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The Functional Advantages of Natural Waxes in Traditional Soaps

A. G. McMahon, B. M. Lemieux

abstract

Humans have been manufacturing soap since at least 2800 B.C., modifying and perfecting their recipes over the centuries. Today's traditional soap bars are made via saponification of triglycerides: the alkaline hydrolysis of fatty ester bonds, leading to mainly C16-C18 fatty acid soaps and glycerol. In this paper, we explore the use of Natural Waxes from Koster Keunen, Inc. as starting raw materials in soap formulations, both alone and as additives to traditional triglycerides. Twelve Natural Waxes were blended with olive oil at 50/50 ratios, each blend was fully saponified, and the reaction products were evaluated for different properties and compared to a standard olive oil soap bar. It was determined through experimentation that each Saponified Natural Wax or Saponified Natural Wax Blend made a chemically complex finished soap, with different properties from the control and from each other. Some of the benefits encountered included improved bar hardness, a longer lifespan, more hydrophobicity, and innovative INCI declarations.

Introduction and Background

Few personal care products can have their history traced as far back as soap. While the historical accounts on soap and soapmaking are rife with legend, experts agree that the earliest evidence dates back to 2800 BC in Ancient Babylon, when a soap-like substance was discovered during an archaeological dig [1].

Other records show ancient Egyptians, Greeks and Romans also made and used soap for thousands of years, perfecting the technique over time. By 1100 AD, soap making was an established practice in Mediterranean countries with easy access to olive oil, a key ingredient in Castille Soap, which was widely traded at the time [2].

Soap was introduced to the Americas in the 17th century, when soap makers arrived in Jamestown, VA [1]. Over the next 200 years, as the United States industrialized, soapmaking evolved into one of the fastest growing businesses, with P&G's Ivory being one of the first to gain national distribution [3]. However, food and fat shortages during World War I led German engineers to introduce synthetic replacements for soap, now known as detergents [4]. American consumers quickly embraced detergents due to their efficiency, availability, and low price; moving traditional soaps over time to niche market segments, particularly "Indie" brands, artisanal soap makers, and crafters [5].

In recent years, and specifically at the onset of the COVID-19 Pandemic, the demand for hand soap has escalated. In fact, the global hand soap market is forecasted to grow by 6.7% from 2020 to 2030, with the household segment accounting for over 70% of the market share [6]. Although it is not clear what percentage of said growth refers to traditional soap bars (versus "syndet" bars or liquid detergents), current consumer trends, such as natural ingredient demand, small business support, and plastic reduction seem to support the persistence of traditional soaps [7,8].

Soap Chemistry and The Role of Waxes

All soaps have in common one core recipe:
Lipid + Alkali = Soap + By-product.

The chemical reaction that takes place is called saponification and can be defined as the alkaline hydrolysis of fatty ester bonds, resulting in fatty acid salts -the soaps- and a by-product (typically glycerol) [9].

Most commercial soaps (and also DIY versions) are variations of "tried and true" recipes, based on well-known saponification reactions of well-known lipids, like tallow or palm oil [10], with the only variations being in fragrance, color, and claim ingredients. This work is meant to encourage chemists and soap makers looking for innovation to vary the lipid source. This can be done by trying new ingredients, trying new combinations of known ingredients, or varying the ratios of these combinations. This will lead to new and innovative soap products, as well as by-products (often overlooked but important as well), which will affect the product performance, sensory aspects, and ingredient listing.

The Lipid Source

Lipids are a diverse group of organic compounds, characterized by their hydrophobicity and their biological importance.

They present either as linear alkyl chains, saturated or with unsaturations, or as isoprene units in varying structures. These molecules may contain oxygenated substituents, such as carboxylic acids or hydroxyl groups [11].

Traditional soapmaking would not be possible without a specific class of lipids, known as fatty acids. Fatty acids are linear alkyl chains, typically C12 - C22, saturated or with unsaturations, and a terminal carboxyl group [12]. In nature, fatty acids are typically found linked by ester bonds to glycerol, called triacylglycerols or triglycerides [11]. Due to this richness in fatty acids and widespread availability, triglycerides are the most prevalent starting raw materials in soapmaking. Some examples of lipid sources rich in triglycerides are tallow, palm oil, or olive oil [13].

Waxes as Lipids

Waxes are a class of lipids with varying definitions and classification options. In a strict sense, waxes are comprised of long chain monoesters: long chain fatty acids esterified onto long chain alcohols with similar chain lengths, mainly saturated. A more practical description of waxes should include that they are blends of many components, including monoesters, hydrocarbons, sterol esters, and fatty alcohols [14].

Many natural waxes are biosynthesized by living organisms to provide a protective barrier against environmental stresses. Due to their protective nature, waxes are widely used in personal care and OTC drugs to provide a barrier to human skin, and are staple ingredients in lip balms, hand salves, and body creams [15]. In this paper, we explore the use of natural waxes as starting materials for traditional soaps.

Waxes Make Complex Soaps

Traditional lipid sources used in soap making, like animal fats (very high triglyceride content, usually centered around C16 - C18 saturated fatty acids) or plant oils (very high triglyceride content, usually centered around C18 unsaturated fatty acids) [13], will yield almost exclusively combinations of saturated and unsaturated C16 –C18 fatty acid soaps, and glycerol as a by-product. While these small differences in carbon number and degree of unsaturation can translate to subtle differences in a finished soap bar, the chemical richness and complexity of each natural wax will create soap bars with vastly different chemical compositions.

In addition to the C16-C18 soaps and glycerol described above, waxes can also yield very high molecular weight soaps (from C24 up to C38) and a broad molecular weight range

of fatty alcohols as reaction by-products, while keeping their unsaponifiable materials intact. These higher molecular weight soaps are more hydrophobic than their traditional counterparts, resulting in harder finished bars with a longer lifespan. In addition, both the newly generated higher molecular weight fatty alcohols, as well as the existing unsaponifiables will also contribute to the overall hydrophobicity of the soap bar, improving the film –forming and skin protecting qualities of the bar [15].

The reaction products obtained from the saponification of each major natural wax component are summarized in **Table 1**.

Major Natural Wax Component	Saponifiable?	By-Product	Natural Wax Example
Monoesters	Yes	Fatty alcohols	Sunflower Wax, Rice Bran Wax
Hydrocarbons	No	N/A	Candelilla Wax
Fatty alcohols	No	N/A	Orange Wax
Free fatty acids	Yes	Water (likely evaporates)	Beeswax
Triglycerides	Yes	Glycerol	Bayberry Wax, Cocoa Butter
Mono- and Diglycerides	Yes	Glycerol	
Triterpenes / Derivatives	Possibly	Various	
Other	Possibly	Various	

Table 1: Saponifiable Materials Found in Natural Waxes.

Objective

The objectives of this paper are as follows:

- Saponify multiple natural waxes and determine the properties of the reaction products.
- Determine the effects of adding different percentages of natural waxes to traditional soap recipes.

Experimental

Materials

The following materials were used to conduct this experiment:

- Natural Waxes as described in **Table 2**, all provided by Koster Keunen, Inc.
- Olive oil (INCI: Olea Europaea (Olive) Fruit Oil) as the control and standard, provided by Columbus Vegetable Oils.
- Sodium hydroxide, purchased from Fischer Scientific, from which a 30% solution was prepared for use in all the saponification reactions.

Wax Name	INCI Name	Source	Broad Chemical Composition	Average Sap Value (mg NaOH/g)	Melt Point (°C)	Appearance	State at Room Temperature
Bayberry Wax	Myrica Cerifera (Bayberry) Fruit Wax	Bayberry Fruit	Triglycerides, C16	155	40-55	Light Brown to Olive Green	Solid
Cocoa Butter	Theobroma Cacao (Cocoa) Seed Butter	Cocoa Beans	Triglycerides, C16, C18:0, C18:1	138	29-35	White to Off White	Solid
Soy Wax	Hydrogenated Soybean Oil	Crude Soybean Oil	Triglycerides, C18	135	62-72	White to Off White	Solid
Castor Wax	Hydrogenated Castor Oil	Crude Castor Oil	Triglycerides, C18:OH	128	85-89	Off White	Solid
Kester Wax K-24	Lauryl Laurate	Crude Coconut and Palm Oils	Low Molecular Weight Monoesters	104	23-30	Off White to Light Yellow	Liquid
Kester Wax K-48	Cetyl Palmitate	Crude Coconut and Palm Oils	Medium Molecular Weight Monoesters	104	45-53	White to Off White	Solid
Orange Wax	Citrus Aurantanium Dulcis (Orange) Wax	Orange Peels	Fatty Acids, Phytosterols, Fatty Alcohols	73	35-45	Orange to Brown	Liquid
Rice Bran Wax	Oryza Sativa (Rice) Bran Wax	Crude Rice Bran Oil	High Molecular Weight Monoesters	70	77-82	Yellow to Light Brown	Solid
Beeswax	Beeswax	Bee Secretion	Esters, Hydrocarbons, Free Fatty Acids / Alcohols	66	62-65	White to Yellow	Solid
Sunflower Wax	Helianthus Annuus (Sunflower) Seed Wax	Crude Sunflower Oil	High Molecular Weight Monoesters	63	74-77	Light Yellow	Solid
Carnauba Wax	Copernicia Cerifera (Carnauba) Wax	Carnauba Palm Leaves	Esters, Free Fatty Acids, Hydrocarbons	62	80-86	Dull Yellow to Light Brown	Solid
Candelilla Wax	Euphorbia Cerifera (Candelilla) Wax	Candelilla Shrub Leaves	Hydrocarbons, Esters, Free Fatty Acids, Resins	43	68-73	Yellow to Brown	Solid

Table 2: Chemical and Physical Properties of Natural Waxes.

Methods and Procedures

- Saponification values for each Natural Wax in **Table 2** were either obtained from the specification sheet provided by Koster Keunen, Inc. or calculated using the USP 401 Method [16]. The saponification values were then used to determine the amount of the 30% sodium hydroxide solution required for each saponification reaction.
- The saponification reactions were carried out as follows:
  - Each Wax or Wax/olive oil blend was heated to approximately 80°C as **Phase A** and allowed to cool below 60°C.
  - The appropriate amount of 30% Sodium hydroxide solution was also heated to 60°C as **Phase B**.
  - **Phase B** was added to **Phase A** with sufficient mixing using a propeller mixer, while maintaining the temperature at 60°C for approximately 25 minutes. The saponified blend was then immediately poured into a silicone mold and allowed to cure for 48 hours.
  - After the 48-hour cure time, a pH testing at or below 10 confirmed the reaction was complete.

Experimentation

Each Natural Wax, as well as the olive oil control, was saponified individually (100%) and the reaction products were evaluated. In some cases, Natural Waxes did not make a commercially acceptable soap at 100% usage, as the bars were composed of a high level of unsaponifiables. To continue the experiment, each Natural Wax was blended with olive oil at 50% ratios, saponified, and the reaction products were evaluated and compared to 100% olive oil (control). We will refer to these blends as "saponified blends" for the remainder of this paper, and each one was evaluated within the following categories:

Quantitative Evaluation: Effect on Bar Rigidity.

Penetration values were measured on a Koehler K19500 penetrometer. The penetration of 100% saponified olive oil was recorded at 57 dmm (needle, 50g). The saponified blends measuring below 57 dmm proved to harden the bar, increasing bar rigidity, while the saponified blends measuring above 57 dmm proved to soften the bar, decreasing bar rigidity.

Tactile Evaluation: Effect on Bar Cleansing Properties and Lather

In a sensory panel performed internally (N=20); individuals were given specific hand washing instructions and asked to evaluate both the lather on the hands and the cleansing properties of each saponified blend against the olive oil control. Panelists rated each saponified blend as "increases," "decreases," or "no change."

Sensory Evaluation: Effect on Bar Color and Odor

The same sensory panel was asked to describe the finished bar colors and odor. These subjective observations were determined by comparing each saponified blend to the olive oil control.

Practical Evaluations: Trace Level, Recommended Use Level, Recommended Applications

Trace Level was determined by a skilled soap maker based on how fast/slow each saponification reaction took place. The Recommended Use Level and Recommended Applications for commercial soapmaking were also determined for each Natural Wax.

Results

All results are presented in **Table 3**.

Discussion

The results in **Table 3** indicate each Natural Wax tested brought forth very distinct characteristics and attributes in traditional soapmaking. It was also determined that certain waxes are not always a suitable addition to these traditional techniques. Specifically, Natural Waxes with high melt points, higher molecular weight components or hydrogenated triglycerides can pose some challenges in processing techniques and are therefore recommended as hardening additives at ≤15% use level. Despite these few specific challenges, we were able to conclude that the following Natural Waxes offer numerous advantages in soap making, whether at 100% usage or as an addition to traditional triglyceride formulations.

Bayberry Wax

Due to its high triglyceride content, Bayberry Wax is fully saponifiable and can be used alone to make textured, artisanal bars, or as an additive to impart a rustic texture, color, and odor.

Cocoa Butter

Presenting a high triglyceride content and a relatively low melt point, Cocoa Butter can also be used at 100% to make a naturally moisturizing soap. As an additive, Cocoa Butter can slow trace, allowing soap makers to achieve swirls and designs, or soften existing bars.

Lauryl Laurate

The saponification of Lauryl Laurate yields sodium laurate, which aids in a stable, fast acting lather. This, combined with the lauryl alcohol generated, allows for soft formulas and unique, jelly-like textures in higher percentages.

Orange Wax

Only partially saponifiable with a high phytosterol content, Orange Wax is recommended as a natural orange colorant (both in the bar and the lather it produces), fragrance additive, and bar softener.

Beeswax

Beeswax is useful in all soap formulations at different usage levels due to its free fatty acids and its ratio of saponifiable/unsaponifiable components. Beeswax adds tack and is primarily a hardening additive, while also maintaining the original soap characteristics like odor, color, or lather.

Wax Name	Quantitative Evaluation	Tactile Evaluation		Sensory Evaluation		Practical Evaluation		
	Effect on Bar Rigidity	Effect on Bar Cleansing Properties	Effect on Bar Lather	Effect on Bar Color	Effect on Bar Odor	Trace Level at 50°C	Recommended Usage Level	Recommended Applications
Bayberry Wax	Hardens	Increases	No Change	Green	Yes	Very Fast	100%	Artisanal
Cocoa Butter	Softens	Decreases	No Change	Light Yellow	Yes	Slow	100%	Layering, Pours
Soy Wax	Hardens	Decreases	Decreases	White/Off White	No	Slow	15%	Molds
Castor Wax	Hardens	Decreases	No Change	Off White	No	Slow	15%	Molds
Kester K-24	Softens	Decreases	Decreases	Ivory	No	Slow	50%	Drop Swirls
Kester K-48	Hardens	Decreases	Decreases	White	No	Average	15%	Molds
Orange Wax	Softens	No Change	Increases	Dark Orange	Yes	Average	50%	Layering, Swirls
Rice Bran Wax	Hardens	No Change	No Change	Light Brown	Slight	Fast	15%	Layering, Designs
Beeswax	Hardens	Increases	No Change	Ivory	Slight	Average	50%	Textured Top
Sunflower Wax	Hardens	Decreases	Decreases	Light Brown	No	Fast	15%	Molds
Carnauba Wax	Hardens	Increases	Increases	Orange	Yes	Very Fast	50%	Artisanal
Candelilla Wax	Hardens	No Change	Increases	Tan	Yes	Average	50%	Embedding

Table 3: Properties of Saponified Natural Wax/Olive Oil blends. 50% of Each Natural Wax was saponified with 50% Olive Oil and evaluated.

Carnauba Wax

The hardening properties of saponified free fatty acids and high molecular weight monoesters present in Carnauba Wax helps to impart a firmer, long-lasting bar of soap with a dense, stable lather. Saponification also occurs much faster due to the higher percentage of unsaponifiables.

Candelilla Wax

Although Candelilla Wax is composed of 50% natural hydrocarbons, the free fatty acids present saponify readily, especially when combined with other triglycerides. Candelilla Wax also produces an excellent lather and an overall hard bar of soap. The natural color and odor are preserved.

Rice Bran Wax

Rice Bran Wax contains mostly high molecular weight monoesters and is an effective and inexpensive hardening agent in soap systems with no real effect on color, texture, or odor. Also high in palmitic acid, Rice Bran Wax also raises the possibility of replacing palm oil in low percentages.

Discussion

Although there is minimal information in current literature regarding the use and effects of Natural Waxes in soapmaking, possibly due to a very triglyceride-focused soap industry, the results of these experiments indicate the beginning of a relationship between the two. The uniqueness of each Natural Wax can be leveraged to create improvements in appearance, performance, and stability, setting the soap maker apart from the competition. In conclusion, incorporating Natural Waxes into traditional soap recipes allows for new formulas and more creativity, while keeping in alignment with a sustainable, natural future.

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Skin and Vascular Fitness

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abstract

Vascular blemishes can lead to unsightly skin appearance. A neglected cause can be oedema leading to puffy eyes and promoting visibility of small blood vessels. By taking advantage of an aqueous extract from *Helichrysum italicum*, puffy eyes and visibility of spider veins could be significantly reduced in an *in-vivo* study. Data from this study and *in-vitro* investigations suggest a negative role of nitric oxide (NO) in the development of these blemishes.

Introduction

Many different parameters can be the cause of unwanted, visible skin changes. One of them is oedema, which causes tissue swelling. Oedema is the result of fluid accumulation in extracellular areas, for example in the dermis. The formation of oedemas can have different causes. Often there is an underlying general connective tissue weakness affecting both dermal structures and vascular structures. Women suffer more often from connective tissue weakness than men as they have a different dermal collagen structure [1]. Chronic oedema leads to swelling of tissues. The legs are particularly predisposed, as hydrostatic pressure is greatest here. Many people suffer from "heavy legs", especially when they have been in an upright position for a long time. The legs feel tired, heavy and can become swollen. Chronic oedema has a negative effect on the skin: it becomes thinner and ulcers (open sores) may even develop; these are weeping wounds that heal with great difficulty. Diabetics are especially affected. This is, of course, the manifestation of a clinical condition and cannot be treated cosmetically. However, the risk of developing oedema increases with age [2]. Thus, early cosmetic support to prevent oedema can be considered a means of anti-ageing body care.

However, oedema can also occur on the face: Periorbital puffiness or puffy eyes are the result of fluid accumulation under the eyes. They are caused by insufficient lymph drainage. They make our eyes look tired and dull. It is not surprising that they can also be engendered by lack of sleep. Lymph can accumulate in the region of the bags under the eyes, especially in those who sleep on their stomach, leading to puffy eyes on waking. This swelling normally recedes in the course of the morning but can persist when individuals become older. During the ageing process, the skin becomes thinner and firmness and elasticity decrease in this delicate organ. Connective tissue weakness, smoking and alcohol intake are factors that promote the permanent appearance of bags under the eyes. An internal survey showed that 86% think puffy eyes are considered worse than wrinkles (n = 91, aged 21–70). Puffy eyes are therefore a significant comfort-reducing factor.

Another unsightly skin change is spider veins. They are dilations of fine veins just below the skin surface. It is a cosmetic issue and do not represent a disorder in the actual sense but can indicate underlying general venous insufficiency. Spider veins (telangiectasis) form preferentially on the legs and the face near the nose. They take the form of dark red to bluish veins with multiple branches. Frequently, spider veins are the result of an underlying venous reflux problem that causes blood stasis, making the vessels visible, and causing oedema in the surrounding tissue. Spider veins on the face can also be caused by sun damage [3]. The breakdown of collagen and elastin (solar elastosis) makes the veins more visible as they get closer to the skin surface. Therefore, skin ageing also plays a role in the development of spider veins on the face.

Other factors involved in spider vein formation include excessive alcohol consumption, smoking, lack of exercise, pregnancy, obesity and frequent standing or sitting causing blood stasis in the veins [4,5]. Spider veins occur more often in women than in men because they often have weaker connective tissue. It is next to impossible to completely prevent the development of spider veins, but if certain basic rules are followed the risk can be minimised. It is best to avoid sitting and standing for too long and thus advisable to move around, to stimulate the vein pump in the calves by means of foot exercises and to elevate the legs. In general, it is important to prevent blood stasis in the veins. There is as yet no tried-and-tested means of preventing the development of spider veins on the face. However, avoidance of excessive alcohol consumption certainly plays a major role here.

Extracellular fluid volume is regulated by peripheral tissue resistance and renal function, among other factors.

