Jungbunzlauer

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facts

Bio-based preservation of home care formulations with lactic acid

Abstract

Natural origin, safety and sustainability range among the top trends in home care. This includes the preservatives used in such formulations. Consumers are becoming increasingly aware of the potential allergenic and sensitising properties of common active substances. Regulatory bodies are reacting to new scientific evidence and restricting the application of these ingredients.

Here, we present L(+)-lactic acid as a viable alternative to synthetic and hazardous preservatives. We demonstrate its efficacy in simple aqueous surfactant solutions and typical home care formulations including softener, surface cleaner and hand dish wash detergent.

Introduction

The trend in consumer products towards more natural, more sustainable, gentler and safer formulations is unbroken. For non-food applications the cosmetics industry is the typical first mover and a good indicator as to which trends and developments will spread to other market segments. In the area of preservation, personal care products have seen the declining use of parabens and isothiazolinones for a long time. Both are suspected, with some evidence, of exhibiting harmful properties, including allergenic and sensitising effects. Among the replacements for these substances we find firstly phenoxyethanol, and, increasingly, preservatives known from and broadly used in the food industry.

In home care, methylisothiazolinone and quaternary ammonium compounds remain the most widely used preservatives. However, in recent years their share in new product launches has started to decrease. Phenoxyethanol, potassium sorbate and sodium benzoate are on the rise. These are certainly less harmful alternatives, but still not completely innocuous and definitively not of natural origin. Here, again, the cosmetics industry is already one step ahead. In this industry we observe a growing use of stabilising ingredients derived from natural processes such as fermentation or directly via plant extraction.

L(+)-lactic acid is one of these ingredients: it is produced by fermentation of renewable resources, i.e. of natural origin. As a liquid it is easy to handle and to dose for home care formulations, which are typically liquid. Lactic acid is non-allergenic and non-sensitising. In the cleaning industry it is known for its descaling and disinfecting properties.

In the present article we will provide some basic efficacy data on the preservation performance of lactic acid and we will show that it is capable of keeping typical home care formulations microbiologically stable.

The need for such an alternative preservative is not just linked to above-mentioned market and consumer trends, but is also driven by regulatory developments. In the European Union, Regulation (EU) No 528/2012, the Biocidal Products Regulation (BPR), sets the legal framework for the approval and use of biocides of all kinds, including in-can preservatives, which fall under product type (PT) 6[1]. The BPR leads to a consolidation of the biocides market, i.e. the number of available actives is drastically shrinking. For PT 6, there are currently only 11 approved substances, a further 37 ingredients are under evaluation. Apart from ethanol, lactic acid is the only ingredient of natural origin. Furthermore, some common actives are being re-evaluated in terms of health hazards; one prominent example is methylisothiazolinone, which will be classified as allergenic starting from concentrations as low as 15 ppm from 1 May 2020[2]. Similar restrictions, stricter classifications and bans of common synthetic preservatives can be observed in other parts of the world, too, and are not linked solely to the preservation of home and personal care products. Paints, inks and coatings are among other categories affected.

Experimental part

To assess the preservation efficacy of lactic acid we tested according to two different norms. European Pharmacopoeia (Ph. Eur.) 5.1.3 describes a protocol to determine the microbiological stability of a given product[3]. It is one of the strictest tests on the market (as compared, for instance, to USP or DIN EN ISO 11930).

Originally designed for topical preparations (cosmetic/pharmaceutical), it is widely used also for other applications. According to Ph. Eur. 5.1.3 the test item is inoculated with 10^5 to 10^6 colony forming units (CFU) of selected microorganisms per millilitre. The microorganisms include Staphylococcus aureus (gram-positive coccus), Pseudomonas aeruginosa (gram-negative bacillus), Escherichia coli (gram-negative bacillus), Candida albicans (yeast), and Aspergillus brasiliensis (mould). The prepared samples are stored at room temperature and the concentration of CFU/ml determined after defined intervals (2, 7, 14 and 28 days).

