inflammatory processes and an extremely important player in loss of health, the ECS can be considered to be its counterpart [5].

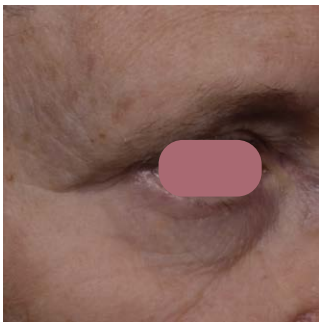
The ECS and EVS are in close contact and this is clearly visible in our skin. Keratinocytes, which make up more than 90% of the top layer of skin, the epidermis, show CB2-receptors but also cellular receptors important for the EVS. TRPV1 (Transient receptor potential channel, vanilloid subfamily member 1) plays a major role in the EVS [6]. TRPV1 can be triggered by heat, UV-radiation, free radicals and pH shifts, among others. Activation of TRPV1 triggers the production of inflammatory mediators which are important in skin inflammation. Activation of TRPV1 on sensory neurons, the cells which make up the sensory nerves in our skin, can lead to itch, pain and burning sensations. TRPV1 is well-described to be one of the key instigators of loss of skin health and well-being [7].

The ECS seems designed to keep the EVS under control and to establish and maintain a healthy situation. Nature has found a tremendously elegant way of managing this through

Synchrolife™



Resynchronising cellular life uplifts skin beauty and well-being



Before

volunteer #30

After **8 weeks** applying **Synchrolife™** eq.2 %

- Realigns circadian rhythms.
- Counteracts the effects of digital pollution.
- Erases signs of fatigue.
- Delivers neuro-beauty benefits with stronger self-esteem and an overall well-being.
- IECIC listed peptide-based active ingredient.

EUROPE - Sederma SAS Tel +33 1 34 84 10 10 email sederma@sederma.fr www.sederma.com
Sederma GmbH Tel +49 21 57 817318 email sederma@sederma.de www.sederma.com
NORTH AMERICA - Sederma Inc. Tel +1 732 417 0800 email sederma-usa@croda.com www.sederma.com
ASIA PACIFIC email pc-asia@croda.com www.sederma.com

Smart Science to Improve Lives™

Follow us on

Scan the code for free* sample Part of Croda international Plc.

*Samples will be sent at Sederma's discretion

the action of CB2; activation of CB2 reduces the activity of TRPV1 [8] and, therefore, leads to a reduction of the negative downstream effects of the activation of TRPV1. This leads to recovery and maintenance of the healthy homeostasis in skin.

EFFICACY STUDIES – *in vitro* assays

Below, the results of the studies relevant to CB2 and TRPV1 are presented in detail. Other *in vitro* studies were performed additionally. It was shown that Cherimoya reduced histamine-induced production of Calcitonin gene-related peptide (CGRP) and the production of Interleukin-6 (IL-6) after application of Tumor Necrosis Factorα (TNFα) and Substance P. These studies were performed on primary human keratinocytes. Another study on these cells was performed to determine the activity of Cherimoya on the production of the Interleukin-31 receptor (IL-31RA). Here it was shown that Cherimoya could strongly reduce the production of IL-31RA, further underlining its importance for reducing skin discomfort [9].

Agonistic effect of Cherimoya on CB2

293T-CB2 cells (CB2-expressing human epidermal keratinocytes) were incubated with different concentrations of Cherimoya, respectively a positive control (WIN55, 212-2 [1 µM]) for 10 minutes. The positive control is a specific agonist for CB2 and can, therefore, induce specific cellular processes induced by CB2. Subsequently the cells were treated with Forskolin (10 µM) for 6 hours. Treatment with Forskolin led to an increase in the production of cyclic adenosine monophosphate (cAMP). Interaction with CB2 by the positive control substance and the different concentrations of Cherimoya had an impact on the Forskolin-induced cAMP production. CB2-induced reduction of Forskolin-induced cAMP production was determined, where the result obtained with the positive control was set at 100%.

Results

At two different concentrations, treatment with Cherimoya showed a strong agonistic effect on the CB2 receptor, where the higher concentration of the two, 0.05%, was even able to outperform the positive control, a specific agonist for CB2 (**Fig. 1**).

Reduction of TRPV1-induced inflammation, co-cultivation of keratinocytes – sensory neurons

cAMP is needed for TRPV1-activity. CB2-induced reduction of cAMP, as shown in the above study, is therefore a potent approach towards deactivation of TRPV1. A CB2-induced reduction of the activity of TRPV1 can elegantly be proven by making use of capsaicin. Capsaicin is a specific agonist for TRPV1 [10] and the determination of changes of capsaicin-induced cellular processes through this pathway was the aim of this study. Primary human keratinocytes were co-cultivated with sensory neurons and incubated with Cherimoya at 0.1%

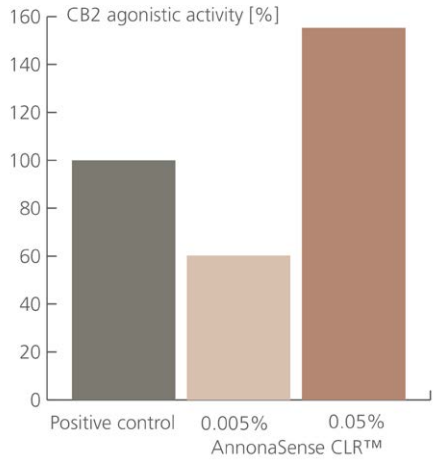


Fig. 1 Agonistic influence on CB2.

and with capsazepine (10 µM), respectively, as an antagonist for TRPV1 for 24 hours. Subsequently capsaicin (10 µM) was applied for 60 minutes. Expression of TRPV-1 induced IL-1β, IL-8 and CGRP was determined.

Results

Reduction of Interleukin-1β (IL-1β), Interleukin-8 (IL-8) and CGRP by positive control (capsazepine) was set at 100%. Reduction of expression of all three inflammatory mediators induced by Cherimoya was close to 80% of what was achieved with the positive control (**Fig. 2**). This clearly illustrates the potency of Cherimoya to reduce the activity of TRPV1 through its action on CB2.

Reduction of TRPV1-induced inflammation, keratinocytes

A similar experiment was performed only using primary human keratinocytes, with IL-8 as the marker. Primary human keratinocytes were cultivated with Cherimoya for 24 hours,

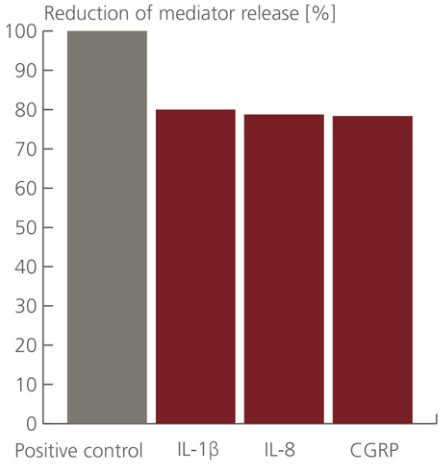


Fig. 2 Influence on IL-1β, IL-8 and CGRP release.

after which capsaicin (0.1 µM) was applied. IL-8 expression was measured at baseline and after treatment with capsaicin, with or without pre-incubation with Cherimoya.

Results

IL-8 production at baseline (untreated) was set at 100%. Treatment with capsaicin led to a 62.5% increase of the production of IL-8. Pre-incubation with Cherimoya, however, led to a clear decrease of IL-8 production, even lower than at baseline and even for the lowest concentration of Cherimoya used (**Fig. 3**).

EFFICACY STUDIES – *in vivo* studies

From the *in vitro* studies it can be concluded that Cherimoya should promote skin health and well-being. Healthy skin, having the ability to stay in homeostasis, is not sensitive, not overly reactive. An objective evaluation of the effect of Cherimoya on the sensitivity/reactivity of skin is therefore one of the prerequisites for providing proof of Cherimoya providing better skin health and well-being.

A growing number of people suffer from skin ailments [11]. Approximately 50% of people perceive their skin to be sensitive. At some point, virtually everybody suffers from skin irritations, redness and itch. In order to provide sufficient proof for Cherimoya to work effectively in daily life, the *in vivo* studies needed to be performed on volunteers who were representative of the general public, of people who want their skin to be healthier. For the subsequent *in vivo* studies more than 40 volunteers were included who reported their skin to be sensitive and dry. Twelve volunteers had atopic dermatitis, eight had type IV allergy, one had psoriasis, and one had diabetes type

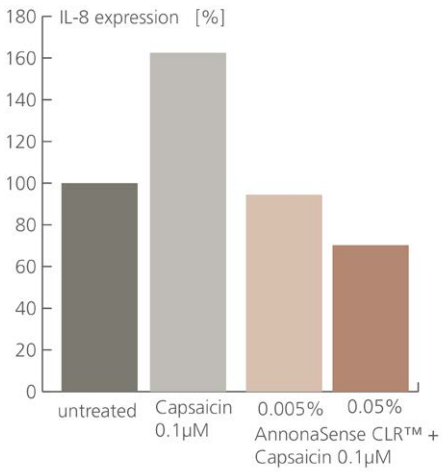


Fig. 3 Influence on IL-8 release.

II. As the general consumer searching for better skin health and well-being, the volunteers showed a regular feeling of skin discomfort (e.g., itch). Both women and men were included, and the age range of the volunteers was 18–70 years.

Assessment of skin sensitivity

One application of test product on 17 (for 250 Hz) and 18 (for 5 Hz) volunteers (including 5 with atopic dermatitis, 3 with type IV allergy, 6 with sensitive skin, 1 with diabetes type II). Skin sensitivity was determined by measuring current perception threshold (CPT) with Neurometer® CPT/C (Neurotron Inc., Baltimore, USA) [12] at 250 Hz and 5 Hz before and 30 minutes after application of the test product. Baseline at t=0 is set at 0%. Values are based on averages of individual results of all volunteers.

Results

The 250 Hz experiment showed that the formulation with Cherimoya outperformed the corresponding placebo formulation by more than 35%. The 5 Hz experiment showed even clearer results: the formulation with Cherimoya performed more than 60% better than the placebo formulation (**Fig. 4**). These results show that Cherimoya clearly makes skin less sensitive.

Skin discomfort

One application of test product after occurrence of itch. 22 volunteers tested formulation with Cherimoya, 20 tested placebo formulation. Itching was scored at t=0, 1 min, 5 min and 24 hrs. Baseline at t=0 is set at 0%. The volunteers were not aware of the nature of the product which they applied on their skin.

Results

Instant, but especially long-lasting relief of itch is an important trait for a cosmetic formulation aimed at sustainably increasing the consumer's health and well-being. Placebo formulations can relieve up to 40% of perceived itch without having any biological effect whatsoever [13]. It is, therefore, important to prove with Cherimoya that it can lead to a relief of itch which is considerably greater than 40%. In this study this was clearly proven. Where the effects of the formulation containing Cherimoya were better than corresponding placebo after 1 and 5 minutes after applying the products, the effect after 24 hours was clearly larger than that of placebo. As could be expected, the placebo formulation was able to reduce itch by almost 37%. The same formulation additionally containing Cherimoya outperformed the placebo formulation by almost 18%, showing an overall reduction of itch by almost 55% (**Fig. 5**).

Skin appearance

In a double-blind test, 22 volunteers tested a formulation with Cherimoya and 20 tested a placebo formulation. Redness was scored at t=0 and after 17 days of twice daily application of test products. Baseline at t=0 is set at 0%.

Results

An unhealthy appearance of skin can have important negative consequences for a person's well-being, i.e. quality of life [14]. Reduction of skin redness in relation to skin health is therefore an important parameter to consider. In this study, 17 days of application of the test substance showed that the formulation with Cherimoya improved skin appearance by reducing redness due to scratching itchy skin. Where the placebo formulation reduced skin redness by 25%, the corresponding formulation containing Cherimoya clearly outperformed the placebo formulation, by reducing redness by more than 36% (**Fig. 6**).

Perception of skin health and well-being

Volunteers (n=22) who applied Cherimoya (3% in an aqueous gel) were given a questionnaire after 17 days of twice daily application.

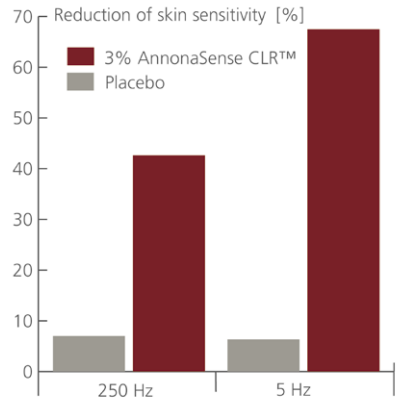


Fig. 4 Assessment of skin sensitivity.

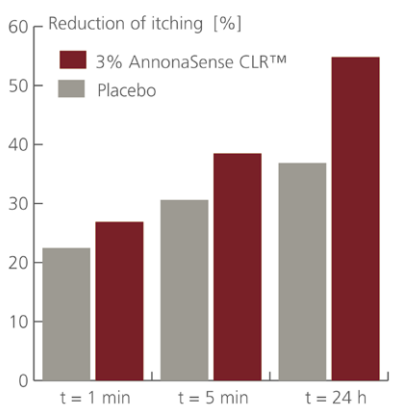


Fig. 5 Assessment of skin discomfort.

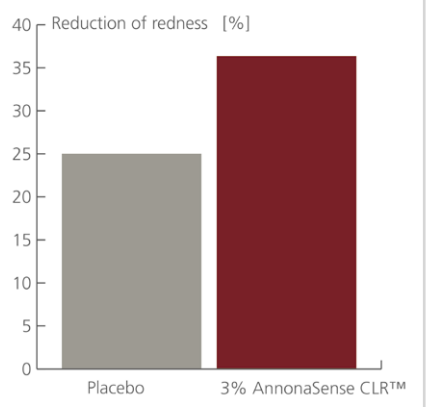


Fig. 6 Assessment of skin appearance.

Since 1997

Fine Chemicals and More...

SLI CHEMICALS

Welcome ! in-cosmetics
Booth no. C10

SLI Chemicals GmbH • info@slichemicals.com • www.slichemicals.com

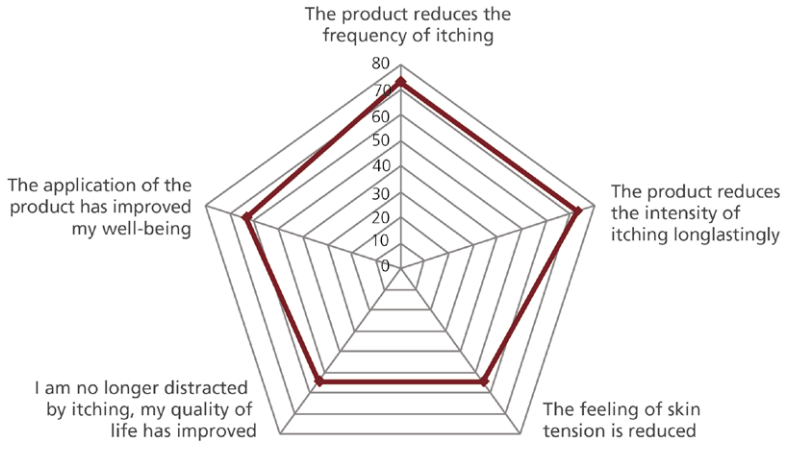


Fig. 7 Perception of skin health and well-being.

Results

In this last study consumers were questioned on whether a simple aqueous gel, showing minimum sensory and biological properties, containing 3% Cherimoya led to improvement of skin health and well-being. After 17 days of daily use of the product the consumers found that both the intensity and the frequency of itching were reduced by almost 73%. A total of 54.5% of the users confirmed that the feeling of tension in their skin – a phenomenon often encountered by people with dry and unhealthy skin – was perceivably reduced. The same number of users reported that the quality of their life was improved, and almost 64% of test persons stated that using this product had improved their well-being after only 17 days (**Fig. 7**).

Conclusion

Cherimoya is a product based on the fruit of Annona Cherimola. Cherimoya acts on the Endocannabinoid System (ECS) and, with that, reduces the activity of the Endovanilloid System (EVS). It establishes a stable homeostatic balance, which is the key to skin health. Mechanistically, Cherimoya activates the endocannabinoid receptor CB2. CB2 is pivotal for the ECS in the skin. With this action the activity of the main receptor of the EVS, TRPV1, is reduced. Reduction of the activity of TRPV1 is essential for reduction of inflammatory processes in the skin. Through Cherimoya a stable balance between the ECS and EVS is achieved.

Cherimoya leads to an improvement of skin health and well-being, which is both measurable and perceivable. This was proven in a variety of *in vivo* studies. The skin becomes less sensitive and more balanced, and even itch could, over a longer period of time, be perceivably reduced. Skin appearance was improved, and a consumer study showed that perception of well-being and quality of life were improved.

References

[1] *V Di Marzo et al.*, Trends Pharmacol. Sci. 27 (3) (2006), 134-140. A brief history of cannabinoid and endocannabinoid pharmacology as inspired by the work of British scientists

[2] *C del Rio et al.*, Biochem. Pharmacol. 157 (2018), 122–133. The endocannabinoid system of the skin. A potential approach for the treatment of skin disorders.

[3] *C Turcotte et al.*, Cell. Mol. Life Sci. 73 (2016), 4449–4470. The CB2 receptor and its role as a regulator of inflammation

[4] *YM Lee et al.*, Exp. Dermatol. 18 (2009), 431–436. Increased expression of TRPV1 channel in intrinsically aged and photoaged human skin *in vivo*

[5] *K Starowicz et al.*, Phil. Trans. R. Soc. B 367 (2012), 3286–3299. Modulation of neuropathic-pain-related behavior by the spinal endocannabinoid/endovanilloid system

[6] *SM Kang et al.*, J. Dermatol. Sci. 88(1) (2017),126-133. A synthetic peptide blocking TRPV1 activation inhibits UV-induced skin responses

[7] *JH Lee et al.*, J Dermatol. Sci. (2018), pii: S0923-1811(18)30204-4. A novel, topical, nonsteroidal, TRPV1 antagonist, PAC-14028 cream improves skin barrier function and exerts anti-inflammatory action through modulating epidermal differentiation markers and suppressing Th2 cytokines in atopic dermatitis

[8] *NA Jeske et al.*, J. Biol. Chem. 281 (2006), 32879–90. Cannabinoid WIN 55,212–2 regulates TRPV1 phosphorylation in sensory neurons.

[9] *IS Bağci et al.*, J. Allergy Clin. Immunol. 141 (2018), 858–866. IL-31: A new key player in dermatology and beyond.

[10] *U Anand et al.*, Pain 138 (2008), 667–680. Cannabinoid receptor CB2 localisation and agonist-mediated inhibition of capsaicin responses in human sensory neurons

[11] *RJ Hay et al.*, J. Invest. Dermatol. 134 (2014), 1527–1534. The global burden of skin disease in 2010: An analysis of the prevalence and impact of skin conditions.

[12] *SJ Kim et al.*, Int. J. Cosmet. Sci. 30(5) (2008), 333-337. The perception threshold measurement can be a useful tool for evaluation of sensitive skin

[13] *AIM van Laarhoven et al.*, J. Invest. Dermatol. 135 (2015), 1234–1243. Placebo Effects on Itch: A Meta-Analysis of Clinical Trials of Patients with Dermatological Conditions

[14] *N Samson et al.*, Int. J. Cosmet. Sci. 32 (2010), 167–184. Visible skin condition and perception of human facial appearance

authors

Harald van der Hoeven
Director Technical Marketing
Dr. Heiko Prade
Director R&D

Chemisches Laboratorium
Dr. Kurt Richter (CLR) GmbH
Berlin | Germany

Forum Cosmeticum 2020

12–13 May
Berlin

www.dgk-ev.de/forum-cosmeticum

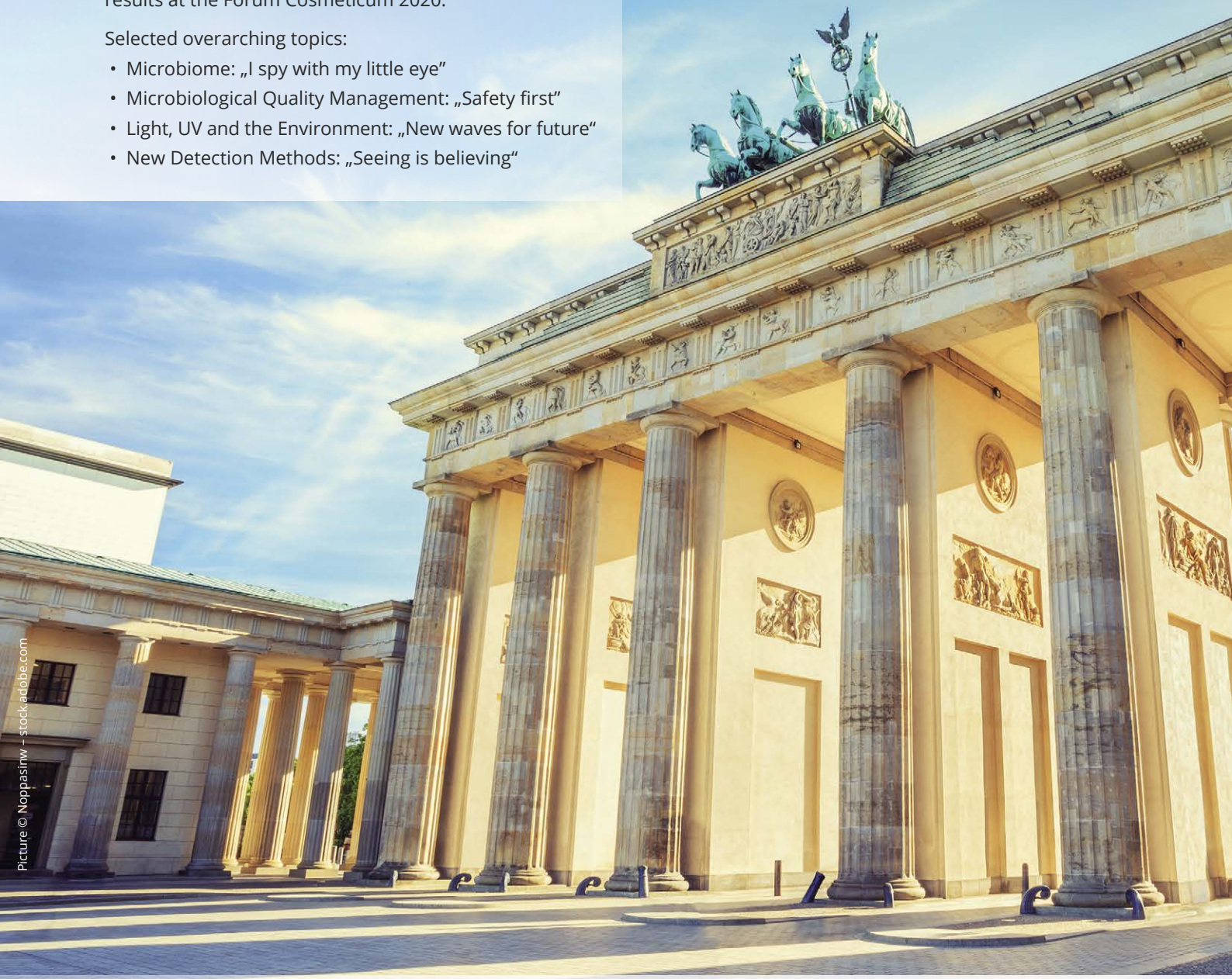
Registration now open

Uncover the Invisible

Learn about the latest scientific research and results at the Forum Cosmeticum 2020.

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"



Picture © Noppasinw - stock.adobe.com

Direct contact:
info@dgk-event.de

Forum Cosmeticum is an event together with

Organized by
DGKeVent GmbH

Clay-like Ingredient for a Better Skin and Well-being

J. Comas, O. Laporta, E. Cañadas, A. Soley, R. Delgado

abstract

Clay-based skin care treatments are one of the oldest skin care treatments still being used today by many consumers. They are known to present multiple cosmetic benefits, as well as well-being-enhancing properties, becoming an ideal ally for people living in busy and urban environments. However, these ancient treatments are also asked to be reinvented to bring fresh air to the market. Uniclay™ biotech ingredient is a fermentation-based extract derived from a clay microorganism that mimics the effects of clays on the skin, offering a cleaner, smoother and more beautiful skin for all ethnicities and improved sense of well-being. The ingredient can also be incorporated into all types of skin care formulations, bringing renovated applications to consumers.

Let's talk beauty

It is hard to define beauty, but it is clear that the standard perception of beauty is shifting towards a more inclusive culture and mindset. Skin care brands are starting to celebrate differences by emphasizing diversity and by offering products specific for different skin types or universal skincare products that represent all. Furthermore, skin care products are not only intended to beautify one's skin, but also to make people feel comfortable in their own skin, making beauty go beyond the physical appearance and starting to focus more on the emotional part and well-being of consumers.

The wellness products designed to promote positive emotions, relaxation or comfort are also expected to be aligned with the clean beauty movement. Clean formulations are considered to be simple and to contain only the necessary ingredients, being these preferably natural and responsibly sourced; they have a strong focus on safety, for both the consumer and the environment; they should contain ingredients with proven efficacy, and they should be transparent in revealing what the product contains and where the ingredients come from.

This clean beauty movement is experiencing a quick growth, most likely due to the rise in environmentally-conscious consumers around the world.

The magic of clays

Muds or clays are commonly used therapeutically, especially in balneotherapy or therapeutic bathing due to their powerful benefits. They are known to offer a wide range of benefits to the skin, as well as emotional benefits, becoming one of the best natural treats to enhance skin health, beauty and well-being.

Clay masks are mainly well-known for their natural purifying properties. A purified skin is often associated to a clean skin free from impurities. One of the markers used to indicate the degree of skin purity is the amount of bacterial porphyrins present on the skin. Skin porphyrins are metabolic products of *Propionibacterium acnes* (*P. acnes*) that can become lodged in pores and can induce the oxidation of sebum, leading to comedogenesis and acne [1]. Therefore, a decrease in porphyrins would be linked to a cleaner skin with less undesired bacteria. Porphyrins can also contribute to the inflammatory reaction around the follicles, which aggravates the presence of inflammatory imperfections [2].

Other benefits associated to clay masks are antioxidant, skin health promoting and smoothing effects, which help obtain a better and younger-looking skin. A strong antioxidant effect helps cope with the reactive oxygen species (ROS) generated endogenously or exogenously by agents such as xenobiotics and UV radiation, for a more protected and younger skin. The health promoting effect would lead to healthy skin cells, meaning that they have properly functional mitochondria with a higher oxygen consumption rate compared to unhealthy cells. Finally, by improving the skin regeneration and healing capacity, unwanted lesions, scars and imperfections on the skin surface can be minimized for a smoother and softer complexion.

Nevertheless, clay masks are also associated with an enhanced sense of well-being. The treatments with clays are usually linked to self-care, since it is known that when we take time for ourselves, we tend to generate emotions that make us feel better, which leads to a greater emotional well-being.

Addressing three current beauty trends: wellness-driven beauty, inclusivity and clean beauty

Uniclay™ biotech ingredient (INCI name: Glycerin, Water (Aqua), Bacillus Ferment) is a biotechnological ingredient

obtained from a microorganism isolated from a clay close to a natural park in the North-Eastern Mediterranean coast of Spain, home of a great variety of plant and animal species. The isolation process of the *Bacillus sp.* from the clay was performed following sustainable practices, which did not involve harvesting high amounts of materials from nature. Furthermore, the ingredient also offers a return to nature, since Lubrizol sponsored a project with a local NGO dedicated to the preservation of the biodiversity in the native region where the microorganism grows.