Nitric oxide (NO), produced by nitric oxide synthase (NOS), promotes venous dilation and varicose veins [6]. It can boost fluid accumulation in tissues by reducing systemic vascular resistance and arterial pressure and increasing vascular permeability [7,8]. A persistent increase in tissue fluid volume leads to the formation of oedema in the form of, e.g., puffy eyes on the face. It has been demonstrated that inhibition of NOS can reduce the formation of NO-induced oedema [9].

Inhibition of NOS and elimination of reactive oxygen species (ROS) have also proved effective in the prevention of sunburn [10]. In this condition, the microcirculation is greatly increased by the activity of NO. This means that inhibitors of NOS can have a soothing effect on the skin. It seems that use of these would therefore represent a general way of attenuating acute and chronic inflammatory vasodilatory responses in the skin in order to reduce redness, oedema and the visibility of spider veins as well as the sensation of "heavy legs". Interestingly, caffeoylquinic acids, such as chlorogenic acid, and especially di-O-caffeoylquinic acids are known to inhibit NOS and to act as antioxidants [11,12].

We at RAHN-Cosmetic Actives discovered that an aqueous extract of the plant immortelle (*Helichrysum italicum* syn. *angustifolium*) is the perfect solution for the above-mentioned issues. Indeed, Helichrysum extract (PERFELINE®-FIT; INCI: Water, Propanediol, Helichrysum Angustifolium Flower Extract, Citric Acid) exhibits a direct inhibitory effect on human NOS *in-vitro*. Besides chlorogenic acid, we were able to detect three different di-O-caffeoylquinic acids in the extract. Helichrysum extract is thus the perfect fit as a body and face care agent when it comes to dealing with oedema-related cosmetic problems like puffy eyes as well as spider veins and heavy legs.

Materials and Methods

Double-blind, placebo-controlled *in-vivo* study, 43 female subjects, aged 30–65 years (average 55.0), application of cosmetic formulations for 28 days twice daily on the legs (5% active ingredient), the eyes (3% active ingredient) and to the face (1% active ingredient).

VECTRA-XT was used to investigate the effect on spider veins (photographic images). The severity of spider veins was evaluated by means of assessment by a dermatologist.

VECTRA-XT was used to investigate red structures in the face. The severity of eye bags volume was evaluated by means of assessment by a dermatologist and quantification with AEVA.

The microcirculation in the eye area was determined using laser Doppler flowmetry. All parameters were measured at days 0, 14 and 28. The immediate effects on skin microcirculation were measured 30 minutes after application of the

formulations. An increase in microcirculation was provoked by heating the tissue with a self-heating probe to 42°C. To calculate the contribution of nitric oxide release to the increase in microcirculation, fast Fourier transforms and wavelet analysis was performed [14]. For this purpose, a baseline was recorded for 8 minutes, followed by the heat stimulation and then measurement for an additional 7 minutes.

To determine the inhibitory effect of Helichrysum extract on nitric oxide synthase, human eNOS enzyme was subjected to reaction with radioactively labelled L-arginine and oxygen to produce nitric oxide and L-citrullin. The resultant amount of L-citrullin was determined by means of scintillation counting. The reaction was performed in the presence of different concentrations of the active ingredient.

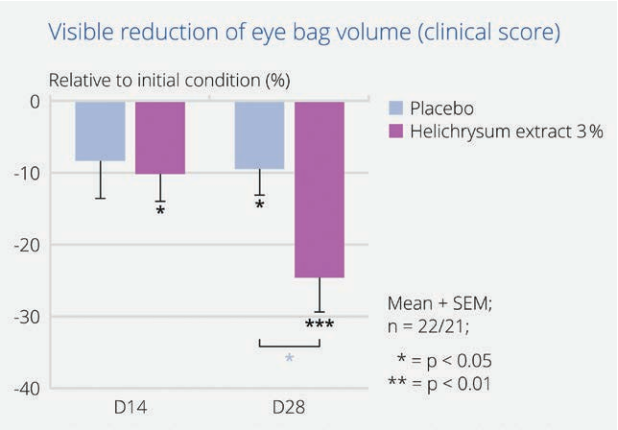

Fig. 1 Reduction of eye bag volume as determined by the dermatologist (clinical score).

Results and discussion

To investigate the efficacy of Helichrysum extract when applied to the whole face, two different types of formulations were used. One emulsion contained 1% Helichrysum extract or none (placebo). The other was a gel for the eye area containing 3% Helichrysum extract or none (placebo). This was applied as a roll-on with a metal ball applicator. One group applied verum only, the other group applied placebo only twice daily for 28 days. Application to the legs was done with an emulsion containing 5% Helichrysum extract.

Fig. 2 Visual representation of the effects of 3% Helichrysum extract on eye bags volume. **Left:** fringe projection with AEVA. **Right:** visual channel of AEVA.

After 14 and 28 days, Helichrysum extract reduced eye bag volume by 10%, which was significant in comparison with baseline **(Figure 1)**. After 28 days, the dermatologist attested a 25% reduction in eye bag volume, significant over baseline and placebo. These effects were confirmed by AEVA measurements (not shown) and clearly visible **(Figure 2)**.

Helichrysum extract significantly reduced red spots by 16% as measured by VECTRA-XT after 28 days (not shown). The images of the red channel clearly show a reduction of visible capillaries as well **(Figure 3)**.

After 14 days, the basal level of skin microcirculation in the puffy eye area decreased by 10.5%, which was significant in comparison with baseline and placebo **(Figure 4, left)**. This value remained in the same range after 28 days (7.4%). Interestingly, this effect was already achieved 30 minutes after product application (not shown). When skin was stressed (heat stimulation), the resulting maximal increase in microcirculation was significantly reduced by almost 20% after 28 days application of 3% Helichrysum extract **(Figure 4, middle)**. The effect was significant in comparison with baseline and placebo. Placebo did not reduce induced microcirculation at all. In contrast, induced microcirculation was significantly increased by more than 20% when placebo was used. It seems that the basic cosmetic formulation reduced the skin's resistance to external stressors, while supplementation with Helichrysum extract actually increased this resistance. Investigation of the nitric oxide contribution to the increase in microcirculation revealed that application of 3% Helichrysum extract significantly reduced the release of this vasodilator **(Figure 4, right)**. The effects were visible already 30 minutes after application and remained significant in comparison with baseline and placebo after 28 days. The microcirculation results suggest that the enzyme nitric oxide synthase (NOS) was inhibited to some extent. Basal activity would involve eNOS (endothelial NOS), which is constitutively, i.e. permanently, expressed. eNOS is also responsible for additional NO release if the microcirculation is provoked [15,16].

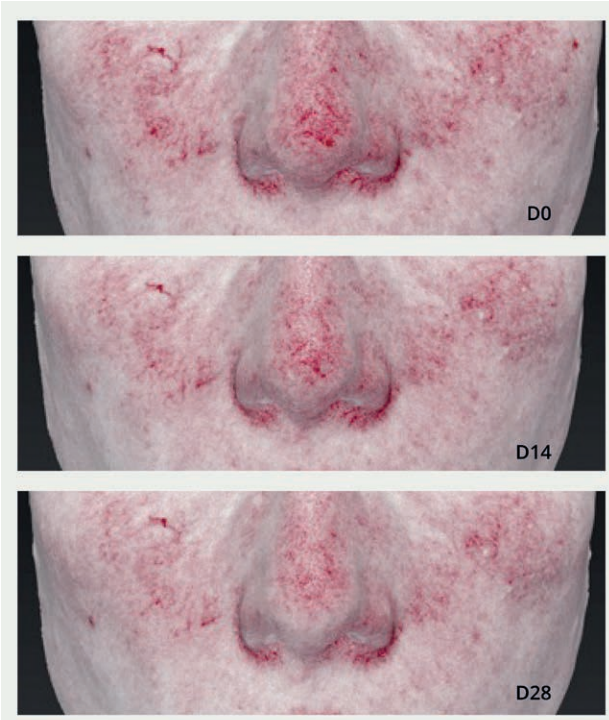

Fig. 3 Reduction of red spots and visible capillaries after application of 1% Helichrysum extract.

As Helichrysum extract reduces microcirculation in stressed skin, it can function as a skin soothing agent as well.

Helichrysum extract inhibits eNOS with an IC_{50} of 0.66 % *in-vitro*. At 1%, the inhibition was 62% (not shown).

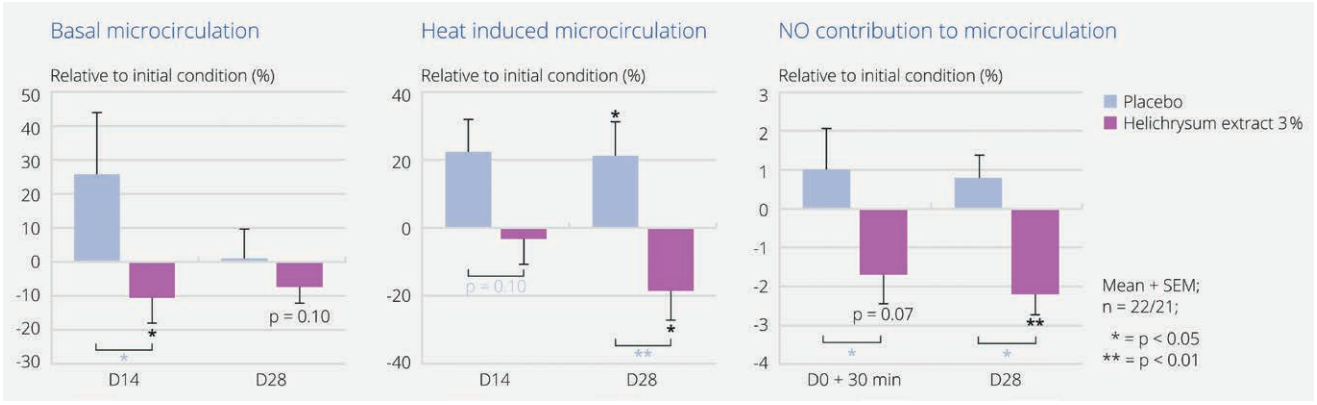

Fig. 4 Reduction in microcirculation. **Left:** Basal microcirculation. **Middle:** Heat induced microcirculation. **Right:** NO contribution to microcirculation.

We tested a beneficial effect on spider veins on the legs as these can be affected by an increased NO content of the surrounding skin. Indeed, the visibility of spider veins on the legs was reduced significantly over baseline and placebo by 10% and 18% after 14 days and 28 days, respectively, as deduced from the clinical score (not shown). The effect was clearly visible as depicted in **Figure 5**.

Fig. 5 Visible reduction of spider veins after application of 5% Helichrysum extract. **Top panels:** knee area. **Bottom panels:** thigh area.

Conclusions

PERFELINE®-FIT contains caffeoylquinic acids, which are known to inhibit nitric oxide synthase [17]. Based on this background, the effects observed in the study seems very plausible. The inhibition of NOS leads to a reduced release of NO and thus prevents excessive vasodilation and increased permeability of the vessels. Permanent vasodilation can lead to the development of visible capillaries and spider veins as well as oedema. We have found that the extract from *Helichrysum italicum* is able to alleviate unsightly puffy eyes and the visibility of spider veins. PERFELINE®-FIT is therefore a perfect solution for taking care of the skin, whether on the face or all over the body. It is a natural cosmetic ingredient that is mild but effective, making signs of stress and fatigue disappear for a fresh look and feel.

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VINCENTZ

Investigate the Hygroscopic Potential of Apiogalacturonans for a Plumped Skin

M. Coirier, L. Verzeaux, H. Muchico, E. Aymard, B. Closs

abstract

Whether dry, mixed or oily, every skin can be subjected to dehydration, characterized by a lack of water. Tightness, lack of suppleness, loss of volume, lines and wrinkles then appear.
To specifically meet the needs of dehydrated skin, SILAB decided to develop APIOSKIN®, a natural plumping active ingredient with outstanding hydrating properties, purified in Apiogalacturonans (APG) from the water lentil (*Spirodela polyrhiza*). Its smoothing and radiance-boosting effects beautify dehydrated skin.

Introduction

Water is indispensable for life. Plants and humans contain 70% water, constituting the natural medium of molecules. In the field of cosmetics, the issue of hydration is one of constant attention, recently boosted by the emergence of new interest focusing on dehydrated skin. This skin is characterized by a lack of water, a state that can be affected by a variety of factors (environmental, emotional, medicinal, etc.). It is very frequent and can involve all skin types.

To address this issue, the use of molecules with elevated hygroscopic capacity is therefore necessary for effective and long-lasting hydration. Pectins are ancestral, structural molecules, capable of binding water molecules according to their organization. SILAB has been studying their natural properties for more than 25 years and decided to use its extensive expertise in glycobiology for the development of APIOSKIN®, its latest natural plumping active ingredient.

1. Apiogalacturonans, unique molecules

Rare and original pectins

Pectins are a family of macromolecules with highly varied compositions and structures. They are present in plant cell walls and, in addition to their structural role, they can bind water, a capacity closely linked to the type of pectin [1,2]. In the context of the development of its latest plumping active ingredient, SILAB paid special attention to pectins found in the aquatic world, in particular those from water lentil. The latter is a plant growing on the surface of ponds. It floats at the water-air interface, enabling it to control temperature variations, thereby preventing deep water from warming.

Among the various species of water lentils, the research teams focused on *Spirodela polyrhiza* that has been used for more than 2,000 years in traditional Chinese medicine for its capacity to promote the metabolism of water. Indeed, the pectinous cell wall composition of *Spirodela polyrhiza* is unique in that it contains Apiogalacturonans (APG). These molecules ensure water exchanges and provide elevated extensibility to the plant when it multiplies [3,4].

A unique hygroscopic potential

APG from *Spirodela polyrhiza* therefore possess specific characteristics, which SILAB decided to study. More specifically, the company wanted to investigate if they possessed an elevated hygroscopic potential enabling water to be taken up and retained.

To this end, a first study was implemented to predict the hygroscopic capacity of APG. This work was conducted jointly with a French laboratory at the University of Reims Champagne-Ardenne (CNRS UMR 7369 - Multi-scale modeling and imaging) and for the first time enabled the three-dimensional structure of an APG and the water shell on its surface to be

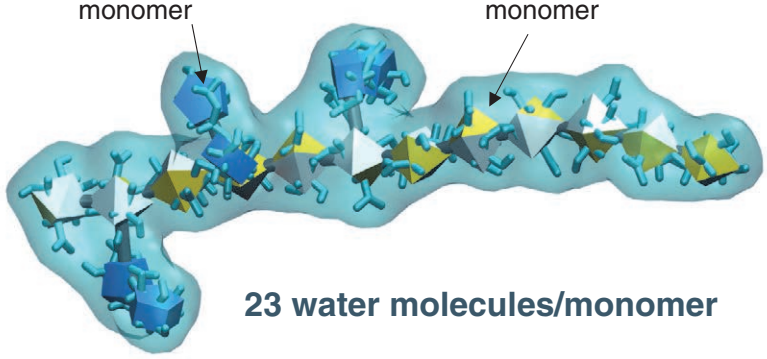

Fig. 1 Visualization of the hygroscopic potential of APG.

simulated. This original model enabled the number of water molecules interacting with APG to be quantified. It was thus shown that each APG monomer can interact with 23 molecules of water **(Figure 1)**. In comparison, each monomer composing hyaluronic acid interacts with 10 molecules of water [5]. These results suggest that APG have a hygroscopic potential twice higher than the reference molecule in the hydration category.

The second part of this work involved validating the capacity of APG to take up water directly in volunteers with dehydrated skin. This was done using Raman microspectroscopy to follow the penetration of a labeled molecule of water (D_2O), brought directly on the skin of volunteers having previously applied a placebo formula or one containing APG. This study showed that in presence of APG, the capacity of the skin to take up and retain water was improved both quantitatively and qualitatively (deeper in the skin) **(Figure 2)**.

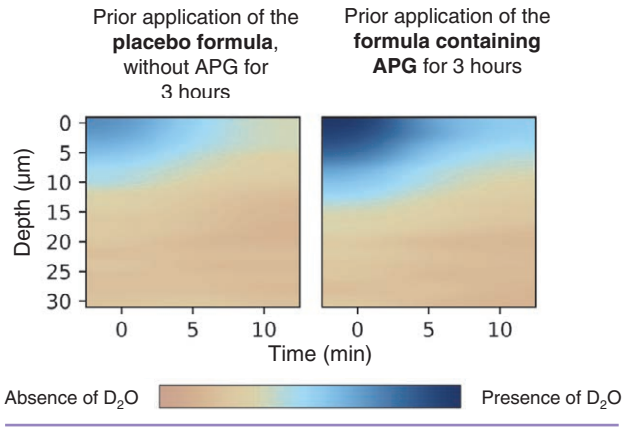

Fig. 2 Visualization of the capacity of APG to take up water.

An additional study demonstrated that APG have a higher hygroscopic capacity compared to other glycocompounds, whether of pectic origin (homogalacturonans) or not (fructosans).

By combining molecular modeling and Raman microspectroscopy, SILAB has for the first time shown the outstanding hygroscopic properties of APG and their value in cutaneous hydration. The company therefore decided to use it as raw material for its plumping active ingredient.

A secure supply and unique process for a qualitative raw material

Even though it is distributed throughout the world, most of today's supply of water lentil comes from Asia as a result of its use. The company thus obtains its supply from South Korea and China. The plant is harvested yearly, from June to September and is then rinsed and sun-dried naturally. In order to have a secure supply chain, an authentication and validation process down to the level of molecular barcoding has been implemented.

Indeed, each plant species has its own genetic fingerprint characterized by unique DNA fragments, like a bar code, that differentiates it from all other species. DNA barcoding of a sample aims to extract, amplify, sequence and compare the specific fragments with those published in databases, in order to identify one or several species. In addition to identification, DNA barcoding detects the possible presence of contaminants.

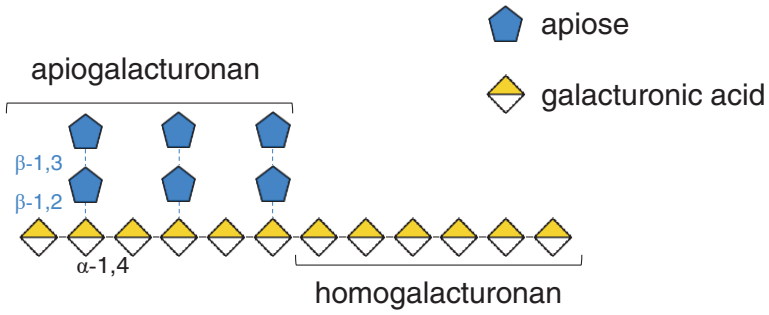

Fig. 3 Structure of the pectin of *Spirodela polyrhiza*.

In this case, usual methods of botanical identification are limited as they can't distinguish the different water lentil species that are received in powdered form. DNA barcoding has therefore been adopted as a method of choice for guaranteeing the validation of the sourcing. Only DNA barcoding can secure the identity and purity of samples, essential for the development of this new hydrating ingredient.

In their native form, the structure of *Spirodela polyrhiza* apiogalacturonans is linear, composed of a skeleton of tightly bound galacturonic acids with ramifications of weakly bound di-apiose sidechains that are more fragile **(Figure 3)**.

To obtain an active ingredient purified and enriched in APG, the challenge was to define extraction conditions enabling molecules of optimal size to be extracted, while preserving their structure. Using a mild acid hydrolysis enabled to develop a hydrating and plumping active ingredient enriched in APG to more than 50%.

2. The ideal response to dehydrated skin

Activation of the biological levers of cutaneous hydration

In addition to its intrinsic hygroscopic properties, the biological activities of the natural active ingredient were evaluated on the hydration levers. Hence, the development of different *in vitro* models demonstrated that the natural active ingredient has an effect on aquaporins, BGT-1 (Betaine/GABA transporter-1) and the NMF (Natural Moisturizing Factor).

Aquaporins

Aquaporins are small channels embedded in cell membranes. They ensure the dynamic and effective circulation of water. Aquaporin 3 is the major type in skin, especially in the membrane of keratinocytes. The extent of hydration of the epidermis depends on the optimal distribution of these channels in all its layers [6,7]. They ensure the flow of 3 billion water molecules per second, thereby guaranteeing that the epidermis is correctly irrigated all the way to the surface [8].

Tested at 0.5% on dehydrated keratinocytes, the active ingredient boosts the expression of aquaporin 3 by 41%.

