Cosmetic
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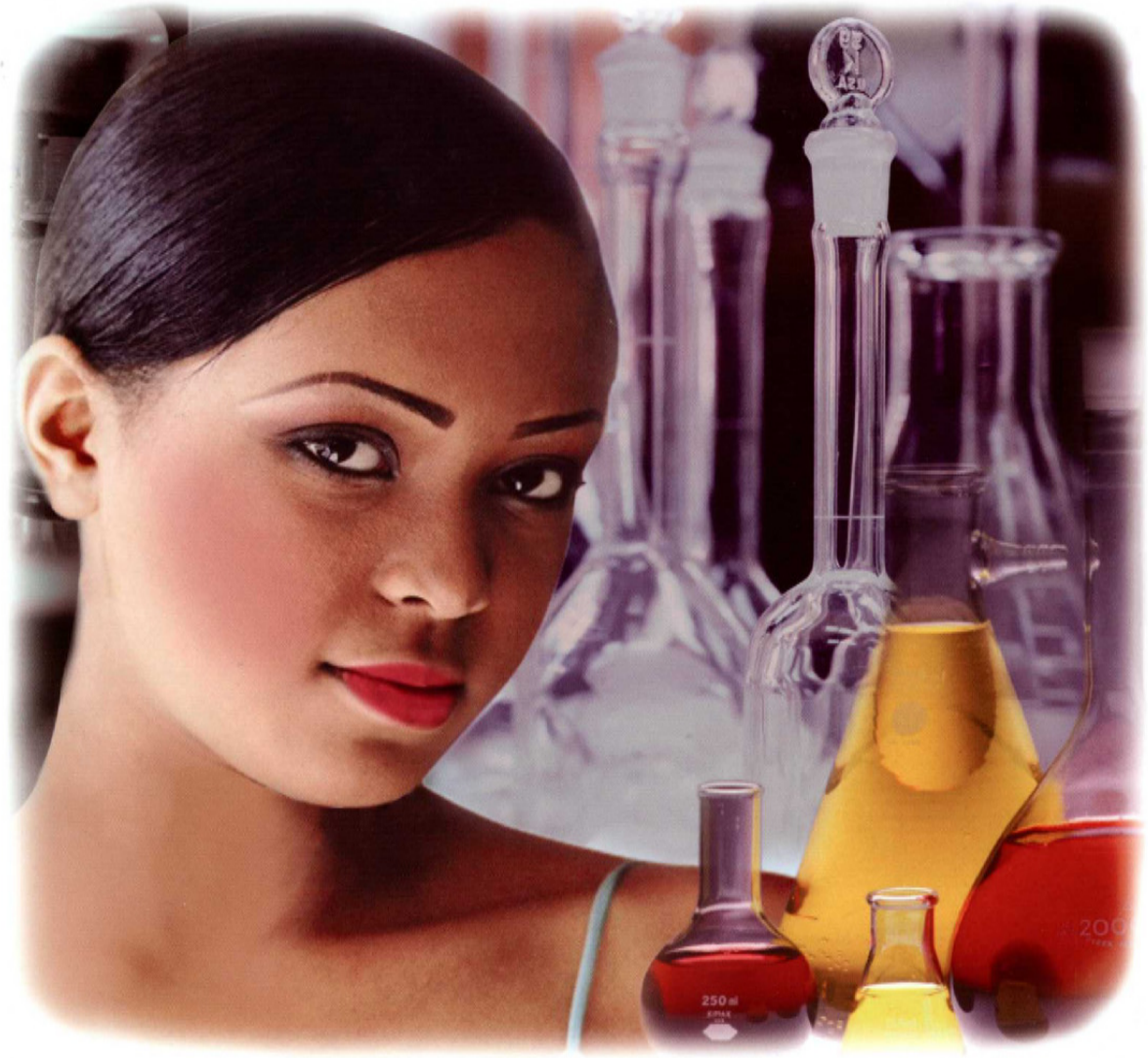

A Multifunctional Ingredient for Leave on Cosmetics

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Keywords: multifunctional ingredient, deodorant, sniff test, ethylhexylglycerin

Abstract

Ethylhexylglycerin is a multifunctional cosmetic ingredient with good deodorising and skin care properties. Several benefits make it very well applicable in different types of cosmetic products. Two main properties of the ethylhexylglycerin – the deodorising efficacy and the efficacy improvement of cosmetic alcohols – will be described. The deodorising efficacy has been proven in sniff tests against triclosan, a widely-used deodorant active. The boosting properties of the antimicrobial activity of several alcohols and glycols will be described exemplarily for phenoxyethanol and pentylene glycol.

Introduction

Ethylhexylglycerin is a glycerol monoalkylether of defined structure and high purity, with a 2-ethylhexyl group bound to the primary hydroxyl function of the glycerol molecule. Due to the fact that it is a crystal-clear, colourless liquid with a slightly characteristic odour it is well suited for the use in cosmetic products. Although it is less soluble in water (< 0.1 % at 25 °C), it is more easily soluble in most common cosmetic alcohols and glycols as well as oils. Ethylhexylglycerin is rather stable, e.g. against hydrolysis and elevated temperatures and compatible with cosmetic ingredients. Ethylhexylglycerin is globally approved and as a new substance accords with the European legislation listed on the ELINCS file (table 1).

