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# home care

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### content

### home care

A. Yarnell, S. Foster, J. Rabiei, J. Blankenship Green Raw Material & Product Preservation Sustainable, readily biodegradable preservation is key for consumer products, as well as their components	2
S. Zhou, M. Chabert, C. Orizet A natural, powerful and biodegradable suspension agent for home care	8
K. Henning Aqueous scouring milk for cleaning hard surfaces with a proportion of an anionic surfactant based on calcium and magnesium salts of linear alkylbenzene acid	14
personal care	
I. Bonnet, T. Clarius, A. Courtois, K. Kulon <b>The science behind effective skincare</b> <b>Three generations of advanced active ingredient release</b>	18
S. Hettwer, E. Besic Gyenge, B. Suter, B. Obermayer Can Fermentation "Preserve" the Skin?	24
Ò. Expósito, A. Guirado, D. Robustillo, A. Gallego, M. Mas, P. Riera, D. Luna, S. Laplana, T. Ruiz, S. Ruiz, M. Gibert <b>A Cell Nectar to Optimize Vitamin D synthesis: The D-Skin</b>	30
H. Shao-yong, Q. Qiu-yue, H. Fang, Z. Li-dan, L. Yi-na A study of <i>prunus persica</i> (peach) resin extract on instant skin firming and anti-wrinkles	36
R. Kräling, M. Ritter, U. Leist, A. Wittersheim, P. Drechsel, CP. Kramer, B. Meinigke, L. Gehm <b>Heavy Metals in Cosmetic Products</b>	42
L. Neumann, B. Fellenberg Data sheets for the evaluation of the efficacy of active substances in cosmetic products	48
advertorials	
Beauty or environmental protection? – Why not both!	50
Natural astaxanthin: the red diamond among radical scavengers!	52
formulations	54-58
interviews	60-63
Index of Advertisers/Imprint	64



### **Green Raw Material & Product Preservation**

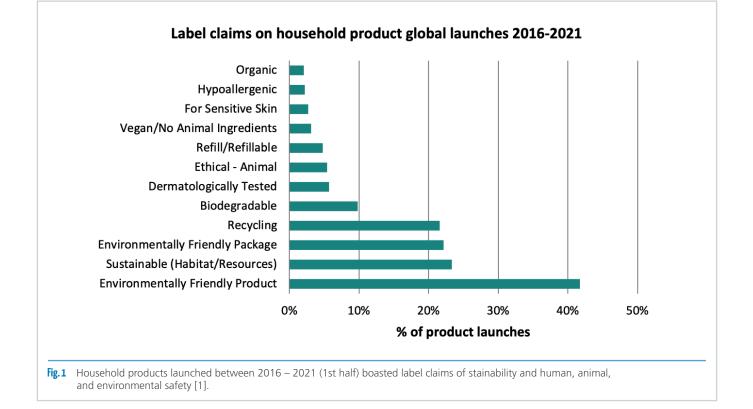
Sustainable, readily biodegradable preservation is key for consumer products, as well as their components

A. Yarnell, S. Foster, J. Rabiei, J. Blankenship

### Introduction

What does the word "sustainable" mean to the everyday consumer? While it's clear that sustainability is growing in importance for home care and consumer products, the term can encompass a number of different considerations; biodegradability, ingredient origin, ethical sourcing, safety, potential hazards, and eco-friendly packaging are all critical aspects that can impact whether a product is perceived as a green choice (Figure 1).

Green labels are increasing in popularity, as they make it easier for consumers to decipher which products align with their personal values and find them on store shelves. While the criteria for each green label certification is different, they each look at certain factors to determine which products meet standards for sustainability. Ingredient selection is an important part of the process of applying for a green label certification, and many programs use approved or restricted ingredient lists to provide guidance to manufacturers seeking certification. Preservatives are an essential ingredient in home care formulations. Not only do they play a central role in preventing products from becoming contaminated by fungi and bacteria, they are also critical for sustainability. A properly preserved cleaning product will have a shelf life in the order of months, while an unpreserved product stored in the same conditions may last only a matter of days. The extended shelf life of preserved products allows them to be shipped, stored, purchased by consumers, and used in their entirety before concern of product spoilage. This reduces waste resulting from disposal of products that spoil before they have been completely consumed. Attempts to prolong the integrity of an unpreserved product may utilize individual packaging or require refrigeration, though both options yield greater environmental burdens versus properly preserved products. Effectively preserved products are essential to sustainability.

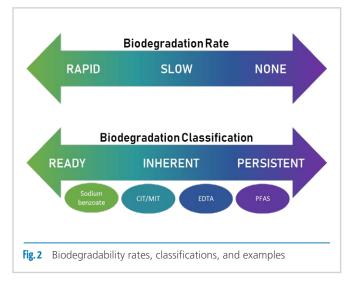


### The importance of ready biodegradability

While effective preservation contributes to the overall eco-friendliness of a product, not all preservatives are equally sustainable. For example, biodegradability is an important consideration for all ingredients used in home care products. Once products have been disposed or washed away, they ultimately make their way to wastewater treatment facilities or the environment, where they can either biodegrade fully or remain persistent in the environment. It is imperative that the chemical components of products can be broken down into innocuous intermediates that are fully degraded in these settings. The use of persistent ingredients with little or no ability to degrade could result in an accumulation of the chemical in the environment, potentially causing harm to plants, animals, humans, or water sources.

On the spectrum of biodegradability, chemicals are classified according to the rate of their degradation (Figure 2). Chemicals that are broken down quickly by microorganisms that are naturally present in the environment are considered readily biodegradable. Sodium benzoate is one such molecule, quickly degrading across a range of test conditions.

In contrast, some materials may require longer times or special conditions to assist in their biodegradation. These ingredients are considered inherently or ultimately biodegradable, though the time in which this biodegradation may occur can vary greatly. Enhanced conditions — including pre-acclimated



microorganisms, pH adjustment, light, or anaerobic settings may be necessary to facilitate the breakdown. Without these accommodations, the presence of the molecules may linger in the environment. Ethylenediaminetetraacetic acid (EDTA) salts are known to require alkaline conditions and long sludge residence times to biodegrade. Metal-EDTA complexes can be quite thermodynamically stable depending on the identity of the metal, with some resisting degradation indefinitely [2]. On the far end of the biodegradability spectrum are molecules that are persistent in the environment. These molecules are often referred to as "forever chemicals", since they lack

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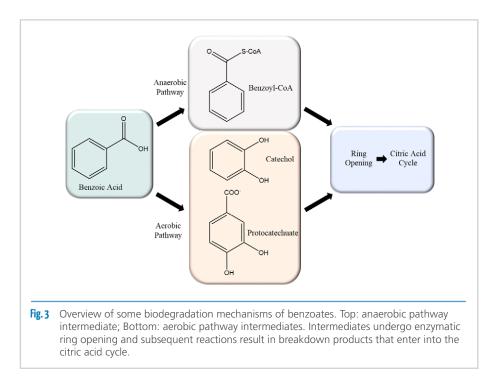
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the ability to be degraded in any capacity. Persistent chemistries, like per- and polyfluoroalkyl substances (PFAS), can accumulate in the environment, water sources, and in living organisms.

Standard test methods, including methods published by ASTM International, International Organization for Standardization (ISO), and the Organisation for Economic Co-operation and Development (OECD), are used to determine the biodegradability of a compound or material. Under OECD 301 methods, the classification of chemicals is achieved by measuring the consumption of dissolved organic carbon (DOC), biochemical oxygen demand, or the CO<sub>2</sub> production. If 70% DOC or 60% of theoretical oxygen demand or CO<sub>2</sub> production can be achieved in a 10-day window, the substance is deemed readily biodegradable [3].

### Sodium benzoate and biodegradability

Sodium benzoate and its parent acid, benzoic acid, have a long history of use as preserving agents in food and personal care. More recently, sodium benzoate has been registered for use in home care products in Europe and U.S. as Kalaguard® SB\*. While sodium benzoate is preferred for its rapid water solubility, the antimicrobial activity exhibited is due to the protonated benzoic acid species. For this reason, the activity of the molecule increases in acidic environments. As an in-can preservative, sodium benzoate offers broad-spectrum protection against a variety of yeasts, molds, and both gram-positive and gram-negative bacteria in applications up to pH 7.

It is essential that preservatives protect products throughout their intended shelf life, but it is equally important that the preservative does not cause harm to the consumer or environment during a product's use or after its disposal. Because it is non-sensitizing and readily biodegradable, sodium benzoate offers product protection without sacrificing consumer safety or negatively impacting the environment.

Once a product has been washed away from the intended application, the formula is diluted upon its introduction to the sewers or groundwater, reducing the concentration of the sodium benzoate preservative to levels below the biocidal or inhibitory concentrations. At such dilute concentrations, the native microbes present in soils, sludge, and water treatment facilities can quickly digest the benzoate through a series of enzyme-catalyzed biochemical reactions. This occurs in both

\* Kalaguard is a registered trademark of Emerald Kalama Chemical, LLC, a LANXESS Group company.



aerobic and anaerobic environments, and studies have elucidated several mechanisms by which the degradation takes place (Figure 3) [4]. Regardless of the initial degradation pathway, the resulting breakdown products are completely digested and converted to energy for the cells by means of the citric acid cycle.

## Preserving raw materials to enhance product sustainability

The preservation of raw materials must be considered when formulating green label products. As consumers' scrutiny of products has increased, so has their awareness of raw materials and hazards associated with particular ingredients. Consumers have increased interest in product certifications by third-party organizations that review in-depth how each ingredient is manufactured, potential health risks, and environmental impact. Examples of these green label certifications include Ecocert, Ecolabel, Natural Seal, and EPA Safer Choice. Meeting criteria for certification can make product formulation challenging, as ingredient selection is limited, and the preservative present in the raw material can interfere with the certification of the final product.

To facilitate formulation in full-scale process setting, liquid surfactant products are available, where the surfactant content is commonly 30-70 percent with water comprising the remaining balance. These pre-dissolved surfactant raw materials often require preservation due to the presence of water. Sodium benzoate is a suitable choice to preserve these raw materials. The amphoteric and anionic surfactant classes demonstrate synergy with sodium benzoate by increasing the apparent pK' of benzoic acid, making effective preservation possible at smaller dosages and higher pH conditions [5]. This makes sodium benzoate an ideal choice to protect raw materials with sufficient water activity for microbial growth while still meeting clean label requirements for downstream users. Sodium benzoate has been an effective preservative for the personal care market in ammonium lauryl sulfate, sodium lauryl ether sulfate, cocoamidopropyl betaine, and other surfactants, where the raw material is typically 30-40 percent active with a pH range of 4-6. Now that sodium benzoate is registered in the EU and US, it is also available to preserve surfactant raw materials intended for the homecare market. Beyond surfactants, there are other raw materials containing water that have need of preservation. Fragrance encapsulation formulations too ask for/require readily biodegradable preservatives satisfying clean label requirements. Every day, Symrise solutions elevate consumer experiences within fragrance, flavor, natural nutrition, and cosmetic ingredients.\* Desiring 100% isothiazolinone-free fragrance formulations for the home care industry, the Symrise formulation team searched for

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a new generation of nature-identical preservation technology. The experts selected sodium benzoate, dosed at 0.4–1 percent by weight, as the appropriate solution for the various high quality fragrance encapsulation offerings provided to the home care industry. Sodium benzoate enabled solutions completely free from isothiazolinones, eliminating skin sensitization concerns while effectively preserving against microbial activity. SAPPI has also followed suit, choosing to incorporate sodium benzoate in their Valida product line\*. SAPPI Valida is natural and biobased rheology modifier and stabiliser suitable for home care and personal care products. SAPPI was searching for a preservation solution that allowed home care customers to switch to isothiazolinone-free formulations. Sodium benzoate satisfies the requirements for shelf life stability and protection against contamination.

### Reformulating for sustainability & biodegradability

Small modifications can make big changes to the overall sustainability of a formula. Consider the following case study for a hand dish wash formulation, initially comprised of both readily and inherently biodegradable chemistries and preserved with sodium benzoate (Table 1). The original formula included tetrasodium EDTA and the unintentional presence of 5-chloro-2-methyl-4-isothiazolineone and 2-methyl-4-isothiazlinone (CIT/MIT), which was the preservative present in the surfactant raw material. Together, these ingredients limit the alignment with clean labels and do not meet regulations in certain countries.

To improve the safety and sustainability of the dish product, some raw materials were substituted with readily biodegradable chemistries (**Table 2**). For the SLES-2 surfactant, the raw material was substituted

Ingredient Name	Function	% (weight)
Water	Carrier	Q.S.
Lauryl Glucoside	Surfactant	4.0
Sodium Laureth Sulfate – EO 2*	Surfactant	11.8
Sodium Coco Fatty Alcohol Sulfate	Surfactant	2.8
Tetrasodium EDTA	Chelating agent	0.5
Sodium Chloride	Rheology Modifier	1.0
Sodium Benzoate	Preservative	1.0
Citric Acid	pH adjustment	To pH 6.5

**Tab.1** A standard hand dish wash formula: Though the product is preserved with readily biodegradable sodium benzoate, the formula included the addition of slow-degrading tetrasodium EDTA, and the unintentional presence of the inherently biodegradable & sensitizing CIT/MIT by way of surfactant.

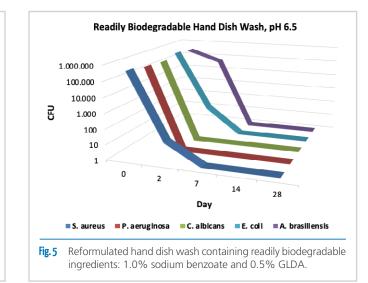
Sodium lauryl ether sulfate contained 30 ppm CIT/MIT as supplied. As a result, the formulated dish product contained 5 ppm CIT/MIT

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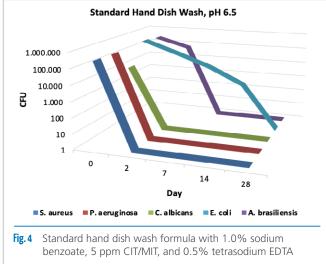
Ingredient Name	Function	% (weight)
Water	Carrier	Q.S.
Lauryl Glucoside	Surfactant	4.0
Sodium Laureth Sulfate – EO 2*	Surfactant	11.7
Sodium Coco Fatty Alcohol Sulfate	Surfactant	2.8
GDLA	Chelating agent	0.5
Sodium Chloride	Rheology Modifier	1.0
Sodium Benzoate	Preservative	1.0
Citric Acid	pH adjustment	To pH 6.5

**Tab. 2** Modified hand dish formula: CIT/MIT and EDTA were removed from the formula, resulting in a formulation comprised of readily biodegradable, sustainable ingredients with a more consumer-friendly label.

Sodium lauryl ether sulfate was preserved with excess alkalinity.



\* SAPPI and Valida are trademarks of SAPPI Limited.



for a comparable product that was preserved with excess alkalinity. The substitution of SLES-2 surfactants decreases risk of adverse effects on the consumer and removes labeling required for the hand dish wash product due to the presence of CIT/MIT. To address the presence of tetrasodium EDTA in the product, GLDA (tetrasodium glutamate diacetate) was selected as the replacement chelating agent. GLDA is readily biodegradable with excellent solubility and performance in the pH range of the dish wash product. Together, these substitutions yield a product that is more friendly for the consumer and the environment, and demonstrates rapid microbial control in preservative challenge testing, as shown in **figures 4 and 5**.

# Conclusions: preservatives protect consumers and brands

It's important to consider preservation throughout a product's life cycle – beginning with raw materials, to formulation, sitting on store shelves, during use by the consumer, and finally, to its ultimate environmental fate. In all stages prior to disposal, preservatives provide a critical role in controlling microbes, which protects consumer safety and product integrity.

Sustainable preservative chemistries go one step further. In addition to providing an effective shield against contamination, they improve the environmental and safety profile of the consumer products we use in our homes and workplaces every day. This includes chemistries introduced through preserved raw materials. Sodium benzoate is a sustainable preservative chemistry now available to manufacturers and raw material producers, which also works hand-in-hand with other consumer-friendly ingredients, such as plant-based surfactants, green chelating agents, and multifunctionals. Together, these sustainable ingredients can create a robust safety network and meet requirements for isothiazolinone-free and green label certifications.

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# A natural, powerful and biodegradable suspension agent for home care

S. Zhou, M. Chabert, C. Orizet

We present an innovative, natural and readily biodegradable suspension agent that opens new possibilities to homecare formulators. When added to detergent formulas, it brings a very powerful suspension capacity without any perceivable impact on viscosity. The agent is based on activated cellulose fibers obtained through the fermentation of starch using specific bacterial strains. We present rheology measurements that allow us to predict the suspension power of a detergent formula supplemented with the agent, based on its rheology at low shear rates. The yield stress value extracted from Bingham plots highly correlates with the density and size of the objects that can be suspended with the agent. We show the application of the agent in multiple home care formulations, in particular the suspension of decorative visual beads and concentrated fragrance in a typical liquid laundry formula. We describe how it is simple to add the suspension agent to a formula due to its pre-activated liquid format. A formula supplemented with the suspension agent is stable for several months at 45°C, with sustained suspension power and no demixion or change in aspect, highlighting the suitability of the suspension agent for commercial consumer products.

### Introduction

Recent years have seen a dramatic shift in consumer expectations towards more ecofriendly, and natural homecare products, yet with maintained or improved performance versus existing formula. Also, the increasingly stringent regulations (e.g. microplastics in Europe) that will come into force in the near future are pushing the specialty chemicals industry to seek innovative solutions to equal the high performance of existing synthetic polymers with sustainable and natural alternatives. In addition to the massive ecoprofile improvement needed for home care polymers, an ever more competitive environment also calls for more differentiation and premiumization of brands serving the western market.

The suspension agent presented in this paper is an attempt to answer these two needs. On the one hand, it is a 100% natural, readily biodegradable cellulose-based product obtained by the fermentation of starch by a carefully selected, non-genetically modified bacterial strain. On the other hand, this product brings new features to the table when added to detergent formula by imparting a powerful suspension capacity with no impact on apparent flow viscosity. This allows home care product formulators to offer new, differentiating features to products and to consumers.

First, we introduce the product and its manufacturing process, together with some typical detergent formula used in this paper, as well as the methods needed to characterize the formulations. We then present rheological measurements designed to control the quality of the finished product. Our rheology method enables the determination of the suspension power of a given formula based on low shear measurements and Bingham plots. We finally show the application of the suspension agent in a typical home care formula, focusing on the concentration of fragrance capsules and the suspension of visual beads in a liquid laundry chassis and the stability study of the formula as a function of time.

### **Materials**

### Solvay standard liquid laundry detergent composition

Table 1 shows the typical composition of the liquid laundry chassis used in this study. In order to study the dose response of Rheozan® BLC to the rheology properties of the chassis, its concentration was varied from 0 to 0.1% wt/wt as the activated cellulose fiber active, which is equivalent to 0 to 10% of Rheozan<sup>®</sup> BLC as is. Our Rheozan<sup>®</sup> BLC is supplied as a flowable and pumpable viscous liquid which will disperse easily in water and in liquid formulations. Hence, Rheozan® BLC can be added and dispersed at any step during the manufacturing of a liquid laundry chassis. In our study, Rheozan® BLC was added toward the end of the chassis formulation steps just before the final pH adjustment. Nevertheless, as Rheozan<sup>®</sup> BLC generates a yield as soon as it is dispersed in a liquid formulation, formulators need to be cautious with the formulation set-up and mixer selection to prevent air bubble formation during the formulation step.