BGT-1

The osmolyte transporter BGT-1 is a regulator of the intracellular betaine content [9,10]. Betaine is one of the major osmolytes in the epidermis. One of the defense mechanisms of cells to combat water stress is in fact osmotic adjustment. Osmolytes are small soluble molecules that participate in protecting cells from a water deficit. In conditions of stress, osmolytes accumulate inside cells by the activity of specific transporters to prevent water losses and maintain cell structures. In addition to preventing cell dehydration, betaine plays the role of chaperon, stabilizing the structure and function of proteins, in particular those involved in tight junctions [11].

Applied at 1% on dehydrated explants, the active ingredient results in 100% restoration of the synthesis of the osmolyte transporter. By regulating osmotic pressure in this way, it has a positive effect on cell volume.

The NMF

The NMF is a family of hydrophilic intracorneocyte substances found in the *stratum corneum*. Resulting primarily from the degradation of filaggrin, they are composed principally of amino acids (40%), the majority of which is serine, of PCA (Pyrrolidone carboxylic acid) (12%), of lactate (12%), of urea (7%) and of inorganic ions. Their presence results in the uptake of free water and its retention in corneocytes, thereby playing an important role in maintaining the physical properties of the *stratum corneum* [12]. Lactate in particular would maintain the acid pH of the *stratum corneum* [13], one of the important parameters for guaranteeing its integrity and cohesion [14].

After 21 and 42 days of twice daily application by Caucasian volunteers with dehydrated skin, the active ingredient significantly increases lactate production by 32.7% and 51.2%, respectively.

Cosmetic benefits beautifying dehydrated skin

Cutaneous hydration increased

By combining the intrinsic endogenous properties of APG with the biological activation of hydration levers, the active ingredient beautifies dehydrated skin. Indeed, these effects

result in immediate and long-lasting hydrating benefits, superficially and in-depth, measured from the *stratum corneum* down to the upper dermis.

The immediate and the long-term hydrating effects of the active ingredient were determined in a Caucasian panel. The long-term effect was also tested in an Asian panel. All volunteers were selected with dehydrated skin on the face, wrinkles on the crow's feet and a dull complexion.

The in-depth effect in the epidermis and upper dermis was determined by measuring the level of hydration with a MoistureMeterD and the superficial hydration of the *stratum corneum* with a CM 825 Corneometer®.

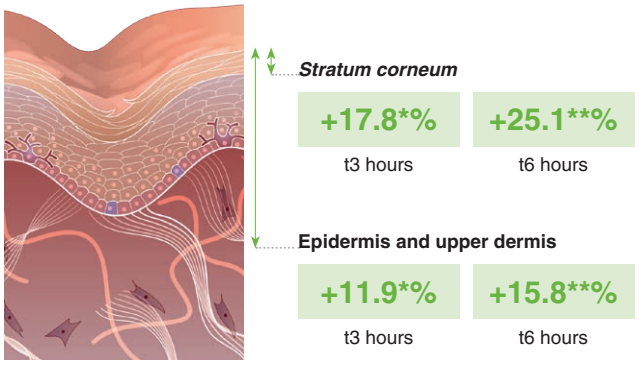

Fig. 4 Representation of the immediate effect of the active ingredient (Result(s)/placebo: **: P < 0.01 --- *: P < 0.05).

Regarding the immediate effect, the results show that as of 3 hours after a single application and compared to the placebo, the active ingredient at 3% in an emulsion increases hydration of the epidermis and upper dermis by 11.9% (P = 0.0416). It also immediately improves superficial hydration by increasing that of the *stratum corneum* by 17.8% (P = 0.0170). These effects continue and intensify after 6 hours of application **(Figure 4)**.

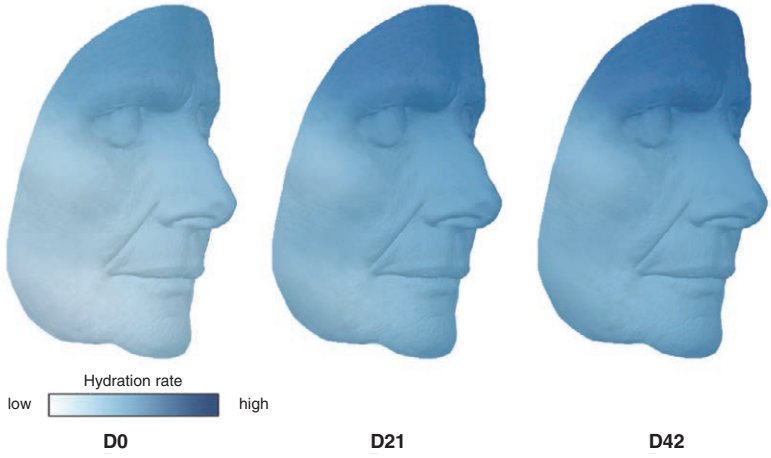

Fig. 5 Map of the long-term superficial hydration effect of the active ingredient in a Caucasian volunteer.

The long-term hydrating effect of the active ingredient was also determined at 3% in an emulsion and the results show that after 21 days of twice-daily treatment, it significantly increases in-depth hydration by 10.6% (P = 0.0004). The long-term superficial hydration of the face is also demonstrated with a significant 5.9% improvement (P = 0.0241). These results are illustrated on a map, which was realized with 23 measurements made on each half of the face, digitized, and placed on an acquisition of the volunteer obtained by fringe projection **(Figure 5)**.

These effects continue and intensify after 42 days of treatment. In the Asian panel, there is also a significant increase in hydration of the stratum corneum, after 21 and 42 days of twice daily application (*data not shown in this publication*).

Plumping effect for a smoothed and radiant skin

As a result of these outstanding hydrating properties, the face is plumped, dehydration lines are smoothed, and complexion radiance is revived. All these parameters were evaluating before and after 42 days of twice daily application of the active ingredient at 3% in an emulsion or the placebo on the entire face.

The plumping effect was determined in Caucasian volunteers by measuring facial volume (EvaFACE3D-S5 system). The results show that, compared to the placebo, the active ingredient significantly improves the parameter of positive volume, characteristic of a plumping effect, as of 21 days until reaching a significant 47.3% increase after 42 days ($P = 0.0230$).

The smoothing effect of the product was determined in Asian volunteers using clinical scoring by a dermatologist. Compared to the placebo, the active ingredient has a smoothing effect on the eye contour area by significantly attenuating the stage of wrinkles by 4.0% ($P = 0.0370$) after 21 days of application. This action continues after 42 days of treatment.

Finally, the radiance-boosting effect of the active ingredient in Caucasian volunteers was determined through clinical scoring by two trained evaluators using photographs. Compared to the placebo, the product significantly improves the parameters characteristic of complexion radiance. Indeed, as of 21 days, it provides a more luminous and fresher complexion by significantly increasing skin reflection (7.8%, $P = 0.0086$) and pink color (21.2%, $P = 0.0040$). It also significantly decreases olive color (13.2%, $P = 0.0127$), as well as the state of eye fatigue (11.1%, $P = 0.0096$). These actions continue after 42 days of treatment and are also seen in the Asian panel (*data not shown in this publication*).

Conclusion

SILAB used its extensive expertise in glycobiology to highlight the hydrating potential of unique pectins: APG. This research work led to the development of APIOSKIN®, a natural plumping active ingredient with outstanding hydrating properties, derived from the water lentil (*Spirodela polyrhiza*). Purified and enriched in APG, which give it its highly hygroscopic capacity, it is able to take up and retain water, from the *stratum corneum* down to the upper dermis, while activating the levers of skin hydration. It provides a flash and long-lasting hydration, superficially and in depth. Its smoothing, plumping and radiance-boosting effects beautify the skin. This efficacy was broadly approved by Caucasian volunteers since 100% of them state that their skin is more hydrated, softer, more comfortable and more luminous. The plumping effect was also noticed by 84% volunteers.

As a true hydration concentrate, APIOSKIN® is therefore the ideal solution for dehydrated skin.

A patented active ingredient, it is recommended in all plumping skin care products at a dose of 0.5 to 3%. Available in aqueous solution, it is easy to formulate. It complies with biodiversity regulations and has a natural origin content of 99.2% (ISO 16128). It also complies with international cosmetic regulations (Europe, United States, China, Japan, and more).

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Unique Active Ingredients Balancing the Skin Microbiome to Solve the Fourth Most Common Skin Issue

C. Zanchetta, M. Fleury

Skin microbiota refers to the living microorganisms present on the skin's surface. As an essential part of the skin's ecosystem, the understanding of skin microbiota is crucial in developing efficient cosmetic products.
Givaudan Active Beauty has developed active ingredients related to the skin microbiota, specifically targeting the four main skin issues faced by the consumers: hyperpigmentation, skin sensitivity, oiliness and skin ageing. In regulating both the skin physiology and the skin microbiome, B-Lightyl™, Mangixyl™, Sensityl™ and Yogurtene® Balance offer strong skin benefits.
These ingredients are part of a larger portfolio dedicated to the skin microbiota. Globally, Givaudan Active Beauty delivers ingredients able to balance, protect and activate the microbiota to enhance the beauty of all skins.

Introduction

Skin microbiota refers to the living microorganisms present on the skin's surface: bacteria, fungi, viruses, and mites. The commensal microorganisms of the skin have many essential roles related to the control of the skin's homeostasis. Among them, to train and maintain the immune system, to produce vitamins essential for the human skin and body and to prevent skin colonisation by pathogens. As an essential part of the skin's ecosystem, the understanding of skin microbiota is crucial in developing efficient cosmetic products.

The Givaudan Active Beauty Centre of Excellence in Microbiomics is fully equipped to build knowledge of the skin microbiome. Microbiota sequencing technologies, molecular biological solutions and bacterial cultural methodologies support a better understanding of the skin microbiota and its interactions with the host's skin. These capabilities unveil the dynamics of the microorganism's communities and their modulations according to their environment.

Based on this understanding, Givaudan Active Beauty develops disruptive active ingredients, acting on the skin in a holistic way. They improve the appearance of the skin by inducing actions on both the skin cells' physiology and the skin microbiota.

Consumer's needs & microbiome understanding

An internal Givaudan Consumer Insight study, conducted in 2021 and dedicated to the skin microbiome, revealed an increased consumer understanding of the skin microbiota over the years. 91% of consumers are now aware that they have microorganisms on the skin, 85% of them note that it is important to maintain their balance and 76% believe that the microbiome plays an important role in the condition of the skin.

Interestingly, consumers also identify a strong link between four of the most common skin issues and the skin microbiota. According to the CMI study, a microbiome imbalance can be related to skin sensitivity for 65% of consumers, skin oiliness for 56%, skin hyperpigmentation for 54% and ageing for 51% of them.

Moreover, consumers heavily rely on cosmetic products dedicated to the microbiome, with 93% of consumers showing an interest in cosmetics acting on the skin microbiota [1].

To better respond to these consumer needs, Givaudan Active Beauty has developed active ingredients related to the skin microbiota, specifically targeting the four main skin issues faced by the consumers: hyperpigmentation, skin sensitivity, oiliness and skin ageing.

Controlling the melanogenesis by rebalancing the skin microbiota - B-Lightyl™

Hyperpigmented spots (HPS) are oval or irregular brown areas of skin. Their emergence is associated with dysregulation of the immune system, leading to perturbed melanogenesis and accumulation of melanosomes close to neighbouring kerat-

inocytes. The link between hyperpigmentation and skin microbiota remains poorly studied but a recent Givaudan Active Beauty study has revealed a specific microbiota composition and network on the skin based on HPS status.

This study was conducted on 38 women aged between 18 and 64 with solar and senile lentigos to determine whether skin with more HPS has a characteristic microbiota profile. The study has been performed using 16S rRNA sequencing methods. Results indicate that skin showing hyperpigmentation disorders also present seven bacterial genera in significantly higher proportions than in skin without hyperpigmentation issues: *Eikenella*, *Xanthomonas*, *Brevibacterium*, *Aerococcus*, *Turicella* and *Klebsiella* [2].

Interestingly, our results indicated a shift in *Eikenella* proportions; from 0.25% on skin with no hyperpigmentation disorders to 0.84% on skin presenting hyperpigmentation issues. This genus is considered an opportunistic pathogen, increasing in ageing and producing proinflammatory molecules. *Eikenella corrodens* (the only species classed in the *Eikenella* genus) produces a molecule that activates the mitogen-activated protein kinase (MAPK) pathway, which is involved in regulating melanogenesis. Thus, constitutive activation of the MAPK pathway inhibits proteasome activity, which is essential for melanin degradation. In addition, *Eikenella* is a Gram-negative bacteria and its increasing abundance will decrease the activation of the stromal cell-derived factor 1 (SDF-1) by the lipoteichoic acid (LTA) harboured by Gram positive bacteria. SDF-1 oversees the communication between fibroblasts and melanocytes throughout the pigmentation process. The synthesis of SDF-1 tends to decrease in ageing and might also be related to microbiome composition as mentioned above. This lower activation of SDF-1 will lead to a loss of control of the melanin synthesis and create pigmentation disorders as brown spots.

B-Lightyl™ is an active ingredient targeting hyperpigmented spots. It demonstrates an antioxidant effect and is able to reactivate the expression of SDF-1 in skin cells. This reactivation passes through a balancing of the skin microbiome. A clinical study has been conducted on 38 volunteers to evaluate the impact of B-Lightyl™ on the skin microbiome. The active ingredient B-Lightyl™ has been evaluated versus placebo. The skin microbiota of volunteers has been sampled before the application of the products and after 28 days and 56 days of application. Very interestingly, we noticed a significant decrease in the proportion of *Eikenella corrodens* after the application of B-Lightyl™, by -84% on day 28 and by -59% on day 56.

This balancing of the microbiome reinforces the control of the melanogenesis by increasing the activation of SDF-1 and the activity of the proteasome. By inducing an action which affects the global ecosystem of the skin, B-Lightyl™ reactivates cellular communication and takes back control of skin pigmentation, getting rid of hyperpigmented spots.

A unique psychobiotic soothing the skin by down-regulating inflammatory bacteria – Sensityl™

Skin sensitivity is characterised by a higher basal level immune system activation. The release of cytokines leads to visible redness and an itching sensation, which can also cause discomfort in social situations due to these visible skin issues. Recently, studies have revealed a specific microbiota composition for sensitive skin [4]. Sequencing of the 16S RNA gene of skin bacteria evidenced higher proportions of some genera such as *Corynebacterium*, *Kocuria* and *Micrococcus* on sensitive skin. A complementary analysis has proven that the proportion of *Corynebacterium* was discriminant in evaluating the sensitivity level of the skin. Skin harbouring the highest proportions of *Corynebacterium* is the most sensitive. On sensitive skin, the *Corynebacterium* genus represents up to 15% of the total bacteria versus only 8% on non-sensitive skin. Many strains of *Corynebacterium* are described as pro-inflammatory, activating some inflammatory pathways (IL-5, IL-8…). This pro-inflammatory activity might be due to the presence of mycolic acids in the cell wall of *Corynebacterium*. *Corynebacterium* is, to date, the only skin bacteria to present inflammatory mycolic acid.

Sensityl™ is an active ingredient dedicated to sensitive skin. Acting through epigenetic regulation and microbiome balancing, it takes control over the whole inflammation process. By using 16S sequencing technologies to study the microbiome of 41 women, we have demonstrated the impact of Sensityl™ on the pro-inflammatory bacterial genus *Corynebacterium*. Before the application, women with sensitive skin presented a proportion of 13.6% of *Corynebacterium* among their microbiota. After the application of Sensityl™ we noticed a significant reduction to only 9.2% (a decrease of -32%, versus a non-significant reduction with the placebo application). This action on the skin microbiota reduces the release of pro-inflammatory mediators and the recruitment of immune cells, restoring a basal inflammation level for an effective soothing effect.

Skin benefits brought through an action on the skin microbiome have led to proven positive effects on well-being. Sensityl™ has been evaluated using neuroscience-based technologies to determine the impact of its soothing effect on consumers' well-being. Two methodologies have been used on 41 volunteers: Mood Portraits®, a non-verbal method to measure consumers' mood and emotions, and Emotion Decoding System® to evaluate unconscious emotions. Results demonstrate a significant increase in positivity and interestingly, an increase in confidence. As people suffering from sensitive skin admit discomfort in social situations, this improvement in self-confidence highlights the global effectiveness of the ingredient both in skin care and in promoting overall well-being.

Sensityl™ is the first active ingredient in the era of Psychobiotics for skin care. Psychobiotics are a new form of microorganism-promoting supplementation after probiotics, prebiotics and postbiotics. Initially, Psychobiotics referred only to probiotics conferring mental health benefits, but the definition has now been broadened to include prebiotics or any molecule affecting the microbiome that leads to mental-health benefits. Until now, all Psychobiotics were ingested products, targeting the brain through actions on the gut microbiome. However, in launching Sensityl™, Givaudan is the first company to offer Psychobiotics for a skin care application. Sensityl™ offers skin care and well-being benefits through its positive balancing of the skin microbiome.

Sebum control and acne prevention by targeting *Cutibacterium* acnes metabolism – Mangixyl™

Sebum is an oily complex protecting the skin and is composed of triglycerides, free fatty acids, wax, esters and squalenes. An increase in sebum secretion will make the skin appear greasy and lead to the development of an acne-prone skin condition.

Cutibacterium acnes is a commensal skin bacteria present in both healthy and acne prone skin. Nevertheless, some specific strains and phylotypes might be specifically linked to acne-prone skin. These specific strains exhibit important lipase activity, using the sebum as a source of energy to colonise the skin. The lipase activity transforms triglycerides into free fatty acids, more viscous and inclined to obstruct pilosebaceous units and to generate inflammation. In response to this increased level of inflammation, the skin will produce even more sebum, leading to a vicious cycle between sebum production, lipasic activity and inflammation.

Mangixyl™ has been designed to prevent acne through the control of sebum production. This *Mangifera indica* extract prompts a specific action on the lipasic metabolism of *C. acnes*. *In vitro* testings have been conducted on a *C. acnes* strain typical of acne-prone skin (phylotype IA1) showing a strong lipase activity. In the presence of Mangixyl™, the li-

pase activity of the strain has been reduced by 23%, highlighting the capacity of the active ingredient to modify the metabolism of acne-associated *C. acnes* strain. This targeted metabolism modification will decrease the inflammatory status of the skin.

To determine if this metabolism modification might lead to an imbalance of the skin microbiome, a 16S rRNA sequencing study was conducted. It involved 45 volunteers comprised of 3 ethnicities (European, Asian, and South African) and was conducted versus placebo. Relative abundance evolution of the bacterial genera showed that Mangixyl™ at 1% protected the skin microbiota's general balance over time.

In combination with this activity on *C. acnes*, Mangixyl™ also triggers sebocytes regulation. This holistic action on the skin physiology and on the skin microbiome leads to visible clinical benefits: decreasing sebum production by 40%, improving sebum quality (by improving the ratio triglycerides/free fatty acids) and also significantly decreasing the production of porphyrins [3].

Rejuvenating the skin microbiota for a global anti-ageing effect – Yogurtene® Balance

The skin microbiome composition in modified in ageing. Some bacterial communities tend to colonise the skin (such as the *Actinobacteria*) while some tend to be less abundant (such as the *Proteobacteria*). More precisely, the reduction of the proportion of *Cutibacterium* and *Staphylococcus* can be observed. This reduction could be linked to the decrease in sebum production in ageing. Simultaneously, potentially pathogenic bacteria such as *Corynebacterium* or *Enhydrobacter* are more abundant on older skin. This shift in microbial communities creates new dynamic interactions between skin and microbiome, with potential deleterious effects due to the increase of opportunistic pathogens.

Yogurtene® Balance is an active ingredient containing inulin, a famous prebiotic metabolised by host microorganisms and conferring health benefits. A clinical study has been conducted to evaluate the hydration properties of Yogurtene® Balance. The ingredient was incorporated into a bar of soap to determine its rinse-off benefits. The hydration level was measured and the skin microbiota was sampled before application, 15 minutes, 3 hours, and 6 hours after the use of the soap. Yogurtene® Balance improves hydration of the skin (significant versus placebo and baseline), conferring to the skin the hydration properties typical of younger skin. Very interestingly, Yogurtene® Balance also balances the skin microbiota. It significantly reduces *Proteobacteria* and upregulates *Actinobacteria*, making the skin microbiome composition closer to that of younger skin. These combined benefits demonstrate a rejuvenation of the skin microbiome, providing skin hydration benefits.