Ethylhexylglycerin is a unique, multifunctional additive for cosmetic preparations which adds value to cosmetic formulations in different aspects (1, 2). It is a globally accepted deodorant active, being a very good alternative to triclosan (3, 4). Ethylhexylglycerin acts as an emollient and mild humectant leading to an improved skin feeling (gentle to the skin) with no "stickiness" after application. Furthermore it makes perfume oils or other ingredients soluble and helps them to stay longer on the skin. Ethylhexylglycerin can lower the surface tension in aqueous systems which may help to improve the antimicrobial efficacy of certain alcohols and glycols.

Deodorant activity

Body odour arises when sweat, odourless in itself, is decomposed by micro-organisms. From the sweat contents, the sebum and skin cell residues, the germs, primarily grampositive bacteria, form substances which have an unpleasant odour.

Antimicrobial deodorants reduce the number of odour-causing bacteria on the skin. They should not affect the normal skin flora and they should be effective for a reasonable time, for example, for about 12 hours. To avoid unpleasant body odour, different active principles are discussed. Sweat secretion is reduced/prevented by astringents, in particular predominantly aluminium salts such as aluminium hydroxychlorides. Astringents denaturise the skin proteins and influence the heat balance of the axilla region.

The microbial flora on the skin are reduced/inhibited by antimicrobial substances. Ideally only those micro-organisms causing the unpleasant odour should be destroyed. Usually the entire microflora of the skin is damaged. Body odour can be concealed by fragrances, although the mixture of body odour and perfume can be unpleasant rather than not.

A well known antimicrobial active ingredient used to reduce the formation of unpleasant body odours is triclosan: 5-chloro-2-(2,4-dichlorophenoxy)phenol. In this investigation we compare the deodorant activity of ethylhexylglycerin with triclosan as a standard substance. To find out the deodorant potential of ethylhexylglycerin sniff tests were performed.

Sniff test

The sniff test is a trial reflecting conditions encountered in practice. The sniff test determines the smell-inhibiting effect of the test products by directly sniffing at the armpits. The test is

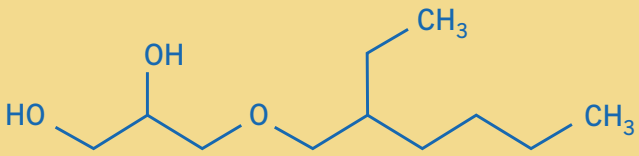
Ethylhexylglycerin	
Molecular formula	• $C_{11}H_{24}O_3$
CAS name:	• 3-[(2-Ethylhexyl)oxy]-1,2-propandiol
CAS-no.:	• 70445-33-9
ELINCS name	• Sensiva® SC 50
ELINCS no.:	• 408-080-2
International Approvals	
Europe / USA:	• INCI name: Ethylhexylglycerin
Australia:	• AICS/NICNAS (NA/966), TGA
Canada:	• DSL
Japan:	• ENCS as glycerin monoalkyl (or alkenyl, C8-C24) ether (No.: 2-414)
	• CLS 1999

Table 1. Ethylhexylglycerin, registration and approval.

performed in accordance with the method of Prof. Heiss of the Heidberg Hospital in Hamburg as described in "dragoco report" 6/76 (5).

Sniff test procedure

The procedure for determination of the smell-inhibiting effect of test products in a comparative study was carried out as described in figure 1.

Volunteers without allergic reactions of the skin, without hypersensitivity to deodorants or antiperspirants and without systemic or skin illness are selected for the test. Subjects must have distinct odour under the armpits after 10 days application of neutral soap. The test subjects are instructed to use an unperfumed soap without antibacterial active ingredients under the armpits over a period of 10 days before the test begins. No

Test panel	10 – 20 volunteers
Sex	female, male
Age	> 18 years
Test area	Armpits
Preliminary phase	10 days
Duration and frequency of application	twice a day (5 days)
Discontinuation of application before rating the initial value	approx. 6 hours after the last wash
Discontinuation of application before rating the final value	approx. 6, 16 and 24 hours after the last application of the product
Testers	3

Figure 1: Description of the sniff test procedure.

cosmetics are used in this body region throughout the test period. Only the unperfumed soap is used, once a day.

The initial values (iv) of body odour are determined on the 11th day, 6 hours after the last wash. The armpits are sniffed by three testers and the value of perspiration odour is rated by means of marks on a scale from 0 to 5:

0 = no. 1 = very low. 2 = low. 3 = moderate.
4 = high. 5 = very strong sweat odour.

A crucial factor in the rating process is the comparison of pairs (left to right armpits) for each volunteer. Noticeable differences in pairs are rated with a difference of at least one mark. The initial values must not be less than 3. The ratings of the three testers are averaged. After sniffing the initial values the deodorant formulations are issued to the volunteers who are instructed to apply the products in a standard manner over a period of 5 days, twice a day in the morning and the evening. Half of the volunteers apply one product to the right armpit and the other product to the left armpit. The other half apply the products the opposite way round. Sniffing of the final values is performed at predefined time intervals and applications. To achieve results that are relevant in practice, there is no standardisation of clothing.

Evaluation

The individual marks are averaged and the standard deviation is calculated. In order to check whether a difference exists between the deo-formulations tested in pairs or whether a difference can be detected between the initial and the final values of each formulation, statistical analysis is carried out. The Shapiro-Wilks test is applied to check whether the results of the study involve data with a normal distribution. Depending on the result of this, either the paired t-test (normal distribution of data) or the paired Wilcoxon test (non-normal distribution of data) is used to determine statistical differences of significance.