Ingredients	Function	Composition as active (% wt/wt)
Rhodapex® ESB70 Sodium laureth (2EO) sulfate	Anionic surfactant	5.0
Rhodasurf® L7/90 Alcohol ethoxylate(Laureth-7)	Nonionic surfactant	10.0
Rhodacal <sup>®</sup> SSA Linear alkylbenzenesulfonate acid	Anionic surfactant	9.0
Coconut fatty acid Fatty acid	Soap	5.5
Repel-O-Tex® Crystal Nonionic polyester	Soil release polymer	1.0
Propylene glycol Solvent	Solvent	6.5
NaOH (40%)	Base	Qs pH
Citric acid (50%)	Acid	Qs pH
Rheozan® BLC Bacteria cellulose	Rheology modifier	0 - 0.1
Water	Solvent	Qs 100
рН		7.50

### **Rheozan® BLC**

Rheozan<sup>®</sup> BLC is a ready-to-use, flowable and pumpable activated cellulose fiber dispersion in water. It is manufactured in

three steps: bacterial fermentation step, bacteria cellulose fiber purification step and activation step as shown in **Figure 1**.

### **Fermentation step**

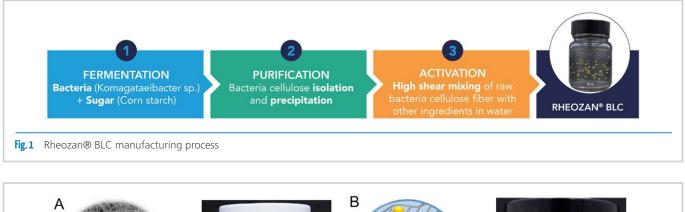
The cellulose fiber is obtained through the bacterial fermentation of starch by an acid resistant bacteria strain Komagataeibacter sp. The bacterial fermentation process is carefully controlled to produce high purity, high crystallinity, high modulus elastis and high aspect ratio bacteria cellulose fibers with a high yield. The yield rate regarding sugar to bacterial cellulose conversion is between 7% to 8%.

### **Purification step**

After the fermentation process, the produced bacteria cellulose is subjected to a further purification process which includes isolation, precipitation and filtration to remove the bacteria and residues from the culture medium.

### **Activation step**

In the final activation step, the obtained raw bacteria cellulose fiber is dispersed in water together with other ingredients such as an organic acid as preservative, a dispersant, etc. The mixture is then activated by subjecting it to high shear rate mixing to expand the very fine and high aspect ratio cellulosic fiber bundles. These expanded and entangled bundle structures form a cross-linked framework that facilitates swelling when in an aqueous solution, thereby providing excellent three-dimensional systems (Figure 2).



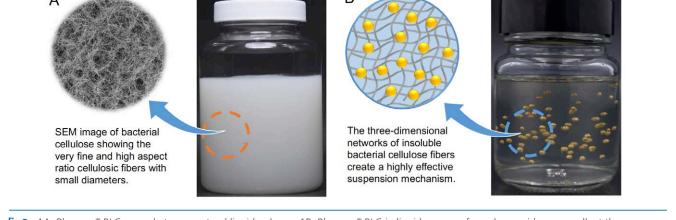
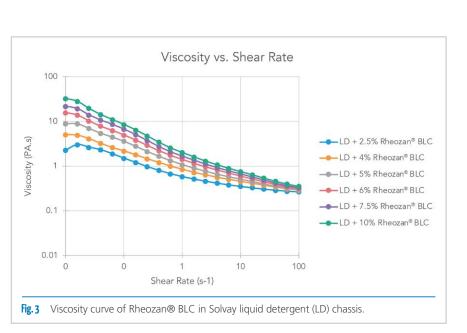
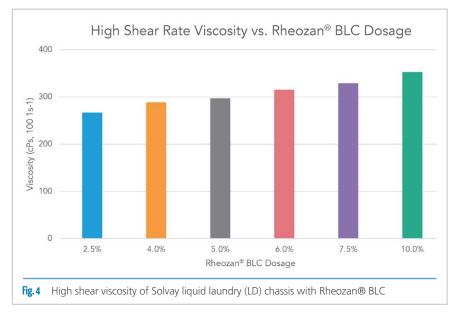
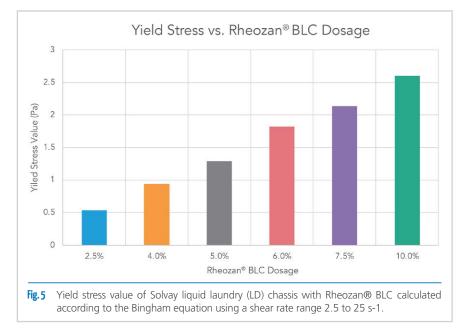


Fig. 2 1A: Rheozan® BLC, a ready-to-use natural liquid polymer. 1B: Rheozan® BLC in liquid aqueous formula, provides an excellent threedimensional network with high suspension power.







### **Methods**

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### Equipment & principle of Lab measurements

The rheological property of Rheozan® BLC in a liquid laundry chassis, according to Table 1, was measured with Discovery HR-2 Hybrid rheometer from TA Instrument using concentric cylinder geometry of the following dimensions: cup radius: 15mm, rotor radius: 14mm and rotor height 42 µm. The measuring temperature was set at 25°C. Prior to measurement, the sample was pre-sheared at a shear rate of 100 s<sup>-1</sup> for 120s and left for equilibrium for 60 minutes. A flow sweep measurement was done from shear rate of 0.01 to 100 s<sup>-1</sup>. The yield stress value was calculated according to the Bingham equation using a shear rate range 2.5 to 25 s<sup>-1</sup>.

### **Stability tests**

To assess the suspending power of Rheozan® BLC, different particles such as fragrance capsules and pearlescent golden coated visual beads from Unispheres were suspended in the liquid laundry detergent chassis supplemented with Rheozan® BLC. To study the robustness and long-term storage stability of the Rheozan® BLC supplemented liquid detergent chassis, the formulations were stored at different storage temperatures such as 4°C, 22°C and 45°C for 3 months and their particle suspending power was compared to the liquid detergent without Rheozan<sup>®</sup> BLC. Furthermore, to study the long-term stability of Rheozan® BLC against enzymatic degradation, 2.5% Medley® Brilliant 300L from Novozyme was added to Rheozan® BLC supplemented liquid detergent chassis and the mixture was stored at 30°C and 45°C for three months and its particle suspending power was compared to the Rheozan® BLC supplemented liquid detergent without the addition of enzyme blends.

### Results

### **Rheology of BLC in LLD**

The flow behavior of the Rheozan® BLC supplemented liquid detergent chassis shows a shear thinning behavior with an increase of shear rates. The low shear rate viscosity of the sample increases exponentially with the concentration of Rheozan<sup>®</sup> BLC in the liquid detergent chassis (Figure 3). However, the high shear rate viscosity (at 100 s<sup>-1</sup>) changes only marginally i.e. less than 100 cPs when the Rheozan® BLC concentration increases from 2.5% to 10.0% (Figure 4). The activated bacteria cellulose fibers form a continuous three-dimensional network in the liquid formulation to create yield stress to suspend particles, represented by the shear thinning rheology flow behaviors. As the bacteria cellulose active content in Rheozan<sup>®</sup> BLC is only ~1%, the active loading in the formulation is very low between 0.025% to 0.1% and for this reason, the high shear viscosities at 100 s<sup>-1</sup> are not significantly impacted by the presence of the Rheozan® BLC network in the formulations.

### **Evaluation of yield stress**

The yield stress value created by the Rheozan<sup>®</sup> BLC continuous three-dimensional network increases linearly with its concentration in the liquid laundry formulations as shown in Figure 5. Although the bacteria cellulose active loading in the formulation is very low, it can generate a remarkable yield stress value ranging from 0.5 to 2.6 Pascal, important for particle suspension performance while the viscosity at high shear rate, important for the final product flow, is not impacted. This yield stress value is sufficient to suspend particles ranging from fragrance capsules, opacifying particles and pearlescent visual beads in the formulation. Depending on the characteristics of the particles (such as size, density) to be suspended in a liquid formulation, formulators can estimate the required yield stress value, hence the required concentration of Rheozan® BLC in order to suspend the particles with long term stability.

### Yield stress vs suspension power

The shear thinning flow behavior and the associated yield stress of the Rheozan<sup>®</sup> BLC supplemented liquid laundry chassis are the result of the continuous three-dimensional entanglement of the high aspect ratio bacteria cellulose fibers in the formulation. In other words, the chassis forms a gel i.e. a viscoelastic system that has both liquid and solid like properties. The solid-like property is associated with the three-dimensional network structure and the strength to hold the network together is represented by the yield stress value [1]. A solid particle in a liquid formulation will exert certain stress to the system, if the exerted stress is less than the formulation yield stress value, the network structure will deform



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elastically and suspend the particle stably in the liquid formulation. With this principle, typical fragrance capsules used in laundry detergents can be suspended with a yield stress value of less than 0.5 Pa. Hence 2.5% Rheozan® BLC is enough to supplement liquid laundry formulations to achieve long term suspension stability of the fragrance capsules. On the other hand, pearlescent golden coated visual beads with a particle size of 1.0 - 1.5 mm and density of 1.4 - 1.6 g/cm<sup>3</sup> will require yield stress higher than 1.0 Pa to suspend, hence it requires 5.0% Rheozan® BLC to stabilize the long-term suspension stability.

### Typical application formula and its stability as a function of time

Rheozan<sup>®</sup> BLC shows not only excellent suspension power in liquid laundry formulation but also robustness and long-term stability at different storage temperatures. We suspended

0.3% by weight of fragrance capsules supplied by Eurofragrance and 0.3% by weight pearlescent golden coated visual beads supplied by Unisperses in Solvay liquid laundry chassis shown in Table 1 with 2.5% and 5.0% Rheozan<sup>®</sup> BLC, respectively. The formulations show excellent storage stability with the fragrance capsules and the visual beads remain suspended even after more than 3 months storage at 4°C, 22°C and 45°C. Without the addition of Rheozan® BLC the fragrance capsules and visual beads would have been quickly sedimented from the liquid laundry chassis. Furthermore, when the Solvay liquid laundry supplemented with 5.0% Rheozan® BLC was added with Medley® Brilliant 300L (a blend of different enzymes, including cellulase) from Novazyme, at 2.5% by weight as supplied and subjected to a long-term storage stability study, its rheological properties as well as its suspension power remained unchanged after 3 months storage at 30°C and 45°C. These results imply that the bacteria cellulose fibers of Rheozan® BLC also have excellent stability against enzymatic degradation, especially by cellulase.



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Throughout this paper we have elaborated the rheological properties, suspension power, storage as well as enzymatic stability and practical applications of Rheozan<sup>®</sup> BLC in a liquid laundry chassis. These interesting features of Rheozan BLC can also be applied to suspended particles in other home care formulations such as laundry sanitizer, hand dish liquid, hard surface cleaner, peroxide bleaching gel, etc.

### Conclusion

3

The new suspension agent described in this paper presents several interesting features that make it perfectly aligned with current homecare market trends and needs. It is a natural and readily biodegradable product manufactured by fermentation of corn starch by specifically selected bacteria strains. It comes as pre-activated ready-to-use cellulose fibers in liquid form that can easily be integrated in liquid homecare detergent formulas, including when continuous processes are used at industrial scale. The additive brings unique suspension properties to homecare formula at low dosage, as evaluated by low shear rheology measurements correlated with the suspension power of the product in typical detergent liquids. This is exemplified in several formulations throughout this paper, such as a liquid laundry detergent with concentrated fragrance that demonstrates long term stability over several months at 45°C.

We believe this suspension agent is one of the first of a new generation of polymers arriving on the homecare market, with improved sustainability credentials from cradle to grave, i.e. natural sourcing and a good biodegradability profile. While the challenge of maintaining or improving the performance of synthetic non-biodegradable polymers with natural and biodegradable alternatives is a tough one, it has to be tackled in the next decade. Biotechnology based polymers, such as this suspension agent, are likely to be a key tool to reaching this ambitious goal.

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### Aqueous scouring milk for cleaning hard surfaces with a proportion of an anionic surfactant based on calcium and magnesium salts of linear alkylbenzene acid

K. Henning

### abstract

The preparation of a storage-stable abrasive cleaning composition without dolomite as a starting material, in which no or only a very small amount of alkylbenzene acid magnesium salt (Mg-LAS) is present, is achieved by using alkoxylated fatty alcohols. Such a cleaning composition contains 1 to 15 wt.% alkylbenzene acid calcium salt (Ca-LAS) at a proportion of Mg-LAS  $\leq$  1% of the amount of calcium salt as well as 0.5 to 10 wt.% alkoxylated fatty alcohol.

### Cleaning agents with abrasive effect

Nowadays, cleaners with an abrasive effect are mainly used as liquid products with a high proportion of calcite in the form of a scouring milk. These have a milder effect compared to the originally developed soda-containing scouring powders with high pH values. Based on common abrasive pastes, which generally contain sodium salt of linear alkylbenzene sulphonic acids, a non-ionic surfactant and an abrasive, milder cleaner compositions with an almost neutral pH value have been developed.

Calcium and magnesium salts of alkylbenzenesulphonic acids are an alternative to sodium salts of alkylbenzenesulphonic acids, which have been used for several decades. Usually, sodium salts are prepared by neutralising the corresponding acids with soda ash or sodium hydroxide. Surfactants prepared with calcium or magnesium salts of linear alkylbenzenesulfonic acids (Ca-LAS and Mg-LAS, respectively), are milder and yet very effective against some of the common stains.

Calcium or magnesium salts of alkylbenzenesulphonic acids are usually obtained by neutralising the sulphonic acid precursors obtained from calcite, magnesium carbonate or other equivalent alkaline substances. Dolomite is a suitable neutralising agent for alkylbenzenesulfonic acids and results in a mixture of Mg-LAS and Ca-LAS. A certain amount of dolomite is also processed to magnesium sulphate and calcium sulphate, which are used as *in situ* structuring agents for cleaning compositions for piece-meal dishwashing detergents.

Indian patent application IN 225/MUM/2000 (Hindustan Lever Ltd., 2005) describes the neutralisation of alkylbenzene sulfonic acid with dolomite. The reaction products obtained, i.e. Ca-LAS and Mg-LAS, serve as surfactants for hard surface or in textile detergent compositions.

Because the proportion of calcium carbonate and magnesium carbonate in dolomites of different grades varies greatly, depending on the deposit, among other factors, the proportion of Ca-LAS and Mg-LAS in the mixture produced also varies. While minor variations are acceptable, major variations are detrimental to the performance of the detergent compositions at end use.

WO2014/044639 (Henkel) describes aqueous hand washing pastes containing Na-LAS, calcium carbonate as an abrasive and a non-ionic surfactant. The compositions do not contain Mg-LAS. The combination of Na-LAS and the additional surfactant produces ductile pastes with a constant total surfactant content. These compositions are highly alkaline due to the presence of a significant amount of soda ash and silicate, a portion of which is consumed in the neutralisation of LAS acid. WO14086634 (Henkel) describes similar detergent compositions.

The use of Ca-LAS as a surfactant is described in alkaline detergent powders (US 4162994 Lever Brothers, 1979) and in non-aqueous scouring powders (US 3772204, Colgate-Palmolive, 1973).

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### Avoiding phase separation

Cleaning compositions with Ca-LAS but without Mg-LAS tend to phase separate easily. This is probably due to the fact that Mg-LAS imparts a certain stability to Ca-LAS. Cleaning compositions containing Ca-LAS and Mg-LAS, obtained by neutralising LAS acid with dolomite, do not give phase separation if only a limited amount of Mg-LAS is present and stabilisation has been achieved by adding non-ionic surfactant in the form of alkoxylated fatty alcohol. However, too high a proportion of Mg-LAS leads to instability.

A scouring milk with a high proportion of abrasives contains

- Calcium salt of linear alkylbenzene sulfonic acid
- magnesium salt of linear alkylbenzenesulfonic acid in a proportion of ≤ 1% of the proportion of calcium salt
- alkoxylated fatty alcohols.

Typically, abrasive cleaners used to clean hard surfaces contain a sodium salt of linear alkylbenzene sulfonic acid (Na-LAS) formed by neutralising linear alkylbenzene sulfonic acid with an alkali such as sodium carbonate, sodium silicate or sodium hydroxide. Soda ash (sodium carbonate) is often used for this purpose and is added in stoichiometric excess to react with a given amount of LAS acid to form Na-LAS. Excess soda ash remains in the formulation and serves as an alkaline builder. Alkaline compositions provide better fat removal but tend to react more aggressively with the skin.

A milder skin alternative to Na-LAS is the use of Ca-LAS, which is formed in a slurry of dolomite and LAS along with Mg-LAS in the usual method of preparation.

The absence of Mg-LAS in compositions that have not been prepared with dolomite as the starting raw material and therefore contain only Ca-LAS leads to unstable products. Compositions without Mg-LAS are particularly prone to separation into solid and liquid phases, and this is particularly observed in the case of products stored at high temperatures at 40°C or at low temperatures at 5°C.

On the other hand, the availability of dolomite with consistent specification is a problem from a raw material procurement point of view.

The production of a storage-stable abrasive cleaning composition without dolomite as a starting material, in which no or only a very low proportion of Mg-LAS is present, is achieved using certain non-ionic surfactants from the group of alkoxylated fatty alcohols.

The aqueous abrasive cleaning composition contains 1 to 15% by weight of Ca-LAS. The amount of Mg-LAS is  $\leq$  1% of the amount of calcium salt. For example, if the total amount of Ca-LAS is 15 wt%, at most 0.15 wt% of Mg-LAS is present, which is not more than 1% of the amount of the calcium salt. However, it is preferred that no Mg-LAS is included.

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Thus, the amount of Mg-LAS is 0 to 1% of the amount of Ca-LAS.

### **Non-ionic surfactants**

Alkoxylated fatty alcohols confer stability to the composition even when Mg-LAS is completely absent or when the Mg-LAS content is minimal. It is particularly preferred that the degree of ethoxylation in the alkoxylated fatty alcohol is 5 to 8 moles of ethylene oxide units. These show particularly good effect in stabilising compositions stored at temperatures above or below normal room temperature.

Suitable alkoxylated fatty alcohols have an HLB value of 10 to 20 and a fatty alcohol carbon chain length of C12 to C16. The amount of alkoxylated fatty alcohol is from 0.5 to 10% by weight. A combination of one or more such non-ionic surfactants may also be used.

### Viscosity

The application and distribution of an abrasive cleaning agent to hard surfaces is facilitated by a certain viscosity. It is therefore preferred that the compositions contain an abrasive with a Mohs index of 0.5 to 7 and a polymer in an amount to give a viscosity of 500 to 2000 cP at 20 °C.

### **Abrasives**

The proportion of abrasive with a Mohs index of 0.5 to 7 is 10 to 50% by weight. Preferably, the abrasive is bentonite, kaolin, calcite or feldspar. The average particle size of the abrasive is 0.5 to 400  $\mu$ m, preferably 10 to 200  $\mu$ m.

