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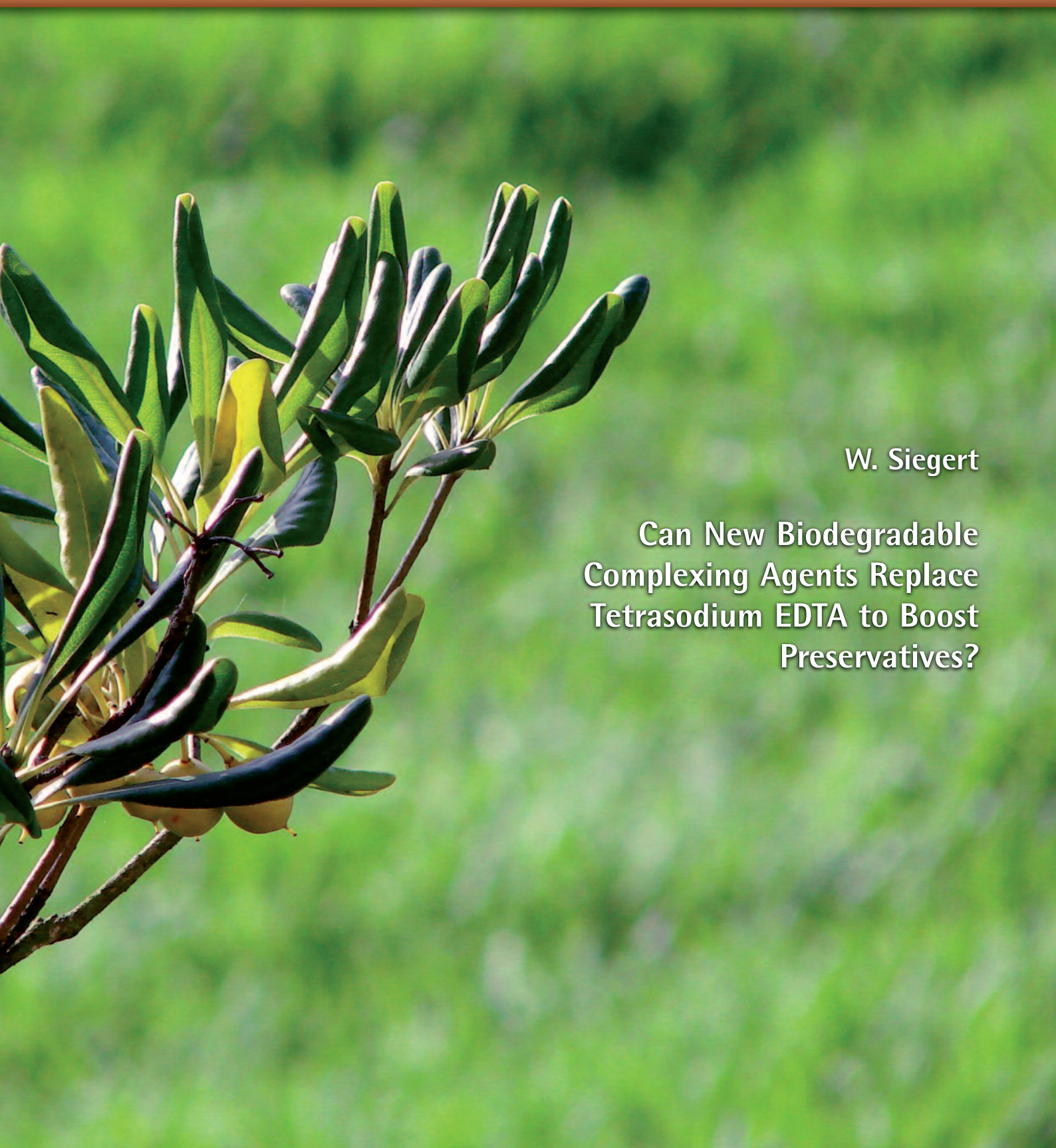

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Can New Biodegradable Complexing Agents Replace Tetrasodium EDTA to Boost Preservatives?