Sniff test results

Sniff tests on two different deodorant formulations were carried out. The first one was an alcohol based formulation and the second one an alcohol-free, silicon-based formulation. In both cases ethylhexylglycerin containing formulas were compared to triclosan containing products because triclosan is one of the most effective deodorant actives used worldwide. The tested concentrations were in accordance to the recommended use concentrations.

Alcohol based deodorant formulation

In the first case the deodorising activity of 0.3 % ethylhexylglycerin was compared with 0.1 % triclosan as bench marking and with the basic alcohol/water formulation respectively (table 2).

	A	B	C
Triclosan	0.1		
Ethylhexylglycerin		0.3	
Propylene glycol	1.0	1.0	1.0
Ethanol, denaturated	40.0	40.0	40.0
Water, demineralised	58.9	58.7	59.0

Table 2. Compositions of the deodorants of the first sniff test (%w/w).

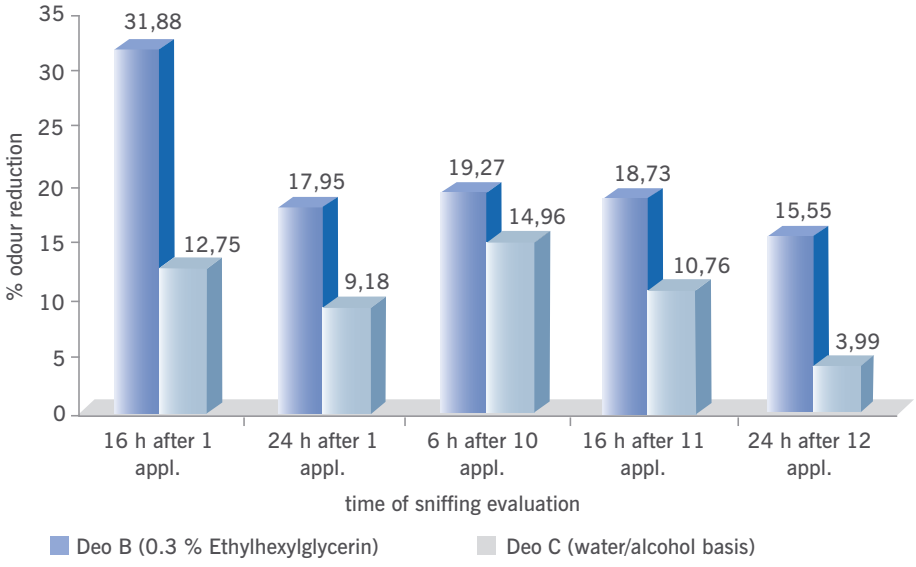


Figure 2. Sniff test Deo B versus Deo C.

Therefore two sniff tests were performed to compare deodorant A versus deodorant B and deodorant B versus deodorant C. Each test was carried out on 20 volunteers. Odour reduction of the test products measured against the initial values (iv) and against each other respectively are given in table 3 and figures 2 and 3. The underlined values in table 3 show a significant difference between initial and final values.

significantly versus the initial values at all times of assessment, up to 24 hours and in both investigations. For deodorant C – the ethanol/water basis – this effect can only be confirmed for the short term investigations.

Figure 3 shows the comparison between the ethylhexylglycerin containing deodorant and the triclosan containing one. Deodorant A with 0.1 % triclosan reduces the perspiration odour of the

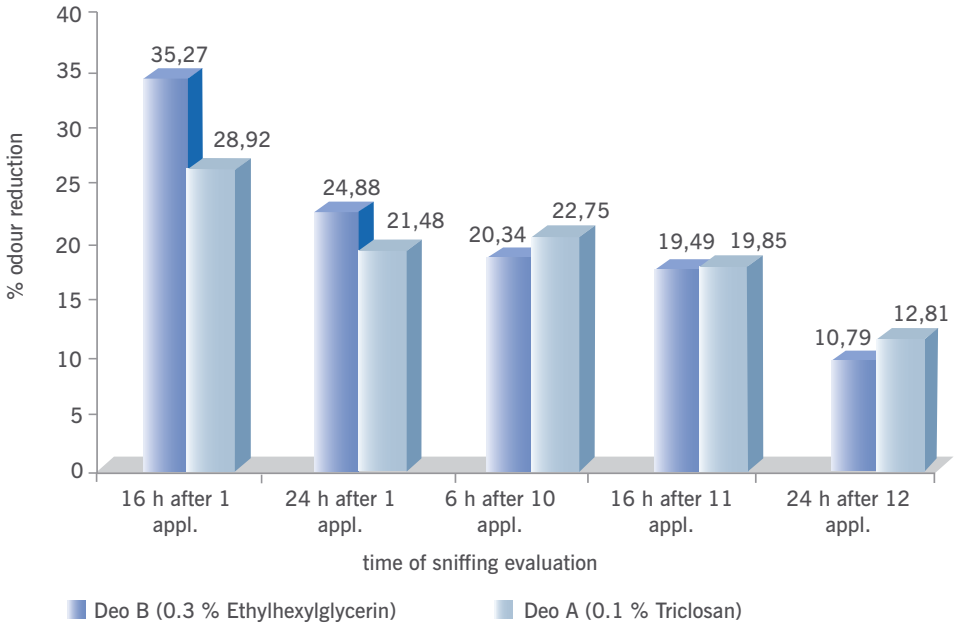


Figure 3. Sniff test Deo A versus Deo B.