### **Polymers**

The polymer used as an alternative or in addition to the abrasive is a water-swellable polymer or an associative polymer. It is preferred that the polymer provides the desired viscosity when the pH of the compositions is from 6 to 8.

Preferred polymers are those of polyacrylic acid, polyacrylates, cross-linked acrylates, guar gum or its derivatives, starch-acrylic grafted copolymers, cross-linked polyoxyethylenes, cross-linked methylcellulose, sodium carboxymethylcellulose or partially cross-linked water-swellable polymers of polyethylene oxides and polyacrylamide or isobutylene/maleic acid copolymers. The proportion of the polymers is 0.008 to 5% by weight, preferably 0.01 to 2.5% by weight. A particularly preferred polymer is acusol 880/882.

The water content of the compositions is 20 to 80 wt%.

It is preferred that the pH of the compositions is in the range of 6 to 8 at  $20^{\circ}$ C.

### **Other components**

The compositions contain < 1 wt% strong alkalis, such as sodium hydroxide, sodium silicate and sodium carbonate. If the pH is > 8, this is likely to affect the stability of the product. Similarly, if the pH is < 6, protonation of the non-ionic surfactants could occur, which may also lead to unstable compositions.

It is preferred that < 1 wt% Na-LAS is present in the composition. Any further amounts of Na-LAS could destabilise the compositions due to the exchange of calcium ions with the sodium ions.

The cleaning composition may further comprise other anionic surfactants, amphoteric and zwitterionic surfactants, provided that they do not affect the performance or stability of the compositions. It is preferred that the amount of these other surfactants is from 0.1% to 20% by weight.

Preferably, the compositions comprise < 1% by weight of cationic surfactant.

It is further preferred that in the compositions the total amount of surfactant is not > 40 wt%.

### Preparation of an aqueous scouring milk for hard surfaces that is free of Mg-LAS

**Table 1** shows the compositions of aqueous abrasive cleaningagents.

For the preparation, 275 g of 65 to 75°C hot demineralised water are first mixed successively with 5 g of coconut fatty acid and 0.2 g of silicone oil, 200 g of calcite, 33 g of LAS acid and a non-ionic surfactant while stirring at 150 rpm. Then another 275 g hot water and 200 g calcite are added to this mixture and mixed for 5 minutes with stirring.

In Example 4, an additional 50 g of bentonite was added to obtain the desired viscosity.

Compositions were also prepared which contained a polymer in addition to the calcite. For this, 0.3 g of polymer was added to 450 g of water while stirring at 150 rpm) and mixed for 5 minutes at 150 rpm. This was followed by the addition of 350 g calcite and the contents were stirred for 20 minutes.

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	Proportion (wt%)							
Ingredients	Comparative example			Abrasive cleaning agents as described				
	А	В	С	1	2	3	4	5
Ca-LAS	3,3	3,3	3,3	3,3	3,3	3,3	3,3	3,3
Mg-LAS	-	-	-	-	-	-	-	-
Calcit	40,0	40,0	40,0	40,0	40,0	40,0	40,0	40,0
Bentonit	-	-	-	-	-	-	5,0	-
Acusol 880/882	-	-	-	-	-	-	-	0,01
Water and ancillary components	55,0	55,0	55,0	55,0	55,0	55,0	50,0	54,9
Span 80	1,7	-	-	-	-	-	-	-
PEG-200	-	1,7	-	-	-	-	-	-
Acconon MC 8-2	-	-	1,7	-	0,5	-	-	-
Lialet 125 5.5	-	-	-	1,7	1,7	-	-	-
Lialet 123-8	-	-	-	-	-	1,7	1,7	1,7

Tab. 1 Compositions of aqueous abrasive cleaning agents.

30 days storage (°C)		Proportion (wt%) Comparative example Abrasive cleaning agents as described						
	A	B	С	1	2	3	4	5
5	unstable	unstable	unstable	unstable	stable	stable	stable	stable
28	unstable	unstable	unstable	stable	stable	stable	stable	stable
40	unstable	unstable	unstable	stable	stable	stable	stable	stable
Initial viscosity (cP)	-	-	-	500	500	520	1150	1400
pH value	6-8	6-8	6-8	6-8	6-8	6-8	6-8	6-8

### **Explanatory notes:**

Span 80 = Sorbitan monooleate from Croda with an HLB value of  $4.3 \pm 1$  and a C24-C26 chain length PEG-200 = Polyethylene glycol with a molecular weight of 200 Daltons, coupled with oleic acid. The polyethylene glycol monooleate has an HLB value of 8 to 9.3.

Acconon MC 8-2 from Abitec is a polyoxyethylene caprylic/ capric glyceride with an HLB value of 13 to 15 and a C8-C10 chain length Lialet 125-5.5 from Sasol is a fatty alcohol polyethylene glycol ether (alkoxylated fatty alcohol) based on Lial 125 and ethylene oxide (5 mol). The HLB value is 11 and the carbon chain length is 12 to 16.

Lialet 123-8 from Sasol is a fatty alcohol polyethylene glycol ether (alkoxylated fatty alcohol) based on Lial 123 and ethylene oxide (8 mol). The HLB value is 12 and the carbon chain length is 12 to 16.

All compositions were stored at 5°C, 28°C and 40°C for 30 days to test stability. The results are given in **Table 2**.

The results show that alkoxylated fatty alcohols (comparison C) with an HLB of 11 to 20 do not give phase stability. It also shows that the combination of HLB value (11 to 20) and carbon chain length (C12 to C16) gives optimal stability for the compositions (compositions 1 to 5).

### Reference

"Aqueous hard surface cleaning composition" Patent-No.: WO 2017/045924 Publication: 23/03/2017 Applicant: Unilever N.V. Weena 455, 3013 AL Rotterdam, Niederlande

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### The science behind effective skincare

Three generations of advanced active ingredient release

I. Bonnet, T. Clarius, A. Courtois, K. Kulon

abstract

As increasing emphasis is placed on the efficacy of skincare products, science is taking on an even more important role in the beauty industry, helping to provide consumers with solutions that help them not only look good, but feel great, too. One of the leading innovations in science-driven cosmetic ingredients trends is BASF's MicroPatch® technology, which offers the opportunity to harness the power and boost the efficacy of active ingredients by controlling the time they are released on the skin, for longer-lasting positive effects. Throughout the years, the research team has continued to refine this technology further and expand its mode of action by integrating different actives that enable ever-evolving claims for skin care products. By now, BASF's portfolio includes four highly effective MicroPatch products – and further innovations are in the works. The technology has the potential to meet an even wider range of consumer needs and applications, making it a leading light in the future of personalized skincare solutions.

Science-based beauty products with strong, proven efficacy are on the rise, as more consumers put their trust in sophisticated technologies to find reliable, personalized solutions for healthy, glowing skin [1]. While the beneficial effects of active ingredients – or "actives" – on the skin are well known, the focus of science-based skincare has now shifted to finding ways to boost the efficacy of these substances through new delivery systems. Leading the way in this field is BASF's MicroPatch<sup>®</sup> technology. By combining their dual expertise in natural macromolecules and the controlled release of active ingredients, the skincare specialists at BASF have created a unique delivery system to entrap actives in a reservoir above the skin and control their release over a longer period, for far greater efficacy.

### The benefit of active delivery systems

Whilst freely applying skincare products ensures a high concentration of actives on the skin at the moment of application, this rapidly diminishes, reducing their effectiveness over a longer period of time. Delivery systems like MicroPatch technology can harness the full power of active ingredients by releasing them over a longer period, prolonging their action on the skin for longer-lasting effects. MicroPatch technology also increases the potential for more personalized skincare solutions by acting as a vessel for different actives to suit various applications and endpoints, from advanced moisturizing to soothing sensitive skin. And it's all thanks to a molecular-level network of natural bio-macromolecules.

### Bio-macromolecules: harnessing the power of active ingredients

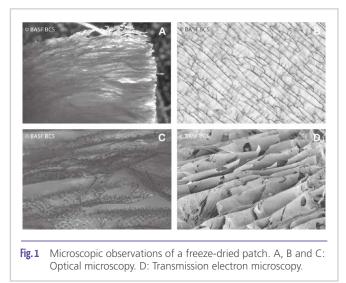
At the heart of MicroPatch technology are bio-macromolecules – natural polysaccharides produced by the cells of living organisms. To create the MicroPatch, two or more biomacromolecules are combined to form a molecular-level, layered, mesh-like structure, which entraps the chosen actives above the skin. This 3D molecular mesh forms an invisible film-like layer – a "second skin" – that serves as a reservoir of actives, delivering them when needed. This improves efficacy by ensuring the slow and prolonged release of the relevant actives, extending their action. When applied, the patch protects the skin from drying out by limiting transepidermal water loss. There are also sensory benefits, as the smooth, film-forming surface provides a pleasant feel on the skin.

### An evolving science

To date, researchers at BASF have created three generations of MicroPatch products, tailoring the science to serve a range of different needs, from moisturizing and anti-pollution, to soothing sensitive skin and boosting well-being. **The first generation** involved a combination of two bio-macromolecules – alginate and acacia gum – combined with the active ingredients caffeine or serine to provide anti-cellulite or moisturizing effects.

<sup>&</sup>lt;sup>1</sup> The trademarks symbolized with a <sup>®</sup> or <sup>™</sup> are either property of or licensed to BASF group and registered and/or applied for registration in relevant countries. Other product names and trademarks mentioned may belong to third parties.

content



**The second generation** of MicroPatch – known as PatcH2O<sup>®</sup> – is a more sophisticated combination of three bio-macromolecules – pullulan, alginate and hyaluronic acid – which are loaded with four hydrating actives (trehalose, urea, glycerine and serine) to provide enhanced skin moisturization [2]. Taking this to an even more advanced level, **the third generation** – known as the Sacred Patch<sup>®</sup> – combines the Micro-Patch technology with Sacran, an ingredient found in a rare Japanese alga that is well known for its soothing properties. Sacred Patch is used to soothe sensitive skin and enhance well-being.

An in-depth look at each generation of MicroPatch technology provides useful insights into the evolving nature of this technology and its potential to shape the future of efficient and personalized skincare.

### First generation: establishing the molecular network

### Composition

The foundation of MicroPatch technology was laid with the formation of a molecular meshwork of alginate and acacia gum macromolecules, within which small molecules of active ingredients could be introduced, which slowly diffuse into the skin. In this first generation of patch, the 3D molecular mesh is loaded with two different active ingredients: caffeine, well known for its lipolytic effects [3], or serine [4], the principal amino acid in the skin's natural moisturizing factor and the synthesis precursor of ceramides responsible for maintaining hydration of the epidermis.

MicroPatch<sup>®</sup> Serine is a serine-filled 3D micro-fleece composed of acacia polysaccharides and alginate (INCI: Aqua (and) Butylene Glycol (and) Pentylene Glycol (and) Algin (and) Acacia Senegal (and) Serine).

MicroPatch<sup>®</sup> Caffeine is a caffeine-filled 3D micro-fleece composed of acacia polysaccharides and alginate (INCI: Aqua (and) Salicylic Acid (and) Caffeine (and) Butylene Glycol (and) Propylene Glycol Alginate (and) Acacia Senegal).

### Efficacy

In a clinical trial, 20 female volunteers with normal to dry skin applied 2% MicroPatch Serine on the forearm. Hydration levels were measured by Corneometer after one, three and six hours. Immediately after application, the hydration level by impregnation of the upper layers of the epidermis increased progressively with time, reaching a maximum at three hours.

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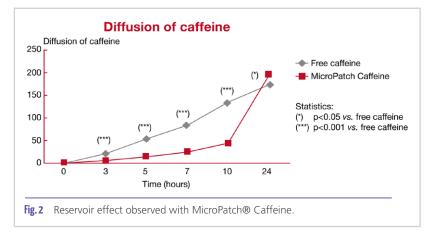
At this measurement point, there was a +25% increase in hydration compared to the control site. This showed that 2% MicroPatch Serine has an immediate hydration capacity, as well as persistence for at least six hours after a single application (data not shown).

The absorption of caffeine when freely applied and when applied via MicroPatch Caffeine was tested on Franz cells with human skin explants. The diffusion kinetics (0-24 hrs), the release (48 hrs) and the storage (72 hrs) were measured by HPLC. Results showed that MicroPatch Caffeine increases the action time of caffeine and has a reservoir effect **(Figure 2)**. At 48 and 72 hours, the release and the storage of caffeine is higher for the MicroPatch application than for the freely applied form (+68% and +114% respectively, data not shown). The MicroPatch technology allows for a slow release of caffeine to exert an intense and long-lasting action.

PatcH2O is a 3D micro-fleece composed of three bio-polysaccharides entrapping a hydration complex (INCI: Aqua (and) Glycerin (and) Glyceryl Polyacrylate (and) Trehalose (and) Urea (and) Serine (and) Pentylene Glycol (and) Algin (and) Caprylyl Glycol (and) Sodium Hyaluronate (and) Pullulan (and) Disodium Phosphate (and) Potassium Phosphate). An alternative version free of Glyceryl Polyacrylate and Phosphate is available.

### Efficacy

In a double-blind, placebo-controlled clinical study, 23 female volunteers with dry skin applied either a placebo formula or a formula containing PatcH2O on each hemi-face, twice daily, for 15 days. A novel measuring device linked to the smartphone enabled the participants to determine their skin moisturization level in real time during the application peri-



od and seven days after the treatment (regression period). Results showed that the day-long moisturizing effect increases the longer the product is used (Figure 3). This moisturization efficacy even lasted into the regression period, after participants stopped using the products. Further performance tests on the anti-pollution effect of PatcH2O, its microbiome-friendliness as well as its suitability for use in hair care products are available (data not shown).

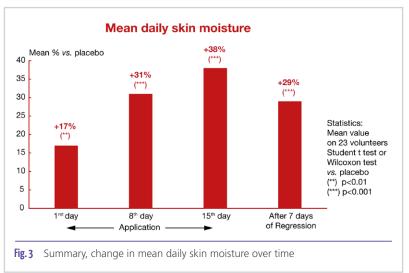
# Second generation: advanced moisturization

### Composition

Second-generation MicroPatch technology, named PatcH2O, focused on finding advanced, longer-lasting moisturization by entrapping a humectant complex with glycerin, L-serine, trehalose and urea in the biopolysaccharide mesh. Here, the 3D molecular mesh was formed from three bio-macromolecules, selected for their filmogenic properties. Hyaluronic acid is a hygroscopic molecule that can hold up to 1,200

times its volume in water [5]. Pullulan is produced by bio-fermentation, and its polymeric structure gives it unique gelling, adhesive and film-forming properties.

Alginic acid is obtained by basic hydrolysis of brown algae (Phaeophyceae) and has the ideal porosity to trap small molecules, while retaining their free diffusion inside and outside the gel.



### Third generation: soothing sensitive skin and boosting well-being

### Composition

Sacred Patch – the most recent generation of MicroPatch technology – enhances the link between emotional well-being and healthy skin, and how our state of mind can impact our skin's appearance [6].

It aims to help those suffering from sensitive skin by soothing irritation, providing advanced moisturization and offering a unique and pleasurable sensorial experience. It combines patch technology with Sacran, a large polysaccharide found in an extremely rare type of blue-green alga found only in Japan [7]. Sacran can offer superior hydration, improve skin barrier function and act on skin irritation, making it an effective way of treating sensitive skin when delivered through a molecular mesh of pullulan, alginate and hyaluronic acid.

Sacred Patch uses molecular patch technology to entrap the precious Sacran biopolysaccharide (INCI: Aqua (and) Glycerin (and) Pentylene Glycol (and) Algin (and) Caprylyl Glycol (and) Glyceryl Polyacrylate (and) Sodium Hyaluronate (and) Pullulan (and) Aphanothece Sacrum Polysaccharides). An alternative version free of Glyceryl Polyacrylate is available.

### Efficacy

The emotional benefits of Sacred Patch were assessed during an *in vivo* test involving 87 Asian women aged 30 to 50, who suffered from sensitive skin. A placebo emulsion and an emulsion containing 2% Sacred Patch were applied twice daily for two weeks on each side of the face. An emotional test was performed immediately after the first application, where participants were asked to correlate their perceived emotions while applying the product to visual stimuli. This non-verbal method is based on nine picture boards, each consisting of several images expressing a specific emotion: surprise, tenderness, curiosity, disgust, desire, anger, ill at ease, happiness, and sadness. The name of the emotion is never mentioned, allowing the volunteers to state how they felt.





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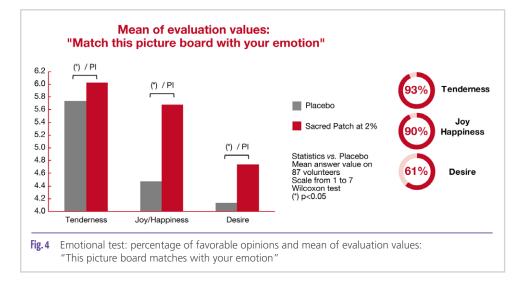
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Copyright © 2021 International Flavors & Fragrances Inc. ("IFF") All rights reserved. IFF, the IFF logo and all products denoted with ® or ™ are trademarks of the affiliates of IFF. The emotional content of the boards had been previously validated with more than 1,200 naïve consumers from eight countries in Europe, the US and Asia. Correlation was indicated on a scale from one (does not match the board at all) to seven (completely matches the board), with positive answers falling within the five to seven range. The results confirmed the positive emotional effect of Sacred Patch immediately after application, with 93% of participants matching the use of the patch



with tenderness, 90% associating it with joy and happiness, and 61% linking it to desire. In all three cases, the active ingredient outperformed the placebo. Other emotions were not selected in a significant quantity.

### Conclusion

### A new future for skincare

The proven efficacy of all three generations of MicroPatch technology shows positive signs for the future of personalized skincare, by offering a customizable active delivery system to suit a range of applications and consumer needs. Alongside proven benefits such as moisturization and soothing sensitive skin, the sensory and emotional benefits of Sacred Patch also helps to meet consumers' growing appetite for cosmetic solutions that not only have beauty benefits, but have positive emotional and well-being benefits, too. With its host of opportunities for continual evolution, BASF's MicroPatch technology clearly has the potential to be a leading light in the future of tailored, personalized skincare solutions – and there is more to come before long.

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### Can Fermentation "Preserve" the Skin?

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

Fermentation preserves food by displacing unwanted microorganisms. Does it work in the same way with skin? Fermented products are becoming increasingly popular in the food sector. Although it is an ancient process for preserving food, fermentation is a very modern topic, including in the cosmetics industry. It is known that certain lactic acid bacteria such as *Lactobacillus sakei* can positively affect atopic dermatitis (taken orally) [1]. Here we show that millet ferment, produced with lactic acid bacteria from sourdough production, can also positively influence atopic skin when applied topically. We demonstrated that bacteria of a healthy skin microbiota can gain a growth advantage and thus lead the skin out of dryness stress. A dermatological assessment of the atopic condition showed a rapid reduction of symptoms even compared to placebo-treated areas.