Supported by several *in vitro* and *in vivo* tests, the ingredient showed to mimic the effects of clays on the skin and to offer a cleaner, smoother and more beautiful skin for all ethnicities and improved sense of well-being.

P. acnes biofilm formation

This test was performed to evaluate the ability of the biotechnological ingredient to inhibit the biofilm formation of *P. acnes*, whose role is gaining prominence in the pathogenesis of acne vulgaris.

P. acnes in culture medium was incubated with 10 µg/mL Bacillus Ferment for 24 hours. Then, plates were washed to remove the bacteria not creating biofilm and were treated again with the active ingredient for 48 hours. Finally, plates were washed again, and the viability of *P. acnes* biofilm was evaluated through an XTT assay.

P. acnes biofilm formation was reduced by 54.5% at the end of the treatment, which could help prevent acne.

Reduced inflammatory response to *P. acnes*

The aim of this test was to evaluate the ability of the active ingredient to reduce the skin inflammatory response in the presence of *P. acnes*.

Human keratinocytes were treated with 10 µg/mL Bacillus Ferment with the presence of 100 µL of *P. acnes* suspension for 24 hours and, interleukin-8 (IL-8) production was assessed by enzyme-linked immunosorbent assay (ELISA).

At the end of the active treatment, IL-8 was significantly reduced by 14.2% ($p<0.01$), helping alleviate acne-prone skin.

Enhanced skin regeneration

The skin regenerative capacity of the active ingredient was evaluated through a cicatrization test.

Human keratinocytes were treated with 10 µg/mL Bacillus Ferment for 24 hours. After performing a mechanical wound on the cells, keratinocytes were treated again with the active ingredient and migrated for 18 hours. Skin cell regeneration activity was evaluated by measuring the wound area of before and after images. (**Fig. 1**)

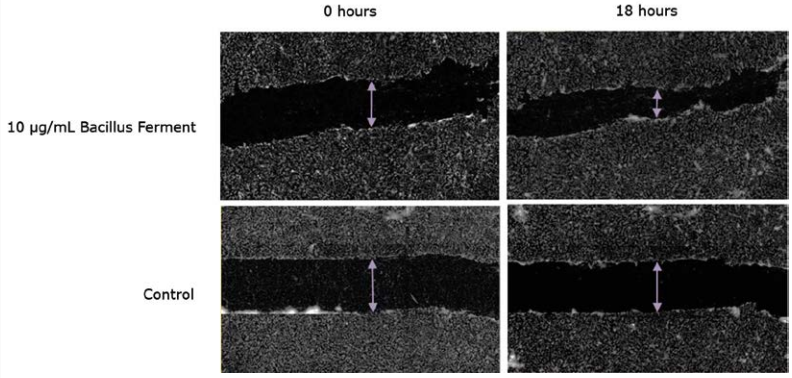


Fig. 1 Mechanical wound in human keratinocytes. The purple arrows show the empty area, corresponding to the injured region.

At the end of the active treatment there was a significant 54.2% regeneration of damaged skin cells culture, suggesting a fast skin repairing effect.

Cleansing effect

The ability of the biotechnological ingredient to clean the skin, by simulating the effect of soaps, was evaluated *in vitro* by testing the emulsifying properties of the ingredient.

10 µg/mL and 100 µg/mL Bacillus Ferment were dissolved in water and were mixed in a test tube with the same amount of an artificial sebum mix. The emulgent activity of the ingredient was assessed by measuring the total emulsified phase.

At the end of the treatment, the active ingredient presented emulsifying properties to clean the skin, with an increased emulsified phase of 33% ($p<0.01$) and 39.8% ($p<0.0001$) at 10 µg/mL and 100 µg/mL respectively.

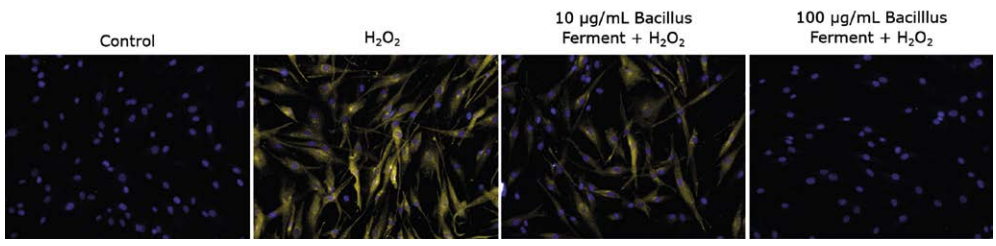


Fig. 2 Images of fibroblasts, with ROS staining shown in orange and cell nuclei in blue.

Antioxidant response determined by ROS

The antioxidant effect of the ingredient was evaluated through the study of the accumulation of reactive oxygen species (ROS) after inducing an oxidative stress in skin cells.
Human fibroblasts were treated with 10 µg/mL or 100 µg/mL Bacillus Ferment for 24 hours. Then, cells were treated with hydrogen peroxide (H_2O_2) for 30 minutes, to induce an oxidative stress, and finally were stained with the ROS orange dye. The levels of ROS were quantified by fluorescence and imaged by confocal microscopy. (**Fig. 2**)
At the end of the treatment, the levels of ROS were reduced by 39.6% ($p<0.0001$) and 94.0% ($p<0.0001$) at 10 µg/mL or 100 µg/mL respectively, suggesting an enhanced antioxidant response.

Cellular oxygen consumption

The aim of this assay was to evaluate the ability of the active ingredient to stimulate the oxygen consumption rate of cells, an important indicator of normal cellular function and cellular metabolism. Unhealthy cells with dysfunctional mitochondria show a lower oxygen consumption rate, compared to healthy cells [3].
Human fibroblasts were treated with 10 µg/mL or 100 µg/mL Bacillus Ferment for 4 hours. Oxygen consumption rate was assessed by using a phosphorescent oxygen probe.
The treatments with the active ingredient showed to increase oxygen consumption rate by 295.1% at 10 µg/mL and by 617.3% at 100 µg/mL compared to the basal control, suggesting a positive effect on the stimulation of cellular functioning and metabolism.

Minimizing imperfections

The aim of this study was to evaluate the ability of the active ingredient to reduce the presence of skin imperfections.
21 female volunteers between 25 and 35 years old applied a cream containing 2% of a solution with the biotechnological ingredient on half face and a placebo cream on the other half, twice a day for 28 days.
The effect on improving skin homogeneity was assessed by the evaluation of red spots through the VISIA complexion analysis system. (**Fig. 3**)
The biotechnological ingredient showed to minimize the appearance of red spots by 6.6% ($p<0.05$) after the treatment, and it visibly demonstrated a reduction of imperfections for a better skin homogeneity and a flawless skin.

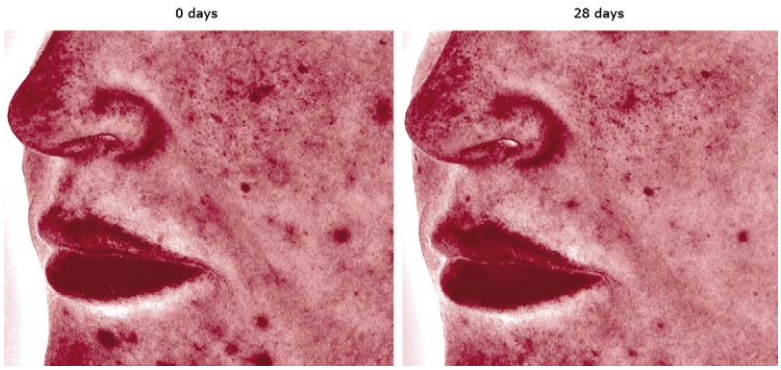


Fig. 3 Images of the red spots of a volunteer before and after the treatment.

Improved skin complexion in all ethnicities

The ability of the active ingredient to mimic the multiple effects of clays on the skin was assessed on a panel of volunteers of different ethnicities.
61 female subjects with different ethnicities between 25 and 55 years old, who presented imperfections and showed uneven skin tone, were split into two different groups. The first group applied a cream containing 3% of a solution with the


CAN YOU
KEEP
A SECRET?
Find out more and visit us at in-cosmetics global,
31 March – 2 April 2020, Barcelona, Booth N20
Contact us for more information:
care@zschimmer-schwarz.com
ZSCHIMMER & SCHWARZ
zschimmer-schwarz.com

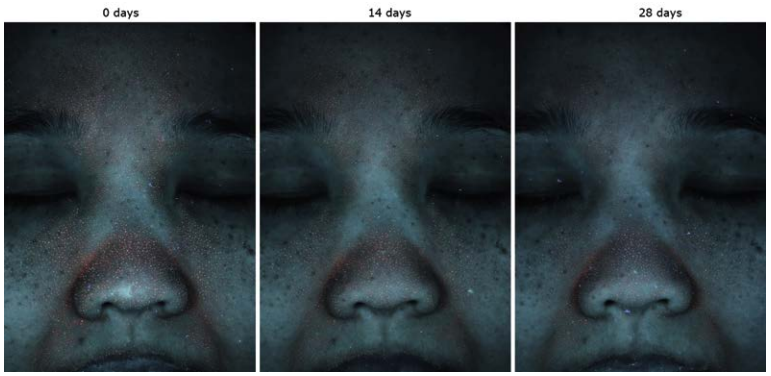


Fig. 4 UV photographs of a volunteer before and after 14 and 28 days of the active treatment. The presence of porphyrins can be seen as colored fluorescent spots.

biotechnological ingredient on the face, while the second group applied a placebo cream, twice a day for 28 days.

The skin purifying effect and the skin texture were assessed before and after the active and placebo treatments.

On the one hand, the cleansing benefits of the active ingredient were evaluated by calculating the area of porphyrins, a marker of skin purity, in a specific region of the face through the VISIA complexion analysis system. (**Fig. 4**)

After only 14 days of the treatment with the biotechnological ingredient, the area of porphyrins on the cheek was decreased by 52.2% ($p<0.001$), with a maximum decrease of a volunteer of 100%.

On the other hand, the skin texture of the volunteers was evaluated by an expert, who scored the skin smoothness and softness parameters through visual and tactile grading, respectively. (**Fig. 5**)

The biotechnological ingredient showed to improve skin smoothness and softness by 9.5% ($p<0.0001$) and 13.5% ($p<0.0001$), respectively, obtaining a softer and visibly smoother skin at the end of the treatment.

Well-being and self-perception enhanced

The aim of this study was to evaluate the ability of the biotechnological ingredient to simulate the wellness-enhancing benefits of clay masks.

58 female volunteers of different ethnicities between 28 and 55 years old applied either a cream containing 3% of a solution with the biotechnological ingredient twice a day on the face or a benchmark rinse off clay mask, once a week, for 28 days. The changes in the emotions of the volunteers were evaluated at the end of the treatments by means of the Mirror Test™ technique, in which the volunteers were confronted to their own reflection in a mirror and were asked questions regarding their image. The responses were evaluated in terms of vocal analysis (prosody) and semantic analysis (verbatim).

Regarding the prosody analysis, the vocal intensity (loudness) and the tone (voice musicality) were specifically evaluated. A low intensity and a high tone are related to a less stressed vocally response. (**Fig. 6**)

The active treatment showed to mimic the well-being effects of clays, since it provided an improved emotional state of the volunteers, similar to that obtained with the application of a clay mask. Besides the emotion expressed vocally, the study also analyzed the verbatim (semantic production) of subjects.

Fig. 5 Image of a volunteer before and after the treatment.

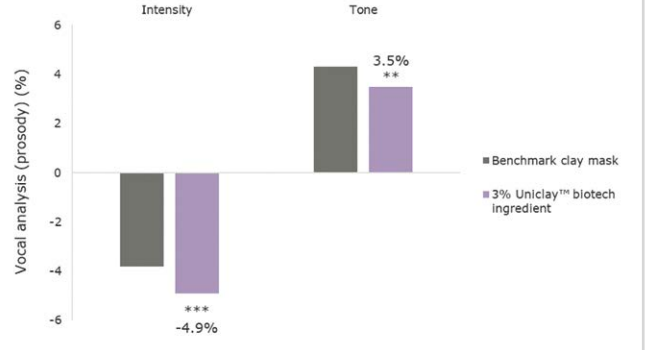


Fig. 6 Changes in the vocal analysis (intensity and tone parameters) of the volunteers after different treatments (**$p<0.01$, ***$p<0.001$).

Similarly, to the treatment with the benchmark clay mask, the application of the biotechnological ingredient resulted in a shift of perception, from a more negative and lower arousal to a more positive and higher excitement feeling, indicating a better self-perception by the volunteers after the treatment.

Conclusions

Uniclay™ biotech ingredient is a biotechnological ingredient, obtained from a microorganism isolated from a clay, that mimics the multiple effects of clays on the skin and has a positive impact on the person's well-being and self-perception.
In vitro, the ingredient showed to reduce *P. acnes* biofilm formation and the skin inflammation that comes along its presence. It also increased cellular oxygen consumption, linked to healthier cells, and provided regeneration, cleansing and antioxidant effects.
In vivo, it showed to minimize skin imperfections, and, in volunteers of different ethnicities, it reduced bacterial porphyrins and improved skin texture for a cleaner and smoother skin. Furthermore, it also enhanced self-perception and the feeling of well-being when applied in a regular cream, obtaining similar effects to the application of a rinse off clay mask.
By mimicking the well-known effects of clay masks in any type of skin care formulation, the ingredient offers renovated skin care applications for consumers to benefit from a clean, smooth and beautiful skin and an enhanced well-being in a more convenient way.

Bibliography

[1] *Saint-Leger D, Bague A, Cohen E, Chivot M.* A possible role for squalene in the pathogenesis of acne. I. *In vitro* study of squalene oxidation. Br J Dermatol 1986. 114(5):535-42.

[2] *Schaller M, et al.* Induction of a chemoattractive proinflmmatory cytokine response after stimulation of keraitnocytes with Propionibacterium acnes and coproporphyrin III. Br J Dermatol 2005. 153(1):66-71.

[3] *Hynes J, Marroquin LD, Ogurtsov VI, et al.* Investigation of drug-induced mitochondrial toxicity using fluorescence-based oxygen-sensitive probes. Toxicol. Sci. 92(1): 186-200, 2006.

contact

Júlia Comas
Olga Laporta
Elena Cañadas
Albert Soley
Raquel Delgado

Lipotec S.A.U.
Gavà, Spain

Moringa oleifera Lam. leaf extract: Anti-inflammatory and Antioxidant Cosmetic Ingredient for Holistic Skin and Hair Care

L. Apaza, L. Sánchez, C. Thiebaut

abstract

The current cosmetic market is shifting towards integrated sustainability, greener and more natural ingredients.
Cosmetic ingredients must not only be efficient but also meet the criteria of environmental, social and economic sustainability.
Along this line, COBIOSA, in its continuous commitment to natural and safe products, has developed CobioPure, a product based on Moringa oleifera leaf extract, with powerful antioxidant and anti-inflammatory properties.
The Moringa tree is cultivated in Paraguay following the biodynamic agriculture norms (DEMETER certification).
Moringa leaf extract holds high antioxidant and anti-inflammatory activities thanks to its high content of polyphenolic compounds used for skin disorders [1].
Although Moringa has gained increasing popularity as a superfood supplement, its cosmetic properties are still not well known in the cosmetic market.
The polyphenolic compounds present in CobioPure protects the skin from environmental damage and fights skin aging, reducing free radicals and inflammation caused by exposure to UV radiation.
The aim of this study was to assess the anti-inflammatory and antioxidant potential of Moringa oleifera leaf extract on skin and hair cells.
Our results show that CobioPure protects both the skin and hair from environmental damage and has shown to fight skin aging, reducing the production of free radicals and inflammation caused by exposure to UV radiation.

Introduction

Moringa oleifera Lam., (family, Moringaceae), is a fast-growing, drought-resistant tree of the family Moringaceae, native to tropical and subtropical regions of Asia, Africa and Latin America [2].
Known as the miracle tree or tree of life, Moringa has been used around the world for its many amazing medicinal benefits and healing abilities.
Due to the richness of its composition, it is considered as a superfood. It is rare for a single plant to contain so many essential nutrients in such high quantities on its own. According to India's ancient tradition of Ayurveda, Moringa leaves prevent more than 300 diseases.
In recent years, many pharmaceutical studies have been published extending the action range of M. oleifera activities such as: antioxidant, antimicrobial, anti-inflammatory, anti-proliferative, antispasmodic, antiulcer, hypocholesterolemic, and hypoglycemic [3,4].
Due to these characteristics, *Moringa oleifera* can be considered as one of the most useful plants in the world as results of its potential uses, and not surprisingly it is also known as the "Tree of life".

Demeter Biodynamic agriculture: Shaping the future

Since its introduction in 1928, Demeter certification has stood for proven quality from production to processing or trading.
Biodynamic agriculture is a system of organic farming. It is based on the concept that the farm is an organic entity made from interdependent organisms.

In addition to not using synthetic fertilizers and chemical pesticides, or artificial additives in further processing, it also includes the principles of the Biodynamic Agriculture:

- Work with the vital energies in nature.
- Restore and support natural cycles and interrelationships.
- Take in consideration the cosmic rhythms in plant production.

Demeter follows a holistic approach to agriculture and its requirements are much higher than what it is required for organic products.
The meaning of "biodynamic" derives from two Greek words: "bios" (life) and "dynamis" (force), meaning the systematized search for the energy that creates and preserves life.
Rudolf Steiner was the founder of the biodynamic approach to agriculture.
Biodynamic farming includes the protection and enhancement of soil to produce high-quality products. To stimulate life in soil and plants, eight specific preparations are used, on the soils and crops and in the composts. Biodynamic agriculture also works in relation to the patterns of the Moon´s movements and the planets of our solar system.
Several studies found that biodynamic farming systems had better soil quality, translating into a better crop quality compared to traditional agriculture [5].
Various scientific publications demonstrate how biodynamic preparations had a significant effect on the content of total polyphenolics in different types of crops [6].
The use of botanical raw materials with Demeter certification guarantees that they are cultivated from the highest possible quality, far stricter than any organic label.

Moringa: natural photoprotective active ingredient

Skin is exposed to a variety of environmental factors such as UV radiation, responsible for the largest percentage of environmentally induced skin pathologies, including erythema, inflammation or degenerative aging.
UV harms basic structures in the *stratum corneum* and is an inducer of many skin disorders like wrinkling, dryness, photo-aging, hyperpigmentation, laxity, sagging, dryness, etc.
Any degree of repeated or chronic disruption of the *stratum corneum* barrier has been documented to activate chronic inflammation [7].
Although the skin is equipped with its own oxidative stress defense mechanisms, daily photo-protection is becoming a very important health issue in view of the increasing number of skin diseases, and it is recommended to boost the amount of natural antioxidants through an external application. Polyphenolic compounds are natural antioxidants found to be one of the most important groups of plant metabolites.
Plants synthesize them as part of their defense mechanisms against mechanical injury, pathogen infection, and UV radiation. They have a broad spectrum of biological properties.
The most common properties of polyphenols are antioxidant, anti-inflammatory and antimicrobial, actions that make them highly effective in the treatment of skin problems.
The polyphenolic compounds present in *Moringa oleifera* leaf extract, mainly phenolic acids, and flavonoids, protects the skin from environmental damage and fights skin aging, reducing free radicals and inflammation caused by exposure to UV radiation.
Among them, we can find gallic acid, chlorogenic acid, ferulic and ellagic acid; and flavonoids: kaempferol, quercetin, rutin.
Supported by a large body of literature, those compounds possess biological activities that confirm the great potentialities of *Moringa oleifera* leaf extracts as an active ingredient in cosmetic products.
Ferulic acid has been demonstrated to have strong antioxidant activity and a protective role against ultraviolet-induced erythema [8]. Likewise, ellagic acid suppresses UVA-induced oxidative stress and may be useful in the treatment of UVA-induced skin damage.
Ellagic acid can also prevent collagen destruction and reduce the inflammatory responses caused by UVB exposure [9,10].
Finally, chlorogenic acid enhances the expression of immune factors and protects the skin from photoaging due to its antioxidant properties [11].
In addition, it has been reported that flavonoids: rutin and quercetin have remarkable antioxidant and UV photopro-

tective activities [12,13]. The use of kaempferol as an agent against skin lesions induced by thermal burns has also been reported [14].

Anti-inflammatory and antioxidant potential of *Moringa Oleifera* leaf extract

Inflammation is the immune system's response to damage caused in cells or tissues by bacterial pathogens or by any other biological, chemical, physical or mechanical aggressor.

The classic symptoms of inflammation are pain, heat, redness, swelling, and loss of function. The first step of inflammation is known as irritation and it is regulated by cytokines. (**Fig. 1**)

Cytokines are a family of proteins and glycoproteins that regulate the inflammatory and immune response, most of them are produced by epidermal cells.

These cytokines act as chemical mediators released during the process, which help to intensify and propagate the inflammatory response; frequently including TNF-α, NF-κB, IL-6, etc [15].

TNF-α is a cell-signaling protein involved in systemic inflammation and is one of the cytokines that make up the acute phase reaction.

IL-6 (Interleukin 6) is a potent inflammatory cytokine that mediates several important physiological functions, most notably the control of the acute phase response at the beginning of acute inflammation [16].

NF-κB (Nuclear factor-kappa B) is a ubiquitous transcription factor that, by regulating the expression of multiple inflammatory and immune genes, plays a critical role in host defense and in chronic inflammatory diseases. Also, NF-κB binds to recognition elements in the promoter regions of inflammatory and immune genes, such as proinflammatory cytokines, chemokines, inflammatory enzymes, and adhesion molecules [Barnes, 1997].

CobioPure skin antioxidant and protective effect

Human skin is repeatedly exposed to ultraviolet (UV) radiation which in turns induces premature skin aging known as photo-aging.

Exposure of human skin to UV irradiation causes sunburn, altered pigmentation, inflammation, immune suppression, and dermal connective tissue damage.

The reactive oxygen species (ROS) load in the skin is higher than in any other organ, and there is a clear correlation between ROS and premature aging.

The goal of this study is to assess the antioxidant and protective potential of *Moringa oleifera* leaf extract on human keratinocytes (HaCaT cells), after treatment and irradiation with ultraviolet A (UVA) light, (1.62 mW/cm^2 during 30 minutes, 3 J/cm^2) through quantification of oxidative stress in form of reactive oxygen species (ROS).

Protocol

HaCaT cells were cultivated during 24h with 0.01% *Moringa oleifera* leaf extract.

When cells were previously treated with 0.01% *Moringa oleifera* leaf extract it significantly reduced ROS levels showing a powerful antioxidant and protective activity as shown in **Fig. 2**.

CobioPure anti-inflammatory effects on TNF-α, IL-6, and NF-kB gene expression in human keratinocytes

The goal of this study is to assess the anti-inflammatory and antioxidant potential *in vitro* of *Moringa oleifera* leaf extract in human keratinocytes.

To evaluate the anti-inflammatory efficacy, we performed an *in vitro* study to measure its capacity to inhibit the production

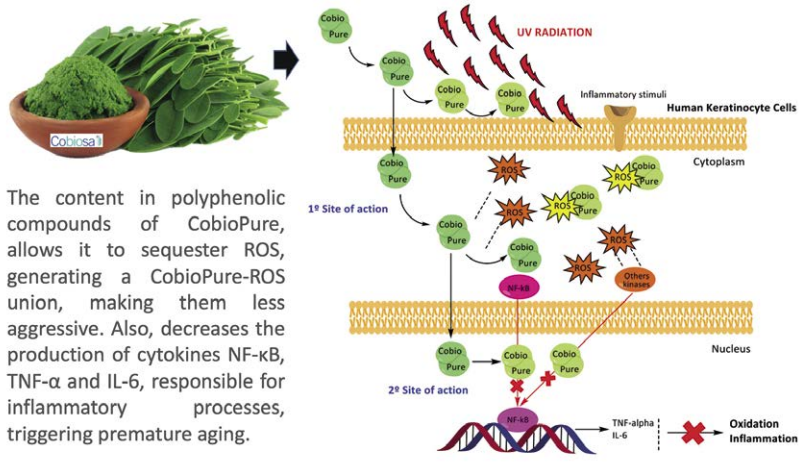


Fig. 1 Mechanism of action of CobioPure during the inflammatory cascade after UV radiation induced damage.

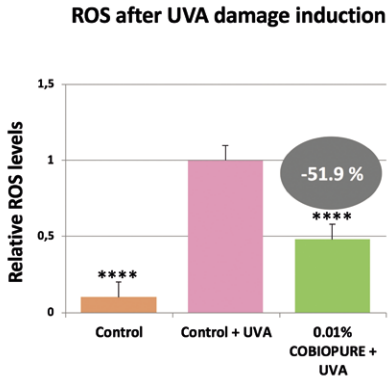


Fig. 2 Bar graphs represent ROS accumulation in samples treated with Moringa oleifera leaf extract at 0.01 % after UVA irradiation for 30 minutes, on HaCaT keratinocytes, normalized to the irradiated group.
**** Represents statistical significance with p value <0.0001

INDUSTRY
SOLUTIONS.

Consumer
Care
Solutions.

CHT
SMART CHEMISTRY
WITH CHARACTER.

NO.
COMMODITIES.

YES.
SPECIALTY SILICONE SOLUTIONS.

The CHT Group offers a wide range of specialty silicones for personal care products. With an international team of highly skilled experts, we are the first choice when it comes to sustainability, performance, product range, and expert solutions for hair care, skin care, and color cosmetics. Our dedicated teams will personally take on every challenge when it comes to finding the best solution for you.

silicone-experts.cht.com

H3i | 3 – 4 March 2020
Milan | stand 212

of pro-inflammatory cytokines TNF-α, IL-6 and NF-kB after *in vitro* treatment on human keratinocytes (HaCaT), through RTqPCR quantification.
The inflammatory response was induced by exposing keratinocytes with LPS (Bacterial Lipopolysaccharide). LPS are large molecules consisting of a lipid and a polysaccharide and are found in the outer membrane of Gram-negative bacteria. LPS are able to produce a strong inflammatory response.
After the treatment, the expression of TNF-α, IL-6, and NF-kB directly involved in inflammatory pathways, was measured.
0.001% *Moringa oleifera* leaf extract significantly reduced gene expression of IL-6 by 97%, NF-kB by 77,5% and TNF-α by 37,8% showing a powerful anti-inflammatory effect:

Protocol

HaCaT cells were cultured in growth medium. 24 hours later, the medium was removed and *Moringa oleifera* leaf extract at 0.001 % was added to cells.
30 minutes later, LPS for inflammatory response induction, at a final concentration of 100 ng/ml was added to the culture medium including *Moringa oleifera* leaf extract.
After 18 hours of the incubation period, cells were collected to proceed with RNA extraction. Total RNA was extracted and quantitative PCR (qPCR) was performed.

Results

Results showed the treatment with LPS significantly increased gene expression of TNF-α, IL-6, and NF-kB, compared to the untreated control, meaning LPS activated the inflammatory response.
When HaCaT cells were treated with 0.001% *Moringa oleifera* leaf extract, results indicated the treatment reduced TNF-α by by 37.8 % (p-value < 0.05); compared to the Control + LPS as shown in **Fig. 3**; Significantly decreased IL-6 gene expression by 97.0 % (p-value < 0.05) as shown in **Fig. 4** and significantly decreased NF-kB gene expression by 77.5% (p-value < 0.05) as shown in **Fig. 5**.