As figure 2 shows, there is at every test time a difference in odour reduction between ethylhexylglycerin containing deodorant B and the basic alcohol/water deodorant C. The highest observed differences for the odour reduction were at 16h after the 1 application and 24h after the 12th application. Deodorant B with 0.3 % ethylhexylglycerin reduces the perspiration odour armpits significantly at all times of assessment. In all cases for both products a significant odour reduction compared to the initial value can be observed.

But, as table 3 shows there was no significant difference between deodorants with 0.3 % ethylhexylglycerin and 0.1 % triclosan.

	Percent odour reduction				
	16 h after 1 appl.	24 h after 1 appl.	6 h after 10 appl.	16 h after 11 appl.	24 h after 12 appl.
Deo B (0.3% Ethylhexylglycerin) vs. iv	31.88 %	17.95 %	19.27 %	18.73 %	15.55 %
Deo C (water/alcohol basis) vs. iv	12.75 %	9.18 %	14.96 %	10.76 %	3.99 %
Deo B vs. Deo C	19.13 %	8.77 %	4.31 %	7.97 %	11.56 %
Significant difference	yes	yes	no	no	yes
Deo B (0.3% Ethylhexylglycerin) vs. iv	35.27 %	24.88 %	20.34 %	19.19 %	10.79 %
Deo A (0.1% Triclosan) vs. iv	28.92 %	21.48 %	22.75 %	19.85 %	12.81 %
Deo B vs. Deo A	6.35 %	3.40 %	-2.41 %	-0.36 %	-2.02 %
Significant difference	no	no	no	no	no

Table 3. Results of sniff tests of water/alcohol based formulations with 0.3 % ethylhexylglycerin (Deo B) and 0.1% triclosan (Deo A) and the basic formula (Deo C) respectively.

The sniff test proves a quantitatively similar smell-inhibiting effect of both test products.

Alcohol free deodorant formulation

As will be described later, ethylhexylglycerin can improve the antimicrobial efficacy of lower alcohols (6). A further sniff test on an alcohol-free formulation was carried out to exclude the possibility that the observed efficacy of the alcohol based deodorant is only due to the efficacy boost of ethylhexylglycerin to the alcohol. Therefore the smell-inhibiting effect of a sodium stearate deo-stick formula containing 0.6 % ethylhexylglycerin compared to 0.3 % triclosan was determined (table 4). The test was carried out on 10 volunteers. Consequently all differences stated as significant can only count as tendencies.

	A	B
Triclosan	0.3	
Ethylhexylglycerin		0.6
Propylene glycol	20.0	20.0
Cyclomethicone and Dimethicone Copolyol	20.0	20.0
Cyclomethicone	52.7	52.4
Polysorbate 20	1.0	1.0
Sodium Stearate	6.0	6.0

Table 4. Compositions of the deodorants of the second sniff test (%w/w).

Table 5 and figure 4 summarise the results of the respective rating times. The underlined values in table 5 show a significant difference between initial and final values.

Applying 0.3 % triclosan deo-stick for five days causes a significant improvement in odour over the initial value, when rated the 10th application after six hours. Otherwise no significant differences to the initial values are found.

Contrary to that, the formulation containing 0.6 % ethylhexylglycerin shows at every rating time a significant odour reduction compared to the initial value except for one. Only 24 hours after the 12th application no significant difference could be observed. At no rating time is a significant difference found between 0.6 % ethylhexylglycerin and 0.3 % triclosan applied as a deo-stick under the armpits. Accordingly, this test indicates a quantitatively similar reduction in perspiration odour for both products.

Beside the effectiveness of a deodorant active against odour-causing germs, it is very important to test whether the deodorant active will affect the natural skin flora. Therefore the antimicrobial activity of three water/alcohol based deodorant formulations, tested in a sniff test, against selected grampositive bacteria found in the axilla region was determined in a dilution test (table 6, next page).

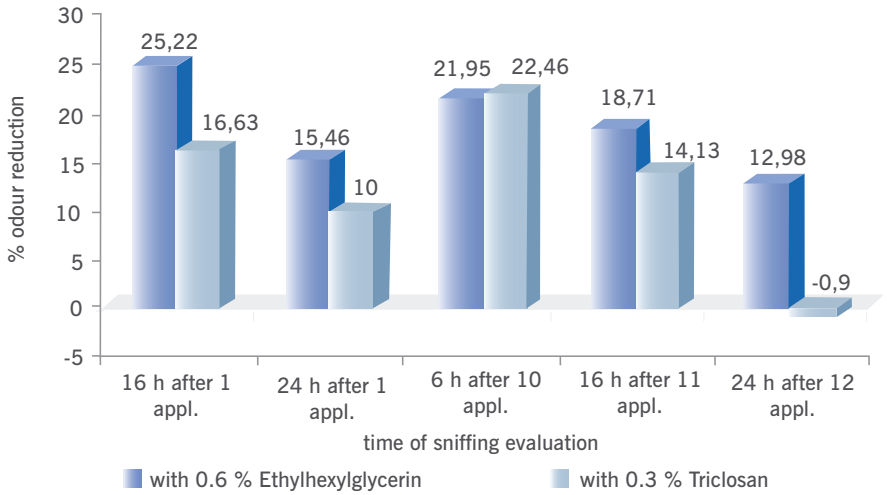


Figure 4. Sniff test: Deo stick with 0.3 % triclosan versus 0.6 % ethylhexylglycerin.