### Introduction

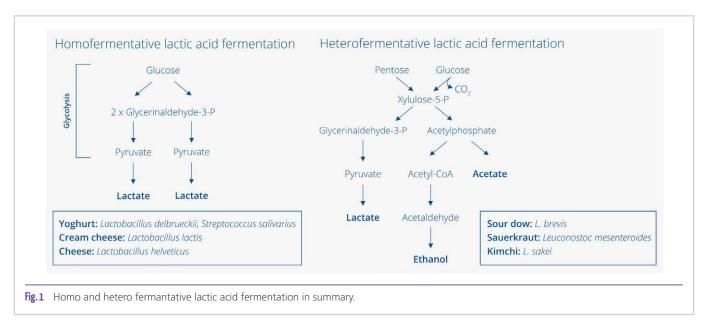
Of course, we cannot ferment our skin, but we can explore the question of why fermentation is a powerful tool to influence the microbiota. Fermentation is one of the oldest preservatives used by humans to preserve food. Microorganisms play a crucial role in this process. Today, fermentation is believed to have played a crucial role in the settlement of humans in the Neolithic period. Initially, cereals were not processed into bread, as was assumed for a long time, but fermented into a beer-like beverage. Here, yeast took over the task of fermentation by converting the starch from the grain into sugar and then fermenting it into alcohol [2]. It was only in times of food abundance that man was able to experiment with other fermentation methods. One of the first is probably the production of sourdough, which is still used today in bread baking and makes the bread more digestible, longer fresh and durable. The oldest sourdough bread found so far is almost 6000 years old and comes from Switzerland [3]. The sourdough uses lactic acid bacteria and yeasts that occur naturally on the cereals. The focus here is not on alcoholic fermentation, but on heterofermentative lactic acid fermentation (Figure 1). In this process, sugars are fermented to carbon dioxide, acetic acid, lactic acid and ethanol. The shelf life is determined less by the alcohol than by the reduction of the pH value. Hence the name "sourdough". A low pH value ensures an environment in which only very few bacteria and virtually no mold can grow. The advantage is that the lactic acid bacteria are not harmful to humans. They colonize the entire "habitat" and displace other bacteria or no longer allow the new settlement of undesirable bacteria. In addition, lactic acid bacteria are probiotic, i.e. they are healthy for the intestinal flora and positively influence the human immune system [4]. It is conceivable that heterofermentative lactic

acid fermentation found its way into vegetable fermentation through the knowledge of sourdough production. It is heterofermentative, here e.g. cabbages are preserved (sauerkraut, kimchi) but also carrots or cucumbers.

The yeast fermentation of berries, here in particular the grape berry, to an alcoholic beverage (wine) enabled a subsequent fermentation, namely the acetic acid fermentation. Although this is somewhat simple, it is also delicate, since special hygiene precautions must be taken to prevent acetic acid fermentation from spreading to a winemaker's wine stocks. However, the preservative properties of acetic acid are undisputed and are the basis for e.g. sour pickles and even sour roast, but of course also vinegar as a seasoning.

With the cultivation of dairy cattle and a surplus of milk, the possibility for another form of lactic acid fermentation was discovered: homofermentative lactic acid fermentation. Here, the sugars are fermented only to lactic acid. Other fermentation products do not occur because specific lactic acid bacteria strains that enter the milk from the animal's udder. These strains have a different metabolism than the heterofermentative lactic acid bacteria. Until the discovery of bacteria and microorganisms in the 17th century and thereafter, everything depended on experience in fermenting natural products. In modern food production we can precisely comply with all conditions to arrive at the perfect product. For generations, stable lactic acid bacteria communities have been cultivated in sourdough production. These could be identified using modern genome sequencing methods. Thus, today it is possible to achieve the same result every time with these cultures. However, instead of fermenting only rye and other cereals, today we have all the possibilities to use any plant material containing starch or sugar. Therefore, traditionally used botanical





ingredients that have been used in cosmetics for decades can be lifted to the next level. An example of this is the golden millet *Panicum miliaceum*, which is particularly promoted for strengthening keratin structures (hair, fingernails), but has overall skin-strengthening effects. Fermentation of the millet seed with a community of specially selected lactic acid bacteria creates a product with completely new properties (DEFEN-SIL®-PURE, hereinafter "millet ferment").

### **Material and methods**

Extract preparation to obtain DEFENSIL<sup>®</sup>-PURE: ground millet seed was fermented with a proprietary mixture of lactic acid bacteria used for sourdough production. The fermentation supernatant was filtered and preserved (INCI: Water, Panicum Miliaceum (Millet) Seed Extract, Lactobacillus Ferment, Sodium Benzoate, Potassium Sorbate).



SLI Chemicals GmbH · Insterburger Str. 7 · 60487 Frankfurt am Main · Germany E-Mail info@slichemicals.com · web www.slichemicals.com Analysis of saccharides and organic acids by <sup>1</sup>H-NMR spectroscopy (Bruker Avance III HD 500 MHz). The same for calcium and magnesium. Silicon analysis by inductively coupled plasma atomic emission spectrometry according to DIN EN ISO 11885:2009-09.

Bacterial growth was determined in liquid cultures. Growth of different strains in buffered chloride-peptone solution (NPP, Biolife 4013952) and transfer of 100 - 1000 cfu/ml into phosphate/citrate buffer (10 mM citrate, 20 mM disodium hydrogen phosphate, 1.09 g/l NaCl, 0.37 g/l KCl, 0.055 g/l CaCl<sub>2</sub>·2H<sub>2</sub>O, 0.011 g/l MgCl<sub>2</sub>·6H<sub>2</sub>O). Growth of the strains was determined after 24 hours by plating and counting the colony forming units (cfu).

Caspase-1 activation was determined by fluorescence microscopy. Primary human keratinocytes (47-year-old donor with Caucasian skin) were cultured for 24 hours and then treated with a cytokine cocktail (TNF- $\alpha$ , IL-4, IL-5, and IL-13, and *Staphylococcus aureus* toxin) to induce atopic conditions for 6 hours. The success of induction was verified with quantification of TSLP. The active was added at 1% before (24 h), during, or after induction of the atopic condition (6 h). Fluorescence staining with 5-carboxyfluorescein-Tyr-Val-Ala-Asp-fluoromethyl ketone covalently bound to activated caspase-1.

The *in-vivo* study was performed in accordance with the Declaration of Helsinki of the World's Medical Association. All study participants signed a written informed consent at the beginning of the study. The active ingredient (3% Panicum Miliaceum (Millet) Seed Extract, Lactobacillus Ferment) or placebo was administered in an emulsion (INCI: Water, Caprylic/Capric Triglyceride, Glyceryl Stearate Citrate, Pentylene Glycol, Cetearyl Alcohol, Glycerin, Sodium Anisate, Sodium Levulinate, Xanthan Gum, Citric Acid, Panicum Miliaceum (Millet) Seed extract, Lactobacillus Ferment, Sodium Benzoate, Potassium Sorbate) was applied to the face and atopic site twice daily for 8 weeks. It was tested on 20 study participants (10 verum, 10 placebo). Skin hydration was determined by corneometry, transepidermal water loss by TEWAmetry. The severity of the atopic site was determined dermatologically using a 100 mm analog scale.

### Results

To obtain the millet ferment, the ground millet seed was broken down in a multi-culture fermentation process. Various lactic acid bacteria are used in a stable ratio to form an ecosystem that effectively displaces competitors and optimally breaks down nutrients. The ecosystem comes from a sourdough base that has been sampled and preserved over decades and must be kept stable by carefully monitoring and adjusting the culture parameters.

A comparison was made between non-fermented millet extract and fermented millet extract. Analysis of (poly)saccharides and AHA acids (alpha-hydroxy acids: malic acid, lactic acid) as well as acetic acid and ethanol showed complete degradation of polysaccharides in the ferment and their conversion into the

Ingreaient	perore termentation	after termentation
Polysaccharides	253 mg/l	0 mg/l
Glucose	452 mg/l	0 mg/l
Malic acid	15 mg/l	63 mg/l
Lactic acid	0 mg/l	1379 mg/l
Acetic acid	110 mg/l	194 mg/l
Ethanol	0 mg/l	1747 mg/l
Mg	18.2 mg/l	40.9 mg/l
Ca	6.3 mg/l	56.6 mg/l
Si	9.0 mg/l	10 mg/l

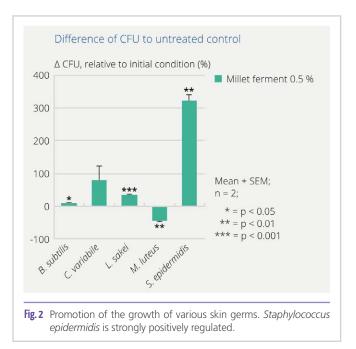
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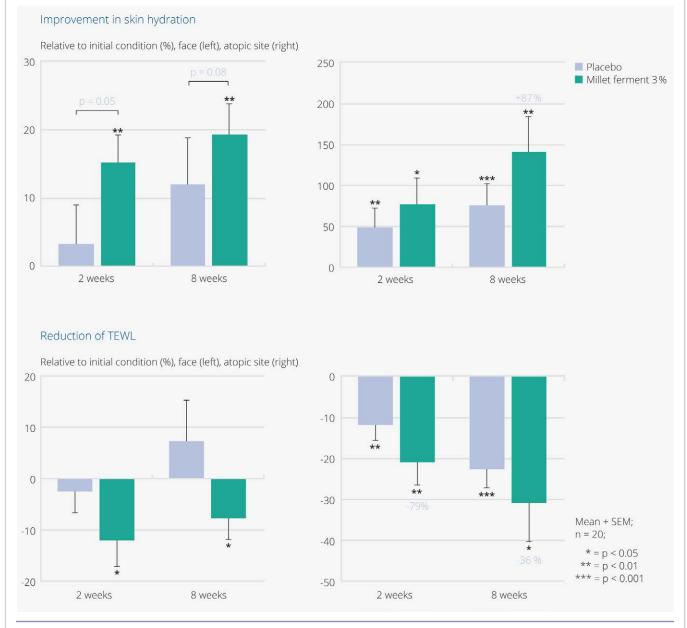
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fermentation products of a hetreofermentative lactic acid fermentation (**Table 1**). The acidic pH between 4 and 5 reflects the accumulation of organic acids. Interestingly, fermentation significantly increased the content of important calcium and magnesium. In the case of magnesium by more than double, and in the case of calcium by 9 times. A similar phenomenon was also observed in the fermentation of moringa leaves [5]. The content of bioavailable silicon remained constant.

acids and ethanol. Minerals can be released more effectively.

To study the effects of the active ingredient towards the skin microbiota, representatives of bacterial strains found on the skin were used. *L. sakei* was used as a representative of the Lactobacili, which is found, for example, in the nose, but is best known for its isolation from kimchi, the Korean sauer-kraut. It was determined how the growth of the bacteria behaves when exposed to a buffer having approximately the composition of human sweat (citrate/phosphate buffer with mineral salts, see Material and Methods) in the presence of the active. It was found that the growth of *Staphylococcus epidermidis* in particular, was stimulated **(Figure 2)**.





content

Fig. 3 Skin hydration and transepidermal water loss on the face and atopic sites. The active ingredient can increase skin hydration at atopic sites by up to 140%.

This germ is crucial for a healthy skin flora by limiting or even preventing the growth of undesirable microbiota, such as S. aureus, a potential trigger of atopic dermatitis. Similarly, it limits excessive growth of Bacillus subtilis, a commensal skin germ that is significantly but only slightly increased in our experiment. Based on current research, neither explicit good nor bad properties are attributed to *B. subtilis*. Nevertheless, it is known that there is quorum sensing between it and S. epidermidis, with S. epidermidis having the upper hand. Bacillus subtilis can be responsible for an unpleasant foot odour. The promotion of the growth of *Corynebacterium variabile* and Lactobacillus sakei suggest that the agent may promote a microbiota found on somewhat moister skin regions. As a consequence, the milieu could change from dry skin to slightly more moist skin. Micrococcus luteus, another commensal germ also known as an "air germ", is negatively regulated. This bacterium can create malodour.

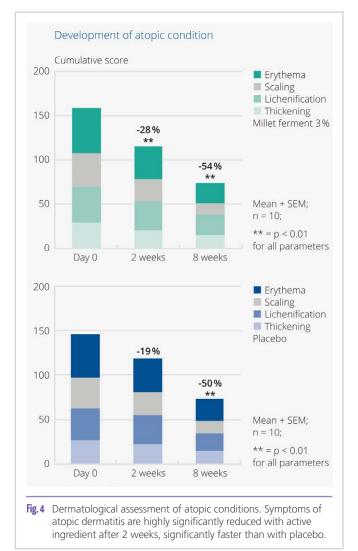
It was shown that 1% of the millet ferment resulted in a significant reduction of caspase-1 expression in a skin model that was exposed to *S. aureus* toxin and a cytokine cocktail to induce an atopic state. Caspase-1 is part of the inflammasome and leads to apoptotic processes (mainly pyroptosis) and strong inflammatory responses. In a preventive application, the expression of this cell death-inducing enzyme was pushed to basal levels. A limit-significant reduction was also observed with immediate (-71%; p = 0.07) or curative application (-61%; p = 0.08) (n = 6, not shown).

Application of the active ingredient to atopic patients in the non-treatment interval showed a skin moisturizing and barrier-strengthening effect both on the face (non-atopic area) and directly on the atopic site (Figure 3). However, while skin hydration in the face increased by about 20% after 8 weeks, it increased by 140% on the very dry atopic site compared to the baseline condition. Transepidermal water loss was similar, but





not quite as pronounced. On the face, TEWL was reduced by a maximum of 12%, and on the atopic site by more than 30% compared to the initial condition. Placebo had no significant effect on the face; at the atopic site, the effect was significantly better with the active substance. This is also reflected in the dermatologist's evaluation of the atopic lesions (Figure 4): the parameters redness, scaling, lichenification and thickening were each reduced highly significantly with the active substance after just 2 weeks, by a total of 28%, which was not the case with placebo. After 8 weeks, the placebo and active ingredient



were similar in their effects, with an advantage for the active. The atopic lesions visibly decreased and the appearance of the facial skin improved significantly **(Figure 5)**.

### Discussion

The active ingredient is of probiotic origin, i.e. the end product of a bacterial lactic acid fermentation of millet seed. The filtered clear culture supernatant contains the fermentation products but no living components of the lactic acid bacteria. Thus, the compound can be classified as postbiotic. Its action is likely to function via correction of the very dry skin environment in atopic dermatitis, where the postbiotic fermentation products may play a crucial role: application may optimize the pH and metabolome of the skin for a healthy skin microbiota. It is known that metabolites of lactic acid fermentation can suppress undesirable germs and promote the growth of desirable ones. Furthermore, it has already been shown in a 3D epidermal model that fermentation products from lactic acid fermentation have a positive influence on epidermal health, e.g. reducing TEWL and increasing the moisture content of the stratum corneum [6], analogous to the results from our in vivo study.

Overall, it can be said that the skin microbiota is positively regulated by the active ingredient, in particular S. epidermidis, a germ that can displace undesirable microbes or prevent their colonization. The promotion of more moisture-loving germs such as C. variabilis and Lactobacilli suggests that the active ingredient promotes an appropriate skin environment, which is of great importance in atopic dermatitis with extremely dry skin areas. The question arises whether the active ingredient itself creates a moister skin environment (significant increase in skin moisturization and reduction in TEWL) or whether this is first brought about by a correction of the skin microbiota. This would require extensive studies on the microbiome and its temporal development over the entire course of the study, which unfortunately was not possible at the time of the study due to the Corona pandemic. It is a fact that the active substance alleviates the symptoms of atopic skin (redness, scaling, lichenification, thickening) more quickly. A more acidic skin environment and fermentation products from lactic acid fermentation have already been described as beneficial for the care of atopic skin

# RAHN

[7-9]. The good effect of the placebo is not surprising, since the formulation used a base that should be suitable for people with atopic dermatitis to achieve appropriate compliance.

The difference between our millet ferment and other fermented cosmetic actives lies in the slow and highly controlled fermentation process similar to sourdough bread. In bread production, this leads to a more digestible product without intolerances, as can be the case with the breads of the wholesale and bakery industry that are optimised for speed. A 9-fold improvement in calcium concentration was shown compared to the unfermented millet extract. The slow and controlled fermentation favours the complete degradation of polysaccharides and thus the release of bound calcium [5]. By that and the use of special alpine millet as the basis for our active ingredient, we take a very sustainable approach, in line with the trend of increasingly popular natural foods. Perfect for providing the right care for sensitive skin such as that of people having atopic skin.

In summary, the active ingredient rapidly moisturizes dry skin areas, strengthens the skin barrier and can compensate for deficiencies, especially on atopic skin. It alleviates atopic symptoms and is particularly well suited for sensitive and dry skin. o return to the initial question: A postbiotic cosmetic active ingredient can indeed "preserve" the skin by making it more resistant to external influences, as evidenced by increased skin hydration and an improved skin barrier.

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Strengthens the healthy skin microbiota



atopic dermatitis





### A Cell Nectar to Optimize Vitamin D synthesis: The D-Skin

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

### abstract

Due to modern lifestyles and the lack of sun exposure, like recently experienced in the pandemic, there is a deficiency of vitamin D. The new active Lithops Pseudotruncatella Callus Lysate stimulates the epidermal cells to stimulate the synthesis of vitamin D and bring beneficial molecules to the skin. Made from *Lithops pseudotruncatella* stem cells, the active ingredient optimizes the skin microenvironment (strong spatial organization of cell membranes, maximization of light use, deep skin hydration boost and vitamin D activation) to strengthen, volumize, revitalize faded skin and get glowing D-Skin. A new cosmetic concept explained in the present article. Various *in vitro* and *in vivo* assays have been carried out to demonstrate the activity and efficacy of the active and the basis of this new concept supported by cosmetic science. A new and healthy way to deal with skin.

### Introduction

### An ignored pandemic: vitamin D deficiency

Vitamin D deficiency is a worldwide pandemic affecting over 1 billion people worldwide [1]. This urgently needs to be addressed to prevent morbidity, mortality and increasing expenses related to the treatment of the involved chronic illnesses [2].

Modern lifestyle is driven by technological advances and social media platforms. A lack of sun exposure on a long-term basis can have detrimental effects on our bodies. The increase of living indoors, office-based jobs, and reduction of socialization in public spaces, along with the rightfully advised and important use of photo-protective products when going outdoors, makes the population especially susceptible to Vitamin D deficiency.

Described as 'the Sun Vitamin', Vitamin D is a steroid with hormone-like activity. It regulates the functions of over 1000 genes and is essential for growth and development. Current research indicates that the vitamin D deficiency is involved in several illnesses e.g. heart and autoimmune diseases [3].

Although there is clinical evidence that links the vitamin D deficiency with different illnesses and physiological malfunctions, lesser is known about the **role of Vitamin D directly on skin**. The vitamin D is synthesized topically and distributed through all the body. This vitamin D has a direct effect on the skin health and appearance and Vytrus Biotech has been deepening focused its research on how to apply it to cosmetics.

### The story of vitamin D and skin environment

Up to 90% of vitamin D is produced by sunshine exposure of skin and the remainder comes through diet. Diet, which

most humans consume, contains hardly anylittle vitamin D. Traditionally the human vitamin D system begins in the skin, not in the mouth [4].

There is a **close relationship between vitamin D**, **sun and skin**. Although this vitamin can be acquired through diet, **the skin is the central vitamin D factory of the body with a minimum of 25 minutes** of sun exposure as the mechanism that starts the production machinery. Vytrus Biotech's study has focused on lesser-known functions at the dermatological level that have recently been related to vitamin D.