Depending on the observed decay of microorganisms and the (non-) occurrence of growth, the test is rated as passed or failed based on the acceptance criteria (table 1). There are two pass criteria, A and B. A refers to the recommended preservation efficacy. However, for many preparations, in particular for gentle formulations with neutral or close to neutral pH value, criterion A is difficult to achieve. It could only be met using rather harsh substances or by increasing the preservative concentration to undesirably high levels. This led to the introduction of criterion B. Here, the required log reduction of the inoculum is either lower than with criterion A or it is tested at a later point in time.

Table 1: Criteria of acceptance for Ph. Eur. test

	Test criteria	Log reduction (Rx), NI = no increase vs. previous			
		2 d	7 d	14 d	28 d
Bacteria	A	2	3	-	NI
	B	-	-	3	NI
Fungi	A	-	-	2	NI
	B	-	-	1	NI

IBRG PDG 16-007.02 describes a method to determine the basic efficacy of biocidal active substances used to preserve aqueous-based products[4]. It is one of the recommended test protocols according to the ECHA Guidance on the BPR[5].
In contrast to Ph. Eur. 5.1.3 it involves not just one initial microbial inoculation, but several consecutive inoculations at weekly intervals to challenge the preservative system. Thus, this approach provides more realistic but also more demanding test conditions. Further, it includes additional microbial species compared to Ph. Eur., namely Streptococcus epidermidis (gram-positive coccus), Klebsiella pneumoniae (gram-negative bacillus), Bacillus subtilis (gram-positive bacillus), Cryptococcus neoformans (yeast) and, instead of Aspergillus brasiliensis, Geotrichum candidum (mould). Target inoculum is 10^5 CFU/ml, inoculation is repeated up to a maximum of three times. The test is passed when the preserved sample shows no microbial growth while the unpreserved sample shows growth.

Results and discussion

In a first step we analysed the basic preservation efficacy of lactic acid in simple aqueous surfactant solutions as a function of pH. The solutions consisted of 3% sodium lauryl ether sulfate (SLES) and 0.9% L(+)-lactic acid (active concentration). Test results according to Ph. Eur. 5.1.3 are displayed in table 2. Up to a pH of 3.5 lactic acid is sufficiently effective to keep the system microbiologically stable. At pH 4 the test fails for the mould. As expected, the lower the pH the better the performance of the organic acid. Nevertheless, with efficacy proven up to pH 3.5, a broad range of acidic home care formulations can be addressed. The concentration of lactic acid has been set below 1% so as not to affect the hazard labelling of the final formulation.

Table 2: Preservation efficacy of lactic acid in basic solutions acc. to Ph. Eur.

	0.94% LA + 3% SLES			
Test germ	**pH 2**	**pH 3**	**pH 3.5**	**pH 4**
E. coli	A	A	A	A
P. aeruginosa	A	A	A	A
S. aureus	A	A	A	A
C. albicans	A	A	A	A
A. brasiliensis	A	A	A	F
Total test results	**A**	**A**	**A**	**F**

Next, we tested the preservation activity of lactic acid in three different acidic home care model formulations: surface cleaner, fabric softener and hand dish wash detergent.

The composition of the surface cleaner is shown in table 3. It is kept very simple with two anionic and one non-ionic surfactant, fragrance, colour and 0.9% lactic acid, at a pH 3. Table 4 provides the test result according to Ph. Eur. – passed according to criterion A, in line with the base data from table 2. The same formulation – but at pH 2 and additionally thickened with 0.2% xanthan gum – was also subjected to the IBRG protocol (table 5 and figure 1). Again, lactic acid passed the test. The detailed graphical representation underlines the powerful biocidal activity of lactic acid, leading to an almost complete reduction of the microbial count at the latest after the second inoculation (actual reported values are < 1.0×10^1). At the same time the unpreserved sample shows not just stable counts, but significant growth.