CobioPure hair protective effects after UV induced damage

UV damages human hair, causing fiber degradation. UV exposure involves considerable changes in the structure of keratin, including the photo-oxidation of amino acids and fatty acids, resulting in rupture of sulfur bridges, decomposition of lipids, decrease in melanin among others.
Both UVA and UVB are very harmful to hair. The effects of UVB irradiation can be severe, resulting in the breakdown of disulfide bonds inside the hair fiber and on the surface of the

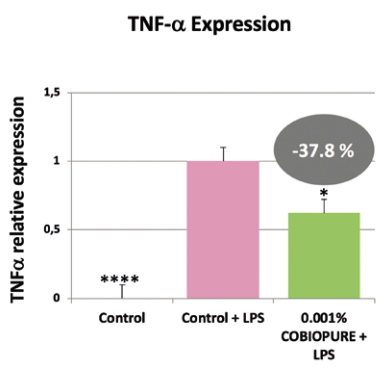


Fig. 3 Bar graphs showing LPS-induced TNFα expression results after treating human keratinocytes with 0.001 % Moringa oleifera leaf extract.
* Represents statistical significance with p value < 0.05.
**** Represents statistical significance with p value <0.0001

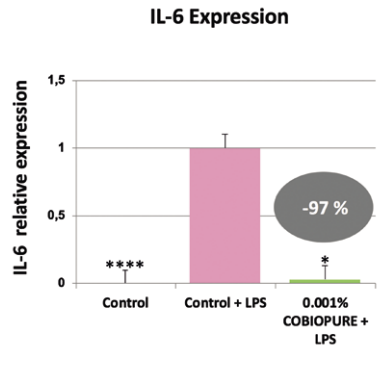


Fig. 4 Bar graphs showing LPS-induced IL-6 expression results after treating human keratinocytes with 0.001 % Moringa oleifera leaf extract.
*Represents statistical significance with p value < 0.05.
**** Represents statistical significance with p value <0.0001

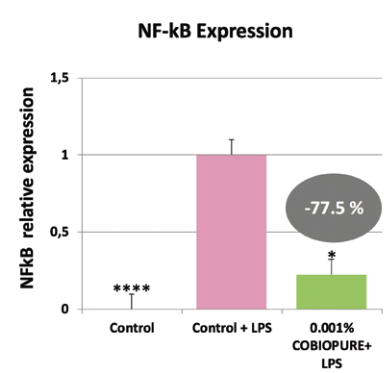


Fig. 5 Bar graphs showing LPS-induced NF-kB expression results after treating human keratinocytes with 0.001 % Moringa oleifera leaf extract.
* Represents statistical significance with p value < 0.05.
**** Represents statistical significance with p value <0.0001

cuticle, and UVA irradiation produces free radical/reactive oxygen species (ROS) leading to hair decoloration.
CobioPure has shown to provide photoprotective activity since it is capable of absorbing UV radiation, acting as a natural sunscreen filter [17].
The goal of this study is to assess the antioxidant and protective potential of CobioPure at 5% in a gel formulation on UVA-induced oxidative stress, after topical treatment on human hair compared to placebo.

Protocol

Cobiopure Gel or Placebo Gel were topically applied on human hair samples through direct immersion in the product. Excess was removed and irradiated with UVA (1.62 mW/cm^2 during 30 minutes, 3 J/cm^2). 1 mg sample was divided in 8 different wells (technical replicates) and ROS concentration was measured by spectrofluorimetry in the samples tested. All data were statistically analysed.

Results

UVA irradiation significantly increased Reactive Oxygen Species (ROS) compared to Control. Treatment with Placebo did not show any significant protection, compared to the Control + UVA.

When hairs were previously treated with CobioPure at 5% in a gel, it significantly reduced ROS levels by 33.2 % showing a powerful antioxidant and protective activity as shown in **Fig. 6**.

Discussion and conclusions

Natural, sustainable, multifunctional and effective products have become increasingly important trends in cosmetic formulations focused on skin and hair daily care against urban pollution and photoprotection. According to this, CobioPure exceeds all expectations.

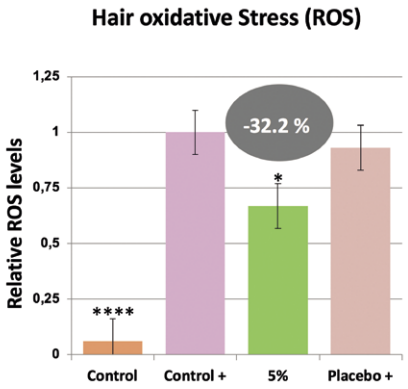


Fig. 6 Bar graphs represent ROS accumulation in human hair samples after treatment with Cobiopure Gel or Placebo Gel, and UVA irradiation, compared to Control + UVA.
* Represents statistical significance with p-value < 0.05.
**** Represents statistical significance with p value <0.0001

CobioPure consists of a *Moringa oleifera* leaf extract, cultivated conforming with DEMETER standard, a holistic approach to farming based on biodynamic agriculture which translates into a better crop quality having a significant effect on the content of total polyphenolic compounds, compared to traditional agriculture.
CobioPure has shown to display potent skin anti-inflammatory and both hair and skin antioxidant protective activity.
In human keratinocytes, 0.001 % *Moringa oleifera* leaf extract decreased the LPS-induced gene expression of pro-inflammatory cytokines TNF-α, IL-6, and NF-kB by 37.8 %, 97.0 %, and 77.5 %.
Regarding the hair protective effects, thanks to its rich composition CobioPure, has also demonstrated to be a potential hair protective active ingredient.
When applied on human hair strands, a 5% CobioPure Gel displays antioxidant and protective effects reducing by 33.2% UVA-induced reactive oxygen species (ROS) preventing adverse effects on the capillary structure.
In conclusion, CobioPure not only has shown to provide anti-inflammatory and antioxidant skin and hair care, but it is also a valuable natural strategy to extend skin and hair protection against UV radiation damage while meeting the criteria of environmental, social and economic sustainability and the highest standards of quality and concentration of bioactive ingredients.

Tables

Table Analyzed	ROS	ROS
Column B	Control + UVA	0.01 % + UVA
vs.	vs.	vs.
Column A	Control	Control + UVA
Unpaired t test		
P value	< 0.0001	< 0.0001
P value summary	****	****
Significantly different? (P < 0.05)	Yes	Yes
One- or two-tailed P value?	Two-tailed	Two-tailed
t, df	t=12.38 df=10	t=6.232 df=10
How big is the difference?		
Mean ± SEM of column A	0.07835 ± 0.01228 N=6	1.000 ± 0.07345 N=6
Mean ± SEM of column B	1.000 ± 0.07345 N=6	0.4808 ± 0.03930 N=6
Difference between means	0.9217 ± 0.07447	-0.5192 ± 0.08330
95% confidence interval	0.7557 to 1.088	-0.7048 to -0.3336
R square	0.9387	0.7953

Tab. 1 Statistical analysis of the results shown in **Fig. 2**.

Table Analyzed	TNFa (Ind)	TNFa (Ind)
Column B	Control + LPS	0.001 % + LPS
vs.	vs.	vs.
Column A	Control	Control + LPS
Unpaired t test		
P value	< 0.0001	0.0042
P value summary	****	**
Significantly different? (P < 0.05)	Yes	Yes
One- or two-tailed P value?	Two-tailed	Two-tailed
t, df	t=8.945 df=30	t=3.096 df=30
How big is the difference?		
Mean ± SEM of column A	-1.164e-009 ± 0.05265 N=16	1.000 ± 0.09862 N=16
Mean ± SEM of column B	1.000 ± 0.09862 N=16	0.6219 ± 0.07202 N=16
Difference between means	1.000 ± 0.1118	-0.3781 ± 0.1221
95% confidence interval	0.7717 to 1.228	-0.6275 to -0.1287
R square	0.7273	0.2421

Tab. 2 Statistical analysis of the results shown in **Fig. 3**.

Table Analyzed	IL-6 (Ind)	IL-6 (Ind)
Column B	Control + LPS	0.001 % + LPS
vs.	vs.	vs.
Column A	Control	Control + LPS
Unpaired t test		
P value	< 0.0001	0.0001
P value summary	****	***
Significantly different? (P < 0.05)	Yes	Yes
One- or two-tailed P value?	Two-tailed	Two-tailed
t, df	t=4.629 df=30	t=4.391 df=30
How big is the difference?		
Mean ± SEM of column A	1.900e-007 ± 0.07564 N=16	1.000 ± 0.2024 N=16
Mean ± SEM of column B	1.000 ± 0.2024 N=16	0.02984 ± 0.08868 N=16
Difference between means	1.000 ± 0.2160	-0.9702 ± 0.2209
95% confidence interval	0.5588 to 1.441	-1.421 to -0.5189
R square	0.4166	0.3912

Tab. 3 Statistical analysis of the results shown in **Fig. 4**.

Table Analyzed	NF-kB (Ind)	NFkB (Ind)
Column B	Control + LPS	0.001 % + LPS
vs.	vs.	vs.
Column A	Control	Control + LPS
Unpaired t test		
P value	< 0.0001	0.0001
P value summary	****	***
Significantly different? (P < 0.05)	Yes	Yes
One- or two-tailed P value?	Two-tailed	Two-tailed
t, df	t=5.822 df=30	t=4.358 df=30
How big is the difference?		
Mean ± SEM of column A	1.211e-007 ± 0.1065 N=16	1.000 ± 0.1348 N=16
Mean ± SEM of column B	1.000 ± 0.1348 N=16	0.2255 ± 0.1158 N=16
Difference between means	1.000 ± 0.1718	-0.7745 ± 0.1777
95% confidence interval	0.6492 to 1.351	-1.137 to -0.4115
R square	0.5305	0.3876

Tab. 4 Statistical analysis of the results shown in **Fig. 5**.

Table Analyzed	ROS	ROS	ROS
Column B	Control + UVA	Cobiopure Gel + UVA	Placebo Gel + UVA
vs.	vs.	vs.	vs.
Column A	Control	Control + UVA	Control + UVA
Unpaired t test			
P value	< 0.0001	0.0331	0.6898
P value summary	****	*	ns
Significantly different? (P < 0.05)	Yes	Yes	No
One- or two-tailed P value?	Two-tailed	Two-tailed	Two-tailed
t, df	t=11.76 df=14	t=2.363 df=14	t=0.4075 df=14
How big is the difference?			
Mean ± SEM of column A	0.06063 ± 0.001535 N=8	1.000 ± 0.07991 N=8	1.000 ± 0.07991 N=8
Mean ± SEM of column B	1.000 ± 0.07991 N=8	0.6686 ± 0.1153 N=8	0.9303 ± 0.1517 N=8
Difference between means	0.9395 ± 0.07992	-0.3315 ± 0.1403	-0.06988 ± 0.1715
95% confidence interval	0.7681 to 1.111	-0.6324 to -0.03060	-0.4376 to 0.2979
R square	0.9080	0.2851	0.01172

Tab. 5 Statistical analysis of the results shown in **Fig. 6**.

Literature

[1] *Fahey,* 2005; *Hsu et al.,* 2006; *Kasolo et al.,* 2010.

[2] *Moringa oleifera* (horseradish tree)". CABI. 17 September 2018. Retrieved 12 October 2018.

[3] *Gopalakrishnan, L.; Doriya, K.; Santhosh Kumar,* D. *Moringa Oleifera*: A Review on Nutritive Importance and its Medicinal Application. Food Sci. Hum. Wellness 2016, 5, 49–56.

[4] *Mishra, G.; Singh, P.; Verma, R.; Kumar, S.; Srivastav, S.; Jha, K.K.; Khosa, R.L.* Traditional uses, phytochemistry and pharmacological properties of *Moringa oleifera* plant: An overview. Der Pharm. Lett. 2011, 3, 141–164.

[5] Soil Quality & Profitability of Biodynamic & Conventional Farming Systems. *John P. Reganold*

[6] Influence of Biodynamic Preparations on the Quality Indices and Antioxidant Compounds Content in the Tubers of Coloured Potatoes (Solanum tuberosum L.). *Elvyra Jarienė, Nijolė Vaitkevičienė, Honorata Danilčenko, Marek Gajewski, Galina Chupakhina, Pavel Fedurajev, Reto Ingold.*

[7] *Elias P, Feingold KR.* Lipids and the epidermal water barrier: metabolism, regulation, and pathophysiology. Semin Dermatol 1992; 11: 176–82.

[8] *Oresajo, C.; Stephens, T.; Hino, P.D.; Law, R.M.; Yatskayer, M.; Foltis, P.; Pillai, S.; Pinnell, S.R.* Protective effects of a topical antioxidant mixture containing vitamin C, ferulic acid, and phloretin against ultraviolet-induced photodamage in human skin. J. Cosmet. Dermatol. 2008, 7, 290–297. [CrossRef] [PubMed]

[9] *Hseu, Y.C.; Chou, C.W.; Kumar, K.J.S.; Fu, K.T.; Wang, H.M.; Hsu, L.S.; Kuo, Y.H.; Wu, C.R.; Chen, S.C.; Yang, H.L.* Ellagic acid protects human keratinocyte (HaCaT) cells against UVA-induced oxidative stress and apoptosis through the upregulation of the HO-1 and Nrf-2 antioxidant genes. Food Chem. Toxicol. 2012, 50, 1245–1255. [CrossRef] [PubMed]

[10] *Young Bae, J.; Choi, J.S.; Kang, S.W.; Lee, Y.J.; Park, J.; Kang, Y.H.* Dietary compound ellagic acid alleviates skin wrinkle and inflammation induced by UV-B irradiation. Exp. Dermatol. 2010, 19, e182–e190. [CrossRef].

[11] *Fukushima, Y.; Takahashi, Y.; Hori, Y.; Kishimoto, Y.; Shiga, K.; Tanaka, Y.; Masunaga, E.; Tani, M.; Yokoyama, M.; Kondo, K.* Skin photoprotection and consumption of coffee and polyphenols in healthy middle-aged Japanese females. Int. J. Dermatol. 2015, 54, 410–418. [CrossRef] [PubMed].

[12] *Kamel, R.; Mostafa, D.M.* Rutin nanostructured lipid cosmeceutical preparation with sun protective potential. J. Photochem. Photobiol. B Biol. 2015, 153, 59–66. [CrossRef] [PubMed].

[13] *Choquenet, B.; Couteau, C.; Paparis, E.; Coiffard, L.J.M.* Quercetin and Rutin as Potential Sunscreen Agents: Determination of Efficacy by an *in Vitro* Method. J. Nat. Prod. 2008, 71, 1117–1118. [CrossRef] [PubMed].

[14] Protection of burn-induced skin injuries by the flavonoid kaempferol. *Park BK1, Lee S, Seo JN, Rhee JW, Park JB, Kim YS, Choi IG, Kim YE, Lee Y, Kwon HJ.*

[15] *Thompson et al.*, 1999; *Chung et al.*, 2003, 2004; *Dollar et al.*, 2006].

[16] *Neurath* and *Finotto*, 2011.

[17] *Moringa oleifera* Leaf Extracts as Multifunctional Ingredients for "Natural and Organic" Sunscreens and Photoprotective Preparations *Anna Baldisserotto, Piergiacomo Buso, Matteo Radice, Valeria Dissette, Ilaria Lampronti, Roberto Gambari Stefano Manfredini* and *Silvia Vertuani*.

contact

Luis Apaza
Lorena Sánchez
Cristina Thiebaut

Cobiosa Industrias Asociadas S.L.
R&D Department

Micellar Formulations – Old Wine in New Skins?

K. Brandt, J. Venzmer

Abstract

An increasing number of "micellar" formulations (e.g. micellar water, micellar shampoo, even micellar laundry detergent) has recently been introduced to the market, causing quite some confusion among both consumers and surfactant experts. Since every aqueous formulation containing surfactants above their so-called Critical Micelle Concentration (CMC) contains surfactant micelles, micellar formulations have been around basically forever. However, a simple soap solution does not get the "micellar" marketing claim because it fulfills neither the performance requirements nor today's mildness expectations. The new feature of modern "micellar" formulations is that they are now both highly effective when it comes to solubilizing ("deep cleansing") and – especially in case of micellar water for skin cleansing – exceptionally mild and gentle. Examples of such modern – nowadays called "micellar" – formulations for both skin and hair cleansing will be given. Contrary to some expectations, there is no direct correlation between micelle size or CMC and mildness or performance; according to latest research, another crucial parameter for mildness seems to be the charge density of the surfactant micelles. All in all, the modern "micellar" formulations should not be considered as "old wine in new skins", but rather as "new wine in old bottles".

Facts about micelles

Micelles have been around forever, at least since the invention of soap thousands of years ago. In aqueous environment, above a certain concentration (= Critical Micelle Concentration, CMC), surfactants form aggregates in order to avoid contact of their hydrophobic tails with water. The existence of such aggregates has been postulated by McBain [1] already in 1913, and the term "micelle" was coined by Hartley [2] in 1936. These aggregates come – depending on the geometry of the surfactant molecules – in different sizes and shapes [3], most often either spherical (few nanometers (nm) in diameter; **Fig. 1A**) or wormlike (few nm in diameter and up to hundreds of nm in length) (**Fig. 1B**). This means they are several orders of magnitude too small to be observed using light microscopy. However, in crystal-clear formulations not containing any hydrophilic thickeners, oil droplets or other objects which scatter light, it is possible to detect surfactant micelles by either Dynamic Light Scattering (DLS), or by just utilizing a (preferably green) laser pointer: In case you can see the laser beam in a clear liquid (see **Fig. 1C**), there must be objects present in the lower nm range – surfactant micelles, if nothing else has been added which could scatter light. In case of thickening was achieved by salt and/or low pH and/or addition of hydrophobic thickener, the presence of worm-like micelles can also be proven by their rheological properties [4]; their behaviour can be described by the so-called Maxwell model.

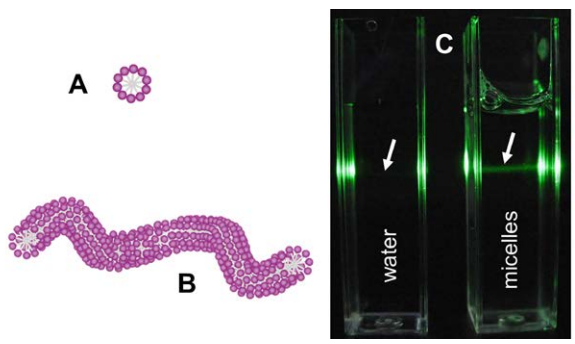


Fig. 1 Schematic representation of A) spherical micelle and B) worm-like micelle; C) Detection of nm-size objects (micelles!) in crystal-clear aqueous surfactant solutions by laser (pointer) light scattering.

Ingredients for mild surfactant formulations

Soap, i.e. sodium or potassium salt of long-chain fatty acids, is comparably harsh and irritating [5] and not the very best solubilizer. On the other end of the mildness spectrum, there are ingredients used in surfactant formulations, so-called hydrophilic emollients, which improve the mildness, but are not that effective as single ingredient when it comes to solubilizing, cleansing or make-up removing. Examples are PEG-7 Glyceryl Cocoate or PEG-80 Glyceryl Cocoate. They are not so much used in shampoo or bodywash formulations because of their solubilizing properties; more important are improvement in skin feel and the fact that they decrease the irritation potential of anionic primary surfactants. The classic way to achieve this is by combining e.g. Sodium Laureth Sulfate (SLES) with amphoteric secondary surfactants such as Cocamidopropyl Betaine (CAPB). A typical

ratio in very basic shampoo formulations is 3:1, e.g. 9% SLES and 3% CAPB (based on active matter). However, in order to become exceptionally mild, the ratio should be reversed and/or other mildness-improving ingredients should be added.

Assessment of mildness

Testing of surfactants for eye irritation is required by the regulatory agencies to ensure consumer safety. Apart from the need to prevent any eye damage, assessment of mildness is also being done to avoid discomfort for the consumers during use of e.g. shampoos (see the "no more tears" claim by Johnson&Johnson). Already in the 1980s, there were approaches to replace the classic Draize rabbit eye irritation test [6] by *in vitro* essays. One successful but experimentally challenging method is the Red Blood Cell (RBC) assay [7], for which a large body of data, for single surfactants as well as for mixtures and complete formulations, has been collected over the last several decades. This assay is based on (1) erythrocyte membrane lysis and (2) denaturation of the hemoglobin released. Both effects are measured photometrically, and the ratio of both parameters (L/D ratio) is used to characterize the in vitro effects of surfactants. Data for surfactants typically used in rinse-off formulations are given in **Fig. 2**. In recent years, a number of other more modern tests using skin models have been evaluated [8]. The validation of new methods is always challenging, i.e. the question whether an in vitro test is able to give a good correlation with the eye irritancy experienced by the consumer. In this context, CON4EI (Consortium for *in vitro* Eye Irritation testing strategy), a Long-Range Research Initiative (LRI) of the European Chemical Industry Council (CEFIC), should be mentioned here. Within this large-scale comparative testing program, a set of 80 chemicals, but unfortunately only few surfactants, has been evaluated using seven different in vitro test methods [9].

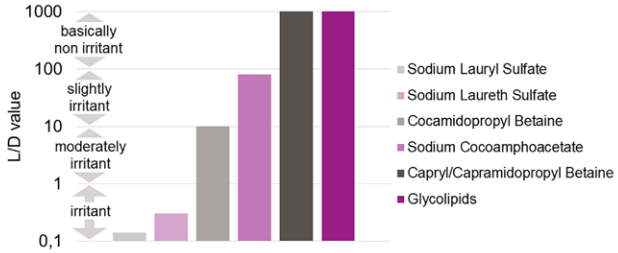


Fig. 2 RBC assay - L/D values of typical surfactants.

Correlation of mildness with micellar properties

Frequently, there are discussions about a possible correlation between mildness and physicochemical properties of the surfactants or their micelles. However, it is not clear on which facts some common beliefs are based and whether these can be generalized. Micelle shape, size and CMC (i.e. the concentration of single surfactant molecules = "monomers") are all thought to be somehow related to the mildness of surfactants. However, this might be true in some cases when comparing homologues having the same overall chemistry (i.e. same structure of the hydrophilic headgroup, e.g. sulfate), but different alkyl chain lengths of the hydrophobic tail. In general, however, such correlations do not hold true [10]. It is often not the concentration of a surfactant, which determines its irritation potential, but its chemical structure. Examples are short chain capryl/capramidopropyl betaines or glucosides, which have a pretty high CMC, but which are known to be especially mild (see **Fig. 2**). A comprehensive overview on the effect of different surfactants on skin has been published recently [11]. The latest research from the group of Ananthaphadmanabhan at the University of Cincinnati has shown that the charge density of surfactant molecules and their micelles seems to be another crucial parameter for skin compatibility [12]. This can also explain why rhamnolipid glycolipids (see **Fig. 3**) are extremely mild. One can expect that the carboxylate group (which could potentially make the surfactant as harsh as soap!) is shielded by the two rhamnose entities. This is also evidenced by the fact that this glycolipid hardly complexes with divalent cations – the carboxylate group is just not accessible.

Modern "micellar" skin and hair care

A typical example of a shampoo formulation from the "pre-micellar" era is shown in **Tab. 1**. Is there any difference to modern micellar shampoos in terms of their total surfactant concentration or micelle content? No – every shampoo always contained surfactants at a concentration well above CMC, otherwise it would not have been able to solubilize any oil (i.e. soil, sebum, dirt) and provide foam. However, the new "micellar" shampoos are based on combinations of surface-active ingredients which are both very effective in terms of cleansing but at the same time especially mild and gentle. An example of such a modern "micellar" formulation is given in **Tab. 2**.

For micellar water, the situation concerning mildness requirements is even more challenging, since the formulations are typically applied with a cotton pad and no rinsing with water should be necessary. At the same time, the expectation of the consumer is that even water-proof mascara can be removed without much mechanical effort. Examples for micellar water formulations with both excellent mildness and makeup removal [13] are shown in **Tab. 3** and **4**.

Conclusion

Using any kind of surface-active molecule in an amount above its CMC would be sufficient to call a formulation "micellar". However, this is not the intention of this mar-

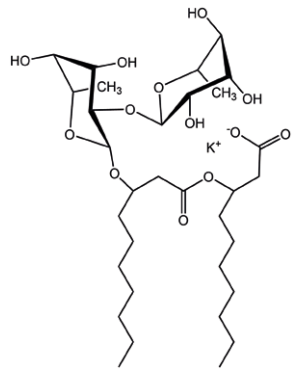


Fig. 3 Structure of rhamnolipid (INCI: Glycolipids).

Classical Shampoo Guideline Formulation

Ingredients	% w/w
ANTIL® Soft SC (Sorbitan Sesquicaprylate)	0.2
Sodium Laureth Sulfate (28%)	37.5
Parfum	0.3
ABIL® Quat 3272 (Quaternium-80, 50%)	1.0
Water (Aqua)	ad 100
TEGO® Betain F 50 (Cocamidopropyl Betaine, 37.5%)	9.3
ANTIL® 171 (PEG-18 Glyceryl Oleate/Cocoate)	2.5
TEGO® Pearl N 300 (Glycol Distearate)	1.0

Tab. 1 Guideline formulation "Classical shampoo" from the "pre-micellar" era.

Purifying Micellar Shampoo (2018-CN-RO-BR-019-0006-3)

Ingredients	% w/w
Sodium Laureth Sulfate (28%)	6.4
TEGO® Betain P 50 C (Cocamidopropyl Betaine, 37.5%)	4.0
RHEANCE® One (Glycolipids, 50%)	2.0
TEGO® Solve 61 MB (Polyglyceryl-6 Caprylate (and) Polyglyceryl-3 Cocoate (and) Polyglyceryl-4 Caprate (and) Polyglyceryl-6 Ricinoleate	5.0
ANTIL® SPA 80 (Isostearamide MIPA (and) Glyceryl Laurate)	0.5
Polyquaternium-10	0.2
Water	ad 100
ANTIL® 500 Pellets (PEG-200 Glyceryl Stearate)	0.5
Sodium Chloride	1.2
Verstatil® SL non GMO (Aqua (and) Sodium Levulinate (and) Potassium Sorbate)	1.5
Perfume	q.s.

Tab. 2 Guideline formulation "Purifying micellar shampoo" with high cleansing efficacy and low level of surfactants.

Micellar Water for Facial Cleansing (UL 5803/9.1)

Ingredients	% w/w
TEGOSOFT® PC 41 MB (Polyglyceryl-4 Caprate)	1.00
TEGO® Betain 810 (Capryl/Capramidopropyl Betaine, 35%)	1.30
TEGO® Solve 61 MB (Polyglyceryl-6 Caprylate; Polyglyceryl-3 Cocoate; Polyglyceryl-4 Caprate; Polyglyceryl-6 Ricinoleate)	1.00
Perfume	0.05
Water	ad 100
TEGO® Natural Betaine (Betaine)	2.00
Hexylene Glycol	1.40
Glycerin	1.00
Preservative	q.s

Tab. 3 Guideline formulation „Pure micellar water", pH 6.0, in vitro mildness test result (RBC test): basically non irritant.

Pure Micellar Water (AM 13/16)

Ingredients	% w/w
RHEANCE® One (Glycolipids, 50%)	5.4
Water	ad 100
TEGO® Natural Betaine (Betaine)	2.0
dermosoft® Pentiol eco (Pentylene Glycol)	5.0
Glycerin	1.0
NaOH (10%)	ad pH
Perfume	0.05

Tab. 4 Guideline formulation „Micellar water for facial cleansing", pH 5.0, *in vitro* mildness test result (RBC test): basically non irritant.

keting claim which has become increasingly popular in recent years. Nowadays, this term is used way beyond its original scientific definition for systems which combine excellent cleansing performance (because of their content of powerful surfactants) with extraordinary mildness. Accordingly, expressions such as "purifying" or "deep cleansing" in combination with "mild", "gentle" and "moisturizing" are used to describe their performance. However, there is no general agreement in the market about which formulations should be called "micellar"; the situation is less clear for instance in case of a laundry detergent. Back to the initial question: Are the recently introduced micellar formulations nothing but old wine in new skins? Not really, it rather seems like it is just the other way around: New wine in old bottles.