A unique strategy for an integral offer dedicated to the microbiome

Thanks to these active ingredients, and to the expertise of Givaudan Active Beauty in the field of skin microbiome characterisation, for the first time in the Beauty industry it is now possible to answer the needs and expectations of consumers by acting on the skin microbiome, solving four of the most common skin conditions: dark spots, hypersensitivity and redness, excess sebum and acne, as well as premature ageing.

This entirely fits with Givaudan's general approach when it comes to ingredients dedicated to the skin microbiome, characterised in three different ways: Balance / Protect / Activate.

- **"Balance"** is a way to describe active ingredients which will directly impact the skin microbiome in terms of general balance and composition, to make more closely resemble that of "healthy" skin, in order to eliminate specific skin conditions.

- **"Protect"** is the function of ingredients which will not disturb the skin microbiota's homeostasis, while offering a cosmetic benefit to the skin. It can also refer to actives with a protective effect against the exposome or external aggression to the skin.

- **"Activate"**, lastly, is a unique strategy, where the skin microbiota is used as a trigger to release the benefits of a super-potent ingredient which has been protected or stabilised for improved formulability or usability in a cosmetic formula (for instance thanks to glycosylation). Specific enzymes produced by the bacteria present on the skin have been proven to be capable of breaking chemical bonds, and therefore "activating" an active ingredient when put in contact with the skin.

Givaudan goes on its exciting journey to explore the skin microbiome through a better understanding of its interaction with the host's skinthe and the development of new active ingredients within the previous three categories.

Leveraging this knowledge, Givaudan Active Beauty also offer the ultimate tool for skincare personalisation (considered to be one of the holy grails of the Beauty industry), thanks to the i-MAPS technology. This tool identifies a link between the composition of the skin microbiome and typical skin conditions, procuring a precise skin diagnosis in a fast, non-invasive, and reliable way to offer beauty consumers the most effective routine according to their needs.

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Characterization and Authenticity Testing of Vegetable Oils for Cosmetics

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1. Introduction

Vegetable oils are frequently used as valuable raw materials in a wide variety of cosmetic products. The oils are said to have positive effects on the skin surface and to strengthen the skin barrier. Furthermore, they counteract excessive moisture loss from the skin.

The decisive factor in the use of vegetable oils as cosmetic raw materials is their purity and impeccable quality. Specific quality and testing parameters are used to ensure quality and safety. This also serves to comply with the requirements of Regulation (EC) 1223/2009 [1]. Furthermore, a reproducible manufacturing process is crucial in order to achieve consistent qualities.

As part of a project, a total of 18 vegetable cosmetic oils (15 different oils) were specified more precisely with regard to the fatty acid spectrum, tocopherol distribution and phytosterols. Sometimes "exotic" oils or oils that are rarely used (e.g. pomegranate seed oil) are also included. The data provide a comprehensive overview of authenticity and the ingredients of the vegetable oils studied.

It must be taken into account that these analytical values may vary depending on provenance, cultivation, production, processing, etc.. Likewise, usual analytical measurement uncertainties must be taken into account when interpreting these data.

2. Analytical methods and examination procedures

The testing of the vegetable oils took place in the period from January to December 2022. The testing was carried out in accredited testing laboratories of the Tentamus Laboratory Group (www.tentamus.com). The coordination of the samples as well as the preparation of the test reports was done by the BAV Institute (part of the mentioned laboratory group). The testing was carried out using the most modern technical testing methods such as HPLC-FLD, GC-FID as well as LC-GC-FID.

The purchase and shipment of the sample specimens were carried out by the company Primavera Life GmbH (www.primaveralife.com). Primavera is an aromatherapy and organic & natural cosmetics manufacturer from the Allgäu region in the South of Germany.

3. Origin of vegetable oils

In addition to other factors, such as the weather conditions during cultivation or the time of harvest, both the area under cultivation of the oil plants and the way in which the fatty oil is obtained can have a significant influence on the composition of the fatty acids and fatty substances in the vegetable oils [2,3]. To increase the comparability of the measurement results presented with other studies, the countries of origin and the quality designations of the vegetable oils investigated in this study are listed in **Table 1** for the reason stated above. For these data, the information provided by the raw material suppliers was used. In addition, the International Nomenclature for Cosmetic Ingredients (INCI) is given in **Table 1**, which provides information on the botany and the plant part processed into oil.

The apricot kernel oil investigated in this study (sample **1**) originates from Uzbek organic farming and was pressed in Germany from the kernels of ripe apricots. The oil was extracted by mechanical cold pressing without any other additives.

In the case of the argan oil (samples **2**, **3** and **4**), the oils studied came from three different suppliers or producers. The country of origin of the seeds from which the argan oil was pressed is Morocco for all three oil samples, to which the cultivation of argan trees for argan oil production is practically limited [4]. Likewise, all three argan oil samples were produced by cold pressing of organic quality argan seeds.

The avocado oil (sample **5**) was produced by malaxation, similar to the production of olive oil. In this process, the avocado fruit is first ground into a paste and the avocado oil is then separated by centrifugation. It is then filtered. The

entire process takes place at low temperatures (45 - 50°C), so the avocado oil studied can be classified as cold-pressed. The avocado fruits used for the production of the avocado oil were cultivated organically in Kenya.

The pomegranate seeds that were cold-pressed in Germany using a screw press for the pomegranate seed oil studied (sample **6**) were also organically grown. The country of origin of the pomegranate fruit processed for this purpose is Azerbaijan.

For the analysis of hemp seed oil (sample **7** and **8**), two samples from different suppliers were used. Both hemp seed oils were produced in a cold pressing process using a screw press and then filtered. The hemp seeds for sam-

Sample-No.	Oil type	Country of origin Oil fruit	Quality designation	INCI
1	Apricot kernel oil	Uzbekistan	Cold pressed, organic	Prunus Armeniaca Kernel Oil
2	Argan oil	Morocco	Cold pressed, organic	Argania Spinosa Kernel Oil
3	Argan oil	Morocco	Cold pressed, organic	Argania Spinosa Kernel Oil
4	Argan oil	Morocco	Cold pressed, organic	Argania Spinosa Kernel Oil
5	Avocado oil	Kenya	Cold pressed, organic	Persea Gratissima Oil
6	Pomegranate seed oil	Azerbaijan	Cold pressed, organic	Punica Granatum Seed Oil
7	Hemp seed oil	Lithuania, Croatia, Finland, Estonia	Cold pressed, organic	Cannabis Sativa Seed Oil
8	Hemp seed oil	Germany	Cold pressed, organic	Cannabis Sativa Seed Oil
9	Jojoba oil	Peru	Cold pressed, organic	Simmondsia Chinensis Oil
10	Almond oil	Spain	Cold pressed, organic	Prunus Amygdalus Dulcis Oil
11	Evening primrose oil	China	Cold pressed, organic	Oenothera Biennis Oil
12	Olive oil	Spain	Cold pressed, organic	Olea Europaea Fruit Oil
13	Sea buckthorn fruit oil	Germany	Cold pressed, organic	Hippophae Rhamnoides Fruit Oil
14	Black cumin oil	Egypt	Cold pressed, organic	Nigella Sativa Seed Oil
15	Sesame oil	Mexico	Cold pressed, organic	Sesamum Indicum Seed Oil
16	Shea butter	Ghana	Raw, organic	Butyrospermum Parkii Butter
17	Wheat germ oil	EU	Cold pressed	Triticum Vulgare Germ Oil
18	Wild rose oil	Chile	Cold pressed, organic	Rosa Moschata Seed Oil

Table 1: Country of origin, quality and INCI of the examined vegetable oils.

ple **7** were organically produced in Lithuania, Croatia, Finland and Estonia and pressed in Lithuania, whereas the hemp seeds of the *Cannabis sativa* USO31 variety for oil sample **8** came from German organic cultivation in Oberschwaben and were pressed and filtered in Germany **(Figure 1)**.

Fig. 1 **Left:** Hemp field in Upper Swabia. **Middle:** Pressing of hemp seed oil using a screw press. **Right:** Filtration of the hemp seed oil.

A screw press was also used to obtain the jojoba oil investigated here (sample **9**). The ripe jojoba seeds (**Figure 2**, **left** and **center**) from organic cultivation in Peru were first ground and then cold-pressed using a screw press (**Figure 2**, **right**).

Fig. 2 **Left:** Ripe female jojoba fruits on the jojoba bush. **Middle:** Drying of jojoba seeds. **Right:** Pressing of jojoba oil using a screw press.

The ripe seeds of the almond tree **(Figure 3)**, the starting raw material for the native almond oil investigated in this study (sample **10**), were obtained from Spanish organic farming in the Barcelona region. Cold pressing of the ripe almond seeds was carried out by means of a stamp press.

The country of origin and production of the analyzed evening primrose oil (sample **11**) is China. The oil quality is a cold pressing of seeds from organic farming using a screw press.

The olive oil analyzed (sample **12**) is from Spanish organic farming. The oil is obtained from the ripe stone fruits by cold pressing.

The raw material for the production of the sea buckthorn fruit oil tested below (sample **13**) comes from organic farming in northern Germany. During oil production, the pulp of the sea buckthorn fruit is centrifuged and filtered, which resulted in the tested orange-red sea buckthorn pulp oil of cold-pressed organic quality.

The black cumin oil analyzed (sample **14**) is also cold pressed and the black cumin seeds used for oil pressing by screw press are of organic origin. The country of production and origin of the black cumin oil is Egypt.

Both the production of the sesame oil investigated in this study (sample **15**) and the organically grown sesame seeds originated in Mexico. The oil was pressed by mechanical cold pressing with a screw press.

Fig. 3 **Left:** Almond tree plantation in the Barcelona region **Right:** Ripe almond fruit on the almond tree.

In the case of shea butter (sample **16**), a raw organic quality was investigated. The starting raw material, shea nuts growing on the shea tree of the genus *Vitellaria paradoxa* subsp. *paradoxa*, originated in Ghana. These were crushed for the production of the shea butter analyzed, mixed with water to separate the fat, heated and filtered.

For the production of the wheat germ oil analyzed here (sample **17**), the germ buds were mechanically separated from the flour and bran of the wheat fruit during flour production and promptly cold pressed in the oil mill. The wheat used for this purpose was from the European Union.

Also produced using a cold pressing process was the wild rose oil tested in this study (sample **18**). The country of origin of the organically grown rosehip seeds from which the wild rose oil was pressed is Chile.

4. Test parameters and results of measurement

Essentially, the vegetable oils differ in their fatty acid spectrum **(Table 2)**, tocopherol content **(Table 3)** and phytosterol distribution **(Table 4)** as well as their composition. For the determination of the authenticity of the oils, depending on the oil fruit, the overall picture of the three parameters is used to determine the purity (typical fingerprint). The values determined within the project were compared with literature data [5–32]. Minor deviations from the literature values can be attributed to natural variations, origin and type of extraction.

With the exception of apricot kernel oil and almond oil, the oils examined in the project are oils whose plant origin does not belong to the same genus.

No sensory abnormalities were found for any of the oils.

In the following section, the characteristics for each of the oils studied are presented:

Apricot kernel oil (sample **1**) is one of the oils characterized by a high content of unsaturated fatty acids. The main part of the fatty acids is oleic acid and linoleic acid [5,6]. Comparable values can be analyzed for almond oil (sample **10**) and can be assumed to be characteristic for seeds of stone fruit plants (*genus Prunus*) [7]. Thus, the test for authenticity cannot be represented exclusively by the determination of the fatty acids. For this purpose, the agreement of the species-specific tocopherol content, as well as the sterol content, is additionally important. For both spectra, agreement with literature data for apricot kernel oil could be achieved.[5,6] Characteristic for almond oil is the high content of α-tocopherol compared to apricot kernel oil, as well as the overall phytosterol content [23]. The results of the study show that almond oil has a high α-tocopherol content.

A total of three different argan oils (sample **2**, **3** and **4**) were analyzed. In all three oils, the four most abundant fatty acids stearic acid, palmitic acid, linoleic acid, and oleic acid were detected in amounts characteristic of argan oil [8]. These four fatty acids can be detected in a similar order of magnitude in sesame oil (sample **15**) [12]. Major distinguishing features can be found in the tocopherol distribution of the two oils [9]. In argan oil, spinasterol and schottenol represent the major fraction of sterols analyzed [10]. In comparison, in sesame oil, the major fraction of sterols is due to β-sitosterol and to δ-5-avenasterol [11]. The main sterols are spinasterol and schottenol. In avocado oil (sample **5**) and olive oil (sample **12**), the three fatty acids palmitic acid, palmitoleic acid and oleic acid were determined in authentic contents [13,28]. In the analyzed olive oil, a higher content of α-tocopherol content was determined than in advocado oil [12]. In argan oil, a higher total content of phytosterols was detected than in olive oil.

Sample-No.	Sample designation	Myristic acid, C 14:0	Pentadecanoic acid, C 15:0	Palmitic acid, C 16:0	palmitoleic acid, C 16:1	Heptadecanoic acid, C 17:0	Stearic acid, C 18:0	Oleic acid, C 18:1 cis	Linoleic acid, C 18:2 cis	Linolenic acid, C 18:3 cis	Gamma linolenic acid, C 18:3 cis	Punicic acid, C 18:3	Stearidonic acid, C 18:4	Arachidic acid, C 20:0	Gadoleic acid, C 20:1	Eicosadienoic acid, C 20:2	Behenic acid, C 22:0	Erucic acid, C 22:1	Lignoceric acid, C 24:0	Nervonic acid, C 24:1 cis	Total saturated fatty acids	Total monounsaturated fatty acids	Total polyunsaturated fatty acids	Unit
1	Apricot kernel oil	n.d.	n.d.	4.8	0.66	0.04	1.2	71.1	21.9	0.08	n.d.	n.d.	n.d.	0.10	0.09	n.d.	n.d.	n.d.	n.d.	n.d.	6.1	71.9	22.0	g/100g sample
2	Argan oil	0.12	0.05	13.1	0.09	0.06	5.3	47.9	32.6	0.07	n.d.	n.d.	n.d.	0.32	0.25	n.d.	0.12	n.d.	0.06	n.d.	19.1	48.2	32.7	g/100g sample
3	Argan oil	0.13	0.05	13.1	0.09	0.07	5.3	48.1	32.3	0.07	n.d.	n.d.	n.d.	0.33	0.25	n.d.	0.11	n.d.	0.05	n.d.	19.2	48.4	32.4	g/100g sample
4	Argan oil	0.15	0.05	13.3	0.11	0.07	5.6	47.8	32.1	0.10	n.d.	n.d.	n.d.	0.33	0.28	n.d.	0.11	n.d.	0.05	0.03	19.6	48.2	32.2	g/100g sample
5	Avocado oil	0.07	n.d.	17.2	6.4	n.d.	0.77	66.2	8.6	0.54	n.d.	n.d.	n.d.	0.07	0.15	n.d.	n.d.	n.d.	0.04	n.d.	18.1	72.8	9.1	g/100g sample
6	Pomegranate seed oil	0.03	n.d.	3.5	0.04	0.06	2.4	9.1	7.5	0.04	n.d.	76.2	n.d.	0.44	0.69	n.d.	n.d.	n.d.	0.06	n.d.	6.5	9.8	83.7	g/100g sample
7	Hemp seed oil	0.05	n.d.	5.7	0.12	0.05	2.7	10.1	55.6	20.3	2.5	n.d.	1.1	0.83	0.39	0.07	0.34	n.d.	0.18	n.d.	9.9	10.7	79.5	g/100g sample
8	Hemp seed oil	n.d.	n.d.	6.7	0.14	0.06	2.3	8.9	55.7	19.3	3.6	n.d.	1.4	0.83	0.41	0.08	0.39	n.d.	0.21	n.d.	10.6	9.4	80.0	g/100g sample
9	Jojoba oil	n.d.	n.d.	2.5	n.d.	n.d.	0.45	13.8	1.3	0.36	n.d.	n.d.	n.d.	n.d.	65.3	n.d.	n.d.	14.4	n.d.	1.8	3.0	95.3	1.7	g/100g sample
10	Almond oil	n.d.	n.d.	6.0	0.43	0.04	1.9	72.3	19.3	n.d.	n.d.	n.d.	n.d.	0.07	0.05	n.d.	n.d.	n.d.	n.d.	n.d.	8.0	72.7	19.3	g/100g sample
11	Evening primrose oil	0.03	n.d.	5.8	0.04	0.05	1.5	5.3	78.4	0.15	8.0	n.d.	n.d.	0.23	0.21	0.03	0.08	0.07	0.04	0.05	7.8	5.6	86.6	g/100g sample
12	Olive oil	n.d.	n.d.	15.1	1.8	0.04	1.9	65.2	14.8	0.53	n.d.	n.d.	n.d.	0.31	0.16	n.d.	0.09	n.d.	0.04	n.d.	17.5	67.1	15.3	g/100g sample
13	Sea buckthorn fruit oil	0.23	n.d.	39.1	33.0	0.04	0.85	23.4	2.3	0.72	n.d.	n.d.	n.d.	0.15	0.14	n.d.	n.d.	n.d.	0.04	n.d.	40.5	56.5	3.0	g/100g sample
14	Black cumin oil	0.20	0.04	12.7	0.88	0.07	2.9	24.9	55.2	0.24	n.d.	n.d.	n.d.	0.18	0.27	2.1	n.d.	n.d.	n.d.	0.23	16.2	26.3	57.6	g/100g sample
15	Sesame oil	0.06	n.d.	9.9	0.19	0.05	5.0	37.2	46.4	0.32	n.d.	n.d.	n.d.	0.55	0.14	0.07	0.13	n.d.	0.08	n.d.	15.8	37.5	46.7	g/100g sample
16	Shea butter	0.06	n.d.	3.8	0.07	0.07	42.4	45.1	6.8	0.15	0.04	n.d.	n.d.	1.2	0.21	n.d.	0.11	n.d.	0.08	n.d.	47.6	45.4	7.0	g/100g sample
17	Wheat germ oil	0.07	n.d.	16.0	0.13	0.04	0.77	15.2	60.8	5.8	n.d.	n.d.	n.d.	0.15	0.80	0.09	0.07	0.10	n.d.	n.d.	17.1	16.2	66.7	g/100g sample
18	Wild rose oil	0.04	n.d.	3.1	0.10	0.04	1.4	13.2	46.0	35.1	n.d.	n.d.	n.d.	0.54	0.25	0.07	0.10	n.d.	0.04	n.d.	5.3	13.5	81.2	g/100g sample

n.d. = not detectable

Table 2: Fatty acid distribution (method: DGF C-VI 11e, GC-FID)

The sterol distribution in olive oil is significantly composed of β-sitosterol and δ-5-avenasterol [28].

The pomegranate seed oil (sample **6**) has authentic contents of punicic acid and of γ-tocopherol and β-sitosterol [14,15,16].

The fatty acids oleic acid, linoleic acid, and linolenic acid represent the majority of the fatty acids detected in the two hemp seed oil samples analyzed (samples **7** and **8**). Hemp seed oil can be distinguished from black cumin oil (sample **14**) by the distribution of oleic acid to linolenic acid and by the detection of stearidonic acid, which is typical for hemp [17,19]. Further distinguishing characteristics of hemp seed oil and black cumin oil are the γ-tocopherol content and the content of β-sitosterol [18,19,20]. The fatty acid content of hemp seed oil was found to be higher than that of black cumin oil.

Analysis of the fatty acid spectrum of jojoba oil (sample **9**) showed the highest content of unsaturated fatty acids. This can be attributed to a high content of gadoleic acid, which is only characteristic for jojoba oil [21]. Furthermore, the sterols β-sitosterol and campesterol, which are specific for jojoba oil, could be determined [22].