	Percent odour reduction				
	16 h after 1 appl.	24 h after 1 appl.	6 h after 10 appl.	16 h after 11 appl.	24 h after 12 appl.
0.3 % Triclosan vs. iv	16.63 %	10.00 %	22.46 %	14.13 %	-0.90 %
0.6 % Ethylhexylglycerin vs. iv	25.22 %	15.46 %	21.95 %	18.71 %	12.98 %
0.3 % Triclosan versus					
0.6 % Ethylhexylglycerin	-8.59 %	-5.46 %	0.51 %	-4.58 %	-13.88 %
Significant difference	no	no	no	no	no

Table 5. Results of a sniff test of alcohol-free deo stick formulations with 0.6 % ethylhexylglycerin and 0.3 % triclosan respectively.

Fomulation	Microorganism	Concentration (Dilution)				
		50 %	25 %	12.5 %	6.25 %	3.12 %
Deo A	SX	-	-	-	-	-
0.1 % Triclosan	MK	-	-	-	-	-
1.0 % Propylene glycol	SE	-	-	-	-	-
40.0 %Ethanol, denaturated	CC	-	-	-	-	-
58.9 % Water, demineralised	CN	-	-	-	-	-
	CF	-	-	-	-	-
Deo B	SX	-	-	+	+	+
0.3 % Ethylhexylglycerin	MK	-	-	+	+	+
1.0 % Propylene glycol	SE	-	-	+	+	+
40.0 % Ethanol, denaturated	CC	-	-	-	+	+
58.7 % Water, demineralised	CN	-	-	+	+	+
	CF	-		-	+	+
Deo C	SX	-	-	+	+	+
1.0 % Propylene glycol	MK	-	+	+	+	+
40.0 % Ethanol, denaturated	SE	-	-	+	+	+
59.0 % Water, demineralised	CC	-	-	+	+	+
	CN	-	-	+	+	+
	CF	-	-	-	+	+
+ = growth / - = no growth						
SX = Staphylococcus xylosus 2		MK = Micrococcus kristinae				
SE = Staphylococcus epidermidis		CC = Corynebacterium collunae				
CN = Corynebacterium nephredii		CF = Corynebacterium flavescens				

Table 6. Inhibitory concentrations (dilution test) of water/alcohol based deodorant formulations (tested in a sniff test) against some grampositive bacteria extracted in the axilla region.

Growth of the grampositive bacteria in this investigation is only moderately inhibited by ethylhexylglycerin and the water/alcohol basis respectively. The triclosan formulation inhibits the growth of bacteria at much lower concentrations. On the other hand, the deodorant activity of the ethylhexylglycerin formulation is quantitatively similar to that of the triclosan formulation – as determined in a sniff test. This result shows that the microbial flora of the skin might be affected to a lesser extent by ethylhexylglycerin than by triclosan.

Ethylhexylglycerin is a gentle to the skin but effective deodorant active. It gives a good protection against unpleasant body odour up to 24 hours after the last application. Based on the results of sniff tests in different formulations it can be recommended

as an active ingredient for all types of deodorants like sprays, sticks and roll-ons. Good deodorant activity in a sniff test is found at a concentration of 0.3 % and higher. As the efficacy of a deodorant product depends on the entire formulation, the use level of 0.3 % - 0.6 % ethylhexylglycerin should be seen as a basic recommendation. Whether the observed deodorant efficacy results from the antimicrobial efficacy of ethylhexylglycerin against grampositive bacteria or on other still unknown effects is not yet finally proved.

Ethylhexylglycerin is commercially available under the trade name Sensiva® SC 50.

Improved efficacy of cosmetic alcohols

Beside the potential activity of ethylhexylglycerin against some odour-causing grampositive bacteria, no antimicrobial efficacy against gramnegative bacteria yeasts or moulds could be found. If ethylhexylglycerin is combined with certain cosmetic ingredients (e.g. alcohols, glycols, lipids, hydro-lipids, lipoaminoacids) a rise in the antimicrobial properties of these ingredients can be observed (7). Some of these formulations might be useful in stabilising cosmetic and personal care preparations against microbial attack. But it should be kept in mind that the stabilising effect of such combinations depends on the whole formula and has to be checked individually.

In the following the phenomenon will be described for phenoxyethanol and pentylene glycol.

Phenoxyethanol

Phenoxyethanol is a well-known and accepted preservative active which is widely used in cosmetic formulations. Ethylhexylglycerin is a multifunctional cosmetic ingredient which can boost the efficacy of several alcohols and glycols.

With Euxyl® PE 9010 a patented combination of 90 % phenoxyethanol and 10 % ethylhexylglycerin was introduced as an innovative cosmetic preservative to the cosmetic market (8). To show the effect of ethylhexylglycerin on the performance of phenoxyethanol as a preservative, germ count reduction tests on Pseudomonas aeruginosa (ATCC no. 15442) and Aspergillus niger (ATCC no. 6275) were performed with dilutions of the combination of both and the single components in sterile tap water. Fifty-ml portions of the end solutions were each inoculated with 0.5 ml micro-organism suspension and stirred. The initial micro-organism count of the test solutions was approximately 10^8 cfu/ml.