Acknowledgment

This paper has benefited from discussions with several colleagues, among them *Uta Kortemeier* and *Jochen Kleinen.*

References

[1] *B. Vincent*, Advances Colloid Interf. Sci. 203 (2014) 51-54.

[2] *G.S. Hartley*, Aqueous Solutions of Paraffin-Chain Salts: A Study in Micelle Formation, Herrmann&Cie, Paris 1936.

[3] *J.N. Israelachvilli, D.J. Mitchell* and *B.W. Ninham*, J. Chem. Soc., Faraday Trans. II, 72 (1976) 1525-1568.

[4] *J. Kleinen* and *J. Venzmer,* J. Cosmet. Sci. 67 (2016) 59-70.

[5] *F.R. Bettley* and *E. Donoghue,* British J. Dermatol. 72 (1960) 67-76.

[6] *J.H. Draize, G. Woodard and H.O. Calvery*, J. Pharmacol. Exp. Ther. 8 (1944) 377-390.

[7] *W.J. Pape, U. Pfannenbecker* and *U. Hoppe*, Molecular Toxicology 1 (1987) 525-536.

[8] How to use new or revised *in vitro* test methods to address Serious eye damage/Eye irritation, ECHA (2018) (https://echa.europa.eu/documents/10162/21650280/oecd_test_guidelines_eye_irritation_en.pdf)

[9] CON4EI: Consortium for *in vitro* Eye Irritation testing strategy, A.v. Rompay and S. Verstraelen (eds.), Toxicology *in Vitro* 49 (2018) 1-116.

[10] *E. Lémery, S. Briançon, Y. Chevalier, C. Bordes, T. Oddos, A. Gohier* and *M.-A. Bolzinger,* Colloids and Surfaces A: Physichochem. Eng. Aspects 469 (2015) 166-179.

[11] *L. Cassiday,* INFORM 27 (2016) 694-699.

[12] *S.A.V. Morris, K.P. Ananthapadmanabhan* and *G.B. Kasting,* J. Pharmaceut. Sci. 108 (2019) 3640-3648.

[13] *K. Brandt, V. Dahl, A. Muss, S. Blaschek, Z. Drljaca, S. Volkmer* and *H.H. Wenk*, SOFW Journal 144 (10/2018) 14-18.

contact

Kathrin Brandt
Joachim Venzmer | joachim.venzmer@evonik.com

Evonik Nutrition & Care GmbH
Goldschmidtstr. 100
45127 Essen | Germany

Sustainable Solutions for Hair Care

M. Franzke, B. Kroon, T. Nuutinen, E.-J. de Feij, L. Kulcsar, X. Qu, C. Alonso, D. Streuli, A. Luschen, P. Antonopoulos

abstract

Sustainability is an area which is permanently growing in importance. Therefore, there is also a growing demand for environmentally conscious cosmetic products.
The current article summarizes a series of solutions for hair care that fit this market need applying different concepts.
A snapshot of varying hair care formulation concepts – shampoos, conditioners, and hair styling – with different strategies to help improve sustainability profiles for the different product types is presented.
Examples include cellulose derivatives that help to reduce raw material use like surfactants in shampoos or conditioners while maintaining the original level of performance. Guar derivatives improve deposition of natural oil or anti dandruff actives on hair and scalp thus optimizing the benefits of such ingredients. Biodegradable guars can also be used in styling products creating high performing systems, with long-lasting hold.

Introduction

We find that the beauty industry is evolving from "green" to "conscious", as noted by a recent Euromonitor study [1]. The notion of conscious beauty is that consumers desire formulas that not only respond to their own needs but to the environment around them as well. Based on surveys conducted by GlobalData, we find that natural/chemical-free and environmentally friendly claims help drive quality perceptions especially among Millennial and Gen. Z consumers [2]. However, they also want products with proven efficacy.
For manufacturers, ingredient selection is an important part of creating formulas that are environmentally aware. One measure that has been used by the industry to source ingredients based on naturalness is the ISO standard-ISO 16128 which delivers guidelines for technical definitions and criteria for natural and organic cosmetic ingredients [3]. Another approach has been published and used by L'Oréal to assess the naturalness of a cosmetic ingredient [4].
For nature-based ingredient derivatives, both systems apply similar assessment principles. Their main difference is the way the naturalness index is calculated-ISO sets molecular weights of natural and non-natural parts into relation. L'Oréal sets the number of carbon atoms from natural and non-natural constituents into relation. Hence, the L'Oréal naturalness index also takes sourcing and processing into account. The ISO standard adds some granularity differentiating between natural and organic thus having an index for both aspects.
Other aspects of raw material sourcing are social aspects, such as the circumstances under which the materials are made: fair-treatment of farmers, exclusion of child labor, etc. Many raw material suppliers meanwhile have established programs with their vendors/suppliers, including running regular social audits with their vendors, to monitor the social aspects of sourcing.
Beside sourcing and processing, ingredients also impact the sustainability of the formula itself. We find that there are a variety of approaches formulators can follow in the hair care space to develop products that meet consumer needs. The Ashland team of hair care solvers have highlighted several examples where the key ingredients lend themselves to being central in the creation of environmentally-friendly formulations. The featured ingredients are all nature-based (% natural carbon >50%) and contain a natural backbone.

Sustainable approaches for shampoo

Reducing surfactant by adding methylhydroxypropylcellulose

A key ingredient group for all shampoos are surfactants. Their main role is detergency and consumers associate cleaning efficacy with a high level of foam. Depending on the formulation, surfactant levels can be up to 16-18%. The main problem for the formulator when reducing surfactant levels is the loss in foam performance, e.g. less foam or change of foam haptics, which in turn is perceived as a lower performing shampoo.
The addition of hydroxypropyl methylcellulose (HPMC) to the shampoo mix allows for a surfactant level reduction of 30%-40% independent of the surfactant base, whether it is sulfate-based or sulfate-free.

LEARN • EXPLORE • NETWORK • CREATE

in-cosmetics® global

Barcelona • 30 June - 2 July 2020

NEW DATE

The leading global event for personal care ingredients

30 YEARS OF INNOVATION

Where personal care **ingredients** and **creators** come together

- **Source** ingredients from up to 150 suppliers
- **Touch** and try the latest innovations
- **Meet** exhibitors from 50+ countries
- **Learn** about future personal care trends
- **Establish** profitable business networks

Organised by: Reed Exhibitions®

Register and learn more at in-cosmetics.com/global

#incosGlobal

Ingredient (INCI)	% active
Sodium Cocoyl Isethionate	1.5
Sodium Lauroyl Methyl Isethionate	4.0
Disodium Laureth Sulfosuccinate	1.3
Sodium Lauryl Sulfoacetate	1.2
Cocamidopropyl Betaine	2.0
Hydroxypropyl Methylcellulose (Benecel™ E10M)	0.3
Water	Ad100

Tab. 1 Sulfate-free surfactant formula w HPMC for foam study.

A comparison between a sulfate-free surfactant mix (see **Tab. 1**) containing 10% surfactant and 0.3% of HPMC (optimized in molecular weight and substitution, Benecel™ E10 methylcellulose) and a commercial sodium laureth sulfate-based shampoo showed similar foam formation (flash foam, foam degradation). The foam stability of the sulfate-free system was even better than the commercial benchmark (as the graphs in **Fig. 1** shows).

The additional benefit of HPMC in the mix is that it helps increase the level of mildness of the overall formulation.

Based on *in vitro* studies, the release of Interleukin1-alpha (as an indicator for inflammatory potential) was measured on reconstructed human skin. The results (**Tab. 2**) indicate a benefit for the surfactant mix with HPMC compared to a pure SLES/CAPB (10/2) system.

The release of Interleukin1-alpha was reduced from 43 pg/ml to 16,6 pg/ml whereas viability increased from 73.8% to 88.4%.

tested products pH 6.5@10% in PBS	[IL-1a] P-[IL-1a] release in PBS pg/mL1	viability %
control formulation 10/2 SLES/CAPB	43.9	73.8
formulation C 10/2 SLES/CAPB 0.2% HPMC	16.6	88.4

Tab. 2 Effect of HPMC in reducing Interleukin1-alpha release in sulfate-free formula.

Guars, nature-derived materials for optimized deposition

Another important aspect of shampoo formulation is the deposition of the benefit agents onto the hair. Here cationic modified guars play a key role.

In the context of hair conditioning from shampoo systems, cationic guars are known for their ability to deposit silicone oils via a coacervate formation on hair. Their performance is mainly driven by charge density and molecular weight. Additional studies have also demonstrated that cationic guars can also effectively deposit natural oils, such as argan, coconut, jojoba, or meadowfoam seed oil, onto the hair.

A special shampoo segment requiring the efficacious deposition properties of guar are anti-dandruff shampoos. Here the deposition of the antidandruff (AD) active onto the scalp is especially important for this type of shampoo. Systems which deposit high level of active allow optimal efficacy. For AD activity, it is not only important that the coacervate is formed but the particle size of these coacervates also plays a role. If the coacervates particle size is too large, they might be filtered out by the hair array or be deposited only in very few domains.

Cationic guars, such as N-Hance™ BF-13 cationic guar (guar hydroxypropyl-

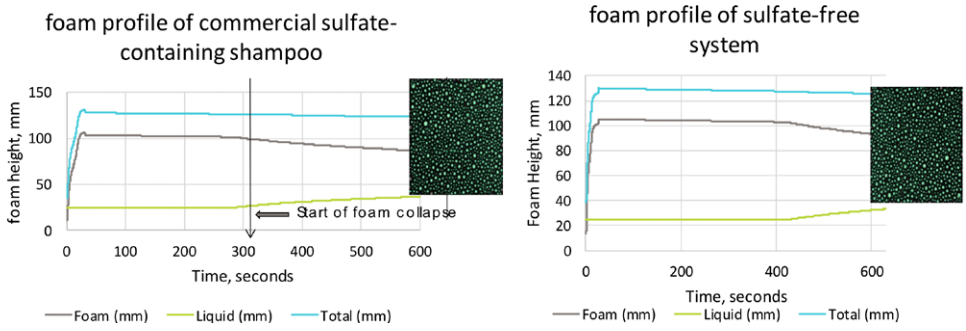


Fig. 1 Foam profile sulfate-free surfactant mix with HPMC vs commercial sulfate-based shampoo.

trimonium chloride), with optimized molecular weight and charge density form a fine coacervate floc which leads to an even distribution of the AD ingredient over the entire head.
Microscopic assessment of the floc formation of a commercial AD shampoo containing cationic guar and zinc pyrithione (ZPT) in comparison to an AD shampoo formulation with N-Hance™ BF-13 cationic guar and ZPT (sulfate and silicone-free base) showed some clear differences. The flocs found in the commercial shampoo are significantly larger than those for the shampoo prototype with N-Hance™ BF-13 (**Fig. 2**).
In vitro efficacy test of the sulfate-free AD formula vs. a commercial benchmark showed similar AD activity/efficacy for the formula with N-Hance™ BF-13. The level of dandruff reduction was measured via reduction of *Malassezia furfur* colonies. The commercial sulfate containing shampoo achieved a *M. furfur* reduction of 76%; the sulfate-free prototype achieved a *M. furfur* reduction of 74%. Further optimisation for the sulfate sytem may be possible by optimizing the ZPT particle size which can have an additional impact on AD activity.

Improving sustainability profile of Rinse off conditioners

Classic conditioners consist of a wax/oil phase (which usually contains fatty alcohol, FA) and a surfactant behenyltrimethylammoniumchloride (BTAC) or/and an appropriate alternate cationic /quasi-cationic surfactant. The mixture of cationic surfactants and fatty alcohol form a three-dimensional structure--the so-called lamellar gel phase (gel phase). In this structure, the cationics and the fatty alcohol align with their hydrophobic resp. hydropilic tails. The microstructure can be influenced by the type of raw materials, raw material ratios but also by the formulation process.
In a conditioner, the surfactant/FA mix delivers the wet conditioning properties and hydrophobisation of the hair, but it is also important for the texture of the product and for the haptics on the hair in the wet state. Dry lubrication is provided by additives like (silicone) oils, emollients and conditioning polymers.
Many cationic surfactants are associated with aquatic toxicity concerns. Significantly reducing use levels of these ingredi-

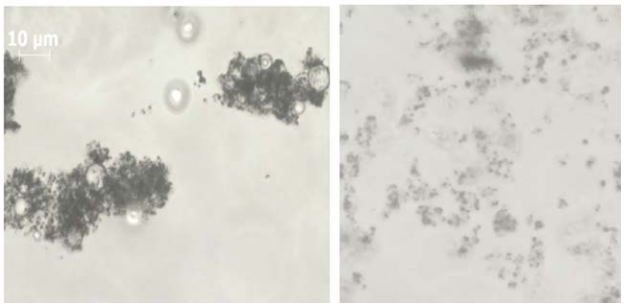


Fig. 2 left: diluted commercial AD w. Guar large flocs formed
right: AD prototype with Guar Nhance BF 13 small flocs formed.

ents can result in conditioning products with more favorable environmental profiles and less surfactants being released to the environment.

Users expect certain textural properties which are associated with product performance. A rich and creamy texture is part of the expected positive product experience. Reduction of the amount of gel phase can result in a disappointing product due to the low viscosity and a runny formula.

When adding just 0.15% hydrophobically modified hydroxyethlcellulose (HMHEC), like Natrosol™ Plus 330 CS cetyl HEC in the formulation, the amount of surfactant/fatty alcohol can be reduced by up to 50% while maintaining the rheological profile.

The base for such a system is laid out in **Formulation 1**.

The associative rheology modifier built up very efficient viscosity and yield stress as it can be seen in viscosity **Fig. 3** . The zero-shear viscosity of the system with HMHEC is similar to a conditioner mix with twice as much surfactant and fatty alcohol. The high shear viscosity is lower than the base without cellulose which should result in a clean feel on dry hair.

This was confirmed by sensory tests on mannequin heads. As seen in **Fig. 4** the cellulose modified system delivered in wet state a similar experience as the high gel phase conditioner- similar level of

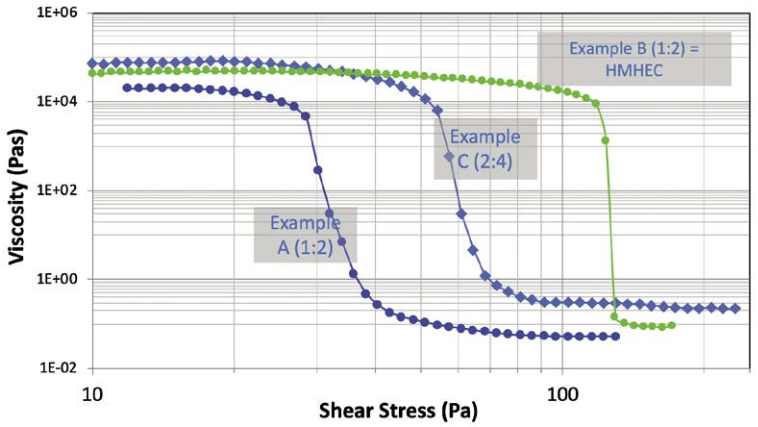


Fig. 3 Viscosity profile of conditioner w. and w/o HMHEC in comparison to conditioner with higher FA/Surfactant ratio. Using Serrated parallel plates (50 mm), 25 deg C, Controlled shear stress, Bohlin Rheometer. All Conditioners were made using identical process, same ageing time.

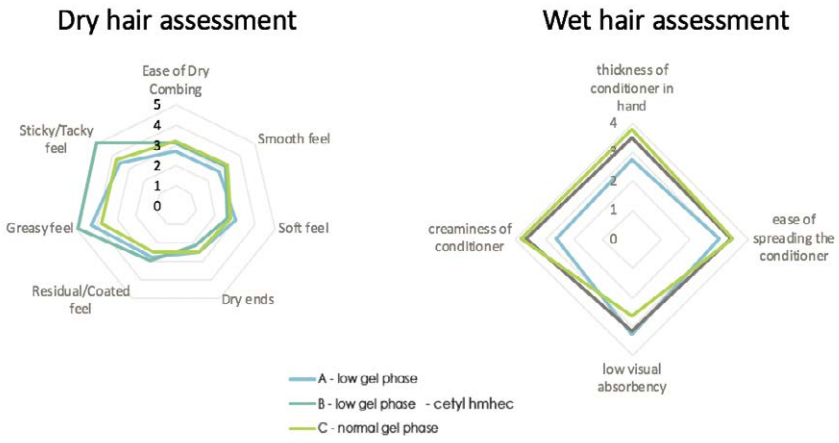


Fig. 4 Wet and dry sensory assessment of conditioner w. and w/o HMHEC in comparison to conditioner with higher FA/Surfactant ratio. Test Mannequin heads with damaged hair.

INCI Name	% w/w		
	A Conditioner (low gel phase LGP	B Conditioner (low gel phase with C16HEC)	C Conditioner (with normal gel phase)
Aqua	ad 100%	ad 100%	ad 100%
Cetyl Hydroxyethylcellulose[1]		**0.15**	
Behentrimonium Chloride	1.45	1.45	2.90
Cetearyl Alcohol	3.00	3.00	6.00
Disodium EDTA	0.10	0.10	0.10
Dimethicone (and) Trideceth-5	3.33	3.33	3.33
Amodimethicone/Morpholinomethyl Silsesquioxane Copolymer (and) Trideceth-5 (and) Glycerin	2.50	2.50	2.50
Benzyl Alcohol (and) Aqua (and) Sodium Benzoate (and) Potassium Sorbate[2]	**0.30**	**0.50**	**0.50**
Parfum	0.30	0.30	0.30
Citric acid	a.n.	a.n.	a.n.
Total		**100**	

[1] Natrosol™ Plus 330 CS cetyl HEC, [2] Optiphen™ BSB-W preservatives

Formulation 1 Base Formulation Conditioner.

creaminess and thickness but also similar spreading behavior. In dry state the cellulose modified system delivered a cleaner feel on hair with less coated feel and less sticky/tacky hair.
With just 0.15% cetyl modified hydroxyethylcellulose (HM-HEC), a 50% reduction of cationic surfactant and fatty alcohol is possible while matching the conditioning performance and the sensorial properties of a high gel phase system.

Hair mousse

In the styling category, fixative properties, setting strength and lastingness are the main factors driving performance.
For various styling applications, including aerosol mousses, guars can be, especially when taking sustainability into consideration, a solution. A cationic guar system with an optimized molecular weight and charge density, like Styleze™ ES-1 polymer (INCI: guar hydroxypropyltrimonium chloride), provides formulators with a nature-derived, biodegradable solution for styling applications that also delivers proven, long-lasting hold.
The polymer provides a series of benefits. First: It has superior lastingness properties. These were demonstrated in classic high humidity curl retention (HHCR) tests.

In **Fig. 5** the HHCR for 0.5; 1.0; and 2.0% for an aqueous Styleze™ ES-1 polymer solution and for a market standard [polyquaternium-4 (PQ4)] are plotted over a 48-hour period. PQ-4, a well-established ingredient in the market, was used as a positive control. The Styleze™ ES-1 polymer solution showed nearly no drop in HHCR, even after 48 hours. The very good lastingness of the hold can be achieved in "guar only" formulas but the polymer can also be used in combination with other styling polymers to boost the lasting hold properties of a given formulation.
Using the formulas summarized in **Tab. 3**, this durability of hold was demonstrated under high humidity conditions (**Fig. 6**). In formula A, 1% of the polymer mix was replaced by 1% cationic guar. Even after 48 hours at 90% relative humidity the curl treated with that mix nearly seemed to be unaffected by the humidity stress.
Beside good lastingness, styling polymers for mousse applications should also give a pleasant feel on hair, not leaving the hair tacky and sticky. Measurements of tack -tack duration and tack maximum via texture analyzer measurements demonstrate a short tack duration and a lower tack maximum

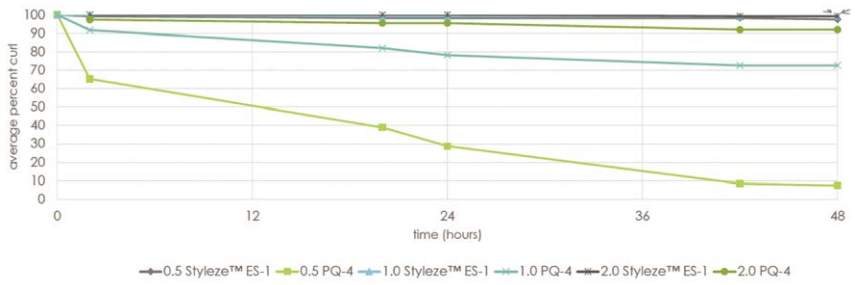


Fig. 5 High humidity curl retention of aqueous Styleze ES-1 solutions in comparison to Aqueous PQ-4 solutions. Cond: 90% rel. Hum./25°C.

Formula #	A	B	C
	12585-195-1	12585-215-1	12730-124
Styleze™ ES-1 polymer	1.00		
Polyquaternium-11 (Gafquat™ 755N)	2.50	4.15	2.50
VP/VA Copolymer (PVP/VA W735)	2.00	3.34	2.00
Polyquaternium-4		1.00	
Polymer	2.50%	2.50%	2.50%

Tab. 3 Mousse Formulations.

Fig. 6 Styleze™ ES-1 delivers long term (48-hour) styling benefits vs. PQ-4 mousse.

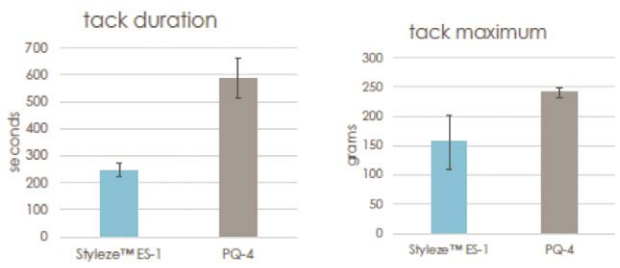


Fig. 7 Tack measurement of Styleze™ ES-1 and PQ-4 with texture analyzer.

the cationic guar chemistry compared to marketed materials like PQ-4 (**Fig. 7**).

Another important aspect is the "definition" a polymer provides to hair especially curly hair. Here cationic guars also deliver excellent results. Applied to curly hair tresses the curls show very good instant definition. This result remains even after the tresses were exposed to high humidity for 90 minutes. The definition did not get lost nor did the level of frizz increase. The results can be seen in **Fig. 8**.

Formulations trials for mousses showed an additional benefit of the cationic guar derivative. Due to its polysaccharide nature, it also boosts the bloom of the aerosol mousse. This is shown in **Fig. 9**. There is a significant and stability difference in foam formation between the formulations with and without cationic guar. This allows formulators to use lower levels of surfactants for the mousse formation.

Summary

Consumers desire hair care formulas that meet their care and styling needs but have a favorable impact to the environment. As a result, the need for sustainable, environmentally-conscious products is a key driver for new formulations and new raw material development.

By incorporating key raw materials, it is possible for formulators to design products with improved sustainable profiles while delivering high performance in various hair applications. An optimized HPMC like Benecel™ methylcellulose allows for the formulation of refined shampoos:

- with 30% reduced surfactant,
- for sulfate-free systems with very good foam properties,
- with improved mildness profile,
- while still maintaining care and cleansing benefits.

An optimized cationic guar like N-Hance™ BF13 cationic guar in shampoos allows for

- effective delivery of natural oils on hair,
- effective delivery of ZPT on scalp,
- without particle size optimization of the AD active,
- improved benefits from a sulfate-free formulation.

The nature-derived cetyl hydroxyethylcellulose like Natrosol™ Plus 330 CS allows for

- formulation of low gel phase conditioners,
- 50% reduction of cationic surfactants,
- maintaining conditioner performance,
- maintaining sensorial properties of high gel phase systems.

Cationic guars like Styleze™ ES-1 polymer allows formulation of styling mousses, gels, and creams with

- enhanced curl formation (definition/shape) & curl integrity,
- improved high humidity hold (48 hours),
- improved foam bloom and stabilization,
- improved style durability.

There is more to expect for the future from existing technologies but also from new ingredients pushing the boundaries of sustainable hair care products further.

Literature

[1] Euromonitor. "The evolution of beauty: from green to clean to conscious, " November 2019.

[2] GlobalData, 2018 Q3 global consumer survey.

[3] ISO 16128-1:2016 GUIDELINES ON TECHNICAL DEFINITIONS AND CRITERIA FOR NATURAL AND ORGANIC COSMETIC INGREDIENTS AND PRODUCTS -- PART 1: DEFINITIONS FOR INGREDIENTS https://www.iso.org/standard/62503

[4] *Michel PHILIPPE**, Blaise DIDILLON, Laurent GILBERT, Annales des falsifications, de l'expertise chimique & toxicologique, N°985 - 2ème semestre 2016, p36ff

Fig. 8 Curl enhancement and maintenance of PQ-4 treated hair vs. Styleze™ ES-1 treated hair.

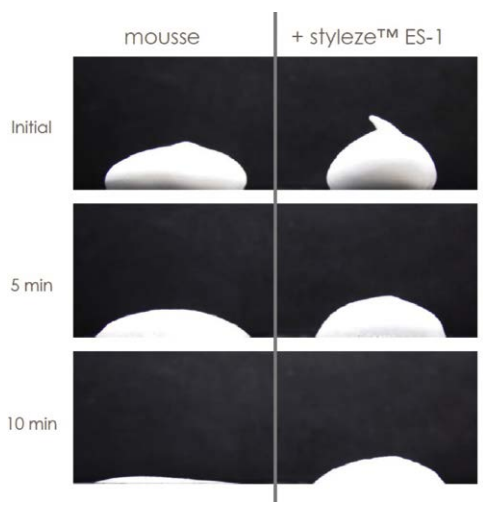


Fig. 9 Bloom and foam stability of foam with and without Styleze™ ES-1.

contact

Michael Franzke
Bert Kroon
Tuttu Nuutinen
Eric-Jan de Feij
Ashland Industries Nederland BV
Zwijndrecht, Netherlands

Lidia Kulcsar
Coralie Alonso
David Streuli
Anthony Luschen
Penny Antonopoulos
Ashland Specialty Ingredients GP
Bridgewater, NJ, United States

Xin Qu
Ashland Specialty Ingredients
Shanghai, China

hpci
SOUTH AFRICA

www.hpci-events.com

IN CONJUNCTION WITH
SOCIETY OF COSMETIC CHEMISTS OF SOUTH AFRICA

EXHIBITION & CONFERENCE

FOR THE COSMETIC AND DETERGENT INDUSTRY

9 - 10 SEPTEMBER 2020

JOHANNESBURG | SOUTH AFRICA

- → brings together suppliers from every point of the production chain
- → gives brands and manufactures of cosmetics and personal and home care products exclusives access to all services required for product development
- → shows new product ideas

Home and Personal Care Ingredients Exhibition
South Africa

Vincentz Network GmbH & Co. KG | P.O. Box 6247 | 30062 Hannover

VINCENTZ

New Technologies for Specific Antiperspirant Actions

J. Welzel, S. Grüdl, B. Banowski, A. Sättler, T. Förster, T. Welss

abstract

Cosmetics support a well-balanced quality of life of consumers by improving their outer appearance or repressing unpleasant attributes such as inordinate sweating or smelling. Especially sweating may be socially stigmatizing when becoming excessive. However, the biological mechanisms behind perspiration are not completely understood. The only effective cosmetic antiperspirant technology to reduce sweating is based on the use of aluminum salts, which lately became publicly misperceived. Henkel Beauty Care developed a sophisticated *in vitro* model of the human eccrine sweat gland to screen for and identify new antiperspirant technologies substituting these aluminum salts. This proprietary cell model combines all the relevant sweat gland cells in a three-dimensional (3D) environment.