Sample-No.	Sample designation	α-Tocopherol	β-Tocopherol	γ-Tocopherol	δ-Tocopherol	Total tocopherol equivalents (vitamin E)	Unit
1	Apricot kernel oil	2.6	n.d.	48.5	1.9	14.8	mg/100g sample
2	Argan oil	3.8	n.d.	31.5	2.4	11.8	mg/100g sample
3	Argan oil	3.6	n.d.	30.3	2.4	11.3	mg/100g sample
4	Argan oil	3.8	n.d.	36.4	2.4	13.0	mg/100g sample
5	Avocado oil	7.6	n.d.	1.3	n.d.	8.1	mg/100g sample
6	Pomegranate seed oil	4.6	n.d.	211.5	6.6	57.5	mg/100g sample
7	Hemp seed oil	4.5	0.52	66.0	2.8	21.3	mg/100g sample
8	Hemp seed oil	6.0	0.68	72.1	4.2	24.4	mg/100g sample
9	Jojoba oil	3.3	n.d.	3.2	n.d.	4.2	mg/100g sample
10	Almond oil	37.4	0.52	1.3	n.d.	38.0	mg/100g sample
11	Evening primrose oil	19.3	n.d.	25.6	4.9	26.0	mg/100g sample
12	Olive oil	21.6	n.d.	0.70	n.d.	22.0	mg/100g sample
13	Sea buckthorn fruit oil	59.1	3.1	n.d.	n.d.	60.7	mg/100g sample
14	Black cumin oil	9.3	n.d.	0.84	n.d.	9.6	mg/100g sample
15	Sesame oil	n.d.	0.60	55.3	1.1	14.2	mg/100g sample
16	Shea butter	8.4	0.55	1.4	n.d.	9.0	mg/100g sample
17	Wheat germ oil	154.3	81.6	18.2	1.0	199.7	mg/100g sample
18	Wild rose oil	22.1	1.2	85.1	7.7	44.1	mg/100g sample

n.d. = not detectable

Table 3: Tocopherols (method: ASU L 00.00-62, HPLC-FLD)

Sample-No.	Sample designation	Sterols (total) in mg/kg	Cholesterol	Brassicasterol	24-Methylene cholesterol	Campesterol	Campestanol	Stigmasterol	delta-7-Campesterol	delta-5,24-Stigmastadienol	Chlerosterol	beta-sitosterol (apparent)	Sitostanol	delta-7-Avenasterol	delta-5,23-Stigmastadienol	delta-7-Stigmastenol	delta-5-Avenasterol	Spinasterol	Schottenol	Stigmasta-8,22-dien3-ol	Stigmasta-8,(14)-en3-ol	Unit *
1	Apricot kernel oil	3253	n.d.	n.d.	0.1	4.6	0.2	0.5	n.d.	1.2	0.9	79.7	2.8	0.3	n.d.	0.3	9.2	n.d.	n.d.	n.d.	n.d.	%
2	Argan oil	1750	0.1	n.d.	n.d.	0.3	0.5	0.1	1.4	1.4	n.d.	n.d.	0.6	4.4	0.6	n.d.	0.1	39.2	44.2	3.1	3.9	%
3	Argan oil	1530	0.1	n.d.	n.d.	0.3	0.5	0.2	1.0	1.3	n.d.	n.d.	0.6	4.4	0.5	n.d.	0.2	40.1	43.7	3.4	3.8	%
4	Argan oil	1414	0.1	n.d.	n.d.	n.d.	0.6	0.4	1.3	1.9	n.d.	n.d.	0.8	4.6	0.7	n.d.	0.3	37.5	44.6	3.3	3.9	%
5	Avocado oil	4485	0.1	n.d.	1.2	5.3	n.d.	0.3	n.d.	0.3	1.7	83.7	0.5	0.2	n.d.	n.d.	6.7	n.d.	n.d.	n.d.	n.d.	%
6	Pomegranate seed oil	5434	n.d.	n.d.	0.1	7.6	0.1	4.5	0.1	n.d.	1.2	86.2	0.4	3.3	0.7	0.5	0.8	n.d.	n.d.	n.d.	n.d.	%
7	Hemp seed oil	4574	0.2	n.d.	0.9	15.5	0.3	2.2	0.4	0.9	0.6	78.8	1	1.2	n.d.	0.6	7.9	n.d.	n.d.	n.d.	n.d.	%
8	Hemp seed oil	5994	0.2	n.d.	0.8	15.2	0.3	1.8	n.d.	n.d.	0.6	80.1	1	8	1.3	0.5	1.2	n.d.	n.d.	n.d.	n.d.	%
9	Jojoba oil	3058	0.3	n.d.	0.2	19	0.3	6.1	n.d.	1	0.9	73.9	0.3	0.2	n.d.	n.d.	5.9	n.d.	n.d.	n.d.	n.d.	%
10	Almond oil	2131	0.1	n.d.	n.d.	2.8	n.d.	0.7	0.2	1.4	1.2	78.3	1	0.8	n.d.	0.4	13	n.d.	n.d.	n.d.	n.d.	%
11	Evening primrose oil	8522	0.1	n.d.	0.3	8.1	0.2	0.3	0.2	0.7	0.8	82.7	1.4	0.5	n.d.	1.1	3.8	n.d.	n.d.	n.d.	n.d.	%
12	Olive oil	1973	0.1	n.d.	0.3	3.3	n.d.	0.6	n.d.	0.7	0.9	81.1	0.4	0.7	n.d.	0.4	11.5	n.d.	n.d.	n.d.	n.d.	%
13	Sea buckthorn fruit oil	6258	n.d.	n.d.	n.d.	2.4	n.d.	n.d.	0.4	1.1	1.2	84.9	1.1	1.3	n.d.	6.8	0.7	n.d.	n.d.	n.d.	n.d.	%
14	Black cumin oil	1857	0.8	n.d.	1.3	11.4	0.5	12.7	0.4	1.8	1.2	52.8	2.7	2.6	n.d.	0.9	10.8	n.d.	n.d.	n.d.	n.d.	%
15	Sesame oil	6340	n.d.	n.d.	3.2	19	n.d.	6.2	0.4	1.2	0.74	53.3	0.2	1.0	n.d.	0.3	14.4	n.d.	n.d.	n.d.	n.d.	%
16	Shea butter	1429	0.2	n.d.	n.d.	1.8	n.d.	0.8	7.2	3.9	n.d.	43.7	n.d.	1.8	n.d.	35.5	5	n.d.	n.d.	n.d.	n.d.	%
17	Wheat germ oil	33422	0.1	0.2	n.d.	23.7	1.2	1.0	1.2	0.9	0.6	61.5	1.7	2.4	n.d.	1.8	2.4	n.d.	n.d.	n.d.	n.d.	%
18	Wild rose oil	4352	1.0	n.d.	n.d.	3.3	n.d.	2.0	n.d.	2.1	1.2	79.0	0.6	1.0	n.d.	1.9	7.9	n.d.	n.d.	n.d.	n.d.	%

n.d. = not detectable
* = based on total content

Table 4: Sterols (method: in-house method, LG-GC-FID)

The fatty acid spectrum of evening primrose oil (sample **11**) was found to have the highest content of unsaturated fatty acids. In evening primrose oil (sample **11**), the content of linoleic acid typical for authentic evening primrose oil was detected [24]. Thus, the fatty acid spectrum differs decisively from that of advocado oil or olive oil. For differentiation, the content of γ-tocopherol as well as the total content of sterols can be used as a supplement [25].

In olive oil (sample **12**), the three main fatty acids palmitic acid, oleic acid, and linoleic acid could be detected [28]. In tocopherol distribution, mainly α-tocopherol was determined in authentic contents [12]. Sterol distribution is mainly composed of β-sitosterol and δ-5-avenasterol [28].

Authentic sea buckthorn fruit oil (sample **13**) is characterized by palmitic acid and palmitoleic acid in combination with α-tocopherol content [26].

The ratio of stearic acid and oleic acid determined in shea butter (sample **16**) was not detected in any other of the vegetable oils studied [27]. Compared to the other oils, shea butter has a high content of δ-7-stigmastenol.

Wheat germ oil (sample **17**) stands out from the other oils due to high levels of tocopherol and phytosterols [29].

In the fatty acid spectrum of wild rose oil (sample **18**), linoleic acid and linolenic acid make up the bulk of the analyzed fatty acids [30,31]. The high content of α-tocopherol and the low content of campesterol, for example, allow wild rose oil to be distinguished from hemp seed oil [30,32].

5. Summary

As part of this project, the authenticity of a wide variety of vegetable oils for use as cosmetic raw materials was tested. These data provide a good overview of the authenticity of these raw materials.

For all samples examined, comparison of the analytical data with literature data and sensory control did not reveal any abnormalities with regard to possible adulteration.

Furthermore, reliable and trustworthy raw material suppliers play an important role. This should be regularly checked within the framework of on-site audits.

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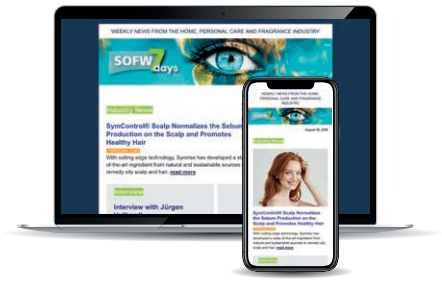

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Image 1. Cherishd. Supreme Superfood Cream packed in mycelium tertiary packaging and seeded paper.

Talking Trash – The Future of Zero-Waste Beauty

A. Crovetto et al.

Introduction

Sustainability is not a trend but a must as the impact of climate change is now felt in real-time. With growing climate anxiety and increasing green legislation, personal care companies are faced with an escalating pressure to improve their environmental footprints, as quickly as possible, while still maintaining financial profits [1]

According to the Sustainable Beauty Coalition, a survey of 23,000 beauty shoppers found almost half (48%) are looking for more information and clarity on brands' values and commitments to the environment [2]. This is supported by the research of Provenance, a global leader in sustainability communications technology, which has shown that 79% of beauty shoppers have doubts about the trustworthiness of sustainability claims, highlighting the issues of greenwashing within personal care [3].

The industrial economy runs primarily on a linear 'take-make-dispose' model. It's no secret that this overconsumption of the earth's resources, coupled with the amount of waste produced, is having a detrimental impact on our planet. But this impact can be mitigated by refocusing efforts on a circular economy, centred on waste prevention and reuse.

Circular beauty is a green business model that focuses on repairing, reusing, and extending a product's life cycle by minimising waste across all aspects of the supply chain. To achieve circular beauty, businesses must look at every step in the creation of a product; from the ingredients used to how packaging is disposed of. Fortunately, this zero-waste movement, which seeks to redesign resource lifecycles, is gaining momentum. Companies of all shapes and sizes are strategizing to eliminate waste across their supply chains to reduce their environmental impact [4]. This zero-waste transition improves transparency throughout the supply chain and helps beauty consumers to identify authentic sustainability actions. With a focus on ingredients and formulas, as well as packaging materials, let's explore the future of zero-waste beauty. Let's talk trash.

Ingredients & formulations

A recent survey by Avery Dennison found that more than 10% of beauty products, worth an estimated $4.8bn, are going to waste throughout the beauty supply chain. Overproduction and excess inventory were attributed the biggest problems, accounting for 6.2% of discarded goods [5].

From rescued products to upcycled actives, we investigate ways to reduce and reuse waste across different stages of the beauty supply chain:

RESCUED PRODUCTS – Formula changes, updated pack design or new branding are just a few reasons leading to wasted inventory, even though the products are still excellent quality. However, brands like Stop the Water While Using Me and Krave Beauty are changing the way waste is dealt with and normalizing the industry conversation around waste.

Stop the Water While Using Me, a Hamburg start-up bought by Beiersdorf in 2020, offers their rescued line of imperfect products which can be purchased at a discount, rather than being thrown away. They say: "*Products with minor flaws should not end up in the garbage can. They deserve a second chance. Whether it's a small dent in the jar, a misprinted bottle, a shorter shelf life or simply overproduced goods: throwing them away is out of the question for us*" [6].

In 2022, "*slow-down skincare*" brand Krave Beauty also launched their "*Waste-Me-Not*" campaign, which showcased a discarded pilot batch transformed into a limited-edition Matcha Hemp Body Wash. Following production of a re-formulated version of their bestselling Matcha Hemp Hydrating Cleanser, the brand was left with more than 4500 litres of bulk that didn't meet specification. Turning lemons into lemonade, Krave Beauty reworked the formula to create a body wash. The product is packed in a pouch that uses less plastic, water, and energy than regular bottles. A "*mistake turned magic*", the body wash retails for a discounted $8, to cover costs [7].

Unused products sitting at home, unsold or returned products, and items that expire in warehouses are all contributing to land fill. An estimated 4% of stock goes to waste due to perishing,

spoiling or damage – a growing concern for both brands and the environment [5].

Arkive is a Beautytech company from the Netherlands with a mission to make the beauty industry more circular through the power of data. They enable beauty companies towards a more sustainable supply chain while reducing waste. One of the Arkive products is a marketplace and solution for surplus beauty inventory focusing on transparent re-commercing towards a purpose-driven beauty citizen, as well as the recycling and repurposing of beauty products. Founder Sinem Tuncer was inspired by the vast quantity and value of unconsumed beauty products ending up in landfill every year. She developed Arkive as a destination to shop unconsumed beauty products, but the platform has since grown into a solution-driven waste fighting movement [8].

FINDING VALUE IN FOOD WASTE – Did you know that one-third of all food produced globally goes to waste? That amounts to approximately 1.3 billion tonnes per year. While this may seem like an issue we can help solve at home, an estimated 40% of fruits and vegetables are discarded simply because of their appearance, not even making it to supermarket shelves [9]. In addition to wasting perfectly good produce, the other looming issue is methane. When food is sent to landfill it decomposes and becomes a significant source of methane, a potent greenhouse gas with 21 times the global warming potential of carbon dioxide [10].

According to Project Drawdown, the world's leading resource for climate solutions, reducing food waste is a key factor to mitigate the climate crisis [11]. Upcycling food by-products and 'imperfect' produce presents an opportunity to create new materials using minimal energy. The personal care industry is finding value in plant-based leftovers with many raw material suppliers creating effective yet sustainable products. By harnessing the power of phytocompounds already present within food waste, these companies are revealing that upcycled ingredients are not just effective, but in some cases, proven to outperform their non-upcycled counterparts.

This zero-waste focus can also help to promote local communities and fosters local partnerships. Collaborating with a local gin distillery in the UK, The Upcycled Beauty Company takes leftover grain ferment from the gin making process. This is captured fresh, straight from distillation, and combined with an upcycled and palm-free glycerine to eliminate the need for any preservative **(Figure 1)**. We're able to upcycle 4 litres of gin distillery leftovers from every bottle of gin. No new materials are created – no existing resources are wasted. This upcycled active quenches thirsty skin by offering long-lasting hydration and delivers three times more moisture than glycerine alone. Shown to reduce the pro-inflammatory cytokine IL-8, this ferment also helps calm and soothe stressed-out skin.

BRING YOUR OWN WATER – Water is often considered a limitless resource, but demand is beginning to outweigh supply, with Global Market Researcher Mintel crowning it "the new luxury" ingredient. "*Water is set to be a precious commodity as consumption outstrips supply*," the company's Global Beauty and Personal Care Analyst explained [12].

As consumers become increasingly aware of the detrimental effect of excessive water usage, more beauty brands are embracing waterless formulations. Waterless skincare and haircare focus on activating solid or powdered formulas with water at home, helping to limit the overuse (and unnecessary shipping) of water within cosmetic formulations. In addition to reducing

Fig. 1 The Upcycling Process - Fermented Grain

water wastage, powder or solid formats can be packed more sustainably, utilising compostable materials, or go one step further by embracing a packaging-free, 'naked' format like eco-brands, Lush, Disruptor London and SBTRCT. This results in more concentrated formulas, fewer carbon emissions and less packaging materials being used and then going to waste.

Packaging & materials

79% of plastic waste from beauty packaging ends up in landfills, dumps, or the environment. If current levels of consumption continue, our oceans could contain more plastic than fish by 2050, warns the UN Environment Programme. In the EU, new packaging legislation highlights the challenges that the personal care industry faces. Since the early 90s packaging volumes have increased due to a decline in reuse and refill schemes and exacerbated by increased eCommerce and on-the-go consumption. According to the European Commission, each European generates ~180 kg of packaging waste per year. Packaging is the primary user of virgin materials accounting for 40% of plastics and 50% of paper. Without action, by 2030, packaging waste would increase by an additional 19% and for plastic packaging waste a 46% increase [13].

Announced in November 2022, the proposed Regulation 2022/0396 on packaging and packaging waste (amending Regulation (EU) 2019/1020 and Directive (EU) 2019/904, and repealing Directive 94/62/EC), aims to stop this trend. For consumers, the regulation aims to ensure reusable packaging options, the removal of unnecessary packaging, limits to overpackaging and the provision of clearer labelling to support correct recycling. For industry, they aim to develop new business opportunities while decreasing the need for virgin materials and boosting Europe's recycling capacity. The proposal applies to all packaging (regardless of the material used) and to all packaging waste, essentially covering the entire product life cycle. The target is to put the EU packaging sector on track for climate neutrality by 2050.

While waste statistics are sobering and legislation can appear limiting, there are plenty of companies already working towards a more sustainable future for packaging with innovative concepts and materials that are helping to divert the use of virgin materials and waste from landfill.

RETURN & REUSE – Refill schemes come with their challenges and a shift in consumer behaviour is required to make them work. Incentives, such as a deposit return scheme, may help to encourage wider consumer engagement. Haeckels is a UK brand that combines design and sustainability; their refillable aluminium deodorant case has a never-ending recyclable life. Instead of buying the case to keep, customers rent it for as long as they need by placing a cash deposit. The case can be reused as often as needed. Once finished, the customer simply returns the case to get their deposit back. The purpose of the deposit is to offer full responsibility for the product – closing the loop.

For decades, The Body Shop has been looking for ways to empower customers to opt out of unnecessary packaging via refill schemes. Long before refillable packaging became popular, The Body Shop founder Anita Roddick encouraged customers to return their bottles to be reused, and in 1993 the company launched an in-store recycling program called '*Bring Back our Bottle*'. 20 years ago, The Body Shop had refill stations in their stores, but at that time, the retail environment was changing, and everyone was moving to plastics. Now, that conversation has evolved to the point that customer demands have come full circle. Hoping a new generation of consumers will adopt the refill practice, The Body Shop have rolled out refill stations across 400 stores globally in 2021 and a further 400 throughout 2022.

UNIQUE MATERIALS – From bio-based polymers to popcorn polystyrene alternatives, there are many unique materials and packaging concepts that provide insight into what's possible when it comes to designing high-impact packaging solutions with minimal waste.

From snacking to packing, popcorn has been given a new purpose. Researchers at the University of Gottingen have reimagined the snack to create an environmentally friendly alternative to polystyrene and plastic packaging. This upcycled plastic alternative is made from the inedible by-product of cornflakes and can be composted after use, leaving behind zero-waste. The University has now signed a license with Nordgetreide, a German cereal manufacturer that specialise in premium milled products, for the commercial use of the process and products for the packaging sector [14].

Instead of polluting the earth, 'bio-contributing' packaging boosts biodiversity by adding nutrients to the soil as it breaks down. Demonstration brand Cherishd. (developed by The Upcycled Beauty Company) showcases a 100% upcycled Supreme Superfood Cream formula housed in secondary and tertiary packaging made from mycelium and seeded paper **(Image 1)**. The mycelium box and the seeded paper can be planted together, with the mycelium encouraging the seeds' growth - a literal seed bomb!

Sprout World is an eco-pencil brand that has launched a patented plantable eyeliner, developed with a microplastic free formula. Designed to be planted after use, the eyeliner contains a cellulose seed capsule at the end that can be placed in soil, watered frequently, and eventually transformed into bee-friendly wildflowers. While the eyeliner is housed in certified sustainably sourced wood, the cap is made from fully recyclable bioplastic to create an eco-friendlier alternative to traditional cosmetic pencils [15].

Proverb is a UK-born beauty brand that is committed to transitioning its entire range to sustainable packaging. Already, they have created a compostable beauty pouch that can hold liquid formulas. Currently being trialled to house skincare emulsions for sampling, the compostable paper-based packaging is said to be the first of its kind, made from layers of paper and a biofilm from wood pulp cellulose. These sachets are designed to be compostable at home and with ambient temperatures, they turn back into water, CO_2 and biomass without ecotoxicity for the soil [16].

Clean Filter Packaging from the USA have a vision to clean up the packaging industry by offering a sustainable alternative to plastic. The company has developed the patent pending BAMBU packaging, designed to be compatible with a range of personal care formulas and with an eco-friendly end of life disposal. This alternative material is made with upcycled bamboo and bio-resins from renewable sources and is free from additives making it non-toxic to the environment when broken down [17].