Table 3 shows the fabric softener formulation. It primarily consists of the esterquat – the actual active component – and some calcium chloride for viscosity adjustment. Test results are displayed in tables 4 and 5. Both Ph. Eur. (for the low-concentration formula, pH 3) and IBRG (for the concentrate, pH 2) were passed with lactic acid, while controls show growth. Again, microbial counts could be reduced significantly, evidencing the strong disinfectant and preservative properties of this bio-based ingredient.

Table 3: Composition of model formulations (active concentrations)

Substance	Function	Surface cleaner	Fabric softener	Fabric softener concentrate	Hand dish wash detergent
		Active substance concentration in the formulation/%			
Sodium Lauryl Sulfate	Anionic surfactant	2			
Sodium Laureth Sulfate	Anionic surfactant	2			15
Decyl Glucoside	Non-ionic surfactant	2			
Coco Glucoside	Non-ionic surfactant				0.5
Cocamidopropylbetaine	Amphoteric surfactant				3
Dihydrogenated Tallow Hydroxyethylmonium Methosulfate	Cationic surfactant		8		
Dihydrogenated Palmoylethyl Hydroxyethylmonium Methosulfate	Cationic surfactant			20	
Calcium Chloride	Viscosity adjustment		0.025	0.075	
Sodium Chloride	Viscosity adjustment				4.5
Potassium Lactate	Moisturising agent				5
Lactic Acid	**Preservative**	**0.9**	**0.9**	**0.9**	**1.9**
Perfume	Fragrance	0.1	0.6	0.25	0.1
Colour	Colour	Qs	Qs	Qs	Qs
Aqua		Qs to 100	Qs to 100	Qs to 100	Qs to 100

Finally, we developed and tested a hand dish wash detergent. Its composition is shown in table 3. Besides the surfactant system we included potassium lactate as moisturiser and sodium chloride as thickening agent. Due to the relatively high pH of 4, we increased the concentration of lactic acid to 1.9% to achieve the preservation target. Indeed, as detailed in table 4, the formulation with lactic acid passed the Ph. Eur. test, while the unpreserved formulation failed for mould. So, even under somewhat demanding conditions, i.e. pH closer to the neutral zone, lactic acid still exhibits reliable stabilising properties against microbial spoilage.

The present test series confirms the capability of lactic acid to preserve different home care formulations. Though by its nature as an organic acid it is limited to the acidic range of cleaning and laundry products, the data show that lactic acid guarantees product stability up to pH 4 as sole preservative. As such, it could replace many common synthetic and hazardous preservatives and contribute to the safety and sustainability of formulations. In an earlier publication we also addressed the use of lactic acid as preservative at an even higher, skin-neutral pH[6]. While it is indeed possible to use lactic acid in this way, a booster is needed for extra preservative power, particularly against moulds. We used 1,2-hexanediol as the booster. Further boosters we evaluated and found suitable include anisic acid and caprylic acid. Such substances allow the efficacy range of lactic acid to be extended into neutral pH levels, which is important for more sensitive applications in cosmetics and personal care.

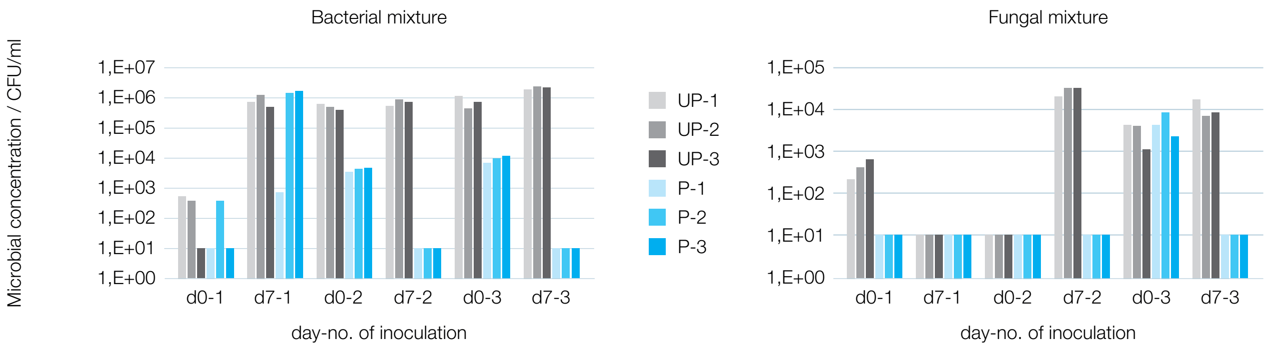
Table 4: Test results according to Ph. Eur. 5.1.3