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Keywords: cosmetic preservative, phenoxyethanol, ethylhexylglycerin, complexing agents

Abstract

The continuing discussion of cosmetic preservatives has limited the number of accepted actives that can be practically used. As a result, a number of different methods and materials are being used to boost the activity of the remaining acceptable preservative. A cosmetic preservative based on a combination of the active ingredient phenoxyethanol and the skin care additive and deodorant active ethylhexylglycerin can be used in many applications. The additional boosting effect of tetrasodium EDTA on preservatives is well known, although the environmental fate of this material has been debated. To avoid the environmental discussion about complexing agents, readily biodegradable alternatives were tested under reproducible conditions.

The Test Model

Dilutions of the preservative and combinations of the preservative/booster are prepared using sterile hard water according to the European standard for testing chemical disinfectants and antiseptics **(Fig. 1)** 50 ml portions of the end solutions are each inoculated with 0.5 ml microorganism suspension (initial microorganism count approx. 10^8 cfu/ml) and stirred. **Table 1** shows the test organisms used. These solutions are streaked out onto tryptone soya agar or sabouraud-dextrose 4% agar after 1, 3, 6, and 24 hours. The cultures are incubated for 48 hours at 37 °C, except for *Aspergillus niger*, which is incubated for 72 hours at 25 – 27 °C. The evaluation is made on the basis of semi-quantitative assessment of the microbial growth of the streaks **(Table 2)**.

Preparation of hard water for dilution of products

Solution A: Dissolve 19.84 g anhydrous magenesium chloride ($MgCl_2$) or an equivalent of hydrated magnesium chloride and 46.24 g anhydrous calcium chloride ($CaCl_2$) or an equivalent of hydrated calcium chloride in distilled water and dilute to 1000 ml. Sterilize in the autoclave. Store the solution at 2 °C to 8 °C for no longer than one month.

Solution B: Dissolve 35.02 g sodium hydrogencarbonat ($NaHCO_3$) in distilled water and dilute to 1000 ml. Sterilize by membrane filtration. Store the solution at 2 °C to 8 °C for no longer than one week.

Hard water: For the preparation of 1 l, place 600 ml to 700 ml distilled water in a 1000 ml volumetric flask and add 6.0 ml of solution A, then 8.0 ml of solution B. Mix and dilute to 1000 ml with water. The pH of the hard water shall be 7.0 ± 0.2. If necessary adjust the pH by using a solution of approximately 40 g/l (about 1 mol/l) of sodium hydroxide (NaOH) or approximately 36.5 g/l (about 1 mol/l) of hydrochloric acid (HCl). The hard water shall be freshly prepared under aseptic conditions and used within 12 h.

Fig. 1 Water for dilution according to European standard

Test organisms	ATCC-N°
Escherichia coli	11229
Pseudomonas aeruginosa	15442
Staphylococcus aureus	6538
Candida albicans	10231
Aspergillus niger	6275

Table 1 Test organisms

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Evaluation	Finding	Germ count/ml
–	no growth	100
+	slight growth	approx. 10^3
++	moderate growth	approx. 10^4
+++	heavy growth	approx. 10^6
C	surface covered	$<10^6$

Table 2 Semi-quantitative assessment of the microbial growth of the streaks

Trade Name	Active Content	INCI Name
Baypure CX 100 / 34	34%	Tetrasodium Iminodisuccinate
Dissolvine GI-38	38%	Tetrasodium Dicarboxymethyl Glutamate
Octaquest E30	37%	Trisodium Ethylenediamine Disuccinate
Trilon B powder	87%	Tetrasodium EDTA