Summary

The zero-waste beauty movement focuses on reducing the consumption of new resources, identifying waste as a valuable resource, and supporting the transition from a linear take-make-dispose system to a circular one. As a global community of beauty citizens, we each have our part to play in the transformation of industrial systems and reducing waste. Green manufacturing, sustainable material selection and ethical ingredient sourcing are all great places to start on the path to zero-waste. By transitioning to upcycled ingredients in personal care we reduce the consumption of primary resources and reduce competition with the food chain. There are also many existing innovations that can be adopted by beauty brands looking to make greener choices and reduce emissions. From refill schemes to packaging that gives back more to the environment than it takes, the increasing availability of sustainable technologies and alternatives on the market today help reduce the production of carbon and take us one step closer to building a circular beauty economy with more authentic sustainability actions. Changes in legislation are also essential to help drive circular innovation and eco-design creating holistic industrial processes which encompass the entire product life-cycle and end-of-life management.

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Recommendation for the Quality Assessment of Paint Care Products for Motor Vehicles

Part 3: Paint Polish (Translation / Original: German)

Section Household Care in the German Cosmetic, Toiletry, Perfumery and Detergent Association (IKW), Frankfurt am Main

1. Foreword

German Cosmetic, Toiletry, Perfumery and Detergent Association (IKW) member companies make their expert knowledge of the products they manufacture available to the general public; this is done in the form of quality assessment recommendations.

The recommendations for the quality assessment are elaborated in working groups and are intended to enable a qualified testing of the relevant products by the manufacturers and test institutes. Quality characteristics are described that need to be fulfilled by the products concerned in order to achieve the effects expected by consumers and manufacturers.

The companies working within the framework of IKW want optimal quality standards for their products. Their aim is a consistent orientation to sustainability as a guiding principle, preparing to successfully face the future in a constantly changing world.

This commitment to sustainability as a guiding principle is built up on experiences expressed in numerous exemplary initiatives. Taking as starting points the declarations of Rio 1992, "92 plus 10" of Johannesburg and the Agenda 21, sustainability is understood as a balanced linking of economic, social and ecological aspects, with a view to meeting the needs of the present without compromising the ability of future generations to meet their own needs.

The member companies of the IKW have long been committed to sustainability under the umbrella of the association and sister federations. This commitment has already resulted in several established industry-specific initiatives, such as:

- Dialogue platform FORUM WASCHEN [1],
- IKW Report on sustainability in the detergents, maintenance and cleaning products industry [2],
- A.I.S.E. Charter for Sustainable Cleaning ("Charter 2020+") of the International Association for Soaps, Detergents and Maintenance Products (A.I.S.E.), Brussels [3],
- voluntary agreements [4].

Furthermore, the member companies are also committed within initiatives of raw material and supplier industries, for example:

- The "Responsible Care" initiative of the chemical-pharmaceutical industry and the chemicals trade in Germany [5],
- "Chemie³", the sustainability initiative of the German Chemical Industry Association (VCI), the Mining, Chemical and Energy Industrial Union (IG BCE) and the German Federation of Chemical Employers' Associations (BAVC) [6]

The constant further development of initiatives and products with sustainability as the guiding principle ensures the future viability of the detergents, maintenance and cleaning products industry in a constantly changing world. The social and societal benefits of these products in terms of hygiene and value-preservation are undisputed. The products make a significant contribution to today's standard of living and health and to the conservation of resources, for example by extending the service life of objects such as motor vehicles.

With this in mind, quality assessment recommendations encourage company staff to act responsibly toward humans and the environment in product development and manufacture. They also serve consumers who can expect efficient, safe and environmentally sound products.

The recommendations describe which qualities are relevant to a given product and how such qualities can be measured. It should be noted that every finished product has a certain efficacy spectrum in its intended use; this spectrum is largely determined by consumer expectations as to each individual quality characteristic – so that in each product some characteristics are deliberately emphasised while others will be less important. Moreover, the desired combination of product properties is subject to constant change, depending on the latest technical possibilities and new consumer habits.

Quality assessment recommendations must not impair such developments. Consequently, for each product only one overall result is valid to determine whether the product meets the quality recommendations or not. Emphasis on isolated test criteria is not admissible and may be misleading.

2. Rules, Standards and Voluntary Agreements

With regard to composition, packaging and labelling, inter alia, the following statutory requirements must be observed in their existing versions or to the extent that they still apply, respectively:

- German Code on Foodstuffs, Consumer Items and Animal Feed (Lebensmittel-, Bedarfsgegenstände- und Futtermittelgesetzbuch – LFGB)
- German Chemicals Act (Chemikaliengesetz – ChemG)
- German Dangerous Substances Ordinance (Gefahrstoffverordnung – GefStoffV)
- Chemicals Prohibition Ordinance [Chemikalienverbotsverordnung – ChemVerbotsV)
- German Detergents and Cleaning Products Act (Wasch- und Reinigungsmittelgesetz – WRMG)
- German Ordinance on Pre-packaged Products (Fertigverpackungsverordnung – FPV)
- German Ordinance on the Transport of Dangerous Goods by Road (Gefahrgutverordnung Straße – GGVS)
- German Ordinance on the Transport of Dangerous Goods by Rail (Gefahrgutverordnung Eisenbahn – GGVE)

as well as the following legislation by the European Union which serves as basis for the German ordinances or to which reference is made:

- Detergents Regulation (EC) No 648/2004
- REACH Regulation (EC) No 1907/2006
- Regulation on Classification, Labelling and Packaging (EC) No 1272/2008 ("CLP Regulation")
- Biocidal Products Regulation (EU) No 528/2012
- Regulation (EU) No 98/2013 on the Marketing and Use of Explosives Precursors

The following international standards were taken into account in respect of individual aspects:

- ASTM D3836-13 (USA): "Standard Practice for Evaluation of Automotive Polish"
- DIN 55660-1:2011-12:"Paints and varnishes – Wettability – Part 1: Terminology and general principles"
- DIN EN ISO 2813 (June 1999, updated 2015): "Paints and Varnishes – Determination of Gloss Value at 20°, 60° and 85°"

Moreover, the following voluntary agreements [4] apply, amongst others, to IKW member companies which can be relevant for paint care products:

- Ban of the Use of Alkylphenol Ethoxylates (APEO) (1986)
- Ban on Ethylenediaminetetraacetic Acid (EDTA) (1991)

3. Introduction

This Recommendation for the quality assessment includes test methods for the assessment of paint care products for motor vehicles. They are applied on larger, painted body components and can be classified in accordance with **Diagram 1** in respect of their polishing, paint conditioning and cleaning properties in a product group triangle. The considered product groups differ in terms of these properties. The boundaries between the product groups are fluid in accordance with the representation in **Diagram 1** and are partly only determined by the application concentration of certain ingredients.

According to **Table 1**, the following typical ingredients and assessment criteria can be assigned to the three properties or corners of the product group triangle in **Diagram 1**.

The paint surfaces are usually two-component paint systems which are used in the automotive industry. The products are applied as a rule with an application medium (e.g. sponge, cloth).

Diagram 1 Representation of the paint care product groups in a "product group triangle". The three product groups covered by this recommendation are shown in circles (*the product group of paint conditioners may also include hard waxes or sealants).

	Property	Typical Ingredient Groups	Assessment Criteria
"Polishing"	Polishing	Abrasives	Gloss
"Preserving"	Paint conditioning	Functional silicones and waxes, polydimethylsiloxanes	Gloss, hydrophobing
"Washing / Cleaning"	Cleaning	Surfactants	Cleaning performance

Tab. 1 Typical ingredient groups and assessment criteria for the properties of paint care products from **Diagram 1**.

The paint surfaces are heavily exposed to daily strains and soiling and are usually cleaned prior to the application of the above-mentioned three product groups (paint cleaner, paint conditioner, paint polish) e.g., by a car wash (car shampoo). The three product groups (paint cleaner, paint conditioner, paint polish) are as a rule used for value preservation and optical upgrading of the paint surfaces and differ in terms of composition of the ingredients and proper use. The products are commercially available in bottles, tubes or glass jars, tin cans or plastics boxes or other containers as well as in other presentations. They are available in a liquid, solid or pasty condition.

Paint cleaners for motor vehicles are used for older, already matt or scratched, pre-cleaned paint surfaces and prepare the paint for the subsequent application of paint conditioners or paint polish. Paint cleaners are products which contain a particularly high share of abrasives. They serve for the removal of weathered, loose pigment and paint particles as well as superficial scratches and scrapes on strongly affected already matt paint surfaces and paint layers. In accordance with their claim in conjunction with Article 2 of the Detergents Regulation (EC) No 648/2004, they are detergents and require labelling, inter alia, in accordance with Annex VII of the Detergents Regulation. In addition, a list of ingredients must be made available on the internet.

Paint conditioners for motor vehicles are usually abrasive-free and contain Gloss, water-repelling components such as waxes and silicones, for the conditioning and sealing of paint surfaces. After their application and subsequent polishing, they provide the paint with a high-gloss look. They protect and condition the paint. Paint conditioners are intended for the care of intact new paints as well as paints pre-cleaned with paint cleaner or paint polish and are, according to their intended use, not to be detached after the single cleaning with a detergent. Consequently, they come neither under the German Detergent and Cleaning Product Act (WRMG) nor under the Detergents Regulation (EC) No. 648/2004.

Paint conditioners, which are, however, mainly detached after a single cleaning with detergents and can then, based on experience, reach waters, come in accordance with § 2 Para 1 Sentence 2 No. 3 under WRMG. In this case, they do not need to be labelled in accordance with the Detergents Regulation, but manufacturers must publish no later than from the placing on the market a list of ingredients on the internet in accordance with Annex VII Section D of the Detergents Regulation. Additionally, it makes sense to print the internet address that leads to the list of ingredients on the packaging.

Paint polishes for motor vehicles are combination products of paint cleaners and paint conditioners and include abrasives as well as paint protecting components. They serve for the optical improvement of already affected paint surfaces. After polishing they provide the paint again with high gloss and protection. Water-repelling components, such as waxes and silicones, serve for the conditioning and sealing of paint surfaces. Paint polishes, which are also claimed for cleaning, are detergents within the meaning of Article 2 of the Detergents Regulation (EC) No 648/2004.

Without a cleaning claim they represent as a rule products in accordance with § 2 Para 1 Sentence 2 No. 3 WRMG which are intended to be applied to surfaces and are primarily detached after a single cleaning with detergents and according to experience can then reach waters. In this case, they do not need to be labelled in accordance with the Detergents Regulation, but manufacturers must publish no later than from the placing on the market a list of ingredients on the internet in accordance with Annex VII Section D of the Detergents Regulation. Additionally, it is recommended to print the internet address that leads to the list of ingredients on the packaging.

4. Aim

In 2014 the Working Group "EQ Paint Care Products" was mandated by the IKW Expert Committee on Cleaning and Care Products to revise the "IKW Recommendations on the Quality Assessment for Car Care and Cleaning Products" of 1992. The work within the working group involved both experts from industrial companies and also from a test institute. The updated recommendation represents a collection of methods which are to permit in their non-binding form a qualified testing of the relevant products for the application at private end consumers by the companies themselves, by the consumers and by the test institutes. The recommendation makes available three separate test methods for the following three products groups *(cf.* ***Diagram 1****)*:

1. **Paint cleaners** for motor vehicles (Part 1 of the Recommendation for the quality assessment of paint care products for motor vehicles)
2. **Paint conditioners** for motor vehicles (Part 2 of the Recommendation for the quality assessment of paint care products for motor vehicles)
3. **Paint polishes** for motor vehicles (Part 3 of the Recommendation for the quality assessment of paint care products for motor vehicles)

PLEASE NOTE:
Part 1 *("Paint cleaners for motor vehicles")* was already published in the SOFW Journal 11/18, volume 144:
https://www.ikw.org/fileadmin/IKW_Dateien/downloads/Haushaltspflege/1811_EQ_Lackreiniger_EN_final.pdf

Part 2 *("Paint conditioners for motor vehicles")* was published in the SOFW Journal 4/22, volume 148:
https://www.ikw.org/fileadmin/IKW_Dateien/downloads/Haushaltspflege/2022_EQ_Lackkonservierer_EN_final.pdf

The three test methods are to fulfil the following criteria:
✓ Practical relevance
✓ Precision and reproducibility
✓ Differentiability
✓ As simple conduct as possible

In order to fulfil these criteria, the tests are to be conducted in blind studies additionally with reference products in respect of which the testers do not know whether they test a reference or a test product. The reference products can be manufactured based on the information in the **Appendix** to the test methods. **Neither the reference products nor the individual chemicals or test specimens, equipment or auxiliary materials can be obtained from the IKW office.**

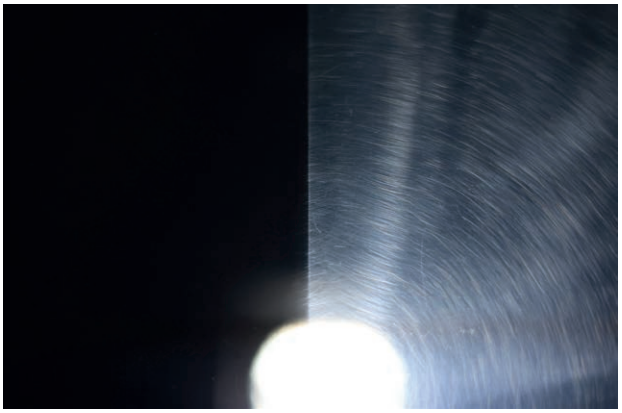


Fig. 1 Polishing effect: A weathered or scratched paint surface **(right)** compared with a paint surface polished with a paint polish **(left)**. *(Image source: SCHOLL Concepts GmbH)*

5. Paint Polish, Paint Surfaces a nd Application Method

After their application and subsequent polishing, paint polishes give the paint a high gloss look and protect it. To obtain reproducible measured values for a gloss increase, a slightly matted paint surface is therefore produced in the test method **(Figures 1** and **2)**.

Fig. 2 Roll-off effect: Paint surface treated with a paint polish on which water droplets roll off. (*Image source: Dr. O.K. Wack Chemie GmbH*)

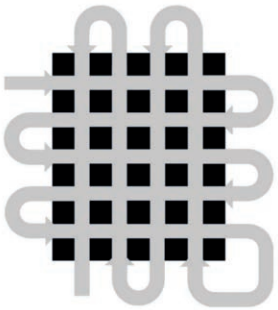


Diagram 2 Schematic representation of a full crosswise application **(grey)** with outline of the treatment surface **(black)**.

The working procedure stated in the test method for the application and/or polishing of the product is based on cross application. The application and/or polishing is carried out in accordance with **Diagram 2** with 50% overlapping of the wiping paths and beyond the edge of the surface to be treated [7].

A video with instructions for crosswise application is available at the following web address: https://www.youtube.com/watch?v=uyYTBKiJi9c&feature=youtu.be.

Note:
In the work procedure, however, it is essential to avoid material carry-over between the different test areas (cf. below **Diagrams 5** and **6**), as otherwise a falsification of the measurement results must be expected.

6. Test method for paint polishes for motor vehicles

One or several paint polishes (in the following "test paint polishes") are tested. For comparison and assessment of the test paint polishes, a specified reference paint polish with a defined formulation is used for certain test parameters (*cf.* ***Appendix***).

The setup (*cf. section 4.3.2 in the following table of the test method*) needed for determining the water run-off behaviour on glossy sheets is shown in the following schematic representation **(Diagram 3)**:

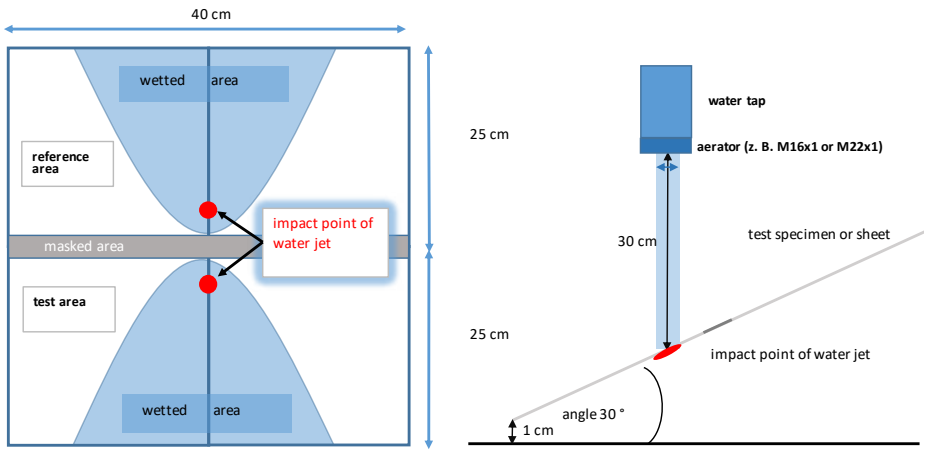


Diagram 3 Schematic representation of the water run-off method with a water jet (**left:** test specimen or test sheet with schematic representation of the parabolic course of the water jet with impact point on the reference or test side; **right:** schematic side view of the impact of a water jet on the test sheet with 1 cm distance from the washbasin or bottom of the water collection vessel for free run-off of the water).

Additionally, a video on the water run-off method using a water jet is available at the following web address: https://www.youtube.com/watch?v=6jiliU4NJ70

Further specifications and information on the test sheets, measuring instruments, equipment, setups, auxiliary materials, formulations, chemicals and supply sources are listed in the ***Appendix***.

Note:

The assessment of the tests is very demanding. Therefore, it should be carried out exclusively by persons who are familiar with the use of paint polishes and the performing of laboratory tests. At least a double determination of the test parameters is necessary to find outlier values among the test data.

In order to secure the results statistically, the tests should be performed ideally by three experienced persons independently from one another. The manufacture of the matted and glossy test sheets (5.1.1 and 5.2.1) should be done by the same person for a test series. The products to be tested should be made available to the testers in anonymised form.

The tests are carried out at a temperature between 20 and 25 °C and a relative air humidity of 20 to 80%. The test conditions should be kept constant for all tests in a test series (same temperature and same air humidity).

Paint polishes are tested on two different types of sheets (matte and glossy). For the testing of different paint polishes, the pre-treatment of the test sheets must take place on the same day. The time interval between pre-treatment and application of the paint polish for subsequent testing must be the same for all products and within the same day. The sequence of tests of the two types of test sheet should be adhered to for efficient work!

In the work procedure, it is essential to avoid material carry-over between the different test areas on one test sheet, as otherwise a falsification of the measurement results must be expected!

As a rule, there are no uniform starting conditions for paints on vehicles. To level out such different conditions for the testing of paint polishes, the tests are carried out on uniformly pre-treated paint sheets. The test conditions are kept as close to practice as possible and are largely oriented to the average consumer behaviour. To facilitate matters and for automated evaluation, a table in Excel format in **Chapter 7** *can be used.*

Test Method for Paint Polish for Motor Vehicles		
1.	**General information on the test paint polish**	
1.1	Product designation (including brand name) of the test paint polish	
1.2	Manufacturer and/or distribution	
1.3	Scope of application	
2.	**Documentation of the test conditions (temperature, air humidity) during the subsequent performance of tests** … °C … % air humidity	
3.	**Properties of the test paint polish in the state as delivered**	
3.1	Form of presentation (e.g. solid, liquid, pasty)	
3.2	Container (e.g. bottle, tube, can)	
3.3	Intended form of application (e.g. using a cloth, a sponge or as spray)	
4.	**Reference formulation; test sheet and special setups for preparation**	
4.1	Reference paint polish	Reference paint polish with defined formulation *(cf.* ***Appendix*** *"Test sheets, measuring instruments, equipment, setups, auxiliary materials, formulations, chemicals and supply sources")*
4.2	Test sheets	In order to cover the paint qualities as they are found in practice, the test is carried out on absolutely flat [8], sufficiently stable and painted test sheets sized [9] 40 cm x 50 cm with original paint (OEM quality) [10] and paint colour „black uni" (no metallic paint, specification *cf.* ***Appendix***). For each subsequent test, a sheet of the same batch and the same history [11] should be used to avoid deviations within the test series. Two different types of test sheet are needed for the following tests: – completely matted sheets: These are divided into four areas for parallel testing to assess the following test parameters for a maximum of 4 paint polishes *(cf. 5.1)*: • Measuring of gloss starting values • Assessment of ◦ distributability ◦ polishability ◦ dust formation 1. Assessment of the surface ◦ surface appearance ◦ paint refreshment ◦ change of gloss ◦ touchability and smear resistance 2. Assessment of surface after degreasing ◦ surface appearance ◦ colour refreshment ◦ change in gloss value – completely glossy sheets [9]: These are divided into two areas for parallel testing to assess the following test parameters for a maximum of 2 paint polishes (*cf. 5.2*): • Determination of water run-off behaviour before application • Water run-off behaviour after application • Wash resistance

4.3	Setups for determining the gloss value measurement and the water run-off behaviour	
4.3.1	Gloss value measurement	Gloss value measurements are carried out as single measurements on the maximum of four different areas evenly distributed in orientation to DIN EN ISO 2813, with a fixed measuring angle of 20 degrees at 5 measuring points (keeping a distance of at least 4 cm from the edge in each case). For each area, the mean value and the standard deviation [12] calculated from this are determined across all 5 measuring points (**Diagram 4**, **Figure 3**).