These solutions were streaked out onto tryptone soya agar (Pseudomonas aeruginosa) or sabouraud-dextrose 4.0 % agar (Aspergillus niger) after 3, 6, 24, 48, 72 and 168 hours depending on the test organism. The cultures were incubated for 48 hours at 37°C or 72 hours at 25 -27°C. The evaluation was made on the basis of semi-quantitative assessment of the microbial growth of the streaks.

As the graphs clearly demonstrate, ethylhexylglycerin, without having any efficacy against Pseudomonas aeruginosa or

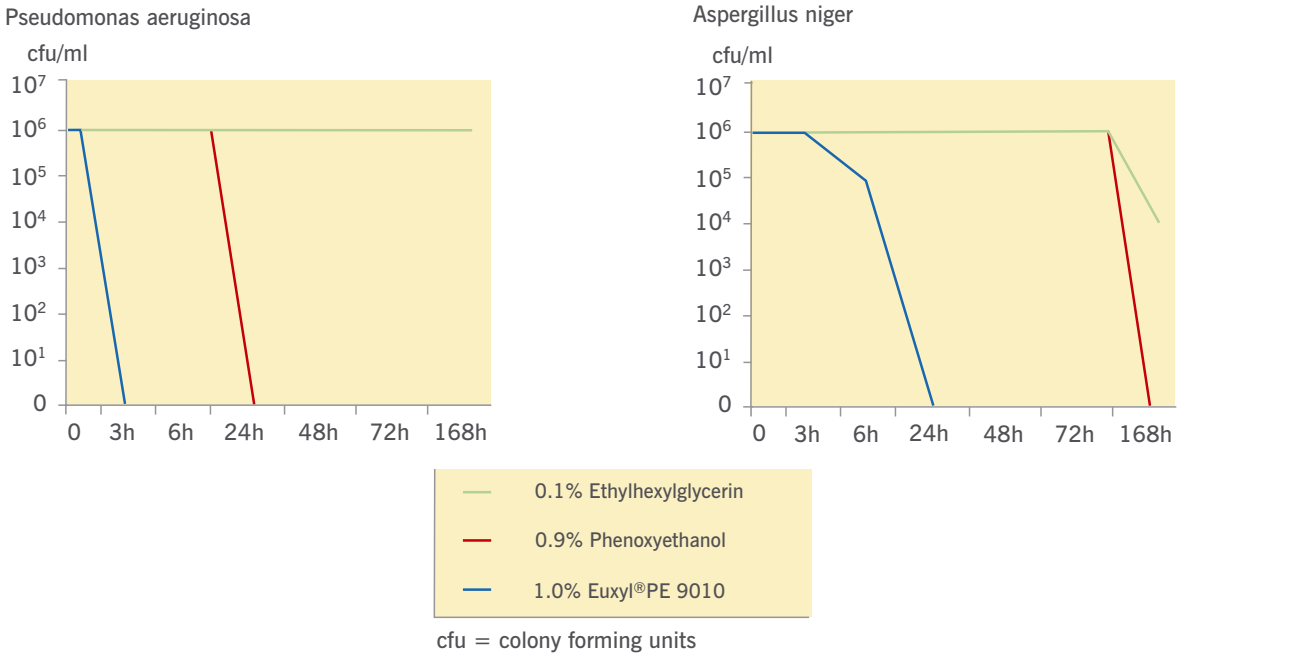


Figure 5. Germ count reduction achieved by a combination of phenoxyethanol and ethylhexylglycerin compared to the single components against Pseudomonas aeruginosa and Aspergillus niger.

Aspergillus niger alone (green line), boosts the efficacy of phenoxyethanol as the preservative active in Euxyl® PE 9010 (blue line) compared to phenoxyethanol alone (red line). The improved efficacy of the combination can be seen after a short period of time (figure 5, previous page).

Mode of action

The chemical structure has to be considered to find an explanation as to how such small amounts of ethylhexylglycerin without having its own efficacy can enhance the preserving efficacy of phenoxyethanol as previously described.

Ethylhexylglycerin has a chemical structure comparable to surfactants. It can reduce the surface tension of water significantly (figure 6a). A calculated HLB-value of 7.4 means that it belongs to the group of wetting agents or water-in-oil emulsifiers.

In combination with phenoxyethanol an additional reduction of the surface tension can be observed. For a 1.0 % solution of Euxyl® PE 9010 in water the surface tension is 32.1 mN/m (water: 72.6 mN/m). This is even lower than the surface tension of 0.1% ethylhexylglycerin, as the contact angles of the corresponding water drops on materials like polyethylene clearly demonstrate (figure 6b).

Although the combination of phenoxyethanol and ethylhexylglycerin significantly reduces the interfacial tension in a foaming test according to DIN 53 902, a 1.0 % solution of it in demineralised water proved to be non-foaming.

It is postulated that the addition of ethylhexylglycerin affects the interfacial tension at the cell membrane of micro-organisms. This improves the contact of phenoxyethanol with the cell membrane resulting in a better interaction of phenoxyethanol at the cell membrane and an improved antimicrobial efficacy.