	Surface cleaner		Fabric softener		Hand dish wash detergent	
Test germ	**Unpreserved**	**Preserved**	**Unpreserved**	**Preserved**	**Unpreserved**	**Preserved**
E. coli	-	A	A	B	A	A
P. aeruginosa	-	A	A	B	A	A
S. aureus	-	A	A	B	A	A
C. albicans	-	A	A	A	A	A
A. brasiliensis	-	A	F	B	F	A
Total test results	**-**	**A**	**F**	**B**	**F**	**A**

Table 5: Test results according to IBRG; table indicates growth, stability or reduction of microbial counts on day 7 after each inoculation.

		Surface cleaner		Fabric softener	
Test germ	**Inoculation**	**Unpreserved**	**Preserved**	**Unpreserved**	**Preserved**
Bacterial mixture	1	Growth	Growth	Stable	Growth
	2	Growth	Reduction	Growth	Reduction
	3	Growth	Reduction	Reduction	Reduction
Fungal mixture	1	Reduction	Reduction	Reduction	Reduction
	2	Growth	Reduction	Growth	Reduction
	3	Growth	Reduction	Growth	Reduction
Total test results		**Failed**	**Passed**	**Failed**	**Passed**

Figure 1: Detailed IBRG test results for the surface cleaner (UP – unpreserved samples, P – preserved samples)



Summary

Global trends point towards the replacement of synthetic and hazardous chemicals by safe and bio-based ingredients. The field of preservatives is still largely dominated by traditional actives, including isothiazolinones and quaternary compounds, while there is an increasing pressure from authorities and consumers to find and implement alternatives. Here, we demonstrated that L(+)-lactic acid is one promising option to comply with this development.

Tests according to the strict and well-established Ph. Eur. and IBRG norms have proven the preservative efficacy of lactic acid in basic aqueous surfactant solutions and model formulations, including surface cleaner, fabric softener and hand dish wash detergent. As a natural-origin ingredient which is readily biodegradable and neither allergenic nor sensitising, lactic acid meets contemporary preservation expectations without compromising on biocidal performance.

References

[1] Regulation (EU) No 528/2012 of the European Parliament and of the Council of 22 May 2012 concerning the making available on the market and use of biocidal products.

[2] Commission Regulation (EU) 2018/1480 of 4 October 2018 amending, for the purposes of its adaptation to technical and scientific progress, Regulation (EC) No 1272/2008 of the European Parliament and of the Council on classification, labelling and packaging of substances and mixtures and correcting Commission Regulation (EU) 2017/776.

[3] European Pharmacopeia (7th ed.), 5.1.3. Efficacy of antimicrobial preservation.

[4] IBRG (International Biodeterioration Research Group) PDG 16-007.02, Tier 1 Method for Determining the Basic Efficacy of Biocidal Active Substances used to Preserve Aqueous-Based Products.

[5] Guidance on the Biocidal Products Regulation: Volume II Efficacy – Assessment and Evaluation (Parts B + C), April 2018.

[6] SOFW Journal 04/2020, Volume 146, p. 22-25: "Combination of Lactic Acid with 1,2-hexanediol – a New Possibility to Stabilise Rinse-off Formulations"; K. von Nessen, F. Weiher, M. Neubauer, T. Kerl, J. Preusche.

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