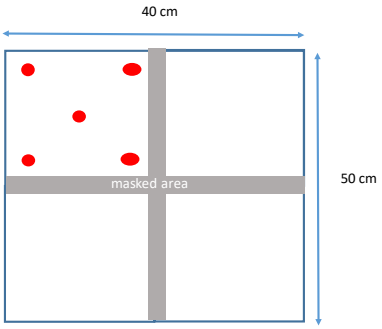


Diagram 4 *Schematic representation of the test sheet with 5 measuring points in one area (side).*

The gloss values before and after treatment with the paint polish and after removal of the product residues are entered in the Excel table *(cf. **Chapter 7**)*.

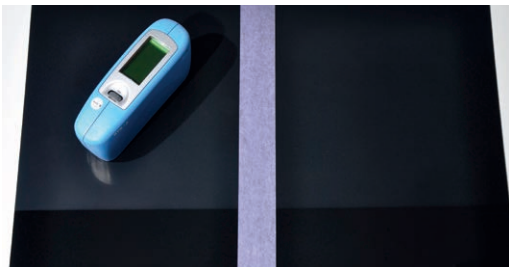


Fig. 3 *Photo of two test areas of the test sheet with gloss meter. (Image source: Chemische Fabrik Dr. Stöcker GmbH & Co.KG).*

4.3.2	Setup to measure the water run-off behaviour	The water-repelling (hydrophobic) properties of a glossy sheet treated with a paint polish are assessed using the water run-off method where the following applies: The faster the water runs off, the better the hydrophobic properties of the paint polish. A jet of water (e.g., tap water) is directed onto one side each of the test sheet, which is divided by an adhesive strip, according to the setup in **Diagram 3** and the procedure shown in the video for **Diagram 3**. Measuring the average run-off time with 1/10-second accuracy is carried out by 5-fold measurement successively on the maximum two test areas of the test sheet. The measuring results are entered in an Excel table *(cf. **Chapter 7**)* and evaluated automatically. For the correct evaluation of the measuring results, it is essential to comply with the test conditions and to observe the test sheet size (40 cm x 50 cm). The 30° inclined sheet is placed on the shorter side (40 cm) in a holding device at a distance of 1 cm from the washbasin or the bottom of the water collection vessel for free water run-off, and the treated areas are exposed in the middle to a water jet from a tap with a flow rate of ca. 8 litres per minute *(cf. **Figure 4**)*. The volume flow should ideally be adjusted with a flow meter. The water temperature is ideally 20 °C. The distance between the water tap and the test sheet (impact surface) should be kept constant at ca. 30 cm; the diameter of the water jet should be kept constant at the aerator *(cf. **Appendix**)*. The water jet is positioned so that the wetted parabola begins exactly at the lower end of the adhesive strip. Here, it is to ensure that the water can run off freely at the end of the sheet. As soon as a parabolic, constantly wet water body has formed on the sheet, the water jet is turned off and the time required until the water has completely run off the sheet surface is measured in tenths of a second (run-off time). All set parameters of the water run-off method must be checked and kept constant throughout the entire test series!

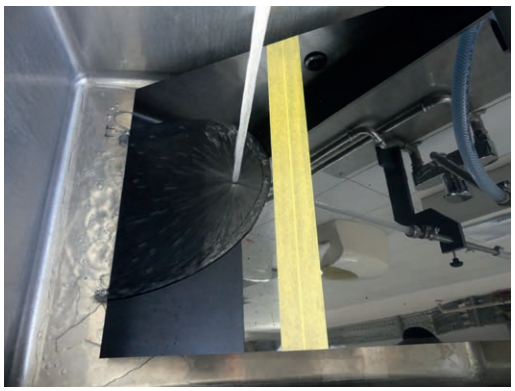


Fig. 4 *Photo of the setup of a 30° inclined test sheet in the holding device at 1 cm distance from the bottom of the washbasin. (Image source: SONAX GmbH)*

5. Pre-treatment of test sheets and test instruction

The sequence of tests on the two types of test sheet (1. matted, 2. glossy) should be adhered to for efficient work.

5.1 Testing on *matted* test sheet

5.1.1 Pre-treat the test sheet with matting agent

(The required chemicals, materials, equipment / *cf.* ***Appendix***: Basic car shampoo, demineralised water, isopropyl alcohol, matting agent, random orbit sander, PUR sponge, weight, gloss meter, if necessary: high-gloss polish, adhesive tape)

The test sheet is matted as follows:

- Clean with the basic car shampoo, rinse with demineralised water (DM water) and then treat with isopropyl alcohol (isopropanol) and dry. Drying is done, for example, by blowing off with oil-free compressed air or using a wiper with a degreased silicone lip.
- Shake the matting agent well before use!
- Work on the test sheet with matting agent, random orbit sander in combination with an excenter pad or by hand [13] in combination with a PUR sponge with the same pressure (1.5 kilogramme weight) in crosswise application *(cf.* ***Diagram 2*** */ video)*. For this, wet the excenter pad or the PUR sponge completely with the matting agent and matt in crosswise application with 50% overlapping:
  - Matting is done up to a gloss level of 70±2 units at a measuring angle of 20 degrees. [Note: If the matting process results in a gloss value < 68 units, the gloss value can be raised by treatment with a high-gloss polish *(cf. 5.2.1)*].
  - Before each measurement is taken, clean with a basic car shampoo. Then rinse with DM water, treat with isopropyl alcohol and dry. Drying is done e.g., by blowing with oil-free compressed air or with a wiper with a decreased silicone lip.
  - The matted test sheet is divided into a maximum of four equally sized areas using adhesive tape (e.g., 48 mm wide) (a maximum of four paint polishes can be tested on one matted test sheet, *(cf.* ***Diagram 5****)*. Additional test sheets for further test paint polishes can be prepared analogously.

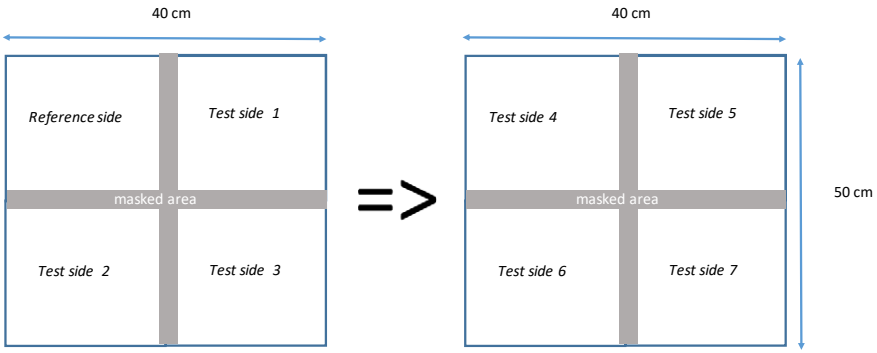


Diagram 5 *Schematic representation of two matted test sheets, each with four areas resp. sides to be tested.*

5.1.2 Measuring of initial gloss values

The initial gloss values are measured according to 4.3.1 on the maximum four matted areas of the test sheet before the paint polish is applied.

5.1.3 Assessment of workability

5.1.3.1 Applying the paint polish

Before applying the paint polish, the paint polish should be thoroughly homogenised by shaking.

For applying the reference paint polish and the test paint polish, in each case a different **unused, new straight from production and dry** PUR sponge should be used *(specification, cf.* ***Appendix****)*. If the manufacturer of the test paint polish recommends another application medium or has enclosed enough of it, this application medium should be used.

If the manufacturer of the test paint polish does not give any instructions regarding dosage and application medium, then **1 ± 0.1 gramme** (if necessary, the optimum quantity must be determined in a preliminary test) [14] of the paint polish is spread thinly and evenly over the entire area of the sponge.

Apply all paint polishes to be tested in a fivefold crosswise application *(cf.* ***Diagram 2* */ video)* with 50% overlapping of the wipe paths and the same pressure with 1.5 kg weight. The sponge must be moved over the test sheet with a weight without manual pressure (Figure 5).**

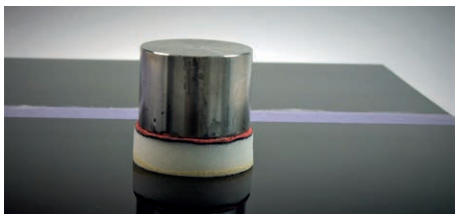
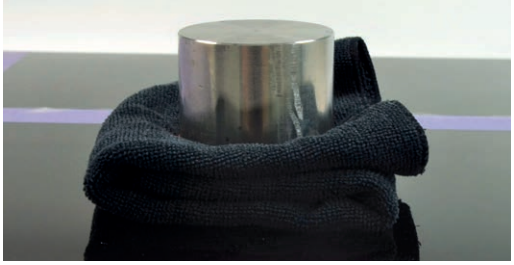
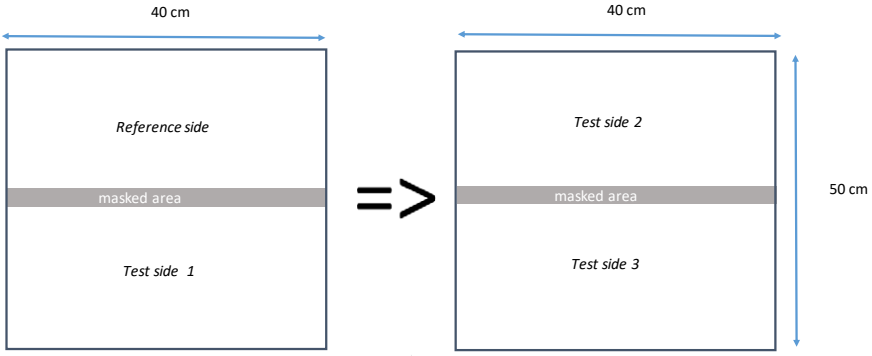


Fig. 5 *Photo of the test sheet with weight and sponge for applying the test polish. (Source: Wigo Chemie GmbH)*

5.1.3.2	Testing of distributability	It should be possible to distribute the test paint polish without effort and without using much force. Squeaky noises can occur which, however, are not considered in the assessment. Distributability is assessed by the amount of force that is needed to move the sponge with a weight according to 5.1.3.1, compared with the reference paint polish (RPP). The values are entered into the Excel table for automated evaluation *(cf. **Chapter 7**)*. 5 points = clearly less force required than with RPP 4 points = less force required than with RPP 3 points = comparable force required as with RPP 2 points = more force required than with RPP 1 point = clearly more force required than with RPP Intermediary marks in 0.5 increments are admissible (e.g., 1.5 points)
5.1.3.3	Drying time	Documentation of the timespan between application and polishability: Polishing out is done directly after the respective product no longer smears and appears to be dry. The testing of the drying of the respective product can be carried out by careful polishing at the edge of the sheet. Information possibly provided by the manufacturer on drying of the product must be considered. (Note: The paint polishes must no longer smear when polishing out. The drying time for the RPP is 10 minutes.)
5.1.3.4	Testing of polishability	Complete and residue-free polishing of the paint polish is carried out directly after the respective product no longer smears and appears to be dry, using a microfibre cloth *(specification, cf. **Appendix**)*. *(Information provided by the manufacturer on drying of the product has to be considered. The testing of the drying of the respective product can be carried out by careful polishing at the edge of the sheet. The drying time for the RPP is 10 minutes. The timespan between application and polishability is documented for the respective product.)* For polishing the different test paint polishes, in each case microfibre clothes of the same specification and the same pre-treatment (pre-washing of the microfibre clothes preferably with an aqueous liquid detergent of low viscosity, without fabric softener) must be used. If the manufacturer of the test paint polish recommends different materials for polishing or has enclosed enough of them, these should be used for polishing. For polishing, the microfibre cloth is moved in fivefold crosswise applications with the same pressure of 1.5 kg weight over the area *(cf. **Figure 6**)* and turned after threefold crosswise wiping paths *(cf. **Diagram 2** / video)*. **The number of crosswise applications up to the full removal of the product (no residues of the paint polish visible on the surface anymore) must be noted.** If the test paint polish can be completely polished with less than fivefold crosswise applications, the polishing should be finished up to the fifth crosswise application. The test paint polish should be polishable without effort. Polishability is assessed compared with the reference paint polish (RPP). The RPP needs threefold crosswise applications to full polishability. In comparison to the polishability to the RPP, only a maximum of two more or fewer crosswise applications are considered for the evaluation. The values are entered into the Excel table for automatic evaluation *(cf. **Chapter 7**)*: 5 points = two crosswise applications less required than with RPP 4 points = one crosswise application less required than with RPP 3 points = the same number of crosswise applications required as with RPP 2 points = one crosswise application more required than with RPP 1 point = two or more crosswise applications more required than with RPP  **Fig. 6** *Photo of a microfibre cloth with 1.5 kg weight. (Image source: Wigo Chemie GmbH)*

5.1.3.5	Colour refreshment (colour strength, intensification of the shade) (before product residue removal)	The test paint polish should hardly produce any dust during polishing. Dust residues on the sheet are assessed after polishing. Scoring scheme for dust formation compared with the reference paint polish (RPP): 5 points = clearly less than with RPP 4 points = less than with RPP 3 points = comparable with RPP 2 points = more than RPP 1 point = clearly more than RPP Intermediary marks in 0.5 increments are admissible (e.g., 1.5 points).
5.1.4	First assessment of the treated surface (before degreasing)	
5.1.4.1	Surface appearance (clouds, veils, streaks)	After the paint polishes were applied and polished out, the product residues (e.g., emulsifiers, auxiliaries) are removed after 24 hours: For removing the product residues, completely rinse the test sheet with DM water. Drying is done by blowing off with oil-free compressed air or using a wiper with a degreased silicone lip. The test paint polish is assessed regarding cloud, veil and streak formation immediately after drying. There should be a uniform surface appearance. The surface appearance is assessed visually, preferably in daylight or corresponding artificial light, and from different angles compared to the reference paint polish (RPP). The values are entered into the Excel table for automated evaluation *(cf. **Chapter 7**)*: 5 points = significantly better than RPP 4 points = better than RPP 3 points = comparable to RPP 2 points = worse than RPP 1 point = significantly worse than RPP Intermediary marks in 0.5 increments are admissible (e.g., 1.5 points).
5.1.4.2	Colour refreshment (colour strength, intensification of the colour shade)	The assessment is made preferably in artificial light corresponding to daylight from different angles. The test paint polishes should produce an intensification of the colour shade and the treated areas should thus appear darker. For better differentiation of the different areas, the adhesive tape should be removed before the assessment. The colour refreshment is assessed visually, compared to the reference paint polish (RPP). The values for each area are entered into the Excel table for automated evaluation *(cf. **Chapter 7**)*. 5 points = significantly better (darker) than RPP 4 points = better (darker) than RPP 3 points = comparable (darker) to RPP 2 points = worse (brighter) than RPP 1 point = significantly worse (brighter) than RPP Intermediary marks in 0.5 increments are admissible (e.g., 1.5 points)
5.1.4.3	Measuring of the change in gloss value	The assessment is made based on the average gloss value increase in gloss units (Δ GU), taking as initial values the gloss values determined in 5.1.2 for a maximum of four areas. The assessment is made in each case via the difference of the average gloss value changes to the determined initial values in gloss units (Δ GU) on the respective areas. The values for each area are entered into the Excel table for automated evaluation *(cf. **Chapter 7**)* and automatically evaluated with the help of the following linear equation: y = 0.357 * x (y: scoring points; x = gloss value increase in Δ GU) The assessment ranges are between 0 Δ GU and 14 Δ GU (scoring scheme: 0 to 5 points).
5.1.4.4	Touchability and smear resistance	Finally, cotton swabs are applied under strong pressure on the areas treated with the product and it is assessed whether traces can be seen on the areas. Touchability and smear resistance are assessed visually. The values are entered into the Excel table for automated evaluation *(cf. **Chapter 7**)*: 5 points = good (no traces to be seen) 3 points = satisfactory (weak traces to be seen) 1 point = bad (strong traces to be seen)

5.1.5	Degreasing	Degreasing is made as follows on the maximum of four areas *(specification cf. **Appendix**)*: – Degrease the test sheet with aromatics-free and paint resistant white spirit (boiling range 80–110°C) and isopropyl alcohol and then wash it with basic car shampoo; – then thoroughly rinse off the basic car shampoo for five minutes with tap water; – rinse off tap water residues with DM water, e.g., with spray bottle or beaker; – dry using a wiper with a degreased silicone lip or a microfibre cloth or by blowing off with oil-free compressed air. Full degreasing is reached at a constant gloss value. Gloss value measurements are carried out according to 4.3.1 to check constant gloss value. Degreasing is carried out to enable an assessment of an effective polishing effect (scratch removal, grey haze removal). Polishes might cause additional scratches on the paint surface.
5.1.6	Second assessment of the treated surface (after degreasing)	
5.1.6.1	Surface appearance (clouds, veils, streaks)	The test paint polish is assessed regarding cloud, veil and streak formation. There should be a uniform surface appearance. The surface appearance is assessed visually, preferably in daylight or corresponding artificial light, and from different angles compared to the reference paint polish (RPP). The values are entered into the Excel table for automated evaluation *(cf. **Chapter 7**)*: 5 points = significantly better than RPP 4 points = better than RPP 3 points = comparable to RPP 2 points = worse than RPP 1 point = significantly worse than RPP Intermediary marks in 0.5 increments are admissible (e.g., 1.5 points).
5.1.6.2	Colour refreshment (colour strength, intensification of the colour shade)	The assessment is made preferably in artificial light corresponding to daylight from different angles. The test paint polishes should produce an intensification of the colour shade and the treated areas should thus appear darker. For better differentiation of the different areas, the adhesive tape should be removed before the assessment. The colour refreshment is assessed visually, compared to the reference paint polish (RPP). The values for each area are entered into the Excel table for automated evaluation *(cf. **Chapter 7**)*. 5 points = significantly better (darker) than RPP 4 points = better (darker) than RPP 3 points = comparable (darker) to RPP 2 points = worse (brighter) than RPP 1 point = significantly worse (brighter) than RPP Intermediary marks in 0.5 increments are admissible (e.g., 1.5 points).
5.1.6.3	Measuring of the change in gloss value	The assessment is made in each case via the difference of the average gloss value changes to the determined initial values in gloss units (Δ GU). Initial values are the gloss values determined in 5.1.2 for a maximum of four areas. The values for each area are entered into the Excel table for automated evaluation *(cf. **Chapter 7**)* and automatically evaluated with the help of the following linear equation: y = 0.4167 * x (y: scoring points; x: gloss increase in Δ GU The assessment ranges are between 0 Δ GU and 14 Δ GU (scoring scheme: 0 to 5 points).
5.2	**Testing on *glossy* test sheet**	
5.2.1	Pre-treat the test sheet with high-gloss polish. (The required chemicals, materials, equipment / *cf. **Appendix***: Basic car shampoo, DM water, isopropyl alcohol, high-gloss polish, sponge, random orbit sander, microfibre cloth, tap water, water run-off setup, gloss value meter)	The test sheet is treated as follows: – Clean with the basic car shampoo, rinse with demineralised water (DM water) and then treat with isopropyl alcohol and dry. Drying is done, for example, by blowing off with oil-free compressed air or using a wiper with a degreased silicone lip. – Apply the high-gloss polish without waxes and silicones (e.g., SONAX Profiline Perfect Finish) with an excenter pad [e.g., SONAX Exzenter pad (medium)], polish with a random orbit sander (ca. 4,500 rotations per minute) in fivefold crosswise applications and let dry. – Then polish out the residues with a soft microfibre cloth without leaving any residues. – Degrease the test sheet with aromatics-free and paint-resistant white spirit (boiling range 80–110°C) and isopropyl alcohol and then wash it with basic car shampoo. – Then thoroughly rinse off the basic car shampoo for five minutes with tap water. – Rinse off tap water residues with DM water, e.g., with spray bottle or beaker. – In order to standardise the test sheets and to remove the basic water-repelling coating, the polishing process is repeated until the maximum gloss level on the dry test sheet according to 4.3.1 is at 86 ± 2 gloss units (GU) at a measuring angle of 20 degrees and the run-off time with the water run-off method according to 4.3.2 is 6 ± 1.5 seconds for all test areas (usually, 15 crosswise applications are sufficient to bring about the desired removal of the basic water-repelling coating). Further crosswise wiping does not result in a further increase of the gloss level.