The effectiveness of this system in cosmetic formulations has been tested in the S&M Koko test (9, 10). This is a multiple-inoculation, preservative efficacy test designed and validated by us. A mixed suspension of grampositive and gramnegative bacteria, yeast and mould is used for inoculation. At weekly intervals, a sample of the test product is streaked out onto nutrient media, incubated and evaluated semi-quantitatively. The longer the time before the occurrence of the first microbial growth, the more effective the preservative. Experience has shown that a well-preserved product should remain growth-free for six inoculation cycles to ensure the shelf-life required in practice (30 months in the original packaging).

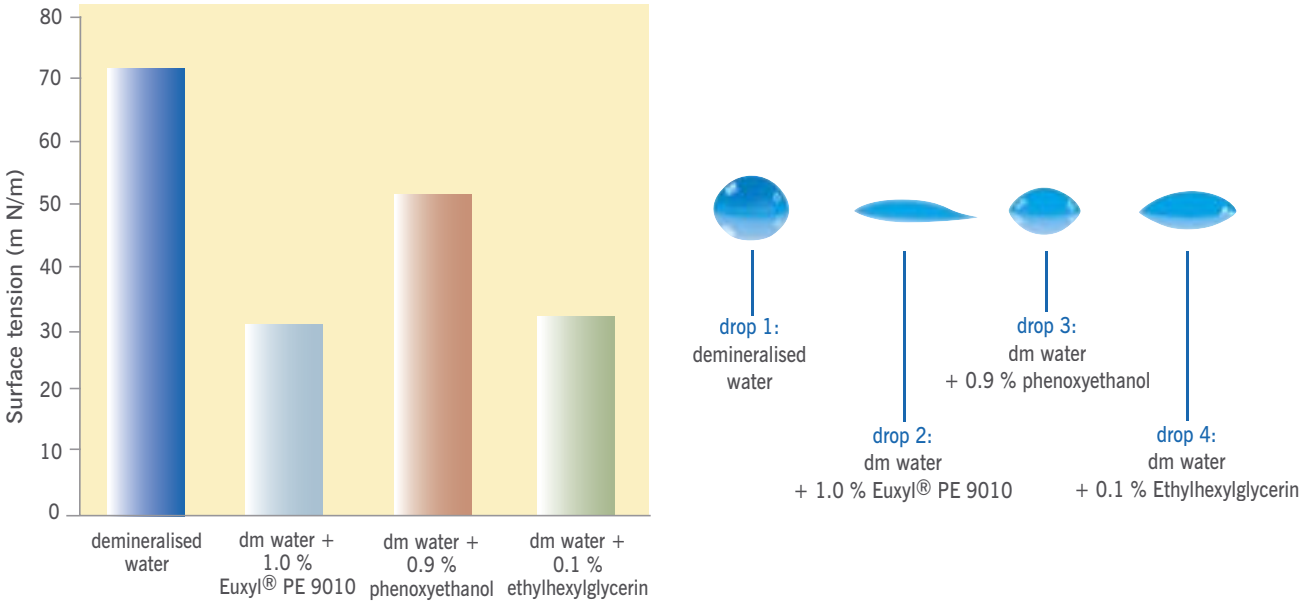


Figure 6a. Surface tension [mN/m] of 1.0 % Euxyl® PE 9010 in water compared to 0.9 % phenoxyethanol, 0.1 % ethylhexylglycerin and water as well as Figure 6b. The corresponding water drops on a polyethylene surface.