Exploring new cosmetic horizons for this vitamin that comes from the sun has led Vytrus to discover its multiple benefits at skin level4 and give it a relevant role in skin care. Amongst its dermal functions, the following stand out:

- **Immuno-protection:** actively participating in the innate and acquired immunity.
- **Photo-protection:** key in the irradiation damage response to dimers and other by-products.
- **Antimicrobial activity:** microbial peptide regulation (TLR-2, defensin and cathelicidin).
- **Protection & moisturization:** maintenance of the epidermal barrier (involucrin, loricrin and filaggrin).
- Healthy ageing boosting: key in cell turnover, maintaining healthy blood microcirculation, antioxidant defences.

Vytrus Biotech has studied the vitamin D biosynthesis and discovered a new cosmetic approach to its production: **the optimization of the skin's endogenous environment**. Although there are still many aspects to explore, Vytrus Biotech has seen that it is possible to optimize the synthesis of vitamin D topically by promoting a favourable skin microenvironment. This skin environment optimization has two main focuses:

- Improving endogenous water levels (skin deep-layer hydration)
- Maximizing light use by optimizing the optical properties of the skin

The vitamin D biosynthesis is a process that begins in the skin with the transformation of provitamin D3 into previtamin D3, and then into vitamin D3. This vitamin D3 is then transported through the bloodstream to the liver and kidneys where it is hydroxylated to obtain its **active form: calcitriol or VITAMIN D**. However, the skin has the metabolic capacity to produce the active form of vitamin D for local action and is **the only body tissue in which vitamin D is produced and used** [5]. Depending on the microenvironment, the previtamin D3 can be transformed into inactive by-products or into lumisterol. This lumisterol can be reversibly transformed into previtamin D3 again. Therefore, lumisterol can be used as a reservoir for vitamin D.

The vitamin D synthesis needs to take place under optimal conditions to obtain the necessary quantity, no more and no less. These optimal conditions include the aforementioned microenvironment optimization: having sufficient hydration at deep skin levels (upper dermis, dermo-epidermal junction and lower epidermis), and having the correct 3D structure which optimizes the transformation of previtamin D3 into vitamin D3, or into lumisterol as the vitamin D reservoir, while preventing the formation of the inactive derivatives of previtamin D3.

This new approach of optimizing vitamin D synthesis emerges through influencing the skin environment, always favoring to the maximum the use and protection of the resources available: water reserves, incidence of light, and spatial organization.



- rig. 1 Optimization of skin microenvironment
- Strong spatial organization of cell membranes: a fully functional and well-structured cell membrane is essential for an optimized vitamin D synthesis.
- **Maximizing light use:** modulating the optical properties of the skin.
- **Boosting water deep reserves:** vitamin D synthesis requires a good water level which modulates the polarity of the medium.

Through the optimization of the skin microenvironment, **the efficiency of vitamin D synthesis is increased** preventing **the generation of inactive by-products while boosting lumisterol as a key vitamin D reservoir**.

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### **D-SKIN: a new concept in Cosmetics**

Vytrus Biotech proposes a new concept that improves both the skin and the vitamin D at the same level: the D-Skin. What does it mean?

- A Dewy skin
- A D-lighted skin
- A vitamin D enriched skin
- A skin Deep-layer hydration
- A healthy Sun care with a D-approach

Associated with well-being and health, the plump and luminous appearance of the skin not only favors, but also radiates youth. We could affirm that there are two factors that directly influence the optical properties of skin and define a D-Skin to capture and radiate light: skin tone and texture or quality of the skin surface.

- **Tone:** the better the skin microcirculation the healthier the skin tone becomes and the better skin's light absorption.
- **Texture:** Skin hydration is basic for maintaining the skin luminous, radiant and volumized, as plumped from the inside.

### Nectaria Lithops: The new cell nectar for a D-Skin

Inspired by nature, Nectaria Lithops (INCI: Lithops Pseudotruncatella Callus Lysate) is a plant stem cell active ingredient that optimizes skin microenvironment and stimulates vitamin D synthesis to strengthen, revitalize, volumize, and glow a faded skin to achieve a D-Skin.

The story behind comes from the plant *Lithops pseudotruncatella* ('Living Stones') (Figure 2). Thanks to its mechanism of action, the active ingredient boosts vitamin D production by epidermal cells to create the optimal skin environment, hydrate the deepest layers of the skin, increases the skin dewiness and cheek volume and improves microcirculation, skin tone and texture.

The product's mechanism of action (Figure 3) consists in the optimization of the skin microenvironment to stimulate the

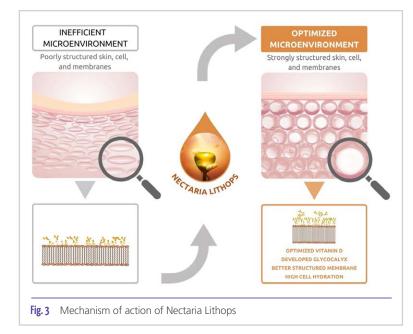




Fig. 2 Optimization of skin microenvironment

vitamin D production by the cutaneous cells. This innovative approach is based on improving the cutaneous structure, creating an adequate structural space for the vitamin D production while boosting deep water reserves. This is possible thanks to the Lithops culture, rich in biomimetic glycocalyx-derived glycoconjugates, organic acids, and polyphenols. This synergy allows to protect and repair the glycocalyx (sugars) of epidermal cells to achieve optimal structure and functionality. Optimized vitamin D levels result in a dewy, radiant, plumped and deeply hydrated D-Skin.

### *In vitro* tests

Several *in vitro* tests were performed to understand and demonstrate the mechanism of action of the active ingredient.

### In vitro 1: Antioxidant activity

The product's antioxidant effect was measured by the enzymatic DPPH assay, a spectrophotometric test which determines the capacity to capture the free radical DPPH (2,2-Diphenyl-1-picrylhydrazyl). A solution of Ascorbic Acid at 16.5 ppm was used as the positive control.

Nectaria Lithops demonstrated to capture a 42% of the free radical DPPH versus the untreated control, showing an antioxidant effect comparable to that of Ascorbic Acid activity (50%).

### In vitro 2: Anti-collagenase activity

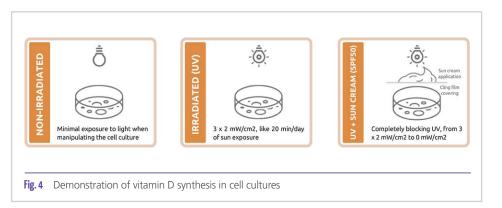
The enzymatic collagenase activity was measured in the absence or presence of the active ingredient. EDTA at 5 mg/ml was used as the positive control. Nectaria Lithops inhibited the collagenase activity by 82% vs untreated control, thus preventing collagen degradation. This activity was comparable to the effect of EDTA (89% inhibition).

### In vitro 3: Inhibition of lipid peroxidation

Another enzymatic antioxidant effect was assessed, in this case to check the product's capacity to inhibit the lipid peroxidation. preventing was shown by the application of the ingredient on the culture. It demonstrated a 82% inhibition vs the untreated control, comparable to vitamin E derivative (31.25  $\mu$ /ml), used as the positive control.

### *In vitro* 4: Proliferation of fibroblasts (HDF)

The regenerating effect of Nectaria Lithops was demonstrated by the time the proliferation of fibroblasts was boosted by 17% at 0.01% dosage, reaching up to 26% at 1% dosage. This improvement of cell viability was observed in a suppressed medium (low in growth factors).



### In vitro 5: Gylcocalyx increase on keratinocytes (NHEK)

The glycocalyx is a layer of carbohydrates (oligosaccharides) bound to proteins, as glycoproteins, and lipids, as glycolipids, of the outer surface of the cell membrane in eukaryotic cells. It envelops the entire cell and provides a semipermeable barrier to the external environment. The glycoproteins and glycolipids, part of the glycoconjugates present in the glycocalyx, contain sialic acid and N-acetylglucosamine, which can be marked with a plant lectin, Wheat Germ Agglutinin (WGA-FITC) and analyzed with fluorescence microscopy.

We were able to increase the number of glycoconjugates by + 50% with an application concentration of 1% Nectaria Lithops on keratinocytes (NHEK). Higher glycoconjugates content means a stronger and a better structured glycocalyx.

### Vitamin D & Lumisterol Synthesis

In the following *in vitro* tests (6 and 7), the active ingredient was applied to cell cultures of fibroblasts and keratinocytes in three different situations (**Figure 4**) to see the behaviour of the active: non-irradiated (minimal exposure to light), irradiated ( $3 \times 2 \text{ mW/cm}^2$ , equivalent to 20 min/day of sun exposure), and UV light + a SPF50 sun cream (completely blocking UV, from  $3 \times 2 \text{ mW/cm}^2$  to 0 mW/cm<sup>2</sup>).

### In vitro 6: Vitamin D synthesis increase

### Activity in fibroblasts

The active ingredient increased the synthesis of vitamin D (+ 77% vs untreated control), more when the Human Dermal Fibroblasts (HDF) were submitted to irradiation with UV (+ 112% vs untreated control). There even was an increase when the fibroblasts were irradiated with UV and an UV blocking SPF50 sun cream was applied on the top of the culture (+ 119% vs untreated control).

### Activity in keratinocytes

An increase of the vitamin D synthesis in keratinocytes (Ha-CaT) versus untreated controls with the active was measured in all three conditions: + 64% when non-irradiated, + 63% when irradiated with UV, and + 57% when irradiated with UV and the SPF50 sun cream treatment was applied.

### In vitro 7: Lumisterol synthesis increase

### **Activity in fibroblasts**

content

It was demonstrated that the active increased the synthesis of lumisterol (reservoir of vitamin D) in Human Dermal Fibroblasts (HDF) in all three conditions (non-irradiated, irradiated with UV and irradiated and treated with the SPF50 sun cream). In the untreated controls, the presence of lumisterol was not detected in any of the three situations.

### Activity in keratinocytes

Finally, an increase in the levels of lumisterol synthesis in keratinocytes (HaCaT) was measured in the three different conditions analyzed. Again, in the untreated controls the presence of lumisterol was not detected in any of the three situations. These assays on vitamin D and lumisterol synthesis boosting lead to a very interesting conclusion: there is no need to choose between having our skin protected from the sun -by using sun creams- and bringing beneficial properties to our skin health by topically optimizing and activating the levels of vitamin D 'the sun vitamin'. A new open horizon to exploring biotechnology advances in the sun care industry.

### **Clinical assays**

Several clinical trials evaluated and demonstrated the efficacy of the active ingredient on the volunteers' skin and appearance.

### In vivo 1:

The first *in vivo* test was performed on a 40-volunteer panel aged 20-64 years old. The study was double-blind and placebo-controlled (half of the volunteers applied the placebo cream and the other half applied the cream containing Nectaria Lithops) and lasted 56 days with two daily applications. The assay was carried **out during the pandemic on volunteers with low levels of vita-min D**. Several parameters were analyzed to see the performance of a cream containing the active ingredient at 1.5% dosage.

### Skin dewiness improvement:

The first marker to be analyzed was the skin dewiness, which represents the ratio between the diffuse brightness (luminosity) and the specular brightness (oiliness).



Fig. 5 Evaluation of skin dewiness improvement

Nectaria Lithops increased the skin dewiness by 17% vs placebo, thus improving the skin luminosity and reducing the skin oiliness (Figure 5). This results in providing a skin perfectioning and mattifying effect, enhancing the skin tone and texture.

### Cheek volume increase:

The volunteers' cheek volume was measured by 3D Volume Face Analysis, where the blue areas indicate the gain in volume versus initial time of the treatment (Figure 6).

After applying a cream containing 1.5% dosage of Nectaria Lithops, the cheek volume of the volunteers significantly increased by 1.95 cm<sup>3</sup> (+ 3%), and up to 3.5 cm<sup>3</sup> (+ 4 %), vs placebo, providing a dermal filler-like effect (\*).

\*Hyaluronic Acid dermal injections increase the volume by at least 5 cm<sup>3</sup>.

### Water retention increase:

The vitamin D deficiency is related to epidermal barrier dysfunction [6]. Subjects with low levels of vitamin D have drier

skin, with lower water content. Furthermore, the assay was carried out during the COVID-19 pandemic. The hydration levels of the volunteers, measured by corneometry, significantly decreased in all the placebo groups (p < 0.001): - 10% in full face; - 7% in the front, and - 11%

However, the treatment with the active Nectaria Lithops prevented the skin dehydration, thus significantly increasing the water retention capacity of the volunteers compared to placebo (p < 0.05) (Figure 7).

in mask zone.

# DERMAL FILLER-LIKE EFFECT

Fig. 6 Evaluation of cheek volume increase

In vivo 2

There was a panel compound of 30 volunteers (21–62-yearold), of which 15 applied the placebo cream while the other applied a 1.5% active dosage in a cream in a double-blind study for 56 days with two daily applications. The assay was performed during the pandemic. Choosing **volunteers with low vitamin D levels** was, once more, a key aspect to analyse the efficacy of the active.

Hyperspectral camera, which allows to study the deepest layer of the epidermis and the dermo-epidermal junction (1 mm depth), was the sophisticated technique to measure several parameters as follows:

### Skin deep-layer hydration:

Firstly, the Tissue Water Index (TWI) was measured to evaluate the deep skin hydration levels where the yellow, orange, and red areas indicate higher TWI (higher deep hydration), as seen in **Figure 8**: The results showed that Nectaria Lithops

demonstrated up to a 17% TWI increase versus initial time, and by 2.2-fold vs placebo (full face measurement).

### **Oxygenation and Microcirculation:**

Skin vitamin D synthesis helps to maintain a healthy blood microcirculation [7]. Subjects with low levels of vitamin D have a worse microcirculation. Furthermore, the assay was carried out during the COVID-19 pandemic.

 ${\rm StO}_2$  (oxyhaemoglobin) analyses the tissue oxygenation (how well oxygenated the skin is), an indirect way to check the microcirculation (better microcirculation means higher oxygenation of the tissue).

While placebo significantly reduced the StO<sub>2</sub> parameter, the treatment with the active ingre-

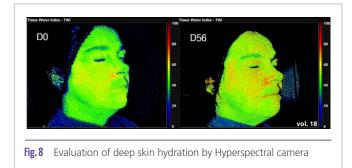
dient did not significantly reduce  $StO_2$ , and in fact showed a 34% increase vs placebo treatment (Figure 9 – Vol. 2).



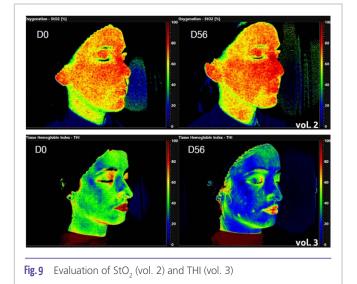
Fig. 7 Evaluation of water retention capacity increase

**sofw**journal | 147 | 10/21

<u>content</u>



The Tissue Haemoglobin Index (THI - total haemoglobin,  $O_2$  and  $CO_2$ , i.e., oxyhaemoglobin plus deoxyhaemoglobin) indicates, when analysed together with the  $StO_2$  parameter, the balance of  $CO_2$  and  $O_2$  (e.g., if  $StO_2$  is higher versus placebo and the THI is lower versus placebo, the main reduction in the THI will be due to less  $CO_2$ , and therefore the better the tissue's microcirculation and oxygenation will be). The active ingredient reduced the THI by 16% vs placebo and confirms its improvement in the oxygenation and in the microcirculation of the skin (Figure 9 – Vol. 3).



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#### Conclusion

The active ingredient Nectaria Lithops stimulates the skin to produce its own vitamin D and synthesize it topically to strengthen, revitalize, volumize, and glow faded skin and achieve a D-Skin. While opening a new D-vision for the whole Sun Care market, Nectaria Lithops approaches cosmetic applications such as volumizing and brightening formulations, tone and texture evening treatments, wellbeing & antioxidant products, formulations for dry/very dry skin, dermal filling applications, moisturizers, skin densifying and structuring formulations, as well as radiance and glow treatments, highlighters, and colour cosmetics, amongst others. This new raw material from Lithops plant stem cells brings a paradigm in Cosmetics and a new way for skin to interact with the sun and its own vitamin D synthesis mechanisms. A new path for not having to choose between being sun-protected and having a healthy skin enriched in vitamin D, also known as 'the sun vitamin'.

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# A study of *prunus persica* (peach) resin extract on instant skin firming and anti-wrinkles

H. Shao-yong, Q. Qiu-yue, H. Fang, Z. Li-dan, L. Yi-na

abstract

**P***runus persica* (peach) is a resin secreted from the bark of rosaceae peaches or mountain peaches. It is a pink or light yellow to yellowish brown translucent solid. The main components of *prunus persica* (peach) are polysaccharides and proteins. *Prunus persica* (peach) polysaccharides are mainly composed of galactose and it is composed of arabinose. And also contains a small amount of mannose, rhamnose and glucose which has the effects of beauty and anti-aging. Subjects of different ages and suitable health were selected for clinical evaluation of the instant wrinkle-removing function of Immedeline PG. It is found that both 5% and 10% Immedeline PG can significantly improve the firmness of finger skin. It was found that 10% Immedeline PG can significantly improve the firmness of beauty at 2 hrs and can be maintained for 8 hrs. After 5 minus of applying, the eye corner wrinkles were significantly improved. The effect was most obvious in 2 hrs and maintained after 8 hrs.

#### Introduction

Skin aging is a complex biological process, which is produced by two basic processes: internal aging (affected by genetic factors, cell metabolism, hormones and metabolic processes) and due to long-term exposure to light, pollution, ionizing radiation, chemicals and toxins External aging. Intrinsic aging, also known as time aging, is observed in the sunscreen skin of the elderly [1-5]. Wrinkles are the result of a natural and irreversible process of change. Aging is the main cause of wrinkles. Moisturized, smooth, and elastic young skin will gradually age after 25 years of age. With age, the function of sebaceous glands and sweat glands declines, the skin loses luster, becomes dry, the subcutaneous fat decreases, and the skin begins to loosen and lose its elasticity. This is called "aging". Around the age of 30, crow's feet began to appear at the end of the eyes, and at the age of 40, wrinkles climbed up to the forehead. After the age of 50, the entire face will appear "rings of life" [6, 7]. Prunus persica (peach) is derived from the resin secreted by the rose family peach or mountain peach[8]. The main component is polysaccharide, whose polysaccharide content is more than 90%[9]. Dried Prunus persica (peach) is crystalline and hard, similar to amber. Prunus persica (peach) has sufficient water solubility and appropriate viscosity. It has anti-wrinkle and firming effects[10-12].

We evaluate the effects of Immedeline PG on skin firming, skin elasticity and wrinkle removal through subjective evaluation and instruments. All data show that Immedeline PG can significantly improve skin firmness, and can effectively improve skin elasticity and improve wrinkles.

#### **Materials and Methods**

#### **Immedeline PG Preparation**

The *prunus persica* (peach) resin extract were mixed with different proportion and dissolved in 1,2-Hexanediol (CAS NO. 6920-22-5), ethylhexylglycerin (CAS NO. 70445-33-9) and water. Finally, Immedeline PG solution was obtained through filtration.