In verification tests of the cell model the *in vitro* simulation of both, stimulation of the sweating process and its inhibition, were shown. Further, this 3D model was used for screening of sweat-regulating actives. Thereby, we focused on the change of certain ion-levels within the cells upon treatment with potential inhibitors. Positively screened potential antiperspirant substances were subsequently tested *in vivo* revealing a striking *in vitro*/*in vivo* correlation.

A new biologically potent antiperspirant technology is on its way to the consumer successfully replacing the common aluminum salts.

Introduction

Wet stains on clothes and a malodorous smell of sweat – that are typical signs for situations under exhaustive physical exercise, high emotional stress or even fear. Both are caused by two different types of sweat glands, named eccrine and apocrine glands, respectively.

Up to four million eccrine sweat glands cover nearly the entire human body opening directly onto the skin surface [1]. Their unique function of body temperature control to prevent death due to overheating is conserved to higher primates mostly [2]. Thus, the release of aqueous sweat from eccrine glands for thermoregulation and under stress results in the formation of wet stains on clothes, especially in the axilla. The second type of sweat glands, apocrine ones, are restricted to certain body parts with hairy skin as they are connected to hair follicles [3, 4]. Constituents of the apocrine secreted oily sebum are decomposed by skin microorganisms into small volatile and partly odorous molecules which are the source of the human body malodor [5]. There exists a huge portfolio of deodorizing actives ranging from antibacterial compounds to malodor covering perfumes [6]. In contrast, the only effective antiperspirant technology of today to reduce axillary wetness is based on aluminum salts and was already described in a patent in 1941 [7]. Together with mucopolysaccharides contained in the aqueous eccrine sweat, aluminum salts form a hydrogel plug which physically blocks the sweat release from the gland [8]. Some research studies investigating the impact of aluminum on health raise concerns by connecting, among other influences, aluminum-containing cosmetics with increased risk of breast cancer or Alzheimer's [9, 10]. These allegations lead to the consumer's rejection of conventional antiperspirants and an increasing demand for aluminum-free alternatives [11]. In connection with this, recommendations of non-governmental organizations as well as advertising claims arouse a trend for "clean products". Pursuing the consumer's demands, we started searching for alternative principles to diminish sweating.

Medical treatment of excessive sweating (hyperhidrosis) with Botox indicates the potential to reduce sweating by biological means [12]. However, to identify new antiperspirant ingredients acting specifically on the biology of the sweating process and as a pre-screening prior to complex *in vivo* studies, sophisticated cell-based *in vitro* test systems need to be developed. Relying on our experiences with the Phenion® human skin models, Henkel Beauty Care developed a three-dimensional (3D) *in vitro* cell model of the human eccrine sweat gland to investigate the ion-based regulation mechanism of sweat production and its secretion from the sweat gland [13]. In a multistage screening process comprised of initial *in vitro* tests on monolayer cells and verification using the 3D *in vitro* eccrine sweat gland model a broad spectrum of chemical structures were tested. Finally, the efficacy of promising substances was cross-checked in volunteer studies *in vivo*.

3D *in vitro* Cell Model of the Human Eccrine Sweat Gland

In a conventional two-dimensional eccrine sweat gland cell-based assay *Ertongur-Fauth et al.* showed, by targeting the TMEM16A ion channel, that specifically active molecules are able to modulate this ion channel in eccrine sweat gland cells [14]. However, the process of sweat secretion is much more complex with the involvement of several other ion channels, proteins and receptors crucial for physiological sweating [15]. Analyzing gene and protein expression patterns we noticed the substantial loss of some of those sweat-relevant proteins in conventional monolayer sweat gland cell cultures. In the following, we successfully established a 3D *in vitro* cell model of the human eccrine sweat gland to circumvent the deficiency of vital proteins and associated loss-of-function within the cells [13].

The generation of these organoids includes several crutial steps: First, required sweat gland cells were isolated from native human eccrine sweat glands out of surgical skin biopsies obtained with written informed consent of the patients. Those tissue explants were transferred into culture medium and cultivated at 37°C until cells started to grow out. When cells covered nearly the whole culture surface, they were separated from the remaining original gland and expanded as monolayer cultures. After reaching a high number of cells, cell suspensions were prepared as the basis for generating the 3D cell models using the Hanging Drop-Technology (**Fig. 1**). Eccrine sweat gland cells aggregate within a small amount of a hanging medium droplet over a few days forming the 3D eccrine sweat gland

Fig. 1 Eccrine sweat gland cells in a suspension aggregate at the tip of the medium droplet in a Hanging Drop Plate forming the 3D eccrine sweat gland model.

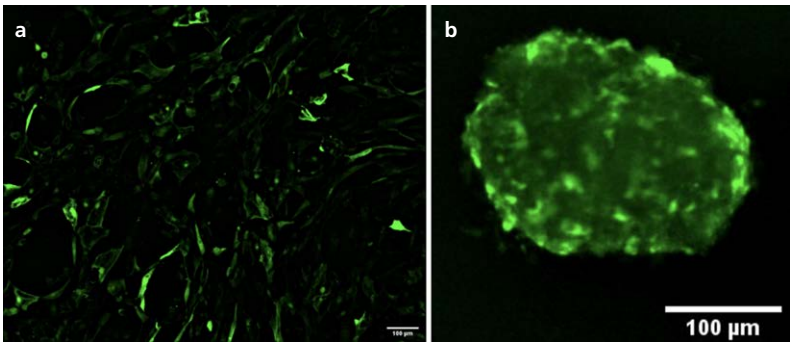


Fig. 2 2D cell culture (a) and 3D eccrine sweat gland model (b) in which the cells are loaded with an ion-sensitive fluorescence dye to measure the changes of intracellular ion contents. Bar represents 100 µm.

model (**Fig. 2**). Due to their intensified cell-cell-contact within the spheroid and the more physiological cell structure as compared to monolayer cells, several proteins essential for sweat generation are re-induced. Most importantly, the muscarinic acetylcholine receptor M3 (CHRM3) and the water channel aquaporine 5 (AQP5) are again expressed, which are nearly absent in monolayer cells. Besides, *Klaka et al.* demonstrated the high resemblance of the 3D cell model to the native eccrine sweat gland in the human skin. By revealing an orientation of the cells within the organoid based on their protein expression pattern the close relation to the *in vivo* situation was established [13].

Altogether, this model promises relevance as a striking tool for screening of potential alternative antiperspirant ingredients.

The *in vitro* Screening

Sweating is a complex mechanism involving the active and passive transport of ions and water throughout the sweat gland cells into the gland lumen. Based on genomic analyses and knowledge from literature, we identified several ion transporters, receptors and water channels as being crucial for normal perspiration [15, 2]. Extracting the most relevant information from these observations, four ions came into focus as being essential in the sweat generation process. Those are calcium (Ca^{2+}), sodium (Na^{+}), potassium (K^{+}) and chloride (Cl^{-}). While calcium is a second messenger released inside the cells upon activation of the sweat gland cells by cholinergic stimulation, sodium, potassium and chloride are the ions with the highest concentration in eccrine sweat gland cells and, consequently, in sweat [16]. To be able to investigate the influence of different substances on the reaction of eccrine sweat gland cells and, figuratively, on sweat production, test systems were developed facilitating the monitoring of these events. First, these analyses were established in common monolayer cell cultures and were subsequently transferred to our 3D sweat gland model. The measurements are based on the quantitation of relative changes in the respective intracellular ion concentration. The assay principles rely on fluorescence dyes, each specific for one of the mentioned ions, which are loaded into the cells. Thereby, each ion variation is measured in one separate assay. After determination of the basic fluorescence intensity, the cells / the 3D models are treated with the test substance (**Fig. 2**). Obtained shifts in the fluorescence intensity upon application of the test solution give a clue on the respective reaction of the cells. Taking all tests together, the recorded changes in the four important ions yield a characteristic ion pattern specific for each test substance.

For validation of functionality of the test system, well-known pharmacological stimulators and inhibitors of sweating were examined and their ion patterns generated. Both, stimulation and inhibition of the cells, induced particular alterations of the intracellular ion contents, observable as a specific ion pattern. As an example, carbachol, an analogue of the endogenous neurotransmitter acetylcholine, simulated the natural stimulation of sweating. In reaction to this stimulus the Na^{+} content within the eccrine sweat gland cells increased.

As the first step of the screening process the highest non-cytotoxic concentration of the substance is determined to ascertain the viability of cells within the assays. In the following, the impact of this test concentration on the cell's intracellular ion levels is specified in monolayer cells. Only candidates with a good pre-screening are then further validated using the more complex 3D eccrine sweat gland organoid to obtain a more valid data set. Substances revealing a clearly divergent ion profile from the one of classified stimulants are considered hits of which the best ones are subsequently chosen for validation in *in vivo* sweat reduction studies.

During the broad screening procedure of multiple chemical structures, treatment of the cells with a new potent candidate, substance 1, unveiled a striking effect: With increasing concentrations of the test substance the intracellular Na^{+}

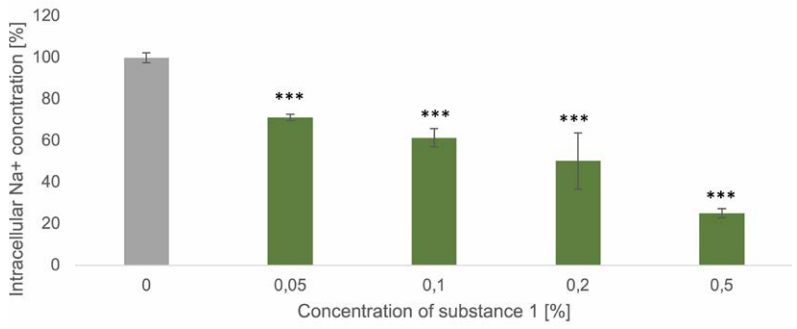


Fig. 3 Concentration dependent reduction of the intracellular sodium content after treatment of eccrine sweat gland cells with substance 1. ***: significantly different from 0% with $p < 0.0001$ (one-way ANOVA with Tukey's post-hoc multiple comparison test). Data are mean ± SD of two independent experiments performed in sextuplet.

level of the cells decreased (**Fig. 3**). As this reaction is contrary to the increase in Na^+ induced by the stimulant carbachol, we expected substance 1 to be a potential efficient regulator of sweating. However, its actual sweat reducing capability had still to be proven in a concomitant *in vivo* study.

In vivo Validation of *in vitro* Observations

To designate an antiperspirant effect a confirming result in an *in vivo* sweat reduction study is mandatory. At a contracted dermatological test institute those studies on the back of volunteers (age 35 to 75) are performed. Due to higher susceptibilities of the *in vitro* cell models resulting from a reduced barrier as compared to the native skin, the concentration of the active was increased by a factor of 10 in this *in vivo* volunteer study.

In the study, a defined volume of an aqueous solution containing the test substance is applied on the back of 18 or 20 study participants. Five minutes after administration the treated areas, and a corresponding control spot on the parallel side of the spine, are covered with an occlusive, non-absorbing foil for two hours (**Fig. 4**). Described procedure is repeated on four consecutive days. 24 hours after the last application absorptive pads are placed on the treated and control areas, respectively. Subsequently, sweating of the volunteers is induced by resting in a sauna at 80°C for about 15 minutes with the pads collecting the locally produced sweat. Gravimetric measurement of the absorbed amount of sweat on the pad and comparison with the respective untreated control enables determination of the sweat reduction efficiency of the test substance. An aqueous solution of aluminum chlorohydrate serves as a benchmark in this study design with a relatively constant sweat reduction of about 50% (**Fig. 5**). Application of different concentrations of substance 1 in these *in vivo* back studies revealed another outstanding effect: A positive correlation of increasing sweat reduction with increasing substance 1 concentrations was determined (**Fig. 5**). Most astonishingly, at a concentration of only 3%, substance 1 was able to diminish the sweat production by well over 60%, making substance 1 as effective as the commonly used aluminum salts. Unfortunately, during following skin compatibility testing some volunteers exhibited incompatibility reactions after application of a cosmetic formulation containing substance 1, rendering its usage as an alternative antiperspirant ingredient problematic. Nonetheless, the striking sweat reducing potential of this compound was demonstrated indicating the general capability of biological actives to effectively diminish sweating.

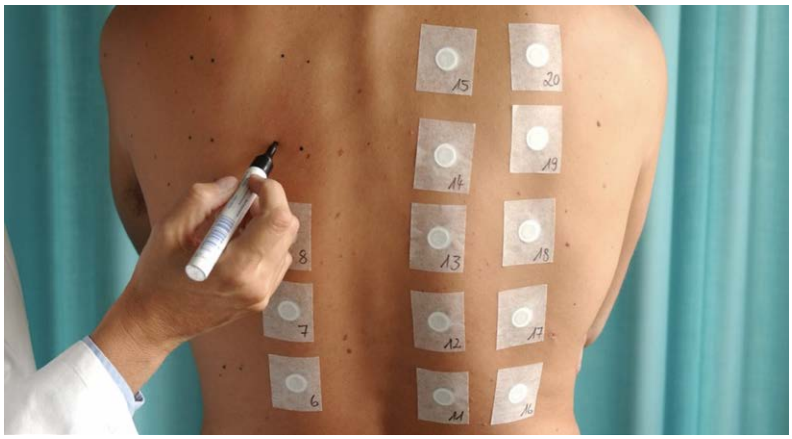


Fig. 4 Photograph of a volunteer's back in a sweat reduction study during occlusion phase.

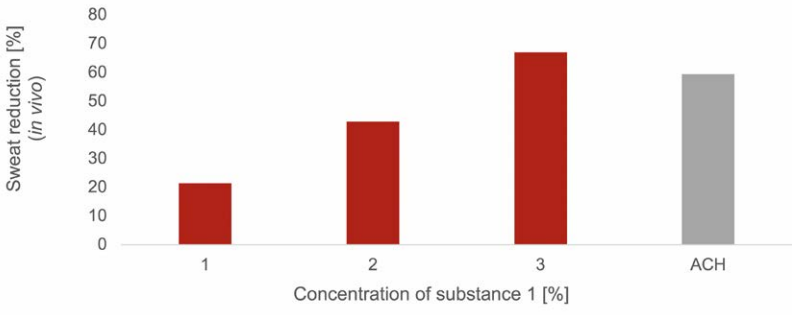


Fig. 5 Sweat reducing effect of test substance 1 determined on the back of volunteers. Increasing concentrations of test substance 1 enhanced the sweat reducing potential up to the efficiency reached with conventionally used Aluminium chlorohydrate (ACH). Data are mean of 15-20 volunteers.

In vitro / in vivo Correlation

The obtained results from both, *in vitro* experiments and *in vivo* studies with substance 1, confirmed the suspected connection between the intracellular changes of the ion equilibrium of the 3D eccrine sweat gland model and the impact of the actives on sweat secretion *in vivo*.

To illustrate this effect, we correlated the remarkable *in vivo* and *in vitro* results: The decreasing intracellular Na^+ concentration *in vitro* corresponds to an increasing *in vivo* sweat reduction as depicted in **Fig. 6**. For better correlation, the lower concentration of substance 1 in the *in vitro* assays is adjusted by a factor of 10.

The discovery of such a correlation is, to the best of our knowledge, the first description of *in vitro* generated data of ion profiles enabling the successful prediction of *in vivo* sweat reducing effects in humans. Further corroborating studies need to be performed to substantiate the read-out parameter of intracellular Na^+ concentrations as a cell-biological indicator for the physiological sweat reducing effect in humans.

Altogether, these results point to substance 1 as an effective antiperspirant ingredient with a performance in the range of the conventionally used aluminum chlorohydrate. Although substance 1 itself may not be used in a cosmetic formulation due to evoking irritative reactions in humans, it definitely gives a lead to further substance screenings. With this eminent *in vitro/in vivo* correlation uncovered, searching for an alternative to aluminum salts will be much more focused and promising.

Conclusion

The consumer's demand for alternative sweat reducing actives replacing aluminum salts in antiperspirant formulations is increasing due to publicly raised health concerns. To facilitate the search for possible substitutes, new, sophisticated but simple methods need to be invented for screening of substances prior to time consuming and expensive *in vivo* studies.

Herein we describe a cell-based system employing isolated primary eccrine sweat gland cells for screening of those potential alternatives to aluminum salts. Using this validated 3D sweat gland model, several assays were implemented enabling the determination of intracellular changes of Ca^{2+}, Na^+, K^+ and Cl^- ions upon treatment with possible sweating inhibitors. The focus on specifically these selected ions is based on their importance for the physiological process of sweat production.

During the broad search for biologically active substitutes including various chemical structures, substance 1 elicited a concentration-dependent decrease of the intracellular Na^+ levels when aqueous solutions were applied to the cell system. Strikingly, these *in vitro* observed results correlated to an actual sweat reducing effect of substance 1 in consecutive *in vivo* validation studies on the back of volunteers. Even more, the diminution of sweating elicited by substance 1 was on the same level as achieved with the commonly used aluminum chlorohydrate.

In conclusion, our developed cell-based screening system using primary eccrine sweat gland cells in a proprietary 3D eccrine sweat gland model turns out as a reliable tool for searching of alternatives to aluminum salts in antiperspirants. Furthermore, the discovery of substance 1 signifies the capability to screen for biologically active antiperspirants using mentioned *in vitro* techniques. With its impact on the intracellular Na^+ concentration in eccrine sweat gland cells and its sweat reducing effect *in vivo*, substance 1 allows the first description of *in vitro* read-out parameters indicating a sweat reducing potential in humans. Further confirmation of this *in vivo/in vitro* correlation will show its relevance for the prediction of *in vivo* antiperspirant effects from cell-based tests. This will drastically reduce the number of necessary studies involving human volunteers and will speed up the identification process to come up with alternative antiperspirant actives.

Altogether, based on these excellent results the first aluminum-free antiperspirant products are currently under development, which will act through a direct biological regulation of eccrine sweat production.

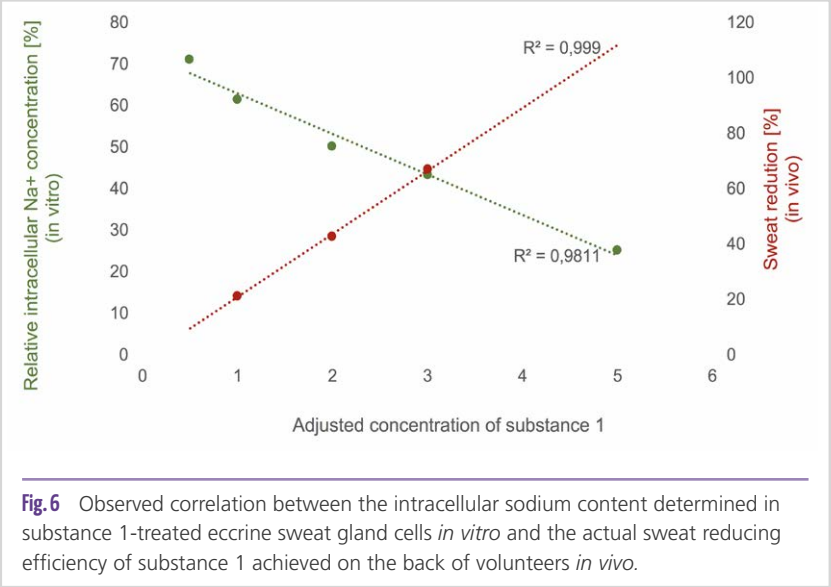


Fig. 6 Observed correlation between the intracellular sodium content determined in substance 1-treated eccrine sweat gland cells *in vitro* and the actual sweat reducing efficiency of substance 1 achieved on the back of volunteers *in vivo*.

Acknowledgements

We thank the Natural Life Excellence Network 2020 (NatLifE 2020) funded by the German Federal Ministry of Education and Research (Bundesministerium für Bildung und Forschung, BMBF) for supporting part of the research.

References

[1] *Sato, K.; Kang, W. H.; Saga, K.; Sato, K. T.* (1989): Biology of sweat glands and their disorders. I. Normal sweat gland function. In: Journal of the American Academy of Dermatology 20 (4), S. 537–563. DOI: 10.1016/S0190-9622(89)70063-3.

[2] *Bovell, Douglas* (2015): The human eccrine sweat gland. Structure, function and disorders. In: Journal of Local and Global Health Science 2015 (1), S. 5. DOI: 10.5339/jlghs.2015.5.

[3] *Schiefferdecker, P.* (1922): Die Hautdrüsen des Menschen und der Säugetiere, ihre biologische und rassenanatomische Bedeutung, sowie die Muscuiaris sexualis. Zoologica - 27_72. Stuttgart: E. Schweizerbart'sche Verlagsbuchhandlung.

[4] *Montgomery, I.; Jenkinson, D. M.; Elder, H. Y.; Czarnecki, D.; MacKie, R. M.* (1984): The effects of thermal stimulation on the ultrastructure of the human atrichial sweat gland. I. The fundus. In: British Journal of Dermatology 110 (4), S. 385–397. DOI: 10.1111/j.1365-2133.1984.tb04652.x.

[5] *Natsch, Andreas* (2015): What Makes Us Smell: The Biochemistry of Body Odour and the Design of New Deodorant Ingredients. In: Chimia 69 (7-8), S. 414–420. DOI: 10.2533/chimia.2015.414.

[6] *Finkel, Peter* (1995): Deodorantien. In: Wilfried Umbach (Hg.): Kosmetik. Entwicklung, Herstellung und Anwendung kosmetischer Mittel. 2. erweiterte Auflage. Stuttgart, New York: Thieme, S. 360–371.

[7] *Montenier, Jules B.* (1941): Astringent preparation am 1941. Anmeldenr: 309,830. Veröffentlichungsnr: US2230084A.

[8] *Darbre, P. D.* (2001): Underarm cosmetics are a cause of breast cancer. In: European Journal of Cancer Prevention 10 (5), S. 389–394. DOI: 10.1097/00008469-200110000-00002.

[9] *Linhart, Caroline; Talasz, Heribert; Morandi, Evi M.; Exley, Christopher; Lindner, Herbert H.; Taucher, Susanne et al.* (2017): Use of Underarm Cosmetic Products in Relation to Risk of Breast Cancer. A Case-Control Study. In: EBioMedicine 21, S. 79–85. DOI: 10.1016/j.ebiom.2017.06.005.

[10] *Mandriota, Stefano J.; Tenan, Mirna; Ferrari, Paolo; Sappino, André-Pascal* (2016): Aluminium chloride promotes tumorigenesis and metastasis in normal murine mammary gland epithelial cells. In: International Journal of Cancer 139 (12), S. 2781–2790. DOI: 10.1002/ijc.30393.

[11] Future Market Insights (2019): Aluminium-free deodorant market poised to rake in $1,200 million (~€1,076 million) in 2019. Health Europa. Online verfügbar unter https://www.healtheuropa.eu/aluminium-free-deodorant/91795/.

[12] *Shelley, W. B.; Talanin, N. Y.; Shelley, E. D.* (1998): Botulinum toxin therapy for palmar hyperhidrosis. In: Journal of the American Academy of Dermatology 38 (2 Pt 1), S. 227–229.

[13] *Klaka, Patricia; Grüdl, Sabine; Banowski, Bernhard; Giesen, Melanie; Sättler, Andrea; Proksch, Peter et al.* (2017): A novel organotypic 3D sweat gland model with physiological functionality. In: PloS ONE 12 (8), e0182752. DOI: 10.1371/journal.pone.0182752.

[14] *Ertongur-Fauth, Torsten; Hochheimer, Andreas; Buescher, Joerg Martin; Rapprich, Stefan; Krohn, Michael* (2014): A novel TMEM16A splice variant lacking the dimerization domain contributes to calcium-activated chloride secretion in human sweat gland epithelial cells. In: Experimental Dermatology 23 (11), S. 825–831. DOI: 10.1111/exd.12543.

[15] *Cui, Chang-Yi; Schlessinger, David* (2015): Eccrine sweat gland development and sweat secretion. In: Experimental Dermatology 24 (9), S. 644–650. DOI: 10.1111/exd.12773.

[16] *Sonner, Z.; Wilder, E.; Heikenfeld, J.; Kasting, G.; Beyette, F.; Swaile, D. et al.* (2015): The microfluidics of the eccrine sweat gland, including biomarker partitioning, transport, and biosensing implications. In: Biomicrofluidics 9 (3), S. 31301. DOI: 10.1063/1.4921039.

contact

Jessica Welzel | jessica.welzel@henkel.com
Sabine Grüdl
Bernhard Banowski
Dr. Andrea Sättler
Dr. Thomas Förster
Dr. Thomas Welss | thomas.welss@henkel.com

Henkel Beauty Care
Henkel. AG & Co. KGaA | Henkelstr. 67
40589 Düsseldorf | Germany
www.henkel.de

Effective Cleaning Through a Second Skin – Why Proteins Improve Cleaning

M. Reihmann, B. Köhler, N. Rittereiser, C. Yüce

abstract

An innovative, easy-to-clean concept with hydrophilic protective layers formed by functional collagen peptides on cleaned surfaces, has been successfully employed in professional vehicle cleaning applications. This has allowed for the reformulation of cleaners to less alkaline and more environmentally friendly conditions. In addition to improved cleaning results, extended cleaning cycles and reduced water consumption are well documented benefits. Meanwhile these protein protection layers are also finding use in hard-surface cleaners.

Here, the current focus is on effective ready-to-use cleaning products with simplified and reduced cleaning processes even at mild alkaline pH levels. For the first time, the mode of action of these proteins was measured in real-time by means of quartz crystal microbalance experiments. The data suggests that during a cleaning process multiple layers of proteins and surfactants are formed on the surface. These layers create an easy-to-clean effect by repeated application of a cleaning formulation. Even after extensive rinsing with pure water, a compact protein layer remains on the surface which acts as effective barrier against resoiling. The easy-to-clean effect of this protective layer was also confirmed macroscopically with several exemplary household cleaner formulations according to the IKW test-method for all-purpose cleaners on a scrub abrasion and washability tester (TQC Sheen).

Cleaning through a second skin – The role of proteins in a formulation

Many cleaning formulations contain additives for the protection of cleaned surfaces. Most of the surface protectants are based on water-repellant products, like waxes, silicones, and other hydrophobic film forming molecules. The basic design of such products is to leave a cleaned surface in a water-repellant state. This allows water to roll off from the cleaned surface, taking the dirt and soil with it (e.g. waxing a car after washing to obtain a beading effect). However, while this might work well for water-soluble stains, it is very difficult to remove any dispersed water-insoluble compounds once this material has been dried on the water-repellant surface.
Furthermore, almost all current surface protectants are based on petrochemical products, which strongly conflicts with the desire of consumers for products with an increased biodegradability and a reduced microplastic footprint.
In order to overcome these challenge, the use of the protein-based additive NOVOTEC® CB800 for surface protection in train cleaning has been described [1-2]. NOVOTEC® CB800 is a concentrated aqueous solution of specific collagen peptides, with an average molecular weight of 3.000 Da and a proline frequency of 14% as well as a hydroxyproline frequency of 13%. When a cleaning formulation is equipped with NOVOTEC® CB800, the polypeptide chains of this natural product will form a protective hydrophilic layer via self-organization during cleaning.
The proposed working mechanism of NOVOTEC® CB800 in combination with cleaners [3] is shown in **Fig. 1**, which represents the cleaning of a train [4]:
First, surfactants are removing soils (step 1). Simultaneously, the protein molecules form a protective layer on the cleaned surface. The protein molecules start to attach themselves to the surface via polar interactions of carboxylic and amino groups within the amino acid chain. A broad range of polar and non-polar side chain-functions assists the protein molecules to form layers on a wide range of surfaces.