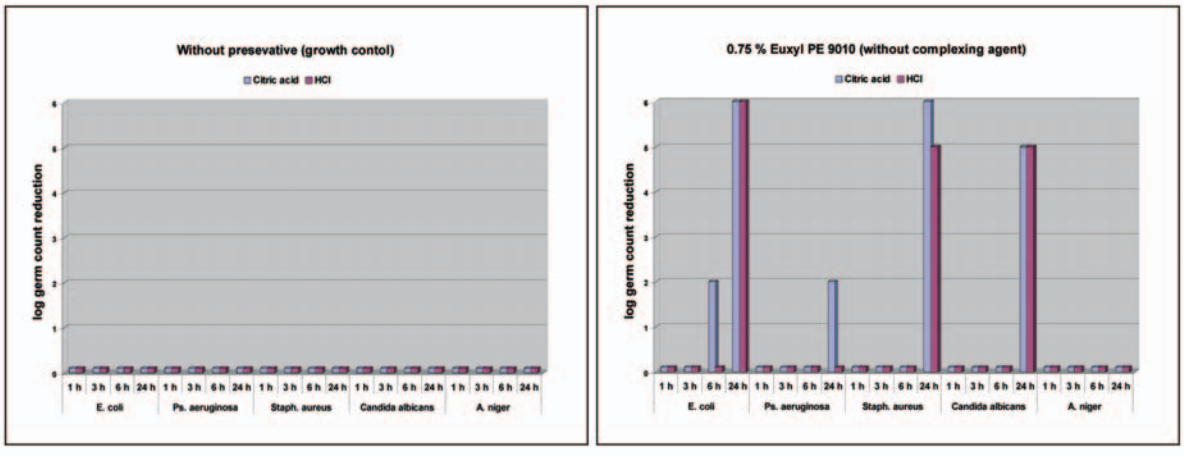
Table 3 Used complexing agents

The Selected Test Concentrations and Complexing Agents

To get a clear differentiation between the preservative with and without complexing agent the use concentration of the preservative was selected at the lowest effective concentration. For the combination phenoxyethanol/ethylhexylglycerin (Euxyl PE 9010) in water this is 0.75%. The complexing agents used are shown in **Table 3**. The complexing agents are dosed at 0.1 and 0.2% calculated as active matter. Due to the alkalinity of the complexing agents, the final solution is adjusted to pH 7.0. Additional, the influence of the acid used for neutralisation is examined. Hydrochloric acid (HCl) is chosen as inorganic acid and citric acid as organic alpha-hydroxy acid. The formed citrates can support the complexing effect. **Table 4** shows the pH values before the adjustment.

	Biocide	Complexing Agent		pH
Hard water for dilution according to CEN	without	without		7.2
	0.75% Euxyl PE 9010	without		8.4
		0.1%	Tetrasodium Iminodisuccinate	9.2
		0.2%		10.1
		0.1%	Tetrasodium Dicarboxymethyl Glutamate	9.4
		0.2%		10.2
		0.1%	Trisodium Ethylenediamine Disuccinate	7.5
		0.2%		7.9
		0.1%	Tetrasodium EDTA	9.4
		0.2%		10.1