#### **Firming evaluation experiments**

15 healthy subjects were selected, including 8 males from 30 to 40 years old and 7 females from 20 to 40 years old. During the test, the temperature is  $(37\pm2)^{\circ}$ C, and the relative humidity is 30%~60%. Before the test, the subjects should wash their hands carefully. Waiting for 5-10 mins in the test environment, and the finger skin is dry to a normal state before proceeding with subsequent experiments. Using 40~50 µl 1%, 3%, and 5% FLEXANII (sodium polystyrene sulfonate, CAS NO. 25704-18-1) as the standard in the area between the first knuckle and the second knuckle of the finger of one hand, corresponding to the firmness of the skin. Recorded as 1, 3, 5 points. Apply 5% and 10% Immedeline PG to the other finger in the same way. Waiting for 5-10 mins, gently bend finger to feel the firming effect and score the results.

# Evaluation of human facial cheekbone skin elasticity test

The skin elasticity was detected by Cutometer® dual MPA 580 (CK, Germany). Briefly, 6 healthy and normal subjects were selected, including 4 males aged 20-40 years old and 2 females 20-40 years old. During the test, the temperature is  $(37\pm2)^{\circ}$ C, and the relative humidity is 30%~60%. The subjects cleaned the face and waited for it to dry in the test environment. Marking the cheekbones on the face with a small label (for accurate positioning during measurement) and applied formula (contained 10% Immedeline PG). After 5 mins of absorbing, the skin elasticity was measured by MPA580. Then after 30 mins, 60 mins, 4 hrs, 6 hrs, and 8 hrs, MPA580 was used to measure skin elasticity parameter R2 **(R2= (e(a)-e(a+b))/e(a) = Ua/Uf).** 

The to tal elastoplasticity of the stretched part was measured 3 times with an interval of 5 mins each time.

# Evaluation of instant wrinkle removal test on human eye corners

The wrinkle parameters were measured by VC98 (CK, Germany) and VISIA (Canfield, America). 11 healthy subjects were selected, including 4 men aged 40-50 years old, 1 man

aged 30-40 years old, 3 women aged 50-60 years old, and 3 women aged 30-40 years. During the test, the temperature is  $(37\pm2)^{\circ}$ C, and the relative humidity is  $30\% \sim 60\%$ . The subjects cleaned the face and waited for it to dry in the test environment. Marking the corner of the eye with a small label (for accurate positioning when taking pictures), and take a picture with VC98, which is recorded as T0 (smear sample Before); Using a cotton swab to take the same amount of formulas (contained 2%, 5% and 10% Immedeline PG) and apply it evenly to the area selected by the left and right corners of the eye, and wait for the sample to be completely absorbed and dry; after it is completely dried for 5 mins, take a photo with VC98 and VISIA, which is recorded as T5. After 30 mins, 60 mins, 4 hrs, 6 hrs and 8 hrs, take pictures with VC98 and VISIA respectively which recorded as T30, T60, T4h, T6h and T8h. The pictures were cut to the same position and the same size, then analyzed the image by VC98 and VISIA software. Calculated the five-segment average R3 and the change rate of skin wrinkle parameter SEw, the calculation formula is as follows:

R3 change rate = (R3<sub>after</sub> - R3<sub>before</sub>) / R3<sub>before</sub> × 100% SEw rate of change = (SEw<sub>after</sub> - SEw<sub>before</sub>) / SEw<sub>before</sub> × 100%

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		2	3	4	5	6	7	8	9	10	11	12	13	14	15	Average score
5% Immedeline PG	2	1	2	1	1	1	1	2	2	1	1	2	3	1	1	1.47
10% Immedeline PG	3	5	3	2	3	3	2	3	5	4	3	5	3	4	3	3.4

content

#### **Data analysis**

GraphPad 5 software was used for statistical analysis of the data, and the paired t test was used for statistical analysis of the data. P<0.05 was regarded as significant difference, P<0.05 and P<0.01 was regarded as extremely significant difference.

#### **Results**

#### Immedeline PG improves the skin firming

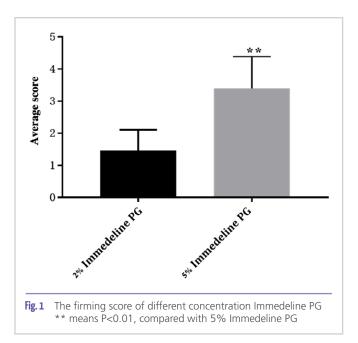
It can be suggested that after applying different concentrations of Immedeline PG and completely drying, 15 subjects all said that they could feel a significant firmness in 5% and 10% Immedeline PG (Table 1). It also can be concluded that the skin tightening score of 10% Immedeline PG is significantly higher than that of 5% Immedeline PG (P<0.01). Therefore, both 5% and 10% Immedeline PG can enhance the skin's firmness.

#### Immedeline PG improves the skin elasticity

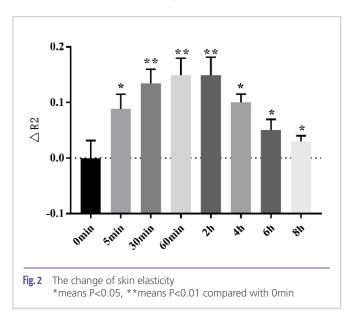
Spread 10% Immedeline PG evenly on the cheekbones of the subjects' face and wait it dry completely for 5 mins, 10 mins, 30 mins, 2 hrs, 4 hrs, 6 hrs and 8 hrs, and then the MPA580 skin elasticity probe was used to detect the skin elasticity parameter R2. It can be demonstrated from Figure 2 that the skin elasticity is significantly improved after 5 mins of application (P<0.05). The improvement of skin elasticity reaches the highest after 2 hrs of drying compared with before application (P<0.01). Until 8 hrs after application, compared with the skin elasticity before application, the skin elasticity was still significantly improved (P<0.05). Therefore, 10% Immedeline PG can significantly improve skin elasticity, and can maintain at least 8 hrs.

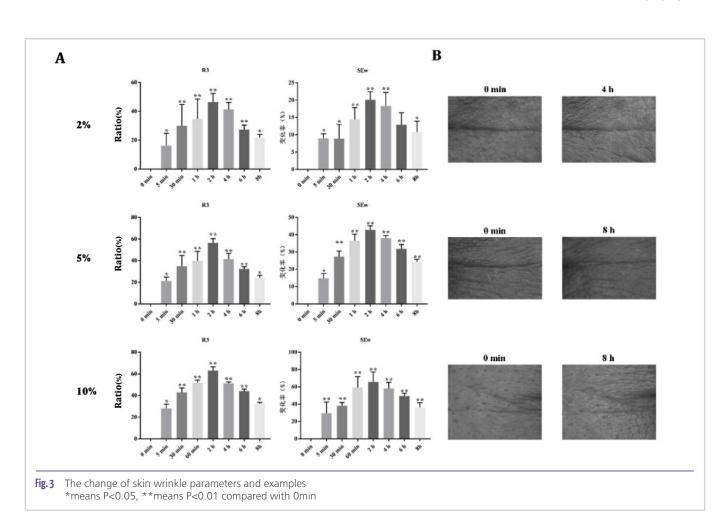
#### Immedeline PG significantly alleviates eye corner wrinkles

2%, 5% and 10% Immedeline PG were applied to the wrinkles at the corners of the eyes and wait for it dried completely for 5 mins, 10 mins, 30 mins, 2 hrs, 4 hrs, 6 hrs and 8 hrs. Then the skin parameters R3 and SEw were detected by VC98. It can be suggested from Figure 3A that the five-segment average R3 of the skin and the change of the skin wrinkle parameter SEw are similar to the change of skin elasticity.



Compared with the change before the application, there is a significant change after 5 mins (P<0.05, P<0.01). The change was highest after 2 hrs (P<0.01), and there was still a significant increase after 8 hrs (P<0.05, P<0.01). With the increase of concentration, the change gradually increased. It can also be demonstrated form Figure 3B that the wrinkles are significantly improved and lightened compared to before application. Therefore, 2%, 5% and 10% Immedeline PG can significantly improve wrinkles, and the higher the concentration, the more obvious the improvement effect.





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The skin is the barrier separating the body from the external environment. In addition to protecting the human body from water loss and microbial infection, it also has an important cosmetic effect. Young and beautiful appearance may have a positive effect on people's social behavior and reproductive status[13]. There is a contradiction between the irreversability of skin aging and people's thirst for eternal young appearance. From ancient to modern times, many efforts were made trying to understand the truth of cutaneous aging and to prevent or even reverse the aging process[14]. As people's demand for cosmetics increases, more research work should continue to fully demonstrate that various raw materials play an important role in the anti-aging process.

In this article, we have verified the positive effects of Immedeline PG on instant wrinkle removal and skin elasticity through experiments. Studies have found that both 5% and 10% Immedeline PG can improve skin firmness. Different concentrations of Immedeline PG can improve the skin elasticity of the cheekbones of the face, and the effect is most obvious at 2 hours, which can last for 8 hours; finally we found that different concentrations of Immedeline PG also can significantly improve eye wrinkles. The improvement effect is consistent with skin elasticity. And as the concentration increases, the improvement effect becomes more obvious. Therefore, peach resin extract can significantly improve skin wrinkles and elasticity.

#### Affiliations

All authors declare no conflict of interest.

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# Heavy Metals in Cosmetic Products

R. Kräling, M. Ritter, U. Leist, A. Wittersheim, P. Drechsel, C.-P. Kramer, B. Meinigke, L. Gehm



Metals are present in various cosmetic products. Metals that are undesirable from a toxicological point of view are of particular interest. They may only be present in cosmetics in technically unavoidable quantities. Reliable analysis of these metals in the low concentration range is therefore essential. In the present study, a proficiency test was performed to determine how comparable the analysis of heavy metals in cosmetic samples can currently be. The results show that the currently valid legal regulations can be monitored and complied to with the current analytical technology.

#### **1. Introduction**

The occurrence of heavy metals in cosmetic products is a continuing discussion topic, also for the Expert Group IX on Analysis of the German Society for Scientific and Applied Cosmetics e.V. (DGK). Heavy metals are expected to be found in decorative cosmetics as those are at least partially coloured with colourants that contain heavy metals. In addition, a large number of tattoo pigments and make-up products such as BB creams contain heavy metals [1]. Sun protection products and antiperspirants may also contain metals as functional ingredients: For example, they may use TiO, or ZnO, which act as UV filters, or aluminium chlorohydrate (ACH) to suppress sweat production. Although these are not strictly heavy metals, the metals mentioned may contain heavy metals as impurities, thereby contaminating the products. Finally, heavy metals can also be found in toothpastes and body lotions. Not least, heavy metals may also be introduced into cosmetic products as impurities of raw ingredients or unintentionally contaminate them as a result of damaged equipment.

Besides the above-mentioned functional metals of TiO<sub>2</sub>, ZnO or ACH, metals may also be present in cosmetic products as colour-providing organic metal complexes or as inorganic pigments [2].

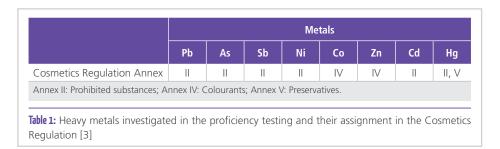
Metals in organic colour complexes used to colour products are usually only found in cosmetics at very low concentrations of < 0.0001% to 0.1%, which makes the determination difficult due to the detection limits.

In contrast, the determination of functional metals such as those in sunscreens, whose contents can range from 1 to 20%, should be analytically easily detectable and quantifiable. In decorative cosmetics such as eye shadows, heavy metals, are present in very high contents of up to 10%, mostly involving inorganic pigments, which should also be analytically detectable without difficulty in these concentrations. Not all metals are of immediate analytical interest, since most of them do not have any side effects. For this reason, the only metals of interest are those:

- that are functional and whose content is relevant for the efficacy of the cosmetic product. They are therefore deliberately introduced into the product.
- metals that are relevant from a toxicological point of view because they may, for example, trigger allergies, mutations or cell changes.

The latter metals are not permitted to be used in cosmetics or their use is limited by a threshold [3, 4]. However, since heavy metals are ubiquitous in the environment, it can be extremely arduous to completely avoid heavy metals in cosmetics, especially those metals that are prohibited. These prohibited metals are therefore subject to a minimisation requirement, which stipulates that these elements may only be present in the products in technically unavoidable quantities [5]. However, this means that precisely the metals specified in Annex II of Regulation (EC) No. 1223/2009 on cosmetic products must be analytically detectable in concentrations that are as low as possible in order to ensure that the product itself does not pose a risk to the consumer.

This is why the **DGK's Expert Group IX on Analysis** decided to push for proficiency testing to determine heavy metals in cosmetics. The aim of this proficiency test was to establish the current state of the art and to be able to make statements on how comparable the detection of heavy metals in cosmetic samples is. Based on the described different relevance of metals in cosmetics, mainly metals with possible toxicological effects should be selected for proficiency testing. As far as possible, the concentration ranges should be defined in such a way that they lie in orders of magnitude which, taking into account the minimization requirement, may still occur in the products. Accordingly, a total of eight different heavy metals were selected for proficiency testing. These are listed in **Table 1**, with details of their assignment in accordance with the Cosmetics Regulation [3].



content

Six of the selected metals belong to the group of prohibited heavy metals. Traces of these prohibited substances may only be present in small quantities if these are technically unavoidable within good manufacturing practice. The samples were doped with these prohibited heavy metals. In addition, cobalt and zinc were also doped. Both are approved for use as a colourant complex or pigment.

#### 2.1 Design of the proficiency test

The planning, organisation and evaluation of the proficiency testing was conducted in accordance with DIN EN ISO/IEC 17043:2010 [6] by the German Reference Office for Proficiency Testing and Reference Materials (Deutsches Referenzbüro für Ringversuche und Referenzmaterialien, DRRR GmbH).

The DGK standard emulsion, an oil-water emulsion, was used as the cosmetic matrix and was produced at the OWL University of Applied Sciences (TH OWL). The undoped oil-water emulsion was the blank sample in the proficiency test, identified as "Sample 1" and is designated RM CP B KOS SME 1. Two further sample materials were also prepared from the provided batch of the DGK standard emulsion: The sample material designated RM CP B KOS SME 2 was doped with the heavy metals specified in **Table 1** in the low concentration range < 1 mg/kg or < 2 mg/kg for zinc. This material was coded as "Sample 2" in the proficiency test. In addition, a sample material was prepared with the above-specified oil-water emulsion and given the designation RM CP B KOS SME 3. This was doped with the heavy metals specified in **Table 1** in the high concentration range > 1 mg/kg. This material was used twice in the proficiency test and coded as "Sample 3" and "Sample 4", both samples contained the same material RM CP B KOS SME 3. This design enabled the laboratories to make a comparative observation of their own laboratory scatter for this sample material.

#### 2.2 Homogeneity testing

The homogeneity test was conducted according to ISO 13528:2015 [7] on 5 representative random samples. If the standard deviation ( $s_{(material)}$ ) of the material homogeneity (HM) is less than 30% of the standard deviation for proficiency assessment (SDPA), then the samples are classified as homogeneous. In-depth stability tests demonstrated and proved the fundamental sample stability. Stability is generally ensured during the proficiency testing period as a result of special manufacturing processes, storage and dispatch conditions as well as regular stability tests.

The materials used in this proficiency test generally fulfilled the homogeneity and stability criteria, with the exception



of the parameter mercury: Mercury was doped together with the other heavy metals, however, after just a few days the analysed concentrations for mercury were significantly lower than expected for the quantity doped. We presume that the homogeneity and stability of the metal in the matrix is not sufficient, which became clear in the homogeneity test. Once the proficiency testing period was over, no mercury could be detected. The homogeneity and stability of the metal in the matrix is therefore not sufficient to perform a statistical evaluation. Various explanations for this are worth considering, which the Expert Group has not been able to prove to date. The DRRR will continue to work with the DGK e.V. to find the cause of the significant lower-than-expected findings.

Metal	ICP-OES	ICP-MS	DIN EN ISO 11885 (ICP-OES)/ 17294-2 (ICP-MS)	Ph Eur.9.0/2.2.58 (ICP-MS)	GFAAS	AFS
Pb	Lab 1	Lab 4 Lab 6 Lab 9 Lab 10	Lab 7 Lab 8	Lab 2	Lab 3	
As	Lab 1	Lab 6 Lab 9 Lab 10	Lab 7 Lab 8	Lab 2	Lab 3	Lab 4
Sb	Lab 1	Lab 4 Lab 6 Lab 7 Lab 9 Lab 10	Lab 8	Lab 2		
Ni	Lab 1	Lab 4 Lab 9 Lab 10	Lab 8	Lab 2	Lab 6	
Co	Lab 1	Lab 4 Lab 9 Lab 10	Lab 7 Lab 8	Lab 2		
Zn	Lab 1 Lab 4	Lab 6 Lab 9 Lab 10	Lab 7 Lab 8	Lab 2		
Cd	Lab 1	Lab 4 Lab 9 Lab 10	Lab 7 Lab 8	Lab 2	Lab 3 Lab 6	

 Table 2: Overview of the different methods of analysis

#### 2.3 Participants

A total of 13 laboratories took part in the proficiency test. Of these, 7 were German laboratories and 6 were international participants, 4 of these were from within the EU. However, not all participants determined all metals in all samples. In the announcement of the proficiency test, the labs were requested to treat the samples as routine samples, which also means that the participants generally investigate only those parameters for which the lab has appropriate methods. Three labs did not submit any results for internal reasons (labs coded with lab codes 11–13).

#### 2.4 Statistical methods

The statistical evaluation of the proficiency test is based on the requirements of ISO 13528:2015 [7]. The proficiency testing data sets were evaluated using four statistical models:

- Sensitive statistics (mean, standard deviation)
- Sensitive statistics with elimination of outliers (according to Grubbs, ISO 5725) and expert outliers (technical outliers)
- Robust statistics (Hampel estimator, Q method)
- Robust statistics (median, MAD (median absolute deviation))

The reason for using four statistical methods is that there is no ideal data set and therefore no ideal statistical evaluation. A  $\chi^2$  goodness-of-fit test [8] is performed to check the quality of the applied statistical methods. The  $\chi^2$ - goodness-offit test is performed for the two sensitive statistics as well as for the robust statistics (Hampel estimator, Q-method). By comparing the different  $\chi^2$  values of the three statistical methods, it is possible to evaluate which of the methods has best identified the normally distributed proportion of the data set. For our purposes, this method helps to evaluate the quality of the three statistical methods. If the  $\chi^2$  value is greater than 7.82 then the data set has not been identified as normally distributed and the statistical method cannot be usefully applied for the relevant data set. If none of the statistical methods are suitable according to the  $\chi^2$  goodnessof-fit test, the robust statistics using median (med) and MAD is used to determine the best estimate as this model does not require a verifiable normal distribution of the data set.

#### 2.5 Summary of the results

The measurement method was chosen by each lab. For instance, various microwave digestion methods were reported. ICP-OES, ICP-MS, graphite furnace AAS and atomic fluorescence spectroscopy (AFS) were used as measurement devices, whereby methods of analysis using inductively coupled plasma were more common (ICP-OES and ICP-MS). **Table 2** shows the different parameter-related methods used by the individual labs.  
 Table 3 summarises the statistical data of the evaluation for the individual metals.

Sample 1 is not listed in this table. As Sample 1 is the undoped oil-water emulsion that was used as the blank sample, the contents of the queried heavy metals are generally below the determination limit of the laboratories, so that no statistical evaluation could be carried out.