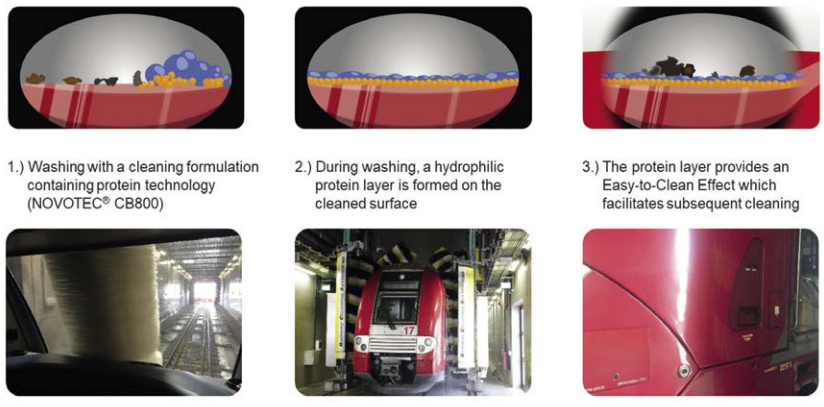


Fig. 1 Scheme of the protective hydrophilic protein layer formation during cleaning of a train surface.

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

Visit us:
in-cosmetics, booth V10

NYSCC Suppliers Day, booth 934

www.jungbunzlauer.com

Next, the proteins form a stable network via self-organization (step 2). Polar proteins with a high proline and hydroxyproline content have been found very effective for this purpose. The protein network also incorporates water from the cleaning formulation. On subsequent drying of the surface, the equilibrium moisture content of the protein film typically reaches between 8% and 12% [5].

Once the layer is established, new soil can no longer reach the cleaned surface and either flows along the water bound by the protein film or is removed as the protein layer is dynamically replenished during following cleanings (step 3). As a result, the surfaces are easier to clean.

Luxembourg National Railway Company (CFL) reported a reduction of their washing time by 30%, combined with a reduction of fresh water consumption by 90% after using protein-based cleaners at their facilities [6]. The cleaners were produced by Reinwerk Solutions, located in Bischheim, Germany. A total cost reduction by 50% displayed that the usage of those protein-based cleaners is also commercially attractive [7].

Example of the protein-induced easy-to-clean effect on a glass surface

The easy-to-clean effect caused by the protein protective layers, can be visualized by the following simple experiment: A glass plate was washed with the same glass cleaning base formulation with and without the protein-based additive NOVOTEC® CB800. The left side was prepared without protein, right side with protein (**Fig. 2**, step 1). The composition of the utilized cleaning formulation (including the protein-based additive) is provided in **Tab. 1**.

Afterwards the glass plate was treated with different permanent markers. Next, the glass plate was washed with 40 °C warm water for 30 seconds. On the right side, which was pre-cleaned with the cleaning formulation that included the protein-based additive NOVOTEC® CB800, the markers could be easier washed away (**Fig. 2**, step 2).

Tracking the protein layer formation in real-time

To evaluate the protein-surface interaction during cleaning in more detail, real-time measurements of the experiment mentioned above have been conducted using a quartz crystal microbalance instrument (QCM-D, QSense Analyzer). This equipment can measure the thickness of the protein layer and potential other layers on variable substrate surfaces via change of frequency (thicker layers will reduce the frequency) as well as their viscoelastic properties by detecting the dissipation factor (higher value corresponds to more softness).

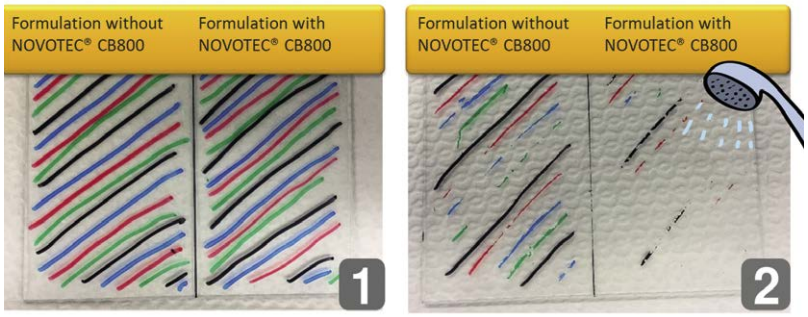


Fig. 2 Glass cleaner with NOVOTEC® CB800 induced easy-to-clean effect
Step 1: Glass plate treated with different permanent markers before rinsing with pure water; Step 2: Glass plate after rinsing with 40 °C warm water.

Glass Window Cleaner (HCI/1001)

Hard Surface Cleaner with a good cleaning an wetting ability. Excellent skin mildness

Phase	Ingredient	INCI	% w/w	Function
A	Deionized water	Water	Qs	
	Carephos® N (ICL)	Sodium Polyphosphate	0.20	Complexing Agent
	GlucoPure® WET (Clariant)	N-C8/10-acyl-N-methyl-glucamin	1.00	Surfactant
	NOVOTEC® CB800 (Gelita AG)	Gelatin Hydrolyzate	2.00	Additive
	Phenoxetol™ (Clariant)	Phenoxyethanol	0.50	Preservative
B	Sodium Hydroxide 10%	Sodium Hydroxide	Qs	pH – Adjuster

Specification:
Appearance: clear liquid
pH-value: 9.20 – 9.80
Viscosity: N/A
Stability test: Stable for 3 months at 4°C, 20°C and 40°C, 1 month at 45°C

Procedure:
I. Mix the components of phase A at room temperature until you have a clear solution.
II. Set pH-value with phase B.

Tab. 1 Glass cleaner with NOVOTEC® CB800 induced easy-to-clean effect.

The QCM-D was equipped with either gold sensors or gold sensors sputtered with soda-lime glass. After equilibrating the sensors with air (step 1, **Fig. 3**) and water (step 2, **Fig. 3**), the glass cleaning formulation equipped with protein as described above (**Tab. 1**) was pumped over the sensor, which resulted in an immediate formation of a relatively soft layer with a calculated thickness of 20-30 nm (step 3, **Fig. 3**). It is hypothesized that this layer consists of the protein layer on the sensor surface and an associated layer of absorbed surfactants. Next, the sensor was taken out of the QCM-D instrument and spin-coated with an ethanol-diluted permanent marker ink (step 4, **Fig. 3**). The ink-soiled sensor was placed back in the QCM-D and the same glass cleaner formulation was pumped over the sensor, but this time without including the protein in the glass cleaner formulation.

The high layer thickness caused by the ink on the soiled sensor vanished within seconds (step 5, **Fig. 3**) and the layer thickness dropped down to almost 5 nm followed by restoring the initial layer thickness of 20 nm. It can be assumed that the ink was washed away together with the associated surfactants from the protein surface at the beginning of step 5 followed by a new surfactant layer formation on the protein surface.

When water was pumped over the sensor, the layer thickness dropped sharply down again to 5 nm level (step 6, **Fig. 3**), as the surfactants were washed away. The dimension of the remaining protein film on the surface did not change on further rinsing with water and stayed on the 5 nm level.

In summary, the QCM-D real-time measurements suggest the following mechanism for the protein-induced easy-to-clean effect:

- Soft multilayers consisting of protein and surfactants are assembled during first cleaning
- Partial dissolving of the layers due to release of surfactants facilitates cleaning
- A thin protein layer remains on the surface as protection layer

Interaction of the proteins with surfactants

In order to better understand the interactions of surfactants with the protein, various combinations of surfactants with

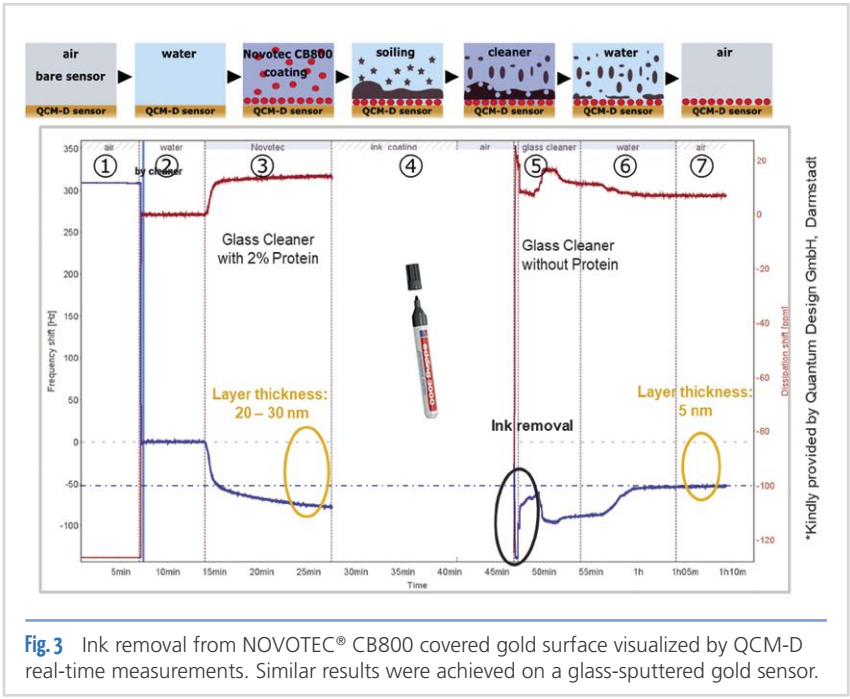


Fig. 3 Ink removal from NOVOTEC® CB800 covered gold surface visualized by QCM-D real-time measurements. Similar results were achieved on a glass-sputtered gold sensor.

NOVOTEC® CB800 have been analyzed regarding their surface tension and contact angle towards cleaned substrates. A previous study [8, 9] included the following surfactants [10, 11]: DODECYLDIMETHYLAMINE OXIDE, SODIUM 2-ETHYLHEXYL IMINO DIPROPIONATE, SODIUM N-LAUROYLSARCOSINATE, SODIUM ETHYLHEXYL SULFATE, SODIUM CAPRYLOYL GLUTAMATE, TIPA-LAURETH SULFATE, N-C8/10-ACYL-N-METHYL-GLUCAMIN, N-C12/14-ACYL-N-METHYL-GLUCAMIN-N-COCO-ACYL-N-METHYL-GLUCAMIN, C10-12 FATTY ALCOHOL EO/PO-ADDUCT, COCO-BETAINE,
COCAMIDOPROPYL BETAINE, ALKYL HYDROXYETHYL DIMETHYL AMMONIUM CHLORIDE, SODIUM LAURETH SULFATE, SODIUM LAURETH SULFATE.
Among all of these surfactants, synergistic interactions – i.e. a lower surface tension with increasing amount of NOVOTEC® CB800 protein – have been observed with glucamin-based surfactants and SODIUM CAPRYLOYL GLUTAMATE; antagonistic results have been found for SODIUM 2-ETHYLHEXYL IMINO DIPROPIONATE. The surface tension of the other surfactants did not change significantly in combination with NOVOTEC® CB800. Concentrations of up to 2.5% protein in a 5% surfactant solution were examined.
The synergistic acting glucamine-based surfactants have been chosen for further analysis of the surfactant-protein interaction during cleaning.

Protein and surfactant concentration and its influence on the cleaning performance

The effect of the surfactant and protein concentration on the cleaning performance was studied by varying the final working dilution of two all-purpose cleaning formulations. One formulation contained NOVOTEC® CB800 and for the other formulation the additive was replaced by water to maintain the amount of the other ingredients (**Tab. 2**). Surface tension and contact angle were determined by optical contact angle measuring and contour analysis system (Dataphysics OCA 50), the reported values are the average of 3 measurements. The cleaning performance was evaluated by using a scrub abrasion and washability tester (TQC Sheen).
For the performance evaluation, the current version of the IKW test-method for all-purpose cleaners [12] was adapted. As studying the protective effect requires protein pretreatment prior to soiling, it was necessary to extend the established IKW test-method by inclusion of an initial cleaning step with the selected formulations. It was decided to simply copy the conditions of the cleaning step for the pre-cleaning.
Following the IKW test-method for all-purpose cleaners, the test soil consisted of 75% peanut oil (Mazola), 23% kaolin and 2% carbon black. The defined test substrate (floor tiles by Villeroy & Boch 3135 30x30cm) was cleaned with ethanol and the test cleaner formulation (5ml) was applied to the surface with the defined towels (Wecovi 02010100, cut to 13x10 cm) by wiping 20 strokes within one minute on a scrub abrasion and washability tester (TQC Sheen). In the next step the test soil was applied on an area of 8x26 cm by screen printing. The soil was burned at 100 °C for 24 h followed by a setting step for 24 h at room temperature. In the final step the tile was again cleaned with the defined towels wetted with 5ml of the test cleaning formulation. This cleaning cycle consisted again of 20 strokes within one minute.
The application of the concentrated cleaner did not show a major difference in the cleaning performance. That was mainly because the cleaner formulation was powerful enough to remove the test soil even without protein pretreatment effectively with 20 strokes (**Fig. 4**). This experiment verified, that the cleaning formulation was adequately formulated to remove the test soil.

Kitchen Cleaner (HCI/1006)
Kitchen Cleaner against grease with an easy to clean effect. Excellent skin mildness.

Phase	Ingredient	INCI	% w/w	Function
A	Deionized water	Water	Qs	
	Carephos® N (ICL)	Sodium Polyphosphate	0.30	Complexing Agent
	GlucoPure® DEG (Clariant)	N-C12/14-acyl-N-methyl-glucamin	2.00	Surfactant
	NOVOTEC® CB800 (Gelita AG)	Gelatin Hydrolyzate	2.00	Additive
	Genaminox® CSL (Clariant)	Coco dimethyl amoneoxide	1.50	Surfactant
	Isopropanol	Isopropanol	1.00	Solubilizer
	Phenoxetol™ (Clariant)	Phenoxyethanol	0.50	Preservative
B	Sodium Hydroxide 10%	Sodium Hydroxide	Qs	pH – Adjuster

Specification:
Appearance: clear liquid
pH-value: 9.50 – 10.00
Viscosity: N/A
Stability test: Stable for 3 months at 4°C, 20°C and 40°C, 1 month at 45°C

Procedure:
I. Mix the components of phase A at room temperature until you have a clear solution.
II. Set pH-value with phase B.

Tab. 2 All-Purpose cleaning formulation with NOVOTEC® CB800 induced easy-to-clean effect.

While the surface tension of both cleaning formulations was almost similar (28.3 and 28.9 mN/m respectively), clear differences were visible regarding the contact angle of the cleaning formulations on a tile washed with ethanol, but without further pretreatment. The protein-based cleaner showed a contact angle of 6.6° while the formulation without NOVOTEC® CB800 had a contact angle of 11.7° (**Fig. 4**). As the surface tension of both cleaners was comparable, the lower contact angle of the protein-based cleaning formulation must be caused by an immediate formation of hydrophilic protein layers once the formulation came in contact with the tile surface.

The impact of the protein protection layers on the cleaning process was clearly visible once the cleaning formulations were diluted with water. The previously mentioned cleaning experiment was repeated, but this time with an aqueous 1:1 dilution of the cleaning formulations (with and without protein) described in **Tab. 2** for the protein based cleaner. Each diluted cleaner was used for the corresponding pre-cleaning and the consecutive cleaning step. The test soil was still effectively removed with 20 strokes from the NOVOTEC® CB800 protection coating, although the pre-cleaning step generating the protein layer was performed with the diluted cleaner. The side of the tile, which was pretreated with the cleaning formulation without NOVOTEC® CB800 remained soiled after 20 strokes (**Fig. 5**).

The analysis of the surface tension and contact angle gave very similar results as received with the non-diluted cleaners. While the surface tension of both cleaners was again almost equal (27.9 and 27.7 mN/m respectively), the contact angle measurement resulted in 4.5° for the protein containing cleaner versus 10° for the protein-free cleaner on a tile washed with ethanol, but without further pretreatment. Thus, obviously even the diluted cleaner was effectively releasing enough protein on the surface of the tile during the first cleaning step, to build a hydrophilic protection layer and make the second cleaning more effective.

In summary, these results are indicating an interesting method for cutting cost of cleaning formulations, especially in applications where cleaners are applied regularly and/or repeatedly.

Differences between cleaning with and without protein protection were also evident at high dilutions (1:5). When the tile was pre-cleaned with the highly diluted cleaning formulations with and without NOVOTEC® CB800, there was still a major difference visible after 20 strokes of the second cleaning step. However, the final cleaning result was not sufficient for both formulations (see **Fig. 6**). This indicates that neither the low protein concentration in the pre-cleaning step nor the surfactant content in the actual cleaning step were sufficient to remove the dirt completely. Interestingly,

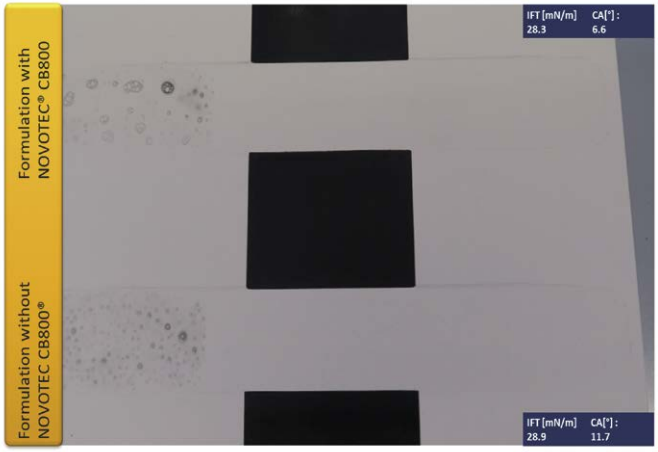


Fig. 4 A tile pre-washed with concentrated cleaner with/without NOVOTEC® CB800 and cleaned again with the respective cleaners shows only a minor improvement due to protein protection of the first cleaning.

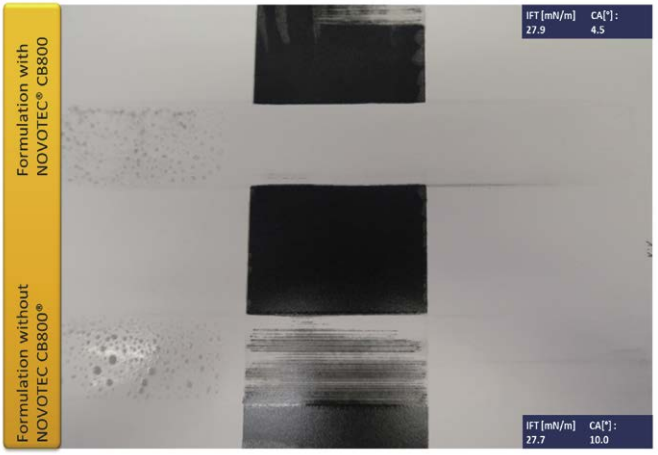


Fig. 5 A tile pre-washed with 1:1 diluted cleaner with/without NOVOTEC® CB800 and cleaned again with the respective cleaners shows a major improvement due to protein protection of the first cleaning.

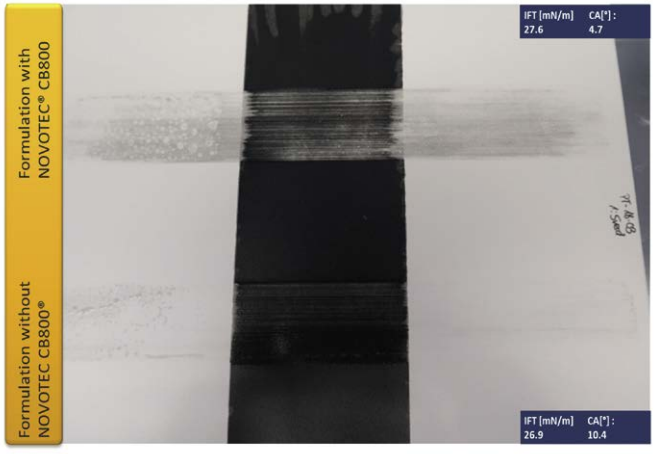


Fig. 6 A tile pre-washed with 1:5 diluted cleaner with/without NOVOTEC® CB800 and cleaned again with the respective cleaners shows only an improvement due to protein protection of the first cleaning but the final cleaning result is not satisfying.

however, the determined contact angle values for the 1:5 diluted cleaners on the untreated tile were quite similar to the values determined for the 1:1 dilutions, which suggests that the hydrophilic protein protective layers are formed even with highly diluted detergent formulations. Thus, the unsatisfactory cleaning result is probably primarily due to an insufficient surfactant concentration.

Conclusion

The presented results show that the performance of water-based cleaners may be boosted by adding small fractions of the protein-based additive NOVOTEC® CB800 to the formulation. During cleaning, the proteins attach themselves to the surface and form stable hydrophilic layers by self-organization. These layers attract water and provide an easy-to-clean effect. Any soil formation on cleaned surfaces can be removed more easily and effectively. QCM-D analyses suggest that if NOVOTEC® CB800 is combined with surfactants, soft multilayers consisting of protein and surfactants are assembled during first cleaning. These layers partially dissolve during subsequent cleaning, releasing surfactants that facilitate the cleaning while a thin protein layer remains.
This opens new opportunities to create more natural cleaners and at the same time provide very cost-effective cleaning formulations by lowering the surfactant concentration.
Moreover, the presented protein-based additive, NOVOTEC® CB800, as a natural polymer, is fully biodegradable, has a very low allergenic potential, protects skin and is not subject of discussions on microplastics in contrast to synthetic polymers.

Acknowledgements

The authors would like to thank *Christopher Schmid*, Quantum Design GmbH for the QCM-D results, *Stefan Wiemer*, Ter Ingredients GmbH & Co. KG for providing the cleaning formulations, *Dr. Paul Pawelzyk*, GELITA AG for surface tension measurements and *Dr. Eric Yezdimer*, GELITA USA for scientific contribution, *Selina Seip*, GELITA AG, for the work with the scrub abrasion and washability tester and *Marina Arnold*, GELITA AG for the work with the optical contact angle measuring and contour analysis system.

References

[1] *Becker, H.*, 2016. Nachhaltiger Lackschutz dank Proteinen. Eisenbahntechnische Rundschau, 3, 48-51

[2] *Becker, H.*, 2016. Sauber bis in die Bilanz. Sauber, 1,22-24

[3] Cleaners produced and distributed by Reinwerk Solutions, Bischheim, Germany

[4] Pictures released by CFL (Luxembourg National Railway Company)

[5] *R. Schrieber* and *H. Gareis.*, 2007. Gelatine Handbook: Theory and Industrial Practice, Weinheim, Wiley-VCH, 107

[6] *Tesch, D.*, 2018. Der perfekte Wasserkreislauf. Sauber, 3, 18-21

[7] *Becker, H.* 2017. Proteine schonen Umwelt und Budget. Eisenbahntechnische Rundschau, 3, 20-21

[8] *Reihmann, M. , et al.*, 2018. Effective Cleaning with a Second Skin. SEPAWA Congress, Berlin, October 12

[9] *Reihmann, M. , et al.*, 2019. Why Proteins Improve Cleaning. SEPAWA Congress, Berlin October 29

[10] Kindly provided by Zschimmer & Schwarz GmbH & Co KG Chemische Fabriken

[11] Kindly provided by Ter Ingredients GmbH & Co. KG

[12] IKW Recommendation for the Quality Assessment of the Product Performance of All-Purpose Cleaners 2014, German Cosmetic, Toiletry, Perfumery and Detergent Association (IKW), Frankfurt, 2014

contact

Dr. Matthias Reihmann | Head of Global Product Management Photo/Technical
Dr. Berthold Köhler | Manager Administration/Research & Business Development
Nina Rittereiser | Product Manager Photo/Technical Europe
Dr.-Ing. Ceren Yüce | Technical Specialist/Technical Product Management Photo/Technical

GELITA AG
Uferstraße 7
69412 Eberbach | Germany

Corresponding author:
matthias.reihmann@gelita.com

GLU – THE HIDDEN CHAMPION

Amino acids are the basis of all life. So why not use the nearly omnipresent glutamic acid as the base molecule for cleaning processes?

The key is intelligent modification to create the desired properties of mild cleaning combined with moderate foaming: ZETESECO HS 100 is the right choice for neutral as well as alkaline cleaning formulations – from laundry to hard surface cleaners.

Contact us for more information:
cleaning@zschimmer-schwarz.com

ZSCHIMMER & SCHWARZ

zschimmer-schwarz.com

Determination of CMCs – Results from CESIO/TEGEWA Working Groups

J. Venzmer

abstract

Because of the trend towards "micellar" formulations, surfactant manufacturers often get requests to prove the existence of micelles and/or provide values for Critical Micelle Concentrations (CMC). Therefore, the CESIO Working Group "Test Methods of Surfactants" and the TEGEWA Working Group "Surface Active Substances" have conducted round robin tests using "technical" surfactants, i.e. surfactants having alkyl chain length distributions: A) fatty alcohol ethoxylates with 9EO and different alkyl chain lengths (C12-C18) and B) two alkylamidopropyl betaines (coco vs. C12). The methods employed were the standard procedures established in industry, e.g. Wilhelmy Plate, du Noüy Ring or Pendant Drop. Two aspects have been the focus of this work: A) the effect of alkyl chain length variations on the CMC values, and B) the influence of the experimental procedures on the results of the surface tension measurements. There is indeed a significant influence of the experimental procedure on the surface tension values – especially for surfactants with broad alkyl chain distribution. Since these differences are mostly below CMC, the values of the CMCs itself are somewhat consistent. However, giving more "precise" values than one significant digit does not make much sense. But this should be sufficient, since in practical applications one is always well above CMC.

Introduction

In the last few years, there was a trend towards "micellar" formulations such as micellar water or micellar shampoos. As a consequence, surfactant manufacturers often get requests by formulators to provide both proof of the formation of micelles and/or values for Critical Micelle Concentrations (CMC) of their products. There are long established procedures and norms how to measure surface tensions (Wilhelmy Plate or du Noüy Ring Method [1, 2] Pendant Drop Method [3]), which can also be applied to determine CMCs by measuring the surface tension as a function of concentration [4]. Nowadays, corresponding computer-controlled equipment is commercially available and well-established in the industry, including autodilution functions to determine CMCs automatically. These norms, however, mainly describe how to perform test methods in terms of equipment, chemicals used for cleaning or calibrating, the exact procedure and how to calculate the results – usually there is less information concerning scope, limitation and the applicability to certain surfactant classes. In scientific studies dealing with CMCs, either ultra-pure surfactants (without homologes) or surfactants with unknown homologe distribution are used. Accordingly, often there is quite a variation in the CMC values reported [5]. Therefore, the CESIO Working Group "Test Methods of Surfactants" and the TEGEWA Working Group "Surface Active Substances" have asked themselves if and how the usual methods to determine CMC values can be applied to "technical" surfactants used in the industry, i.e. surfactants based on broad alkyl chain length distributions (e.g. coco C8-C18) instead of e.g. pure C12.