		– Drying of the test sheet between the measurements and at the end of the pre-treatment is done with oil-free compressed air or using a wiper with a degreased silicone lip. – The glossy test sheet is divided in length (50 cm) with the help of an adhesive tape (48 mm wide) into a maximum of two equal-sized areas (a maximum of two paint polishes including the reference paint polish can then be tested on one glossy test sheet). Additional test sheets for further test paint polishes can be prepared analogously *(cf. **Diagram 6**)*. – **Note: The pre-treatment of the test sheets for the testing of different paint polishes must take place on the same day. The time interval between pre-treatment and the application of the paint polish for subsequent testing must be the same for all products and be on the same day.**  **Diagram 6** *Schematic representation of two glossy test sheets with two test areas resp. sides each.*
5.2.2		Assessment of the water run-off behaviour
5.2.2.1	Measuring of the initial value for the water run-off behaviour before applying the paint polish	The water run-off behaviour of the untreated areas is measured according to the setup described in 4.3.2. The run-off times are noted as target values for the wash resistance under 5.2.3.2.
5.2.2.2	Application, drying, polishing out and removal of product residues	**Before applying the paint polish, the paint polish should be thoroughly homogenised by shaking**. If the manufacturer of the test paint polish does not give any instructions regarding dosage and application medium, then **2 +/- 0.1 gramme** (if necessary, the optimum quantity must be determined in a preliminary test) [14] of the paint polish is spread thinly and evenly over the entire area of the **unused, new straight from production and dry** PUR sponge. Except for the application quantity, application is made according to 5.1.3.1. Drying (5.1.3.3), polishing (5.1.3.4) of the paint polish and removal of product residues (5.1.5) on a maximum of two areas follow the rules in 5.1 without assessment or documentation of the parameters. **The time interval between polishing and testing of the water run-off behaviour is at least 24 hours**.
5.2.2.3	Assessment of the water run-off behaviour	Next, the water run-off behaviour of the treated sheets is assessed according to the setup in 4.3.2. For the assessment of the run-off behaviour and automated scoring, a linear equation# according to the spreadsheet "Assessment of run-off time" in the Excel table *(cf. **Chapter 7**)* is used with the following scoring scheme:* 5.4 to 4 points: 0.1 to < 1,6 seconds 4 to 3 points: > 1.6 to < 2.7 seconds 3 to 2 points: ≥ 2.7 to < 3.8 seconds 2 to 1 points: ≥ 3.8 to < 4.9 seconds 1 to 0 points: ≥ 4.9 to < 5.9 seconds 0 points: ≥ 6 seconds The values of the run-off time are entered in the Excel table for automated evaluation. # *y = 0.909 * t + 5.4545 (y: points; t: run-off time in seconds)* * *Because of the y-axis intercept of the straight-line equation used in the Excel table, a maximum of 5.4 points can be achieved with a theoretical minimum run-off time of 0.1 seconds (accuracy of the stopwatch)!*
5.2.3	Wash resistance (paint preservation / long-term effect)	Wash resistance of the dried, polished and product residue-free test paint polish should be given over as many wash cycles as possible. One wash resistance cycle consists of washing according to 5.2.3.1 and the subsequent assessment of the water run-off behaviour according to 5.2.3.2. The wash on one test area is repeated until the initial value of the run-off time before treatment with the paint polish, as documented under 5.2.2.1, is reached or a maximum of 25 washes have been carried out. The assessment is made up to and including the fifth wash after each wash. From the fifth wash, assessments are made only after all five washes. When the initial values of the run-off time are reached, the assessments of the following washes carry zero points *(cf. **Chapter 7** "Excel assessment table")*.

5.2.3.1	Washing of the test specimen (test sheet)	The areas for the paint polishes on a test sheet need to be washed separately to avoid carry-over of the paint polishes. In addition, separate sponges and separate containers of basic car shampoo should be used *(cf. **Appendix**)*. Wash the test sheet with basic car shampoo: Completely soak the PUR sponge for treatment in fivefold crosswise applications *(cf. **Diagram 2** / video)* at the same pressure (ca. 1.5 kg) (corresponds to one wash). After each crosswise application, squeeze out the sponge and completely soak it anew with shampoo. Before the assessment, the test sheet is rinsed completely with DM water to remove the shampoo residues. Drying is done, for example, by blowing off with oil-free compressed air or using a wiper with a degreased silicone lip [before this, degrease the pull-off lip with aromatics-free and paint-resistant white spirit (boiling point 80 – 110°C) and isopropyl alcohol].
5.2.3.2	Assessment of the water run-off behaviour after the wash ("wash resistance")	The water run-off behaviour of the test paint polish is assessed each after 1, 2, 3, 4, 5, 10, 15, 20 and 25 washes or until complete removal of the paint polish (run-off time ≥ 6 seconds or until the initial value in 5.2.2.1 is reached) according to the setup in 4.3.2. The measurement of the run-off time is stated in tenths of a second and in each case separately, e.g., on the test and reference sides (areas). For the assessment of the run-off behaviour and automated scoring, a linear equation[#] according to the spreadsheet "Assessment of run-off time" in the Excel table *(cf. **Chapter 7**)* is used with the following scoring scheme: 5.4 to 4 points: 0.1 to < 1,6 seconds* 4 to 3 points: > 1.6 to < 2.7 seconds 3 to 2 points: ≥ 2.7 to < 3.8 seconds 2 to 1 points: ≥ 3.8 to < 4.9 seconds 1 to 0 points: ≥ 4.9 to < 5.9 seconds 0 points: ≥ 6 seconds The values of the run-off time are each entered in the Excel table for automated evaluation (if the paint polish has already been completely removed before the 25th wash and a water run-off time of ≥ 6 seconds has been achieved, no further wash cycles nor inputs in "run-off time after treatment" in the Excel table are required. The scoring of the subsequent wash cycles is automatically set to zero.) and a mean value between 0 and 5.4 points is determined for the entire test item "Wash resistance" *(cf. **Chapter 7**)*. [#] *y = -0.909 * t + 5.4545 (y: points; t: run-off time in seconds)* ** Because of the y-axis intercept of the straight-line equation used in the Excel table, a maximum of 5.4 points can be achieved with a theoretical minimum run-off time of 0.1 seconds (accuracy of the stopwatch)!*

7. Assessment of the test results

The assessment of the test results of the product group is carried out in a weighted point system **(Table 2)**. The total score can be calculated automatically using an Excel table. The assessment of the average wash resistance of the paint polishes is made by assessing the water run-off behaviour in each case after 1, 2, 3, 4, 5, 10, 15, 20 and 25 washes. The test procedure for wash resistance ends after a maximum of 25 washes. Evaluation and scoring for wash resistance are done using a linear equation provided in the spreadsheet "Assessment of run-off time" in the Excel table.

Under the following internet address an Excel table can be downloaded for the assessment of the test method for a paint polish (assessment scheme), including the following assessment table and calculation of the overall result:

https://www.ikw.org/fileadmin/IKW_Dateien/downloads/Haushaltspflege/2023_01_26_Assessment_scheme_Paint_Polish.xlsx

Test Criterion	Scoring points from the assessment scheme of the test method	Weighting (%)	Weighted Score
Matted test sheet			
Assessment of usability			
5.1.3.2 Distributability	1 to 5	2.5%	
5.1.3.4 Polishability	1 to 5	10%	
5.1.3.5 Dust formation	1 to 5	2.5%	
First assessment of the treated surface			
5.1.4.1 Surface appearance	1 to 5	5%	
5.1.4.2 Colour refreshment	1 to 5	5%	
5.1.4.3 Change in gloss value	0 to 5	15%	
5.1.4.4 Touchability and smear resistance	1 to 5	5%	
Second assessment of the treated surface			
5.1.6.1 Surface appearance	1 to 5	5%	
5.1.6.2 Colour refreshment	1 to 5	5%	
5.1.6.3 Change in gloss value	0 to 5	15%	
Glossy test sheet			
5.2.2.3 Water run-off behaviour before the wash	0 to 5.4	10%	
5.2.3.2 Wash resistance (water run-off behaviour after the wash)	0 to 5.4	20%	
Total score	**8 to 60.8**	**100%**	**0.4 to 5.12**

Tab. 2 Assessment table of the weighted test results of a paint polish for motor vehicles.

8. Members of the Working Group

Claudia Figulla-Kroschel, Hartmut Hauber, Heiko Kaufmann, Oliver Kerp, Thorsten Kessler, Arend J. Kingma, Stephan Kollig, Thilo Kunst, Carmen Manhart, Manfred Pitsch, Andrea Thole, Shute Ye.

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[1] https://www.forum-waschen.de/

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[5] https://www.vci.de/themen/nachhaltigkeit/responsible-care/uebersicht.jsp

[6] https://www.chemiehoch3.de/

[7] Applying / polishing beyond the edge of the area to be treated is intended to ensure an even treatment of the area.

[8] Measurement on curved vehicle surfaces does not make sense, as this does not provide correct measuring results in gloss value measuring.

[9] The size of the test sheets is crucial for the correct evaluation of the water run-off method on the glossy test sheets, using the Excel table in Chapter 7.

[10] Original Equipment Manufacturer – OEM.

[11] Unless no new sheets are used, the sheets should have a comparable condition regarding preparation and treatment. The history of the sheet can be noted down e.g., on its back.

[12] For a homogeneously matted area, the standard deviation should be < 1 gloss unit. DIN EN ISO 2813 (June 1999, updated in 2015) "Paints and varnishes – determination of the gloss value at 20°, 60° and 85°".

[13] When applying by hand, up to 20 crosswise applications might become necessary. Ideally, do not replace the sponge and add matting agent if needed. Too little matting agent can make matting more difficult.

[14] If necessary, the ideal application quantity should be determined in a pre-test. The application quantity should completely wet the test areas. It should be noted here that during the tests on the glossy test sheets, twice as large an area has to be wetted and therefor twice the amount of the paint polish should be needed as for the matted test sheets. If the application quantity is not sufficient for this, the areas must be completely cleaned with isopropyl alcohol and the process must be repeated.

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Appendix

Test specimens, equipment, auxiliary materials, formulations and supply sources

Test sheets (test specimens)

- For example, plain steel or aluminium sheet [8,11]
- Original paint layering in OEM quality (no repair and special paint) [10], base paint plain black, e.g. clear paint PPG APO 1.2 (e.g. Thierry GmbH, Motorstraße 30, 70499 Stuttgart, Germany)
- Size of the test sheet: [9] 500 mm x 400 mm x 1 mm
- Use of the test sheets three weeks after manufacturing at the earliest or expose test sheets to accelerated ageing: e.g., 16 hours in the drying cabinet at 60 °C including at least one day at ambient temperature for conditioning. The relative air humidity should amount to ca. 30 to 80%.

Measuring instruments, equipment and setups

- Gloss meter with measurement geometry and measurement conditions based on DIN EN ISO 2813 (e.g., company Byk-Gardner GmbH, Lausitzer Strasse 8, 82538 Geretsried, Germany, phone.: +49 (0)8171 3493-0, fax +49 (0)8171 3493-140, email: info.BYK.Gardner@altana.com, www.byk.com or ERICHSEN GmbH & Co. KG, Am Iserbach 14, 58675 Hemer, Germany, phone: +49 (0)2372 – 9683-0, fax: +49 (0)2372 – 6430, email: info@erichsen.de, https://www.erichsen.de/erichsen-de)
- Setup for the water run-off method with water jet according to **Diagram 3**:
  - Rack for 30 °C installation, so that the sheet at the underside is at least 10 mm above the bottom of the washbasin (free run-off)
  - Water tap
  - Aerator: e.g., Neoperl Strahlregler Perlator (TT, IG M16x1 V, Art. No.: 1562145 or M22x1 DL, Art. No.: 40460395, Neoperl GmbH, Klosterrunsstraße 9-11, 79379 Müllheim, Germany, phone: +49 (0)7631-188-0, fax: +49 (0)76 31-188-287, email: info@neoperl.de)
  - Flow meter 60 - 600 l/h: e.g., PVC-U flow indicator with 2fold bonded socket 20 mm (Art. No.: AA461; HT CONNECT GmbH & Co. KG, Norisstraße 4, 91257 Pegnitz, Germany; phone: 09241/9109100, email: info@ht-connect.de; https://www.pvc-welt.de/PVC-U-Durchflussmessgeraet-2fach-Klebemuffe)
  - Stopwatch for measuring the water run-off with a 1/10 second measuring accuracy
  - Thermostat
  - Washbasin or water collection vessel
- Balance for weighing the paint polish samples with 0.1 gramme measuring accuracy
- Random orbit sander: free-wheeling or forced rotation eccentric polisher for matting and polishing: e.g., ECCENTRIC POLISHER - BIGFOOT LHR15 MARK III (Rupes S.p.A. a socio unico, Via Marconi 3A loc. Vermezzo, 20071 Vermezzo con Zelo (MI) – Italy, phone: +39 02946941, fax: +39 0294941040, email: info_rupes@rupes.it, https://www.rupes.com)

Auxiliary materials

- Adhesive tape (residue-free): e.g. adhesive tape 5959, width 48 mm (3M Deutschland GmbH, Carl-Schurz-Str. 1, 41453 Neuss, Germany)
- Felt pad for matting (e.g. SONAX Felt Pad Art. No. 493 300, SONAX GmbH, Münchener Str. 75, 86633 Neuburg, Germany, phone:+49 (0)84 31 53-0, email: info@ sonax.de, www.sonax.de)
- Sponge for eccentric polisher for polishing and matting: e.g. Eccentric pad (medium) 143 (Art. No. 04933410), SONAX GmbH, Münchener Str. 75, 86633 Neuburg, Germany, phone: +49 (0)84 31 53-0, email: info@sonax.de, www.sonax.de)
- Wiper with silicon lip: e.g. Flexiblade (Art. No. 04174000, SONAX GmbH, Münchener Str. 75, 86633 Neuburg, Germany, phone: +49 (0)84 31 53-0, email: info@sonax.de, www.sonax.de)
- Polyuethane sponge (PUR sponge), round, for applying and matting; 7.5 cm diameter and 2 cm thick: e.g., T28065 (Oskar Pahlke GmbH, Linzer Straße 95, 53562 St Katharinen, Germany, phone: +49 (0)2645 9523-0, fax: +49 (0)2645 9523-40, info@pahlke-schaumstoffe.de, http://www.pahlke-schaumstoffe.de/)
- Microfibre cloth for polishing, e.g. microfibre cloth black (weight: 300 g/m², dimensions 40 x 40 cm, Art. No. 615.900.337, De Witte SA, Kluizenmeersen 7, B-9170 Sint-Gillis-Waas, Belgium, phone: +32 (0)3 766 46 83, fax: +32 (0)3 766 46 84, email: info@dewitte.biz, http://www.dewitte.biz /Dewitte /index.html)
- Cotton swabs: e.g., CLASSIQSwabs™ (Copan Flock Technologies Srl, Via Perotti 18, 25125 Brescia, Italy, phone +39 030 3666100, fax: +39 030 2659932, email: info@copanflock.com, www.copanflock.com)
- Metal weight to be placed on sponge and/or microfibre cloth: ca. 1.5 kg (cf. **Figures 5 and 6**)
- Containers with basic car shampoo for the testing of wash resistance

Formulations and chemicals

- Reference paint polish (RPP):
  - 10% by weight Mipri LK2 polishing grain (Mipri GmbH / 55543 Bad Kreuznach)
  - 8% by weight Korasilon NPF 60 silicone oil emulsion (Kurt Obermeier GmbH & Co. KG)
  - 5% by weight paraffin hydrocarbon (e.g., test white spirit 190-245 or Exxsol D80] / ExxonMobil)
  - 5% by weight paraffin wax emulsion (e.g., Hansa Care 4670 / CHT Germany GmbH)
  - 0.80% by weight xanthan gum (e.g., Hammonia Gum FG / Hammonia Oleochemicals GmbH, 20457 Hamburg, Germany)
  - 0.20% by weight preservative, at own choice
  - Ad 100% by weight water (DM)

- Matting agent (start with the liquid components and then stir in the abrasive):
  - 20% by weight abrasive (e.g., Silitin V 85 / HOFFMANN MINERAL GmbH, 86633 Neuburg, Gemany)
  - 15% by weight of 28% sodium lauryl ether sulphate (2.5 EO) solution (e.g., Emal 228D / KAO or Texapon® NSO / BASF SE)
  - 15% by weight complexing agent [e.g., Trilon® M liquid (40%) BASF SE]
  - 8% by weight glycerine
  - 0.2% by weight preservative, at own choice
  - Ad 100% by weight water (DM)

- Basic car shampoo:
  - 0.5% solution of 28% sodium lauryl ether sulphate (2.5 EO) solution (e.g. Emal 228D / KAO Chemicals Global or Texapon® NSO / BASF SE)
  - Ad 100% by weight water (DM)

- Demineralised water (DM water)
- Degreaser: Isopropyl alcohol (isopropanol alcohol), ≥99.8% purity
- White spirit: aromatics-free and paint-resistant (boiling range 80–110°C)
- Abrasive: e.g., Silitin V 85 (HOFFMANN MINERAL GmbH, Postfach 14 60, 86619 Neuburg, Germany, phone: +49 (0) 8431 53-0, fax: +49 (0) 8431 53-3 30, www.hoffmann-mineral.com, email: info@hoffmann-mineral.com)
- High-gloss polish without waxes and silicones (e.g. SONAX Profiline Perfect Finish (Art. No.: 224 141, SONAX GmbH, Münchener Str. 75, 86633 Neuburg, Germany, phone: +49 (0)84 31 53-0, email: info@sonax.de, www.sonax.de)

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Phase	Ingredient	% wt./wt.	Functions
A	Aqua	qs	Diluent
	SYMPARE MES (as 100%)	**3.0**	**Surfactant**
	Maltooligosyl Glucoside (and) Hydrogenated Starch Hydrolysate	2.0	Binding Agent
B	PALMERA Glycerine*	3.4	Humectant
	Lauryl Glucoside	2.0	Co-Surfactant
	Sodium Lauroyl Sarcosinate	6.0	Co-Surfactant
	Cocamidopropyl Betaine	7.0	Co-Surfactant
	PALMOCOL CMMEA 85%*	3.0	Viscosity Modifier
C	Preservative	0.4	Preservative
	Lactic Acid	pH 6.0 - 7.0	pH Adjuster

* Ingredients offered by KLK OLEO

PROCEDURE:

Heat aqua to 70°C, dissolve SYMPARE MES, add the remaining Part **A** ingredients, then cool to 40°C. Add Part **B** and blend under gentle stirring, cool to room temperature. Add Part **C**, stir. Adjust pH to 6.0-7.0 with Lactic Acid.

TYPICAL PROPERTIES:

Appearance: Clear Liquid
pH: 6.0 - 7.0

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SYMPARE

Hand Wash

Phase	Ingredient	% wt./wt.	Functions
A	Aqua	qs	Diluent
	SYMPARE MES (as 100%)	**2.5**	**Surfactant**
	TENSAGEX SLES 70%*	3.4	Surfactant
	Cocamidopropyl Betaine 30%	1.0	Co-Surfactant
B	PALMOCOL CMMEA 85%*	1.2	Viscosity Modifier
	PALMERA Glycerine*	1.0	Humectant
C	Fragrance	As Needed	Fragrance
	Preservative	As Needed	Preservative
	Sodium Chlroride	As Needed	Viscosity Adjuster

* Ingredients offered by KLK OLEO

PROCEDURE:

Heat aqua to 70°C, dissolve SYMPARE MES, add the remaining Part **A** ingredients, then cool to 40°C. Add Part **B** and blend under gentle stirring, cool to room temperature. Add Part **C**, stir. Adjust viscosity with Sodium Chloride.

TYPICAL PROPERTIES:

Appearance: Clear Liquid
pH: 6.5 - 7.5

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