#### 3. Discussion

content

The interlaboratory test evaluation has shown that the laboratories quantify the heavy metals comparably within the determined standard deviations. When looking at the submitted results for Sample 1, it is striking that the limits of quantification of the individual labs vary greatly even though they used the same method of analysis. The reported limits of quantification for the individual labs and heavy metals are summarised in **Table 4**.

Metal	Sample	Best estimate (mbest) [mg/kg]	Uncertainty (95.5%)	Standard deviation (sbest)	Number of values for calculation mbest	Number of outliers	χ² value	Statistical method used
Pb	2	0.46	0.07	0.10	10	0	0.48	Robust statistics (Hampel estimator, Q method)
	3+4	1.59	0.13	0.28	20	0	0.33	Sensitive statistics
	2	0.51	0.05	0.07	9	0	0.54	Sensitive statistics
As	3+4	1.75	0.18	0.36	18	0	2.75	Robust statistics (Hampel estimator, Q method)
	2	0.63	0.09	0.11	8	0	0.17	Sensitive statistics
Sb	3+4	2.24	0.28	0.52	16	0	0.70	Robust statistics (Hampel estimator, Q method)
Ni	2	0.39	0.13	0.14	7	0	0.24	Robust statistics (Hampel estimator, Q method)
INI	3+4	1.17	0.20	0.37	16	0	1.02	Robust statistics (Hampel estimator, Q method)
	2	0.30	0.05	0.05	6	0	0.98	Sensitive statistics
Со	3+4	1.10	0.14	0.25	14	0	2.94	Robust statistics (Hampel estimator, Q method)
Zn	2	1.60	0.18	0.17	6	1	0.98	Sensitive statistics with elimination of outliers
	3+4	5.13	0.45	0.78	14	0	3.53	Sensitive statistics
Cd	2	0.48	0.07	0.10	10	0	0.32	Sensitive statistics
Cu	3+4	1.79	0.21	0.45	20	0	0.95	Sensitive statistics

Table 3: Summary of the statistical evaluation of the proficiency testing results

	Pb	As	Sb	Ni	Co	Zn	Cd	Hg
Lab 1	Quant. in BS 0.59 mg/kg	Quant. in BS 0.11 mg/kg	Quant. in BS 0.12 mg/kg	Quant. in BS 0.04 mg/kg	<0.03	Quant. in BS 1.78 mg/kg	Quant. in BS 0.02 mg/kg	Quant. in BS 0.52
Lab 2	<0.0125	<0.0125	<0.0125	Quant. in BS 0.04 mg/kg	Quant. in BS 0.02 mg/kg	<0.0125	<0.0125	<0.0125
Lab 3	<0.3	<0.5	-	-	-	-	<0.02	-
Lab 4	<0.01	<0.05	<0.01	<0.05	<0.01	<0.1	<0.005	Quant. in BS 0.01
Lab 5	<0.05	-	-	-	-	-	<0.02	-
Lab 6	<0.08	<0.02	<0.01	<0.21	-	0.0	0.0	<0.08
Lab 7	<0.1	<0.1	<0.1	<0.1	<0.1	<1	<0.01	<0.01
Lab 8	<0.3	<0.5	<0.5	<0.5	<0.5	<1	Quant. in BS 0.05 mg/kg	Quant in BS 0.05
Lab 9	<0.05	<0.05	<0.05	<0.1	<0.05	<0.1	<0.05	<0.05
Lab 10	<0.2	<0.5	<0.5	<0.2	<0.2	<0.5	<0.05	<0.05

 Table 4: Limit of quantification [mg/kg] (BS= blank sample) violet figures AFS and GFAAS / black ICP-OES; ICP-MS

In some cases, quantifiable amounts of the heavy metal to be detected were found in the blank sample by individual laboratories; this may indicate carryover or contamination of the analytical instrument during the test.

The limits of quantification and detection given by the participating labs are presented here for information. At this point, no further comment can be made on the specified limits of determination and detection, since the working group has no further knowledge, such as the validation data of the individual participants.

In this context, there is an interesting publication of the BVL [9] about the concentrations of heavy metals that are considered technically avoidable in the future **(see Table 5)**.

Instead of technically unavoidable amounts in the finished product, the German Federal Institute for Risk Assessment (BfR) [10] states that the heavy metal contents should be regulated by purity requirements of the starting products, so that correspondingly lower contents are then present in the finished product.

Irrespective of this, the heavy metal contents discussed as being currently unavoidable are in concentration ranges that were covered by the proficiency test and could be determined at a relative standard deviation of reproducibility in the testing of < 30%, or < 36% for nickel. This excludes mercury, as no statistical evaluation could be performed for this parameter within the framework of this proficiency test. The test thereby shows that the currently valid regulations can be monitored and complied to using current analytical technology.

The different limits of detection could be a result of different sample preparations. Not all participants provided this information. For a complex matrix such as an oil-water emulsion, sample preparation is particularly important. The methods "Microwave digestion" and "acidic digestion" were indicated.

In this context, Lab 2 also stands out as it gave a limit of detection of 0.0125 mg/kg for all metals and differed from the other labs, especially for zinc. There, the other detection limit is 0.1–1 mg/kg depending on the lab. However, no clear trend can be identified with regard to which method of analysis produces the lower limit of detection.

Element	General cosmetic product (mg/kg)	Toothpaste (mg/kg)
Pb	2.0 <sup>a)</sup>	0.5
Cd	0.1	0.1
Hg	0.1	0.1
As	0.5 <sup>b)</sup>	0.5
Sb	0.5	0.5

a) For make-up powder, blusher, eye shadow, kajal eyeliner, stage, fantasy and carnival make-up: 5 mg/kg

b) For stage, fantasy and carnival make-up: 2.5 mg/kg

**Table 5:** According to the BVL publication [9], heavy metal contents in cosmetics will be considered technically avoidable in the future if they exceed the following concentrations.

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# Data sheets for the evaluation of the efficacy of active substances in cosmetic products

L. Neumann, B. Fellenberg

Labelling and advertising of cosmetic products are comprehensively regulated in the European Cosmetics Regulation 1223/2009 [1] in conjunction with the "Claims Regulation" VO (EU) 655/2013 [2].

The protection of the consumer from being misled by advertising claims of a cosmetic product is essential in the regulations. Misleading advertising in cosmetics may result from the use of only traces of certain ingredients whose efficacy is generally known to consumers and which are intensively advertised on the product, in the media or on the internet.

The following questions need to be addressed:

- When is such advertising of a supposed active substance in the product dishonest?
- Which substances are perceived by consumers as active substances and can actually give rise to consumer deception?
- At what concentrations can effective concentrations be assumed?

The Cosmetic Products Working Group of the Lebensmittelchemische Gesellschaft (LChG), a sub-society of the Gesellschaft Deutscher Chemiker (GDCh), has been dealing with these questions for about 30 years. Results are data sheets for the evaluation of the efficacy of active substances in cosmetic products.

These data sheets are available free of charge on the website of the GDCh [3].

Individual cosmetic ingredients such as vitamins can have certain functions, for example anti-oxidant efficacy, which is only achieved by these ingredients alone in an overall formulation. If such an ingredient is used in an extremely low concentration and only its indication in the list of ingredients (INCI list) is required by law, a boastful claim of its anti-oxidative efficacy would be a deception of the consumer. The prominent indication of the substance in the product name (substance X cream) or in special prominence on the product (now with substance X) is also a deception, because the efficacy known to be associated with this substance is thereby implied and advertised in a deceptive manner. If, on the other hand, a substance is emphasized in advertising, the concentration of which is low, while the efficacies generally known for this substance are also achieved by other substances in the formulation, a possible deception is less obvious as a result. This is because unlike medicinal products, whose efficacy is usually based on a single active ingredient, the efficacy of cosmetic products is usually the result of the interaction of all substances in the formulation. In order to assess a possible misleading effect, the overall presentation of the product must be taken into account in each case (case-by-case decision).

The aim of research and development by cosmetics manufacturers is to achieve effective overall formulations. The effectiveness of these overall formulations is investigated and documented using a variety of methods. A cosmetics manufacturer does not need to know the exact contribution of individual ingredients to the overall effect. In order not to fall under the suspicion of deceptive advertising of ingredients known for their efficacy, such as vitamins, hydroxy acids or propolis, manufacturers can orientate themselves on the information in the data sheets.

When paying attention to the information in the data sheets, manufacturers should also think about the stability of the ingredients. If one of the ingredients described in the datasheets is used in sufficiently high concentrations during production, but is degraded to a certain extent during storage due to stability problems, a concentration that is too low for efficacy claims may be found when the concentration is checked by control authorities months after production. This can result in a complaint, and this can happen even if the corresponding advertising emphasis is only to be found in the small print on the reverse side. Just like stability, possible antagonistic effects of other components of the formulation that may adversely affect efficacy must also be considered.

To date, data sheets on the following substances have been published by the working group:

- Allantoin •
- Urea
- Honey .
- Hydroxy acids and other organic acids • with comparable effects
- Camomile .
- Panthenol
- Propolis
- Selected proteins •
- Vitamin A and its esters
- Vitamin E
- Vitamin C .
- Niacinamide (Vitamin B3)

Another data sheet provides guidance on claims regarding pH values for cosmetic products.

Each of the data sheets must be considered in conjunction with the general notes on the application of the data sheets. These general notes can also be found on the GDCh website [3].

The data sheets have not only been repeatedly reviewed and revised. New data sheets on additional substances are also compiled and published by the working group through literature searches in scientific publications as well as in information from raw material manufacturers and compilation of the data. Aloe vera is the most recent substance in the collection, for which a data sheet has just been published and posted on the working group's website.

#### LITERATURE

- [1] Regulation (EC) No 1223/2009 of the European Parliament and of the Council of 30 November 2009 on cosmetic products, as amended
- [2] Commission Regulation (EU) No 655/2013 establishing common criteria for substantiating claims relating to cosmetic products of 10 July 2013, as amended
- [3] www.gdch.de/netzwerk-strukturen/fachstrukturen/lebensmittelchemische-gesellschaft/arbeitsgruppen/kosmetische-mittel.html

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# Beauty or environmental protection? Why not both!

Liquid polyurethane film formers are a powerful alternative for the production of more sustainable cosmetics.



The trend towards increased environmental protection and sustainability has long been present in the cosmetics sector. Nowadays, consumers expect cosmetics that are both safe to use and have a reduced impact on the environment.

One term in particular has recently fallen into disrepute: Microplastics. This refers to solid polymer particles that are used in many cosmetic and hygiene products to improve their performance. These non-soluble particles are considered difficult in terms of biodegradability and can thus have a negative impact on the environment.

More sustainable alternatives to microplastics are therefore urgently needed. More and more cosmetics manufacturers are turning to biodegradable polymers to meet the dual demands of performance and increased sustainability for their products. But there are also still major reservations and a lack of knowledge about the biodegradability of polymers.

#### Not all polymers are microplastics

The fact that microplastics are always polymers often leads to the opposite conclusion: All polymers are microplastics and must therefore be classified as harmful to the environment. But is that really the case? The answer is: No! Because besides polymers in solid form, there are also polymers in liquid form. There are also differences in the environmental compatibility of liquid polymers: Some are not biodegradable whereas some are, including liquid polyurethanes (PU) from the Baycusan<sup>®</sup> product family by Covestro. This makes them an interesting alternative for more sustainable cosmetics, both from a manufacturer's and a consumer's perspective.

For most cosmetics manufacturers, doing without polymers altogether is hardly an option. After all, their customers not only want more sustainable products, but also high-performance ones. Baycusan<sup>®</sup> makes an important contribution here with high-performance formulations. This includes polymers such as polyurethane that form a thin and elastic film and protect hair from moisture and heat or ensure water resistance in make-up formulations.



#### It all depends on the chemical structure

From an environmental point of view, however, the decisive moment comes after application. This is because the ingredients of hair styling and other cosmetic products end up with the wastewater in a sewage treatment plant or, in some cases, directly in the natural water cycle. There, liquid polyurethanes bring considerable advantages compared to microplastics: Thanks to their composition, they are ultimately biodegraded in the water by microorganisms to carbon dioxide, water and mineral salts.

The advantage is clear: While microplastics and some liquid polymers accumulate as waste in the oceans, liquid polyurethanes decompose and re-enter the biological cycle. For example, some of the PU film formers from the Baycusan<sup>®</sup> product family achieve average biodegradability rates of up to 82 percent within 28 days according to the OECD 301 standard for easy biodegradability.



Baycusan<sup>®</sup>: polyurethanes for biodegradable and high-performance formulations

It is not surprising that the cosmetics industry is increasingly using biodegradable liquid polymers such as polyurethanes for hair styling products, in make-up or in sun protection products. After all, the substances combine two properties that until now seemed contradictory to many in the industry: improved sustainability and high performance.

Hair care products with Baycusan<sup>®</sup> C 1001 (INCI: Polyurethane-34), Baycusan<sup>®</sup> C 1008, (INCI: Polyurethane-48), and Baycusan<sup>®</sup> eco E 1000 (INCI: Polyurethane-93) achieve curl definition and anti-frizz. The biodegradable polymers leave no residue, are non-sticky and vary in hold from light to strong. For skin care and sun protection as well as make-up products, Baycusan<sup>®</sup> C 1000 (INCI: Polyurethane-34) ensures water resistance and active ingredient boosting.

Biodegradable liquid polymers offer cosmetic manufacturers the opportunity to meet growing consumer demand for more sustainable products without compromising on performance. High-performance and biodegradable cosmetic products for the future!

Learn more about the Baycusan<sup>®</sup> portfolio:



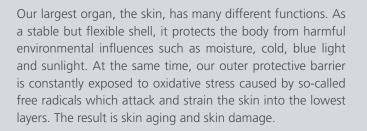
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# Natural astaxanthin: the red diamond among radical scavengers

#### For a natural balance of the skin

Katharina Dokulil



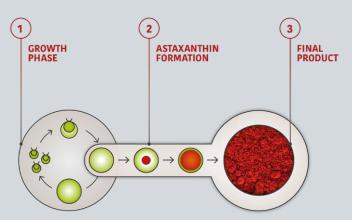
Therefore, the cosmetics industry increasingly relies on the power of antioxidants. By neutralizing reactive oxygen species (ROS) in the body, antioxidants can help reduce oxidative stress. Oxidative stress is considered to be partly responsible for the aging process and associated with the development of diseases and skin changes.

The radical scavengers, thus, protect and support the skin's health and prevent the skin's aging process. The most powerful natural antioxidant is astaxanthin, which belongs to the group of carotenoids. The highest concentration of the active ingredient is obtained from the microalga *Haematococcus pluvialis.* Due to its unique chemical structure and the numerous conjugated double bonds, astaxanthin can fully develop its antioxidant power in all layers of the cell membrane. Hence, for cell protection, astaxanthin is an ideal ingredient in cosmetics. Its naturalness, enormous efficiency and uniqueness make it the "diamond amongst radical scavengers."

Numerous clinical studies have proven the positive effect of natural astaxanthin on the skin's aging process. Due to its hydrophilic and lipophilic properties and its antioxidant power, it provides protection within the lipid bilayer of the skin, reduces wrinkles and pigmentation spots and supports skin health. The power antioxidant from the microalga is 6,000 times more potent than vitamin C and easily outperforms Co-EnzymQ10 and vitamin E. At its self-constructed industrial plant using its own technology, the Austrian company BDI-BioLife Science produces natural astaxanthin of the highest quality. Production under the highest qualitative standards is certified by ISO9001:2018 and FSSC 22000. In a complex, self-contained cultivation process, the natural active ingredient is gained in two phases from the microalga *Haematococcus pluvialis* and then harvested. In addition to the highest product quality, sustainability of the used resources is always ensured.

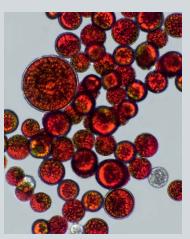
The two-stage cultivation process is divided into:

- **Growth phase:** in this phase, an ideal supply of light, CO<sub>2</sub> and nutrients guarantees optimal conditions for the cultivation of the algae – they grow magnificently, while biomass is built up.
- Astaxanthin production: When enough biomass has been produced, the alga's living conditions (nutrient deprivation, light intensity etc.) are massively altered, causing stress in the alga. Hence, it activates a unique survival mechanism, carbonizes and starts to store astaxanthin in the liposomes.



In a complex harvesting process, the algae biomass with high astaxanthin content is harvested and gently processed. The fat-soluble  $CO_2$  extract obtained from the raw biomass by  $CO_2$  extraction forms the basis for the cosmetic active ingredient AstaCos<sup>®</sup> OL50. With the ingredient AstaCos<sup>®</sup> OL50, which

was specially developed for the cosmetics industry, BDI-BioLife Science meets the zeitgeist of ecologically sustainably produced cosmetics and produces the ideal active ingredient for the entire industry up to natural cosmetics. The oleoresin, based on certified organic jojoba oil and with an astaxanthin content of 5%, acts as a biological



cell protection in cosmetic formulations and keeps the skin in oxidative balance. The vegan sustainably produced active ingredient was certified with the renowned Cosmos standard in 2021.

The positive effects of the high-quality AstaCos<sup>®</sup> OL50 on the skin have been conducted, verified and confirmed in numerous *in-vivo* and *in-vitro* studies by renowned institutes and medical universities. AstaCos<sup>®</sup> OL50 is the new trend ingredient in natural cosmetics and satisfies customer demands in numerous formulations and applications.



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Ingredients	%
REWOPOL® TS 35	30.0
TEGO Sorb <sup>®</sup> Conc. 50	3.0
Triethanolamine	0.5
Lactic acid, 88%	0.5
Phosphonic acid (DTPMP), 32%	1.0
Water	65.0
Manufacturing: Blend ingredients in the given order and stir.	

# Gloss up your bath!

# High Gloss Bathroom Cleaner

Ingredients	%
Water	92.3
TEGO <sup>®</sup> PP 1027	1.0
GLDA, 40%	0.5
REWOPOL <sup>®</sup> SC 200	2.0
REWOPOL® D 510 NC	2.0
NaOH, 50%	0.9
Citric acid monohydrate	1.3

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Anti-fogging efficiency also for wet wipes

Ingredients	%			
Water	84.4			
TEGOTENS® DO	0.2			
TEGOPREN <sup>®</sup> 5840	0.8			
Isopropanol	15.0			
Preservative	qs			
Manufacturing: Blend ingredients in the given order while stirring. pH: 9.0				



# Grill Monster Concentrate for Barbecue Season

Clean your grill with the new benchmark for heavy duty cleaning

Ingredients	%			
Water	78.0			
REWOQUAT <sup>®</sup> CQ 200	12.0			
Sodium metasilicate	2.0			
Tetrasodium Glutamate Diacetate (GLDA), 40%	8.0			
Manufacturing: Blend ingredients in the given order while stirring until clear. pH: 12.8 Dilute: 1:10 REWOQUAT® CQ 200 for Powerful Heavy Duty Cleaning and Best in Class Re- sults!				

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Ingredients	%
Water	91.8
TEGOTENS <sup>®</sup> DO	6.0
Triethanolamine	0.6
TEGO Sorb <sup>®</sup> Conc. 50	1.0
Lacid acid	0.6
Preservative	qs

Manufacturing:

Blend ingredients in the given order while stirring. Add lactic acid to adjust the pH value to pH 7. Temporarily the solution appears cloudy. Stir until clear.