For the experiments in this study, which were conducted as Round Robin tests, two types of model surfactants having different chain length distributions were examined, i.e. a series of fatty alcohol ethoxylates with 9 EO (C12, C12/C14, C12-C18, C10+C18) as well as two alkylamidopropyl betaines (coco vs. C12). The equipment was commercially available standard tensiometers typically used in the industry, i.e. DuNoüy ring or Wilhelmy plate (mostly using the autodilution feature), as well as Pendant Drop Tensiometry of individually prepared solutions. Two aspects have been the focus of this work: On the one hand, the influence of the experimental procedures on the results of the surface tension measurements. On the other hand, the effect of alkyl chain length variations on the CMC values determined.

Materials & Methods

The nonionic surfactants were all laboratory products prepared at Schärer&Schläpfer, basically variation on the theme C12E9 with different alkyl chain length (distributions). Since the same ethoxylation conditions have been used for all products, it can be expected that the EO chain length distribution is pretty much identical, and there is only a variation in alkyl chain length distributions: C12E9 (>98% C12), C12/14E9 (70% C12, 30% C14), C12-18E9 (55% C12, 22% C14, 11% C16, 12% C18), C10E9 (>98% C10) and C18E9 (>98% C18).

As amphoteric surfactants, Cocamidopropyl Betaine (CAPB; 7% C8, 7% C10, 50% C12, 18% C14, 10% C16, 8% C18) and Lauramidopropyl Betaine (LAPB; >99% C12) have been used; both were laboratory products prepared at Evonik under

the same experimental conditions; the levels of residual amidoamine was quite low (0.037% for CAPB; 0.057% for LAPB), and the active matters were 31.1 and 30.0%, respectively.
Samples of these surfactants have been sent to the seven participating laboratories; not every laboratory possesses or has used all methods discussed here. Also, we did not agree on an experimental protocol - every laboratory used its inhouse standard procedures. For the Du Noüy Ring or Wilhelmy Plate method there exist three different standard procedures, namely (i) stepwise addition of surfactant stock solution (e.g. 5 g/L) to water, (ii) preparation of individual solutions, and (iii) the autodilution feature in the inverse mode, i.e. stepwise dilution of a surfactant stock solution with water. The latter option, often called "inverse" CMC determination, seems to be the most frequently used for routine measurements, since there is less cleaning of equipment required – the titration apparatus is only filled with pure water rather than a surfactant stock solution. In the Pendant Drop method, the surface tension is recorded as a function of time. The values reported here are the equilibrium values, either after 600 s or until a constant surface tension has been reached. The water used to prepare the surfactant solutions was either doubly-distilled or MilliQ water.

Results & Discussion

The results of the surface tension measurements using the nonionic surfactant C12/14E9 (**Fig. 1**) might look confusing at first sight. The data points with filled brown symbols are all from individually prepared solutions using the Pendant Drop method. Especially close to the CMC and above, the surface tension values are pretty consistent, and independent of the laboratory in which the measurements have been conducted. One advantage of the Pendant Drop method is that the surface age is well defined, and hence the values detected are equilibrium surface tension values. Since the surface tension is determined as a function of droplet lifetime, in principle the diffusion kinetics of the slower, more hydrophobic components to a freshly created air/water interface can be observed as well. This, however, goes beyond the scope of this study. In the Du Noüy Ring or Wilhelmy Plate method, using stepwise dilution or addition of surfactant, the surface age is not defined. Hence, the surface tension values (below CMC!) are somewhat lower, most probably because of the accumulation of the most hydrophobic components at the surface over time. Another reason, why the Pendant Drop values are higher, could be depletion effects [6]. Looking at the open symbols, it is obvious that the „reverse" CMC method (i.e. stepwise dilution of concentrated surfactant solution) is especially problematic. Above CMC, the data are quite consistent, but there are large deviations below CMC. This is not totally unexpected, since the most hydrophobic components are enriched at the surface, and upon dilution, their tendency to leave the surface is low. There is obviously no real mixing, but just an exchange of the "subphase", and the more or less insoluble monolayer of surfactants persists. Independent of the method, the CMC value for C12/14E9 is ≈ 0.02 g/L; giving a more precise value is hardly possible and does not make sense, also considering the log scale of the concentration axis.

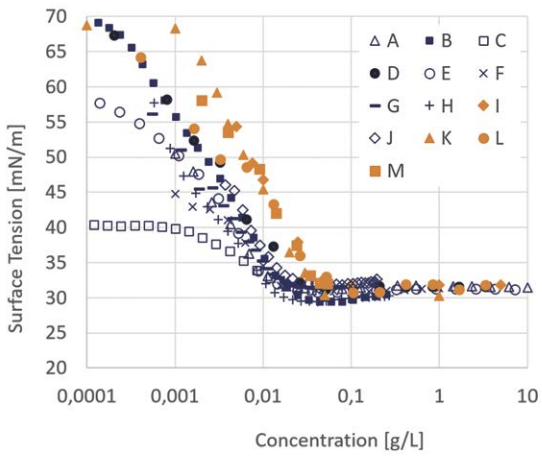


Fig. 1 Surface tension of C12/14E9 as a function of concentration; filled symbols in blue: Automated CMC by stepwise addition of surfactant stock solution to water; filled symbols in brown: Pendant Drop method of individually prepared surfactant solutions; open symbols: Automated „reverse" CMC. (Different shapes are a code for the different participants, e.g. data D, E and L have been determinded in the same laboratory).

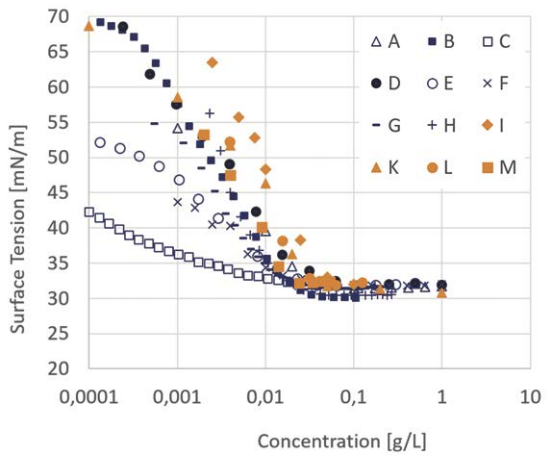


Fig. 2 Surface tension of C12-18E9 as a function of concentration; filled symbols in blue: Automated CMC by stepwise addition of surfactant stock solution to water; filled symbols in brown: Pendant Drop method of individually prepared surfactant solutions; open symbols: Automated „reverse" CMC. (Different shapes are a code for the different participants, e.g. data D, E and L have been determinded in the same laboratory).

There was initial hope to obtain more consistent CMC curves and hence more "precise" CMC values using C12E9, the "purest" ethoxylate in our series of nonionics, but these results look pretty much identical (data not shown). This means that the variation in the alkyl chain length (C12-C14) is not the main cause for the "confusing look" – it is rather the distribution in the hydrophilic headgroup, i.e. the presence of homologes carrying different numbers of EO. As a consequence, there is still a mixture of species of different hydrophilicity present, despite the well-defined alkyl chain length.

The results for the ethoxylate with a rather broad alkyl chain length distribution, C12-18E9, are given in **Fig. 2**. As expected, the broader alkyl chain distribution makes the situation worse – especially using the autodilution feature („reverse" CMC). The most hydrophobic components (C18) really have problems leaving the surface upon dilution. Also, some Pendant Drop data below CMC are quite high; the diffusion of the most hydrophobic components to the surface seems to be challenging. The CMC value itself, however, is quite similar (≈ 0.02 g/L) as compared to the one of C12/C14E9.

The results for the shortest, i.e. most hydrophilic nonionic surfactant, C10E9, look more like what can be found in a surface chemistry textbook; even the "reverse" CMC gives quite comparable results, except at concentrations much lower than CMC (**Fig. 3**). The surface tension values seem to be more or less independent of the method used, and corresponding to the considerably higher hydrophilicity and/or solubility, the CMC is significantly higher than for C12E09, ≈ 0.8 g/L instead of 0.02 g/L.

For the most hydrophobic nonionic in our test series, C18E9, the surface tension values depend a lot on the method used, and no reliable CMC values could be obtained (**Fig. 4**). Especially the reverse method fails; a CMC, i.e. a break in the surface tension curve, is hardly detectable. One additional complication using this surfactant is the cloud point [7] (CP); according to the norm [4] on CMC determination, the method is only applicable to nonionic surfactants which are soluble in water and have a CP at least 5°C above the testing temperature. Since CPs are strongly concentration dependent, it could well be that very dilute solutions are clear (<CP), whereas more concentrated solutions are turbid (>CP). In the latter case, the concentration of dissolved surfactant is unknown and the sample should not have been measured at all.

Summarizing the results using the nonionic surfactants, it can be stated that the CMC curves obtained by different laborato-

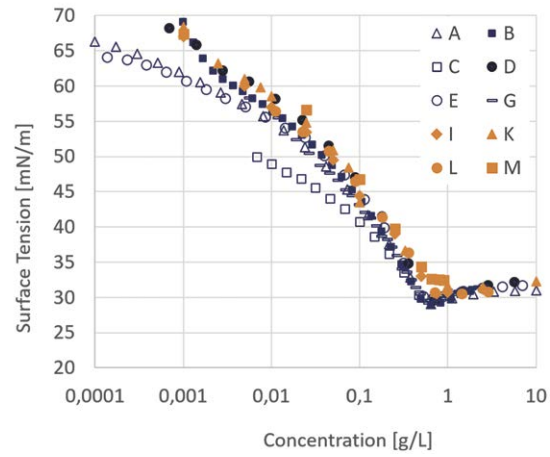


Fig. 3 Surface tension of C10E9 as a function of concentration; filled symbols in blue: Automated CMC by stepwise addition of surfactant stock solution to water; filled symbols in brown: Pendant Drop method of individually prepared surfactant solutions; open symbols: Automated „reverse" CMC. (Different shapes are a code for the different participants, e.g. data D, E and L have been determinded in the same laboratory).

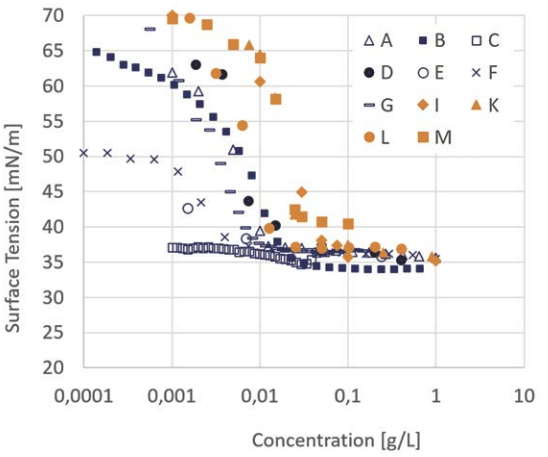


Fig. 4 Surface tension of C18E9 as a function of concentration; filled symbols in blue: Automated CMC by stepwise addition of surfactant stock solution to water; filled symbols in brown: Pendant Drop method of individually prepared surfactant solutions; open symbols: Automated „reverse" CMC. (Different shapes are a code for the different participants, e.g. data D, E and L have been determinded in the same laboratory).

ries using different methods are quite comparable; the main differences are below CMC and are most probably caused by differences in surface age. The CMC values of C12E9, C12/14E9 and C12-18E9 are pretty similar (≈ 0.02 g/L); more "precise" CMC values can hardly be obtained, since there is no sharp break in the surface tension curve detectable and considering the logarithmic scale of the concentration axis. The chain length dependencies of CMCs which are published (for a good overview see Supporting Info of [5]) could not be verified with the technical surfactants used in our study; in addition to the variations in alkyl chain length, a distribution in the number of EO units of the hydrophilic headgroup (which is almost unavoidable) seems to have a major effect on the results. Most importantly, the automated "reverse" CMC data should be treated with care, especially for more hydrophobic surfactants. Unfortunately, in brochures and data sheets typically only CMC values are given, without mentioning how the values have been generated.

For the two betaines, LAPB and CAPB, the surface tensions of individually prepared surfactant solutions using the Pendant Drop method are shown in **Fig. 5**. The shape of the curves (in terms of curvature) is pretty much as expected for a pure (i.e. single chain length) surfactant (LAPB) and a surfactant with alkyl chain distribution (CAPB) and therefore overlapping CMC curves. The CMC values according to these measurements are about ≈ 0.2 g/L for LAPB and ≈ 0.1 g/L for CAPB. The dip in the LAPB curves, as it is usually observed for Sodium Lauryl Sulfate (SDS) [8], is somehow surprising, as it indicates the presence of a hydrophobic impurity. The reason could be the amount of residual amidoamine; although it is quite low, for LAPB it is higher (0.057%) than for CAPB (0.037%).

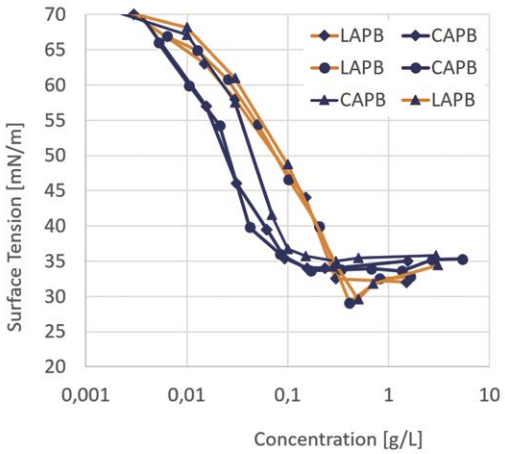


Fig. 5 Surface tension of LAPB (brown) and CAPB (blue) as a function of concentration using the Pendant Drop Method; (Different shapes are a code for the different participating laboratories).

Looking at the plot summarizing all methods for LAPB (**Fig. 6**), there are quite some discrepancies between the different methods, but hardly between different laboratories using the same method. Pendant Drop gives somewhat higher values than Ring or Plate; the „reverse" CMC method is again problematic, although there is neither an alkyl chain distribution (>99% C12) nor differences in the hydrophilic headgroup. Again, it is hardly possible to give a more precise value than ≈ 0.2 g/L.

For the most popular secondary surfactant, CAPB, the curves measured with different methods do not match at all (**Fig. 7**); however, the values obtained by different laboratories but using the same method are quite consistent. The curvature of the CMC curves is as expected for surfactant with alkyl chain length distribution, but a well-defined CMC value can also not be given – the CMC is somewhere around 0.1 g/L or slightly below. The surface tensions are somewhat higher than for LAPB, and there is hardly any „dip", i.e. no or much lower level of hydrophobic impurity present. Again, Pendant Drop gives slightly higher values than Plate or Ring. Using the „reverse" CMC is even more problematic; considering the presence of hydrophobic species up to C18, this is not unexpected.

There are several potential reasons for the differences in surface tension values determined, among them the material of the flask (plastic, glass) to prepare the surfactant solutions and to perform the measurement, as well as of the syringe used for the Pendant Drop method, and last but not least the pH value of the surfactant solution. Since the pH values were not adjusted and considering the isoelectric point of CAPB is at pH 6.25 [9], a small fraction of CAPB will already be protonated and in its cationic form. Cationic surfactants are known to be challenging during surface tension measurements as they adsorb to most solid surfaces; especially below CMC, there is a lot of surface as compared to the amount of surfactant.

Conclusions

It has been shown that – depending on the alkyl chain length distribution – the influence of the experimental procedure on the surface tension values below CMC is indeed significant. CMC values shown on data sheets are typically determined by using methods corresponding to a norm; however, the applicability of the procedures described there to "real"

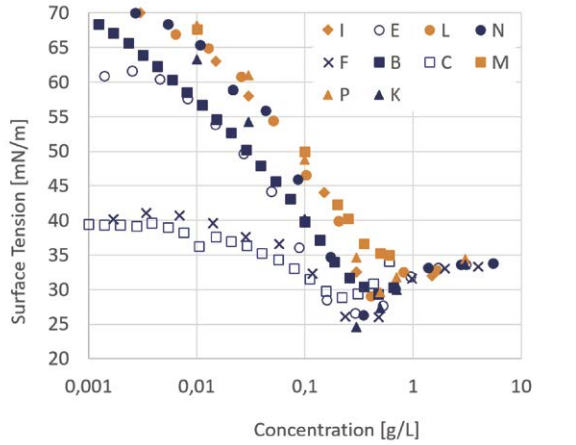


Fig. 6 Surface tension of LAPB as a function of concentration; filled symbols in blue: Automated CMC by stepwise addition of surfactant stock solution to water; filled symbols in brown: Pendant Drop method of individually prepared surfactant solutions; open symbols: Automated „reverse" CMC. (Different shapes are a code for the different participants, e.g. data E, L and N have been determinded in the same laboratory).

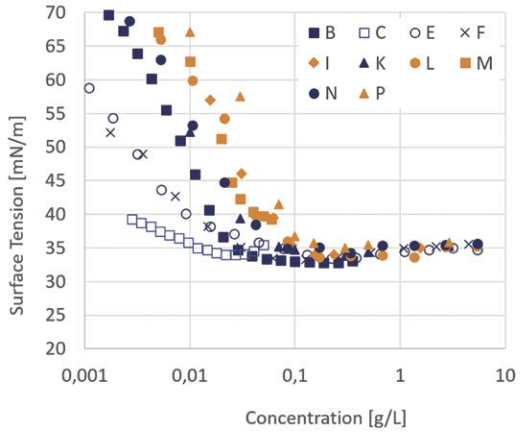


Fig. 7 Surface tension of CAPB as a function of concentration; filled symbols in blue: Automated CMC by stepwise addition of surfactant stock solution to water; filled symbols in brown: Pendant Drop method of individually prepared surfactant solutions; open symbols: Automated „reverse" CMC. (Different shapes are a code for the different participants, e.g. data E, L and N have been determinded in the same laboratory).

surfactants is not part of a norm. Depending on the surfactant, its hydrophobicity and homologe distribution (both in the alkyl chain and the hydrophilic headgroup), the surface tension curves might look more or less good. Nevertheless, the CMC values are surprisingly consistent, but considering the logarithmic concentration scale and the fact that there is often no sharp break, more than one significant digit does not make sense. Since practical applications are always well above CMC, one digit should be sufficient.

All in all, surface chemistry is a delicate beast! Automation should not replace thinking and critical evaluation of the results; especially "reverse" CMC results should be treated with care. Before doing any CMC measurements, one should always ask why a CMC value of a single surfactant is needed at all. In most surfactant mixtures, there is a synergistic interaction between the different surfactants, also affecting the CMC values [10]. Therefore, the relevance of CMC values of single surfactants for formulations containing several surfactants is rather limited.

Acknowledgments

This study was a joint effort of the members of the CESIO Working Group "Test Methods of Surfactants" and the TEGEWA Working Group "Surface Active Substances", i.e. *Roland Borner* (Schärer&Schläpfer), *Wolfgang Brennich* (Zschimmer&Schwarz), *Carmen Pey* (Kao), *Renke Rommerskirchen* (Sasol), *Natascha Schelero* (Clariant), *Kati Schmidt* (BASF), *Louis Schwarzmayr* (Nouryon) and *Michael Stapels* (Kao).

References

[1] EN 14370:2004 Surface active agents. Determination of surface tension.

[2] ISO 304:1985 Surface active agents – Determination of surface tension by drawing up liquid films.

[3] ISO 19403-3:2017 Paints and varnishes – Wettability – Part 3: Determination of the surface tension of liquids using the pendant drop method.

[4] ISO 4311:1979 Anionic and non-ionic surface active agents – Determination of the critical micellization concentration – Method by measuring surface tension with a plate, stirrup or ring.

[5] *W.C. Swope, M.A. Johnston, A.I. Duff, J.L. McDonagh, R.L. Anderson, G. Alva, A.T. Tek* and *A.P. Maschino*, J. Phys. Chem. B 123 (2019) 1696-1707.

[6] *T. Kairaliyeva, N. Mucic, L. Spasojevic , S. Bucko , J. Katona , E. Aksenenko , S. Aidarova, V. Fainerman, A. Makievski, Y. Tarasevich* and *R. Miller*, Colloids and Interfaces, 1 (2017) 1-11.

[7] ISO 1065:1991 Non-ionic surface-active agents obtained from ethylene oxide and mixed non-ionic surface-active agents – Determination of cloud point

[8] *H. Fruhner* and *G. Czichocki,* Tenside Surf. Det. 33 (1996) 310-314.

[9] *J. Kleinen* and *J. Venzmer,* J. Cosmet. Sci. 67 (2016) 59-70.

[10] *A. Shiloach* and *D. Blankschtein*, Langmuir 13 (1997) 3968-3981.

contact

Joachim Venzmer | joachim.venzmer@evonik.com

Evonik Nutrition & Care GmbH
Goldschmidtstr. 100
45127 Essen | Germany

Energizing Serum with IceAwake™

mibelle GROUP biochemistry

Phase	Application/ Trade Name	INCI	% w/w
A	Solvent	Aqua/ Water	Add 100
	Chelating agent/ EDTA	Disodium EDTA	0.02
B	Emulsifier/ Moisturizer	Methyl Gluceth-20	2.00
	Emulsifier/ Humectant	Methyl Propanediol	5.00
	Rheology modifier/ Thickening agent	Xanthan Gum	0.15
C	Preservative[1]	Phenoxyethanol (and) Triethylene Glycol	0.80
	Non-ionic o/w emulsifier[2]	Polyglyceryl-6 Distearate (and) Jojoba Esters (and) Polyglyceryl-3 Beeswax (and) Cetyl Alcohol	2.50
	Base fluid feeling agent[3]	Dimethicone	5.00
	Emulsifier-free/ Gelling agent[4]	Isostearyl Alcohol (and) Butylene Glycol Cocoate (and) Ethylcellulose	2.50
D	Thickening agent/ Stabiliser[5]	Hydroxyethyl Acrylate/ Sodium Acryloyldimethyl Taurate Copolymer	0.10
	Thickening/ Moisturizer[6]	Ammonium Acryloyldimethyltaurate (and) Hyaluronic Acid (and) Glyceryl Ester	5.00
E	**IceAwake™ [7]**	Succinic Acid (and) Maltodextrin (and) Aqua/ Water	2.00
	Solvent	Aqua/ Water	5.00
F	Perfume	Fragrance	qs
	pH regulator, citric acid (50 % sol.)	Citric Acid (and) Aqua/ Water	qs

Manufacturing Procedure:

1. Dissolve EDTA in water.
2. Disperse components of phase B in phase A under strong agitation and stir until completely hydrated.
3. Heat up to 70 °C.
4. Add components of phase C under strong agitation and stir to homogeneity.
5. Add components of phase D under strong agitation and stir to homogeneity while cooling down.
6. Premix IceAwake™ in water and add to phase A–D .
7. Add perfume under agitation.
8. Continue stirring and verify pH after 15 min. Adjust pH to 5.0 – 5.5 – 6.0 if necessary.

[1] Iscaguard® PEG by ISCA / [2] Emulium® Mellifera MB by Gattefossé / [3] XIAMETER™ PMX- 200 Silicone Fluid F 50 cS
[4] Emulfree® CBG by Gattefossé / [5] Feligel 305s by Guangzhou Dongxiong Chemical /
[6] Hydrogel by Shanghai Leasun Chemical Co., Ltd. / [7] IceAwake™ by Mibelle Biochemistry

Relaxing Face Cream | SC-FR-19-BC-50810-07

Phase	Ingredients	INCI	% by weight	Function
A	Emulgade® Sucro Plus	Sucrose Polystearate, Cetyl Palmitate	1.00	Emulsifier (O/W)
	Cetiol® LC	Coco-Caprylate/Caprate	7.50	Emollient
	Cetiol® Sensoft	Propylheptyl Caprylate	2.50	Emollient
	Cetiol® 4 All	Dipropylheptyl Carbonate	2.00	Emollient
	Lanette® O	Cetearyl Alcohol	4.00	Consistency agent
B	Water, demin.	Aqua	45.02	
	Preservative*		q.s.	Preservative
	Glycerin	Glycerin	0.70	Humectant
C	Water, demin.	Aqua	35.80	
	Rheocare® C Plus	Carbomer	0.20	Rheology modifier
D	Sodium Hydroxide (18% solution)	Sodium Hydroxide	0.28	pH Adjustment
E	Inolixir™ BC10079	Glycerin, Aqua, Inonotus Obliquus (Mushroom) Extract	1.00	Active ingredient

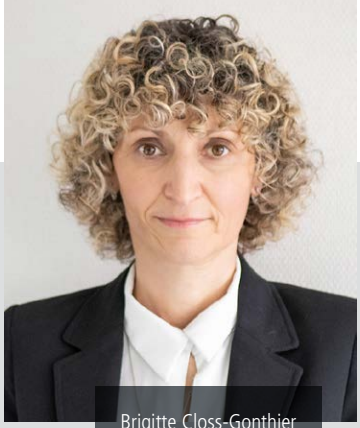
Specifications
pH value (23°C): 6.35
Viscosity (Brookfield; RVT; spindle TD, Helipath; 5 rpm; 23°C): 96 000 mPa s

Performance
Additional performance has not been evaluated

Manufacturing Process
1. Heat up phase A and B separately to about 80°C.
2. Add phase B to phase A whilst stirring.
3. After 2 minutes add phase C to phase A/B whilst stirring.
4. Stir slowly and start to cool to 50°C. Don't homogenize.
5. Add phase D and E one after another at a temperature below 40°C whilst stirring.
6. Continue to cool to room temperature whilst gently stirring.

Stability test
Stable 3 months at 4°C, RT, 40°C, 45°C

Additional information
Preservative system: Elestab™ 388 at 2.5%



Brigitte Closs-Gonthier

Interview with Brigitte Closs-Gonthier

Deputy General Manager, Innovation, SILAB

Mrs. Closs-Gonthier, SILAB is a company that researches, develops, produces and markets natural active ingredients for the cosmetic and dermo-cosmetic markets. What role do biotechnologies have in your product development?

Since its creation more than 35 years ago, SILAB has been developing natural active ingredients from 100% natural raw materials. Historically, these raw materials are from plants origin. However, we detected very early the huge potential of biotechnologies (yeasts, microalgae, bacteria) as an additional source of raw materials for the cosmetics market and became one pioneer in 1999, launching our first anti-aging active ingredient obtained from a yeast. With the aim of strengthening our bio-sourcing commitment, we developed a real in-house expertise in the cultivation of microorganisms, first at the laboratory scale, then transposed at the industrial scale. To this end, and in line with our strategy of independence, we self-financed as of 2014 our own fully automated Production Unit for Biotechnologies onsite, an investment of 5 million euros.

What are the advantages of using biotechnologies? What are their characteristics?

Biotechnologies are likely to play a much larger role in the future as they can enable companies to diversify their supplies and offer additional levers to drive innovation. Indeed, they open up a wide range of possibilities as a new source of raw materials, since microorganisms have three branches (yeasts, bacteria, microalgae), each with thousands of species, compared to plants that have only one branch.

In addition, at SILAB, we place all our efforts in meeting consumers' needs as well as market and legislation requirements for naturality, efficacy, traceability and safety. In this context, we value the use of biotechnologies, which is fully sustainable and respectful of biodiversity. It offers the opportunity to obtain, on demand, a large quantity of biomass and supernatant without using agricultural land, thus significantly reducing the ecological footprint usually related to plant crop. Only one initial sample of a strain of natural microorganisms is needed, as it can be reproduced almost indefinitely.

In addition, using biotechnologies as raw material is completely in line with our commitment to traceability and safety, as all the R&D and production phases are conducted in-house. Secure strains, qualified as Biosafety level 1 and GMO-free, are used. An external expert partner confirms their genetic identity. A quality biomass and supernatant, rigorously analyzed and validated, is obtained. It is repeatable and reproducible over time, thanks to the robustness of the processes in place.