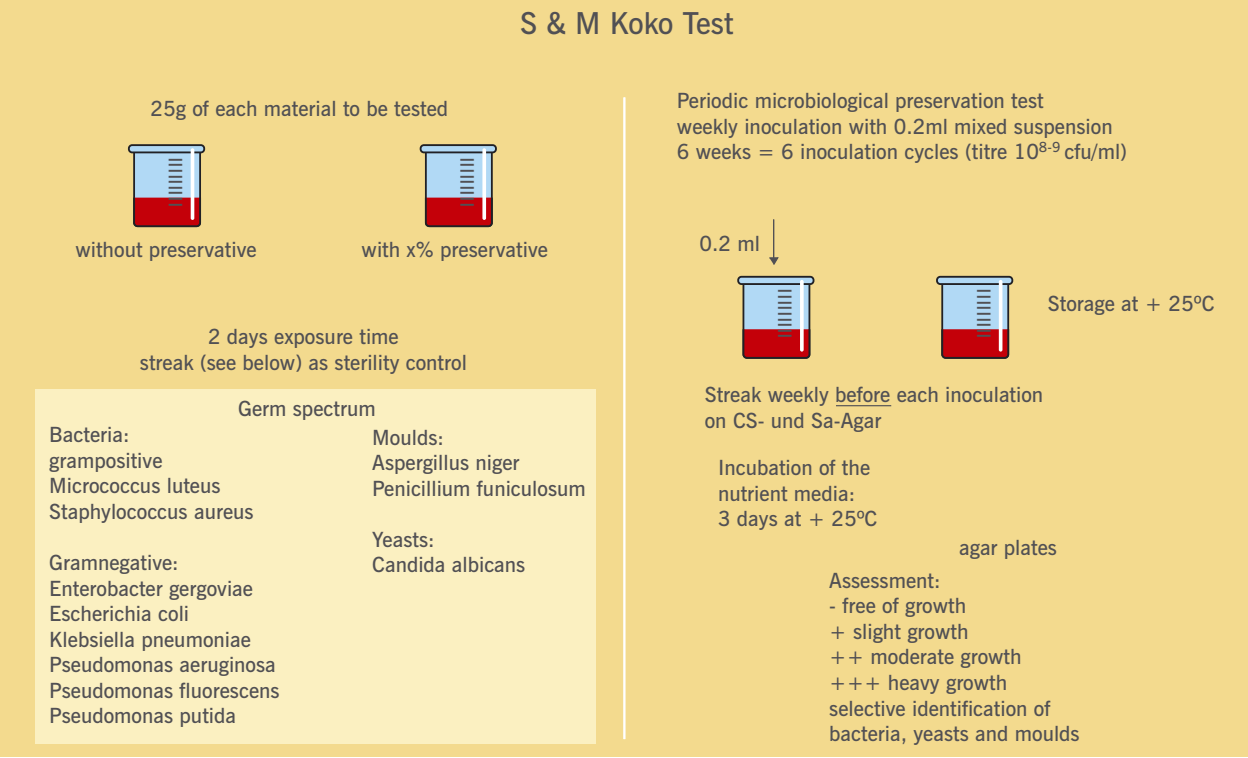


Figure 7. Evaluation of the microbial stability of cosmetic formulations with the S&M Koko test.

The results of those tests show that Euxyl® PE 9010 is most suitable for the preservation of leave-on cosmetics such as creams, lotions, wet wipes, hair care and hair styling products. The recommended use concentration in those types of product is 0.5 – 1.0 %. The evaluation of several challenge tests has shown that it is as effective as phenoxyethanol based paraben mixtures, if used in the same use concentration.

Pentylene glycol

A similar action compared to phenoxyethanol can also be described for ethylhexylglycerin in combination with glycols like butylene glycol, pentylene glycol or caprylyl glycol. This will be demonstrated exemplarily for the combination pentylene glycol / ethylhexylglycerin. Pentylene glycol is a multifunctional cosmetic ingredient for which a certain antimicrobial efficacy is known. It is often used in systems claimed “preservative free”. How the efficacy of pentylene glycol in those formulations can be improved by combining it with ethylhexylglycerin will be shown exemplarily on an o/w lotion (table 7). Therefore to the basic formula A 5.0 % pentylene glycol (formula B), 0.5 % ethylhexylglycerin (formula C) and the combination of both (formula D) was added.

o/w lotion					
		Formula			
		A	B	C	D
		% w/w	% w/w	% w/w	% w/w
Phase A	Aqua	83.5	83.5	83.5	83.5
	Propylene Glycol	2.0	2.0	2.0	2.0
Phase B	Paraffinum Liquidum	9.5	9.5	9.5	9.5
	Cetearyl Alcohol	3.0	3.0	3.0	3.0
	Ceteareth-25	2.0	2.0	2.0	2.0
Phase C	Pentylene Glycol	-	5.0	-	5.0
	Ethylhexylglycerin	-	-	0.5	0.5

Table 7. Compositions of the o/w lotion tested with a combination of pentylene glycol and ethylhexylglycerin.

Test material	Inoculation cycles						
o/w lotion	0	1	2	3	4	5	6
Formula A	-	+++ BYM	+++ BYM	./.			
Formula B	-	+++ M	+++ M	./.			
Formula C	-	+++ BM	+++ BM	./.			
Formula D	-	-	-	-	-	-	-
O	Sterility control			-	free of microbial growth		
B	Bacteria			+	slight growth		
M	Moulds			++	moderate growth		
Y	Yeast			+++	massive growth		

Figure 8. Result of challenge test on the o/w lotion during 6 inoculation cycles with pentylene glycol / ethylhexylglycerin.

The results of the challenge test are given in figure 8. As the picture shows, the basic formulation A, as well as the formulations containing only pentylene glycol (B) or ethylhexylglycerin (C), fail the challenge test after two inoculation cycles. Compared to that, the combination of 5.0 % pentylene glycol with 0.5 % ethylhexylglycerin is free of growth even after 6 inoculation cycles.

Those formulations may be claimed “preservative free” or self preserving systems.

Some trials were done in using a combination of pentylene glycol and ethylhexylglycerin for self preserving systems. Most formulations showed self preserving properties with a combination of 3.0 % pentylene glycol and 0.5 % ethylhexylglycerin. But due to the fact that this effect depends on the whole formulation, it is recommended to test every new cosmetic formulation separately.

Conclusion

Ethylhexylglycerin is a multifunctional cosmetic ingredient with excellent deodorising properties. It gives a good protection against unpleasant body odour up to 24 hours after the last application, without strongly influence the natural skin flora.

Because of its varied benefits it can be used in cosmetic products for different reasons as these are:

- emollient and mild humectant, leaving a pleasant feeling on the skin
- deodorant active with additional skin care properties
- solubiliser for certain cosmetic ingredients e.g. perfume oils which may help them to stay longer on the skin
- enhancer for the antimicrobial efficacy of certain cosmetic alcohols and glycols.

Ethylhexylglycerin is a versatile additive for cosmetic and personal care products with multifunctional, especially deodorant activity, which can be used in different types of cosmetic products like deodorants, sun care products, baby care products or face creams.

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Author’s Biography

Dr. Marion Leschke, born in 1974, has studied chemistry in Chemnitz (Germany) in the field of organometallic and complex chemistry. After studying she worked for 3 years in the R&D department for a contract filler for aerosols in the cosmetical, household and technical area. Since 2005 she works as product manager for Ethylhexylglycerin at Schülke & Mayr.