#### Disclaimer:

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# Sanitizing Hand Gel

### with PURAC<sup>®</sup> Sanilac 80

Phase	Ingredients	INCI	Function	wt %
Α	Natrosol <sup>™</sup> 250 HHR¹	Hydroxyethylcellulose	Thickener	0.7
	Propylene glycol <sup>2</sup>	Propylene glycol	Flow regulator	1.00
	Glycerin	Glycerin	Humectant	2.00
В	Demi Water	Water		q.s. to 100
С	Ethanol 99+%	Ethanol Absolut 99+%	Antibacterial active & solvent	40
	PURAC <sup>®</sup> Sanilac 80 <sup>3</sup>	Lactic Acid (and) Water	Antibacterial active	1.88
	pH adjuster	pH adjuster	pH regulator	q.s. to pH 3.5

Suppliers:

Ashland | <sup>2</sup> Acros | <sup>3</sup> Corbion

Antibacterial hand soap I Cleaners

Other applications with PURAC<sup>®</sup> Sanilac:

Appearance: transparent, low viscous liquid

Viscosity Brookfield (100s-1): 1.1 - 1.5 mPa·s

Stable for 2 months at RT, 5°C (41°F)

pH Value: 3.0

and 40°C (104°F)

Stability:

#### Manufacturing procedure:

Mix the ingredients of part A and premix until a homogenous mixture is obtained. Add part A to part B and mix by using an overhead stirrer at 500-600 RPM or higher if necessary. When fully mixed and a gel is obtained, add part C slowly under stirring. Adjust the pH if necessary to pH 3.5-4.0.

#### **Product characteristics:**

Appearance: Transparent Liquid Gel Viscosity: 3000 cP, (22.0°C; Brookfield Model DV-I; 50 RPM; Spindle 2)

pH: 3.5-4.0

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# Multipurpose cleaner

### with Lactic Acid and Citric Acid for superior descaling

# Jungbunzlauer

From nature to ingredients.

Phase	Ingredients	INCI	Supplier	Quality
А	Water	Aqua		Qs to 100%
В	Texapon <sup>®</sup> LS30	Sodium Lauryl Sulfate	BASF	6.70%
	Texapon® N70	Sodium Laureth Sulfate	BASF	2.90%
	Plantacare <sup>®</sup> 2000 UP	Decyl Glucoside	BASF	4.00%
С	Lactic Acid 90%	Lactic Acid	Jungbunzlauer	3.20%
	Citric Acid	Citric Acid	Jungbunzlauer	2.90%
D	Perfume, Colour			Qs
E	NaOH 30%	Sodium Hydroxide	Merck	Qs
Directions:		·	Technical Data:	

#### Directions:

1 Dissolve phase B into water (phase A), a propeller stirrer is suggested

2 Add phase C and mix at room temperature until homogeneous

3 Add phase D and stir well until product is homogeneous

4 Set pH value with phase E

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The information contained herein is meant to demonstrate how our products can be used. This formulation has been subjected to limited stabilitytests and has been shown to perform well. The given data are suggestions without any guarantee aimed to support customers' development.





# An eco-friendly, biodegradable liquid dish soap with CELLULON<sup>®</sup> Cellulose Liquid



Compatible with even the most concentrated formulation, CELLULON<sup>®</sup> Cellulose Liquid is a unique and readily biodegradable cellulose that stabilizes components with minimal impact on viscosity and dispersibility. It comes fully activated, ready to use, and is not sensitive to temperature or pH.

Order of Addition	Ingredients	% by Weight
А	De-ionized water	QS to 100
	CALFOAM <sup>®</sup> SLS 95 <sup>1</sup>	17
В	AMMONYX <sup>®</sup> LO SPECIAL <sup>2</sup>	13
	GLUCOPON® 625 UP <sup>3</sup>	2.2
	Glycerin 99%	5.8
С	Citric Acid	0.12
	TROYGUARD B20F <sup>4</sup>	0.10
	Fragrance	0.30
	Microsphere beads	0.10-0.30
	Dye (optional)	QS
D	CELLULON® Cellulose Liquid	4.0

#### Suppliers and trademark owners:

<sup>1</sup> Pilot Chemical Holdings, Inc., <sup>2</sup> Stepan Company, <sup>3</sup> BASF Company, <sup>4</sup> Troy Technology Corporation, Inc.

#### Specifications:

pH: 8.0 – 9.0 Viscosity 25C (LV-2 @60 rpm): 800 – 1000 mPa.s. Yield Stress Value: 1.7 Pa

#### Procedure:

- Heat de-ionized water to 65 70°C
- Add CALFOAM® SLS 95 and mix for at least 10 minutes until completely dissolved
- Cool to 35 40°C and add Step B ingredients
  Mix for 5 minutes using propeller mixer. Cool to 25°C and add ingredients in Step C
- Mix for 5 minutes
- Add CELLULON® Cellulose Liquid from Step D. Mix for 20 -30 min. at 500-700 rpm with propeller mixer. Try not to aerate the product.

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# Love & tenderness soft cream SC-FR-20-BC-50681-09

Phase	Ingredients	INCI	Function	% by weight
A Water, demin.		Aqua		74.25
	Preservative		Preservative	q.s.
	Hispagel <sup>®</sup> 200 NS	Glycerin, Glyceryl Polyacrylate	Rheology modifier	10.00
	1,3-Butanediol	Butylene Glycol	Emollient	5.00
	Edeta <sup>®</sup> BD	Disodium EDTA	Complexing agent	0.05
В	Cetiol <sup>®</sup> CC	Dicaprylyl Carbonate	Emollient	4.00
	Cetiol <sup>®</sup> Sensoft	Propylheptyl Caprylate	Emollient	4.00
	Cosmedia <sup>®</sup> SP	Sodium Polyacrylate	Rheology modifier	0.70
С	Sacred Patch™ BC10022	Aqua, Glycerin, Pentylene Glycol, Algin, Caprylyl Glycol, Glyceryl Polyacrylate, Sodium Hyaluronate, Pullulan, Aphanothece Sacrum Polysaccharides	Active ingredient	2.00

#### Specifications:

Performance:

pH value (23°C) 5.8 Viscosity (Brookfield; RVT; spindle TD, Helipath; 5 rpm; 23°C) 62 000 mPa s

#### Manufacturing Process:

1- Mix ingredients of A homogeneously at room temperature.

2- Add Phase B into A while stirring.3- When A+B is homogeneous, add Phase C while stirring.

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

Additional performance has not been evaluated

# Silky Moisturizing Hair Vial HB-IT-19-001-4-1

Phase	Ingredients	INCI	Function	% by weight
Α	Water, demin.	Aqua		76.32
	Rheocare <sup>®</sup> HSP-1180	Polyacrylamidomethylpropane Sulfonic Acid	Rheology modifier	12.50
	D-Panthenol 75 W	Panthenol	Active ingredient	0.50
	Glycerin	Glycerin	Humectant	3.00
	Sodium Hydroxide (10% solution)	Sodium Hydroxide	pH Adjustment	3.08
	Sodium Benzoate	Sodium Benzoate	Preservative	0.50
В	Eumulgin <sup>®</sup> CO 40	PEG-40 Hydrogenated Castor Oil	Solubilizer	1.50
	Copherol® 1250 C	Tocopheryl Acetate	Active ingredient	0.50
	Perfume	Parfum	Fragrance	0.10
С	PatcH2O™ A00297	Aqua, Glycerin, Glyceryl Polyacrylate, Trehalose, Urea, Serine, Pentylene Glycol, Algin, Caprylyl Glycol, Sodium Hyaluronate, Pullulan, Disodium Phosphate, Potassium Phosphate	Active ingredient	2.00
D	Citric Acid (50% solution)	Citric Acid	pH Adjustment	qs

**Specifications:** pH value (23°C) 4.90 Appearance-Opaque, slightly blue

#### Manufacturing Process:

Performance: Additional performance has not been evaluated External Suppliers: Perfume: Care 754743 - Symrise Dissolve Rheocare HSP 1180 in water and adjust pH to ~6 with sodium hydroxide. Add other components of phase A. Dissolve phase B seperatly and add to phase A. Stir until homogeneous. Add phase C under stirring and stir until homogeneous Adjust pH with citric acid <5.

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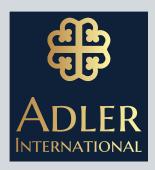
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# Persian Rose Oil *(Rosa Damascena)* – Millennia of history

Interview with Dr. Alireza Azimzadeh, Managing Director, Adler International GmbH



#### Dr. Azimzadeh, you are the founder of Adler International GmbH, whose aim is to present high-quality essential oils, but especially Persian rose oil, internationally with a new approach.

How did this goal come about and what drives you?

Persia is the origin of rose cultivation. Later, in the 17<sup>th</sup> century, roses were also cultivated in India, North Africa and Turkey. In Europe, rose cultivation first developed at the beginning of the 18<sup>th</sup> century in Bulgaria, which at that time was part of the Ottoman Empire.

Among the many oils in Persia, rose oil and rose water have always had a special place. This tradition was passed on and established in all Islamic countries, especially after the Islamization of Iran, but the Persian scent of the rose, just like the Persian carpet, always remained the secret number one among connoisseurs.

Among other things, it is thanks to the special ecosystem, the cultivation location and the special sunlight on the edge of the desert that Persian rose oil has this unique scent and the special greenish colour. Characteristics that have always been decisive for all lovers of rose products. This even goes so far that traditionally, still, the holiest site of the Muslims - the Kabaa in Mecca - has to be sprayed annually only with Persian rose water from Kashan, which is self-explanatory.

In Iran itself, rose oil is now cultivated in almost all parts of the country (Kashan, Shiraz, Kerman etc.), but the Kashan region is and remains the region with the most exotic and breathtaking rose oil scents.

Unfortunately, at some point, the age-old tradition of rose oil production, which was passed down from father to son, was neglected to be updated rapidly enough, so that Persian rose oil no longer plays the leading role in the present, but only a very tiny one. Therefore, from Hamburg, one of the most traditional trading metropoles in the world, we decided to tackle the initiated goal of reorientation even more professionally and later to include special oils from other parts of the world in the assortment.

# How did Persian Rose Oil experience the decline on the international trading platform?

Well, somewhere in the 20<sup>th</sup> century, with a growing interest in high quality oils, came a new trade and manufacturing culture. The traditionally produced rose oil, which no longer met the higher demands and not least the new criteria of the companies, could not meet the new conditions of the cosmetics manufacturers and their customers, who were looking for even more purity and better, consistent quality. On top of that was the failure of the Iranian producers, who were mostly farmers of the respective region and whose inherited business was mostly a secure rose water trade, and who did not feel compelled to adapt to the new international standard criteria and conditions, or even to think about export and world trade, due to the lack of facilitating government regulations.

These are just some of the most important parameters, which is why steam distillation only gained a foothold in Iran much later than in other countries and why Iran only became internationally known very late and in an unsystematic way.

# You have brought rose water into focus. What role does rose oil production play in Iran today?

That is correct. One of the reasons why Iran is very sluggish in the international arena for rose oil is without question the great domestic demand for rose water. This is an important part of the food and natural healing culture not only in Iran, but also in the neighbouring countries of the Persian Gulf. Therefore, they do not feel compelled to switch to oil production, but consider rose oil only as a by-product that has buyers from time to time. This culture is now gradually taking a turn towards rose oil and is part of our commitment.



# What role does your company play in the international placement of Persian Rose Oil and other oils?

Adler International GmbH in Hamburg has set itself the goal as a German company and member of SEPAWA e.V. to address the above-mentioned weaknesses in its international presence. The selection of suppliers and permanent control of the few Iranian producers in question, their training and constant communication on the ongoing needs and changes of the world market, but last but not least to be the interface for professional marketing, sales and above all logistics, is our main task.

A major concern of international buyers, especially when purchasing rose oil from Persia, was and still is the inconsistency in quality at higher purchase quantities, the failure to guarantee the agreed purchase quantities, contractual concerns, but also logistical difficulties. We have eliminated all this and much more with hard work and persistence and are trying to build trust.

The aim is, among other things, to awaken the dormant or sluggish private industry and at the same time to be a focal point in the heart of Europe to ensure quality and communication and try to fill the very large gap between the production culture in the country and the international buyers in an increasingly diversifying and competitive world.

#### What other oils do you provide?

The culture of oils and herbal potions has always been an integral part of Persian culture and has spread from there to a great many countries over the centuries. This culture reached its peak at the time of the Persian scientist and physician "Avicenna", who compiled and strongly emphasised these aspects in his books, which were taught for centuries at universities all over the world.

Based on this culture, we had planned to start with the oils and extracts that are specific to Persia and subsequently offer other interesting oils with potential on an international level. We have the possibility of 25 different oils so far and the trend is growing (see http://adler-international.com/handel-2/).

However, rose oil, which we started with more than 25 years ago, is leading the way. But also saffron oil, galbanum oil, lavender oil, etc. are available.

We offer the rose oil both organically certified and in 100% pure quality. The 100% pure rose oil is itself offered in three different variations:

- 1) From 1<sup>st</sup> distillation
- 2) From 2<sup>nd</sup> distillation
- 3) Mixture of 1<sup>st</sup> and 2<sup>nd</sup> distillation

We hope that with the support of partners and loyal customers we can realise these goals, while contributing to the sustainable development and the increase of cultivation and standards in different growing regions of the world.

#### www.adler-international.com



# Interview with Amber Yarnell

Ph.D., R&D Applications Scientist, LANXESS



#### Sustainability has a growing importance for home care and consumer products. What does "sustainability" mean to the everyday consumer?

Most people can agree that sustainability is important and plays a role in the products we purchase and use in our homes every day. We want to protect our health, safety, and environment, and these core values drive consumer buying habits. This is especially true in 2021, with new United Nations scientific findings being released about climate change and a global pandemic drastically increasing usage and exposures to cleaning products. People are more conscious of sustainability than ever before.

For consumers, there are two main ways of thinking about sustainability. One is to evaluate the specific product on the shelf. What claims or certifications are present on the label? Is the formulation readily biodegradable? Does the packaging reduce waste? Does the product contain any concerning ingredients, such as allergens, environmental hazards, or bio-accumulative materials? Is it safe to wash down the drain?

The other way consumers think about sustainability is by repeatedly purchasing from brands that they trust. Does the manufacturer promote consistent and specific objectives for sustainability? Do they ethically source their ingredients? Can they certify that their operations meet global industry benchmarks for sustainability? Do they rigorously test their products? All of this translates to a lot of complexity for manufacturers, who have to navigate a wide variety of consumer needs. However, by consistently adopting more sustainable practices and ingredients, they can make their product labels and brand ethos more attractive.

# What is an important factor for formulators to receive green label certification?

Ingredients are one of the most important considerations that impact whether a product can be certified to meet green label standards. For example, Ecolabel and Ecocert restrict ingredients that are believed to represent specific hazards. The manufacturer must work with their suppliers and be able to demonstrate that their raw materials are sustainably sourced. In addition, the certifying body will look at overall product design and eco-friendly or recyclable packaging. To achieve these goals, manufacturers should consider which green certifications they would like to attain before beginning formulation.

# Why are effectively preserved products essential to sustainability?

If a product is not effectively preserved, it greatly reduces product lifespan. Sustainable cleaning products often have a high concentration of water and naturally derived ingredients. These ingredients could support microbial activity as they provide an attractive environment for the growth of fungi and bacteria. Such microorganisms can threaten consumer health, reduce product effectiveness, or impact product aesthetics like color, odor, viscosity, or appearance.

To avoid contamination without preservatives, products would need energy-intensive solutions to prevent contamination, such as refrigeration. Even in carefully controlled, refrigerated conditions, the product may only last weeks before spoiling instead of months or years. Then, the unused portion of the spoiled product would need to be disposed, wastefully sending materials down the drain or to the landfill.

In contrast, properly preserved products can last for much longer, so they can be fully used and minimize waste. They can be stored and transported easily in a wide variety of environments, even hot or humid areas where microbes thrive more readily. Proper preservation means that preservative concentration is optimized – the levels are limited to minimize hazards, while also using enough to protect the product, the consumer and the environment.





Biodegradability is an important quality for all ingredients used in home care products. What is your solution for sustainable preservation without negative impact on the environment?

There are multiple levels of ingredient biodegradability, which is determined by the rate of degradation and the conditions required. At the top of the spectrum, readily biodegradable materials can be easily broken down by microorganisms that are present in the environment, without special conditions. Ready biodegradability it the most sustainable, since it means that the ingredients may have less environmental impact. Organic acids are increasingly preferred in consumer products due to their ability to readily biodegrade. Of the chemistries in this class, sodium benzoate is the most attractive option, as it is frequently cited as a reference material in testing guidelines. This is due to its capacity to readily and completely degrade in a variety of environments. High quality sodium benzoate is necessary for consumer applications. It is available for personal care as Purox<sup>®</sup> S, and for BPR applications such as household care, it is available as Kalaguard<sup>®</sup> SB.

Some materials require more heat, light, oxygen, time, or pre-acclimated microorganisms to break down. These materials are considered inherently biodegradable, as long as they will eventually degrade. If they aren't expected to break down fully or partially, they are considered persistent, and they may accumulate in the environment or even inside the human body. Persistent chemistries should be avoided whenever possible.

# How difficult is reformulating for sustainability & biodegradability?

Many of our customers come to us for guidance on how to create more sustainable, well preserved, higher performing products. Our applications lab is very focused on this, as it can be quite challenging to design formulations that meet all of the consumer's needs for sustainability, while also being effective and easy to use. The best time to start thinking about sustainability is at the beginning of the formulation process, so the manufacturer can carefully select ingredients that meet the requirements. For example, if an Ecolabel certification is desired, you can design the product around those needs. In some cases, it may be possible to tweak existing formulas to eliminate certain ingredients, but the manufacturer will need to understand how that omission or substitution could impact the product. For example, does the pH need to be adjusted? Is there any change in product performance? Could new ingredients increase the needed preservative concentration, which could be the case for ingredients with high bioburden? Are there multifunctionals that could do the job of multiple ingredients?

#### Recently, LANXESS completed the acquisition of Emerald Kalama Chemical. How will this affect your business sector?

As a global chemical company, LANXESS has substantial business in the preservation and stabilization field, not only in the food and beverage industry but also in the personal and home care industries. With the completed acquisition of Emerald, LANXESS has formed a new business unit under the Consumer Protection segment called Flavors & Fragrances. This new business unit will be focused on delivering sustainable, high quality ingredients used in consumer products, such as home care, personal care, foods, pharmaceuticals, feed, and agriculture. Together Emerald Kalama and LANXESS can offer a broader portfolio to their customers.

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# MossCellTec<sup>™</sup> Aloe Harmonizes the skin's moisture flow

MossCellTec<sup>™</sup> Aloe is an unequaled aloe-moss extract sustainably obtained through our MossCellTec<sup>™</sup> technology. Thanks to an intensified connexin-mediated cell-to-cell communication, MossCellTec<sup>™</sup> Aloe optimally evens the moisture distribution in the skin and reduces the volume and depth of wrinkles.

- Improves hydration evenness
- Activates cell-to-cell communication
- Fades away signs of aging
- Comforts dry skin

SWISS QUALITY PRODUCT

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