Table 4 pH values before the adjustment to pH 7.0



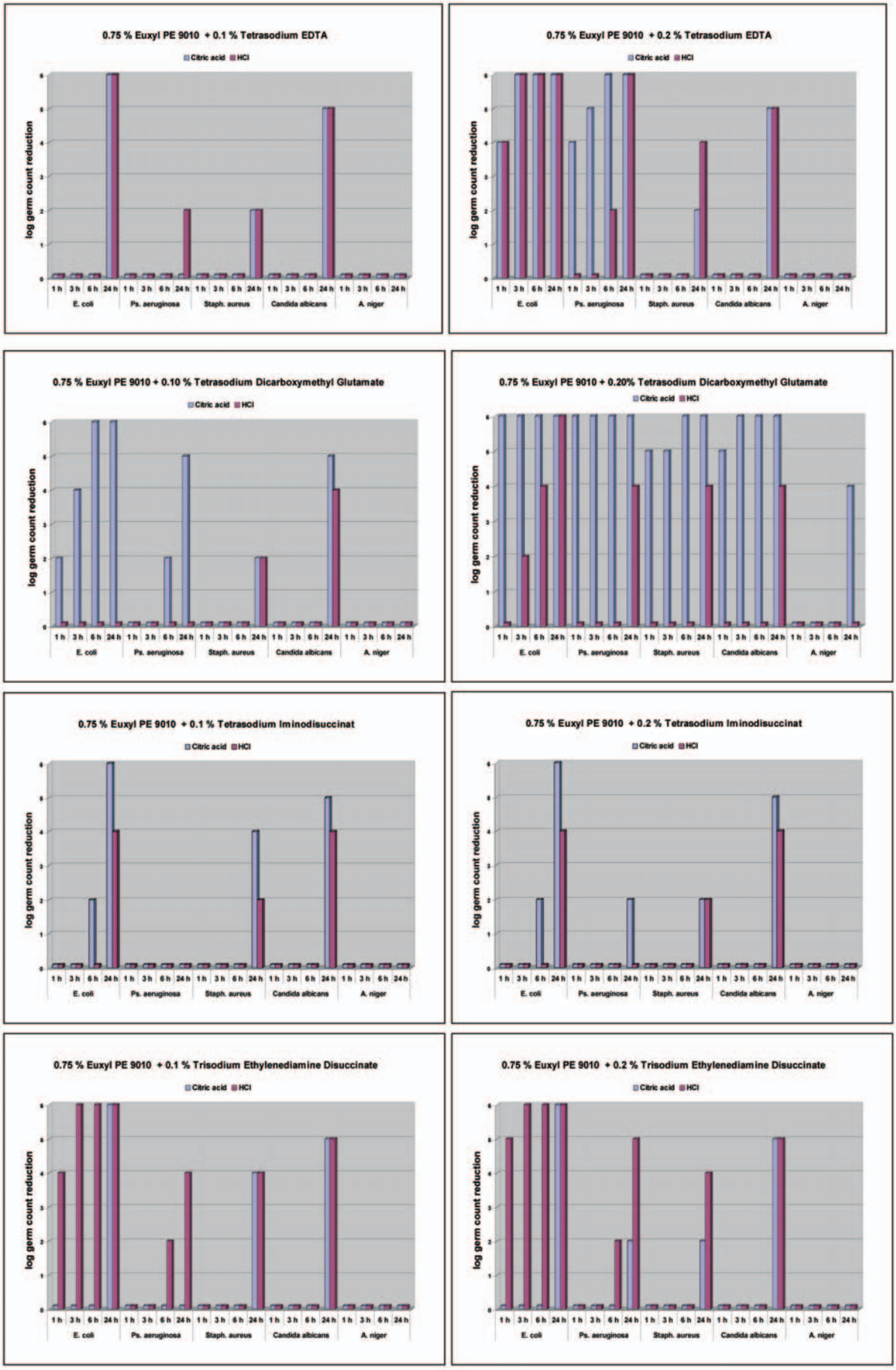


Fig. 2 Boosting effect of the complexing agents on Euxyl PE 9010

Results

Fig. 2 shows the boosting effect of the complexing agents on Euxyl PE 9010.
All used complexing agents can boost the effect of the preservative. Noticeably, tetrasodium dicarboxymethyl glutamate in combination with citric acid gives an even better effect than can be achieved with tetrasodium EDTA. Particularly unexpected is the good effect of tetrasodium dicarboxymethyl glutamate in combination with citric acid against fungi.

Practical Experience

It is well known that phenoxyethanol and parabens are inhibited in the presence of higher amounts of ethoxylated emulsifiers due to their becoming encapsulated in micelles. Unfortunately, this cannot be avoided by adding complexing agents. A test series similar to the one described above, but performed in a shampoo containing 8 % sodium laureth sulphate, failed completely when tested with either parabens or with Euxyl PE 9010.
Several challenge tests performed in our customer service department showed the benefit of using complexing agents together with Euxyl PE 9010 in leave-on formulations. This included some difficult to preserve sun-care formulations.

Literature

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