Do you think consumers are ready to accept product derived from microorganisms?

We do think so indeed. In recent years, there has been an emerging trend related to pre-, pro- and postbiotics. The consumers first discovered these terms related to food industry and gut health. These microorganisms are now an area of research in the cosmetics industry. As a precursor company, we got interested in the idea of transposing their beneficial effects to the skin for the development of our latest active ingredient. At the crossroads of various fields of expertise (mastering natural, microbiota and biotechnologies), LACTOBIOTYL® is a natural postbiotic active ingredient specifically designed to restore hydration and luminosity to dry skin, while preserving the equilibrium of the cutaneous microbiota. For this development, our research teams drew inspiration from the adaptation properties of a plant probiotic (Lactobacillus arizonensis) to a desert climate. This good bacterial species colonizes one of the shrubs most adapted to the extremely dry desert conditions of Arizona, jojoba. It bioconverts the molecules of its host in order to ensure its survival. Our teams have reproduced in industrial bioreactors the natural environment of this bacterial species specific to desert climates by growing it in association with its host, jojoba. This targeted bioguiding technology has led to the selective production of bioactive molecules (= postbiotics) that constitute LACTOBIOTYL®.

LACTOBIOTYL® development was clearly bioinspired. Do you consider this approach as being an important lever driving innovation?

At SILAB, we are deeply convinced that Nature already has all the answer. As a result, our products are aiming at reestablishing the homeostasis of the skin and the concepts supporting their developments are mostly bio-inspired.

To provide another example, let us focus on the case of ECOBIOTYS®. SILAB studied the very particular microbiota of floral nectar, the Nectarobiota®, and more specifically the one of a refined and highly nectariferous plant (Hoya carnosa or porcelain flower), which ensures its defense, development and regeneration. Within this Nectarobiota®, we performed a customized sampling and specifically isolated the yeast Metschnikowia reukaufii to use it as a raw material for the development of our novel active ingredient able to rebalance the microbiota of mature skin and revive complexion radiance: ECOBIOTYS®.

For the first time in the cosmetics industry, natural molecules were extracted from the microbiota of plants to influence the skin microbiota. This unprecedented bio-inspired approach resulted in three awards for ECOBIOTYS® (the 1st prize at PCHi 2019, the Gold award at in-cosmetics Asia 2019 and the Bronze award at in-cosmetics Global 2019), which is a true recognition that bioinspiration leads to innovation.

Biotechnologies will account for a growing part in the cosmetics industry. What are the perspectives?

These two examples perfectly illustrate that biotechnologies are fully integrated in SILAB's product development strategy, combined with many other fields of expertise. For the moment, 16 active ingredients out of our standard catalogue of around 100 actives are coming from biotechnologies. In order to meet the growing need of the market, we are already extending our production capacity, multiplying it by 4 with a new production unit.

www.silab.fr

OBITUARY

A good name is worth more than great wealth.

Proverbs 22:1

Dr. Klaus Henning

After a long and serious illness, which he endured patiently and with great discipline, our highly esteemed colleague, partner, honorary chairman and friend Dr. Klaus Henning passed away on December 22, 2019 at the age of 78.

In addition to his professional commitment as managing director of the Hakawerk H. Kunz GmbH, Dr. Klaus Henning had been involved in SEPAWA e.V. on a voluntary basis since 1977 and headed the association for many years as first chairman and member of the board. His knowledge, diligence, diplomacy and planning foresight have contributed to making SEPAWA e.V. the most successful association in the detergent, cosmetics and perfumery industry in Europe. His tireless efforts were largely responsible for the establishment and development of the SEPAWA e.V. congress into the leading event of this industry. He had been honorary chairman of the association since 2011.

In addition, Dr. Klaus Henning proved his extraordinary expertise, specialist knowledge and talent as an author in many publications and created a lifetime achievement for himself.

Dr. Klaus Henning left lasting impressions. We are grateful to have known him and to have been part of his life. We will always honour his memory.

Our thoughts are with his family and relatives.

The Executive Board of SEPAWA e.V.
on behalf of all members

Arctic Tundra: the icy cold charm of the north

The Arctic, the region north of the polar circle, is an area of many facets. It's a place where temperatures can drop as low as minus 70° C and winds blast across the vast expanses of tundra and polar deserts; where the winter nights are eternally dark and the days never end in summer. But a new lifestyle trend? Let us explain…

North of latitude 66° 33' 55'', a different world unfolds: the Arctic. This extreme world may seem inhospitable at first glance, with its permafrost, the vast icebergs, but also the mosses, grasses and stunted shrubs of the bleak tundra, the sparse woodlands where small, squint trees do battle against the wind and ice.

But at the same time it is precisely the uniqueness, remoteness and pristine expanses, the fascinating colour palette from white to classic blue that intrigue people. And also, as bleak and barren as it may seem, nearly 6000 plant species flourish in the Arctic, and every year thousands bloom, adding a magical wash of colour to the tundra. Thousands of species of animals, including many insects, are native to the region. In early summer, when the migratory birds return, the Arctic tundra comes alive.

The Arctic is a region that for centuries has fascinated and attracted people. First, it was the promise of riches that lured people north, to catch whales, seals and fish and then to explore the promise of new, shorter shipping passages for trade. It wasn't until the 19th century that scientists became interested in the Arctic. Research into the animal and plant world and the climate continues to this day, with a particular focus on tapping into the unique powers of the unusual plants and flowers and the secret of their exceptional resilience to the harsh conditions, and using these findings for new product ideas.

If you google "Arctic" and "expedition", you will no longer be taken to pages describing historic explorers or modern scientists, but more likely to websites for state-of-the-art cruise ships, which in keeping with the trend for sustainability are now also available as hybrid versions.

DÜLLBERG KONZENTRA

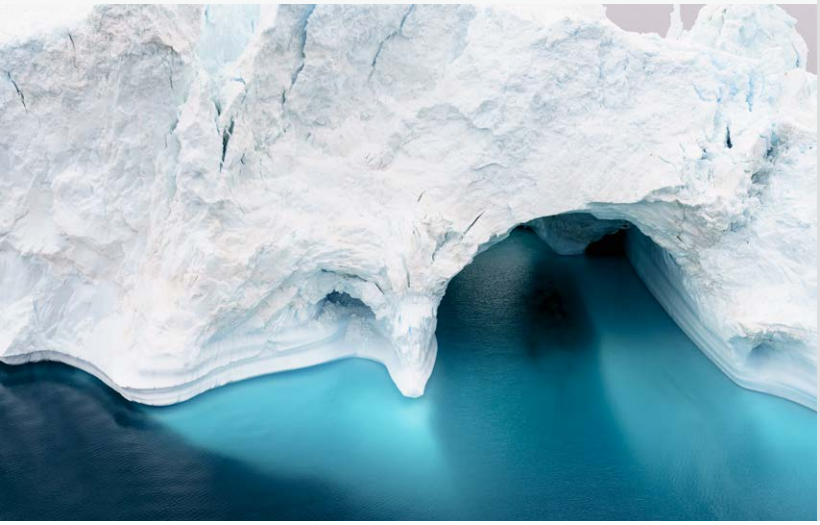
And if you navigate through the modern world of consumer goods with open eyes, chances are that you'll make the odd Arctic discovery of your own. "As well as being a natural phenomenon, the Arctic is also a cultural phenomenon with influences that range as far as fashion and especially tourism", says Lisa Achilles, Marketing DK, describing the trend. "Even if the term 'exotic' is usually associated with warmer regions, the Arctic is exotic. It is far away, difficult to get to, unique and exclusive. It is a place that people dream of visiting and it conjures up visions of pristine nature, of utter peace, remoteness and tranquillity."

The "Arctic lifestyle" isn't nearly as far away as you might initially assume. "Aromatic infusions of pine and Siberian spruce in the sauna or bath products with these ingredients have been around for a long time. And extracts of Arctic berries such as cloudberry or the 'ginseng of the north' – the rose root, are popping up as ingredients in skin and body care products", says Lisa Achilles.

Perfumery has also started harnessing the appeal of the polar circle. Jimmy Choo's "Ice", Azzaro Chrome's "Under the pole", the new Michael Kors' "Extreme Sky" or the Cliff Energy Shower "Arctic Spirit" – all of these products employ visually and olfactory attributes that conjure up coldness, clarity and purity.

Evocative as it is, the trend – which Düllberg Konzentra has named "Arctic Tundra" and has filled with inspiration and insights – is far from being a mass phenomenon. "The Arctic is a relatively new contender as a lifestyle topic. It is literally the antithesis of the tropical or Mediterranean theme worlds – which are also geographical regions that the industry has appropriated and uses for product creations. The Arctic evokes many positive associations, and it has its own icy magic with the perfect balance of longing and fascination, but it also reveals nature's fragility. As a trend concept, it should be treated like the sensitive region itself: with respect – and with curiosity."

www.duellberg-konzentra.de



RAHN
Your partner for excellence

REFORCYL®-AION Garb'Ageing Clean-Up

NEW PRODUCT LAUNCH FROM RAHN AT IN-COSMETICS 2020

ZURICH/Switzerland, January 2020. REFORCYL®-AION rejuvenates and purifies the skin by activating the key parameters of a healthy autophagy process: Golgi vesicle formation, mitochondrial fitness and LC3 gene upregulation.

Autophagy is a natural cellular cleaning program. Degradation products are enveloped by vesicles, enzymatically upcycled and then reused. Impaired autophagy weakens the skin barrier and promotes skin "garb-ageing".

REFORCYL®-AION triggers vesicle formation by disassembly of the Golgi apparatus, the cellular parcel service. Furthermore, it maintains mitochondrial health and promotes their fusion to form an energy-rich network. It also upregulates the LC3 gene that is responsible for the fusion with lysosome, the enzymatic waste degradation system.

Clinical studies have shown that activating this form of cellular spring-cleaning markedly improves skin tone and leads to smooth, redensified skin.

Pumpkins from Styria, a region in Austria renowned for its healthy pumpkin seed oil, are used to produce REFORCYL®-AION. It is carbon-neutral, conforms to the principles of a circular economy and provides for a holistic upcycling concept for skin, soul and our planet.

EFFICACY

- Induces autophagy by upregulation of LC3A (in-vitro study)
- "Spring cleans" to produce healthy cells and strengthens mitochondria under stressed conditions (in-vitro study)
- Provides an upcycled look to ageing skin (in-vivo study)

For further information, please contact cosmetics@rahn-group.com

www.rahn-group.com

in-cosmetics 2020: Berg + Schmidt Presents Natural Products from Own Development

Hamburg/Barcelona, February 6, 2020. Berg + Schmidt, the specialist in functional raw materials and cosmetic specialities based in Hamburg, will demonstrate its more than 60 years of expertise in oleochemicals at in-cosmetics 2020 (Stand No. Q80) in Barcelona. Above all, the oleo experts attach great importance to naturalness: the novel B+S product BergaCare FG Olive is a natural alternative to silicone for use in skin and hair care formulations. Other new items in the range include "BergaCare SmartLipids Ceramide", a lipid-encapsulated mix of skin-identical components for strengthening the natural skin barrier. In a technical lecture on the subject of GlyAcid®, Dr Sabilla Digel will present the latest findings on the beneficial effects of glycolic acid in hair care.

Olives as an alternative to silicone

Cosmetics manufacturers that address their products specifically to environment-conscious consumers are seeking alternatives to synthetic raw materials. In order to meet the increasing demand for natural ingredients, Berg + Schmidt will show BergaCare FG Olive at in-cosmetics 2020 as an alternative to the use of D4 and D5 silicones. The product is a mixture of ester oils and unsaponifiable substances obtained from olives. The feel is similar to that of cyclopentasiloxane, and BergaCare FG Olive can be used as an emollient in skin and hair care formulations. The colourless and odourless liquid contains no preservatives or other additives and is biodegradable. BergaCare FG Olive can be worked into the formulation in a cold state and is compatible with the usual cosmetic ingredients. Moreover, BergaCare FG Olive offers consumers the same smooth and silky skin feeling as they are used to from silicones.

Ceramide complements the BergaCare SmartLipids range

Berg + Schmidt is now adding Ceramide to its BergaCare SmartLipids range and exhibiting it in Barcelona. The valuable active ingredients help to create a stronger and healthier skin barrier. SmartLipids is a high-tech transport system for active substances in which the latter are embedded in lipid particles. These consist of a matrix containing a wide diversity of liquid and solid lipids that protect the active ingredient against physical effects and prolong its stability. Through diffusion, the active molecules leave their lipid matrix continuously when used on the skin and thus take effect over a long period. The lipid matrix itself serves to increase the skin penetration of the active substance and strengthen the natural skin barrier. Since the lipids adhere well to the skin, they prolong the release of the substance additionally.

SmartLipids Ceramide: natural protection against environmental influences

BergaCare SmartLipids Ceramide is ideal for dry, aging skin and persons prone to atopic dermatitis. Consisting of ceramides, sterols and fatty acids, it is a mixture of the skin's own barrier substances. The high concentration of valuable ceramides helps to restore the natural skin barrier and combats the loss of moisture and elasticity. The ready-to-use aqueous suspension is based on a patented technology and can be added easily to a formulation. All the ingredients are natural, odourless and white. The new Berg + Schmidt product will also be exhibited in the Innovation Zone.

Glycolic acid has positive effects in hair care products

On the exhibition stand and in a technical lecture given by Dr Sabilla Digel, Berg + Schmidt will present research results of its own to demonstrate why GlyAcid® is highly suitable for use in hair care, too. Glycolic acid is probably the best known and smallest representative of the alpha-hydroxy acids (AHAs) and has so far shown itself to be especially effective in chemical peels. The highly purified glycolic acid from the company CrossChem is sold in Europe by Berg + Schmidt. Thanks to a unique saponification and purification process it is free from formaldehyde and other harmful impurities. Experiments in Berg + Schmidt's research laboratory have shown that the use of GlyAcid® has positive effects in hair care, too, in the form of easier combing, better temperature resistance and increased strength and gloss.

The technical seminar "The new hair care beauty routine with glycolic acid – GlyAcid® for beautiful hair" with Dr Sabilla Digel will take place on Wednesday, 1st April 2020, from 14:00–14:30, in Theatre 1.

www.berg-schmidt.de

COLORS & EFFECTS

A brand of BASF – We create chemistry

BASF Colors & Effects Website Brings Customer Benefits

New website and online service platform from Colors & Effects® offers customers a convenient and personalized digital hub

Ludwigshafen/Germany, February 12, 2020. Colors & Effects® launched a new corporate website and highly-innovative online service platform designed to offer customers a robust, one-stop portal for interacting with the Colors & Effects® brand and product portfolio.

The new Colors & Effects® website serves as the landing page for the online service platform. "We redesigned the company website to streamline the user journey and bring relevant content to the forefront," said Caroline Syms, Digital Marketing Communications Specialist for Colors & Effects®. "We want to give customers the information they're looking for within a few clicks."

The Colors & Effects® Pigment Finder product tool, which launched during 2019, has been rolled into the new service platform and is the single location for all product- related content. "Customers can now enjoy a more centralized and personalized experience when interacting with our products digitally," said Anna Herbst,Digitalization Project Lead for Colors & Effects®.

A newly created registration functionality provides customers the ability to create an account with Colors & Effects where they can access relevant product-related information and see contact information for their specific Colors & Effects® representatives. In addition, state-of-the-art data search capabilities and convenient access to product-related documentation contribute to enhanced user activity.

"We went down a completely new path by first developing and testing a prototype for the online service platform with several focus groups," said Martin Fischer, Senior Manager Digitalization and IT Strategy for Colors & Effects®. "The new digital services available in the platform were created based on the market's needs, giving us the opportunity to really improve the customer journey and offer value to our customers each step of the way."

Users can register to create an account with the online service platform and will only need one ID and password to access all Colors & Effects digital services in the future.

The online service platform can be accessed:

www.colors-effects.eu

EVONIK POWER TO CREATE

Evonik Launches New "intoCleaning" Customer Platform

- Evonik launches centralized customer platform for cleaning industry
- Digital customer interaction is key success factor for Evonik
- Platform will help customers meet sustainability and performance targets

Essen, Germany/Orlando, USA, January 27, 2020. The new platform – operating under the theme 'Cleaning begins with knowledge' – makes it easier for customers to access product details, including regulatory information. It also serves as an in-depth resource for product videos, marketing concepts, and formulations for customers who develop cleaning-oriented products for the home care, industrial & institutional, and vehicle care markets.

"Digital customer interaction is a key success factor for our business. Our customers expect to find information of interest fast, anytime and anywhere," says Tammo Boinowitz, head of Evonik's Care Solutions business line. "With intoCleaning we focus on bundling all product information, sample orders and interactive online tools in a web-based portal," he continues.

Among its features, the intoCleaning portal features a detailed product selection tool that allows customers to quickly examine chemistry solutions meeting a variety of application, registration, and use criteria. The system also has a module enabling participants of Evonik workshops and events to access presentation materials. Among its first featured market concepts is one focused on Water Responsible Cleaning, which addresses the challenges posed by limiting water use in the cleaning industry as well as providing different approaches to meet these challenges.

To access the new system, please register for access at

https://intocleaning.evonik.com/

Gattefossé Comes into the Microbiome Discussion with a New Study on its O/W Emulsifier Emulium® Mellifera MB

Lyon/France, February 12, 2020. Emulium® Mellifera MB is a natural O/W emulsifier that allows the formulation of a wide range of textures suitable for all skin types and climates. It creates natural emulsions with white and luxurious appearance, while leaving a light and smooth afterfeel. Its moisturizing and anti-pollution properties, its capacity to visibly improve skin texture and its high tolerance even for sensitive and hyper reactive skin make Emulium® Mellifera MB an active emulsifier.

To go further in understanding the attributes of a healthy skin, Gattefossé demonstrated that Emulium® Mellifera MB is "microbiome-friendly", meaning that it maintains, even improves the cutaneous microbiota balance.

New clinical study

The objective of this research was to study the effect of an emulsion formulated with 4% of Emulium® Mellifera MB on moisturization, skin barrier function and to understand its impact on cutaneous microbiota. Several evaluation techniques have been used:

- Study of cutaneous microbiota by amplification and sequencing of ribosomal 16S DNA
- Biometrological measures: corneometry, TEWL
- Self-assessment by the volunteers on various criteria
- Questionnaire to understand what volunteers know about cutaneous microbiota
- Overall satisfaction questionnaire

Maintaining the cutaneous integrity and balance of the skin flora

Results showed an increase of moisturization, a reduction of TEWL reflecting a better barrier function and an improvement of the skin quality of volunteers.
More importantly, the study of cutaneous microbiota after application of an emulsion containing Emulium® Mellifera MB revealed that the taxonomic diversity and richness of the skin flora have been at least maintained or even improved for 81% of volunteers. These parameters are directly linked to a healthy skin, able to defend itself against external aggressions.

A natural and flexible emulsifier

Emulium® Mellifera MB is a natural and PEG-free O/W emulsifier based on the transformation and functionalization of natural waxes, a patented technology developed by Gattefossé oleochemists.
From beeswax and jojoba wax, the wax butter obtained by esterification and interesterification brings a unique sensoriality and moisturizing properties to this emulsifier.
Flexible, it allows the creation of a wide range of textures, from sprayable lotions to thick butters, and applications, such as skincare, suncare or makeup.

www.gattefosse.com

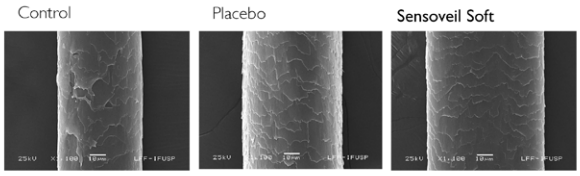
CHEMYUNION

Chemyunion's Launches at in-cosmetics Global 2020

Sorocaba/SP, Brazil, February 18, 2020

Category Hair Care: Sensoveil Soft is a proprietary technology applied to hair care formulations, which delivers conditioning and lipid reposition by intermolecular attraction forces. It forms a biofilm on the hair surface that acts as a bridge between the hair fiber and oils present in its composition. In this way, we can say it is a delivery system. This mechanism of action evenly spreads over the hair fiber, improving lipid replacement and conditioning properties, including hair softness. In vitro results have shown in relation to silicon and other natural ingredients: Film-forming capacity without build-up; 16.7% reduction of frizz; 2.8 times improvement of hair combability. Sensoveil Soft is a natural and versatile technology and provides noticeable conditioning in the first application on the hair. It is 100% biodegradable, which may be used in vegan products and as an alternative to silicone-free and lanolin derivatives-free claims.

Evaluation of cuticles throught SEM (Scanning Electron Microscopy).
Sensoveil Soft treatment provided cuticle alignment , repair after bleaching process and film formation without build-up effect.



Category Skin Care: Searching for a non-invasive alternative of skin fillers, Chemyunion has developed Cellfie, a vegetable ingredient with a precise and efficient delivery system to stimulate adipogenesis by stimulating adiponectin in order to reduce the signs of aging, promoting a remarkable rejuvenation with natural looking. This new active eases structural aging due to lipoatrophy, upregulating adiponectin by 122%, being significantly superior to the benchmark (27%), despite being at a concentration 10 times lower. Clinical studies have shown that Cellfie performed on different regions of the face. In vivo results have shown:

- reduction on area, length and depth of perioral wrinkles up to 28%, 30% and 9%
- reduction on area, length and depth of smile lines up to 60%, 47% and 29%
- reduction on area, length and depth of nasolabial lines up to 23%, 25% and 8%

Effect of **Cellfie** on perioral, periorbital, smile line and nasolabial wrinkles after 60 days of treatment.

www.chemyunion.com

HALLSTAR BEAUTY

BRIGHT Oléoactif is the 100% Natural, Safe Whitening Solution for Skin

BRIGHT Oléoactif® has superior skin brightening efficacy, without the side effects

Chicago (Illinois), USA, February 25, 2020. Totally safe and highly stable active BRIGHT Oléoactif® promotes skin brightening, whitening and reduction of hyperpigmentation.The 100% vegetal active ingredient is extracted from three plants screened for their complementary activities: marshmallow root, rice bran and licorice root. The combination of these plants as well as its oil-based form make BRIGHT Oléoactif® a uniquely safe, tolerable alternative to other well-known brighteners.

- Recent data demonstrates that a dose of 0.12% of BRIGHT Oléoactif® inhibits 90% of tyrosinase activity, which is equivalent to the effect of 0.01% of pure hydroquinone. BRIGHT Oléoactif® is thus a safe, natural solution to minimizing the quantity of hydroquinone in a cosmetic formulation. It can be used in day care and sun care products for all skin types, even the most sensitive.
- BRIGHT Oléoactif® has also been shown to be 24% more effective at brightening than pure glabridin. Compared to licorice extracts, it is cost effective and easier to fomulate with.
- At 0.1% dose, BRIGHT Oléoactif® is more than twice as effective at brightening as ascorbic acid (Vitamin C).

To learn more, be in touch with Nathalie Lefebvre, Active Naturals Product Manager, at nathalie.lefebvre@hallstar.com -- or visit us at in-cosmetics Global 2020 (Boooth B60). Click below to visit our website where you can download the informative brochure detailing BRIGHT Oléoactif®'s complementary mechanisms on four key stages of the cutaneous pigmentation process.

www.hallstarbeauty.com

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)

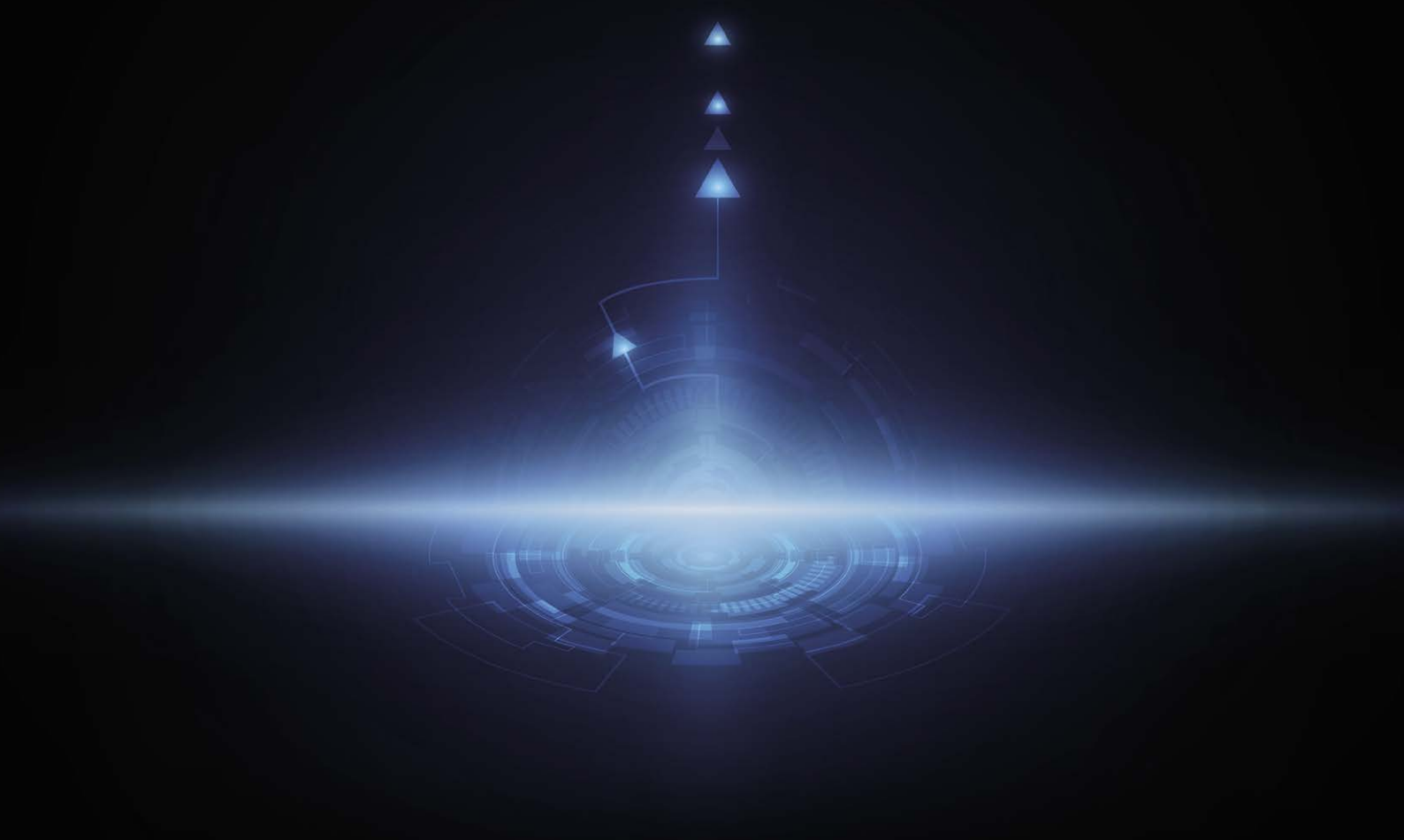
Online Subscription
› EUR 195.00 (+ VAT where applicable)

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

Picture Credits
Cover: Marina Goncharova/shutterstock.com
p. 70 Gregory A. Pozhvanov/shutterstock.com
p. 71 murattellioglu/shutterstock.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

Active Beauty
Synchronight™
The microbiome-activated digital stress defender



The beauty filter for happier nights & healthier skin

- Fight against the damages of digital stress
- Protect melatonin's natural cycle
- Reverse visible signs of ageing
- Improve the sleep quality
- Crafted by green fractionation

To find out more, contact us at global.cosmetic@givaudan.com

Givaudan

engage your senses