

Amino modified silicon microemulsions for textile softening made with non-ionic surfactants from BASF

Performance Chemicals
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Amino modified silicones are perfectly suited for textile softening

The ammonium group ...

... exhibits good affinity to the fabric, thus the wash permanence is good.

... offers the opportunity to make silicon oil microemulsion concentrates with perfect shelf stability.

... offers the opportunity to make silicon oil microemulsion concentrates which upon dilution form fine nanoemulsions. Those are able to penetrate the fiber bundle very uniformly.



Making the microemulsion concentrate

The literature cites a large number of methods for making amino modified silicon oil microemulsions. Those include high- and low-energy input, cold, hot and sometimes even multi-step processes, with cofeed of water, emulsifier or acids in between.

All these processes are optimized to overcome gel formation and to achieve stable emulsions with small droplet sizes. However, the simplest processes can be sketched roughly as follows:

Method 1

- dissolve the nonionic surfactant with water.
- add the amino silicon oil and emulsify as finely as possible
- upon vigorous stirring add acid to charge the amino oil. The microemulsion will form automatically.

Method 2

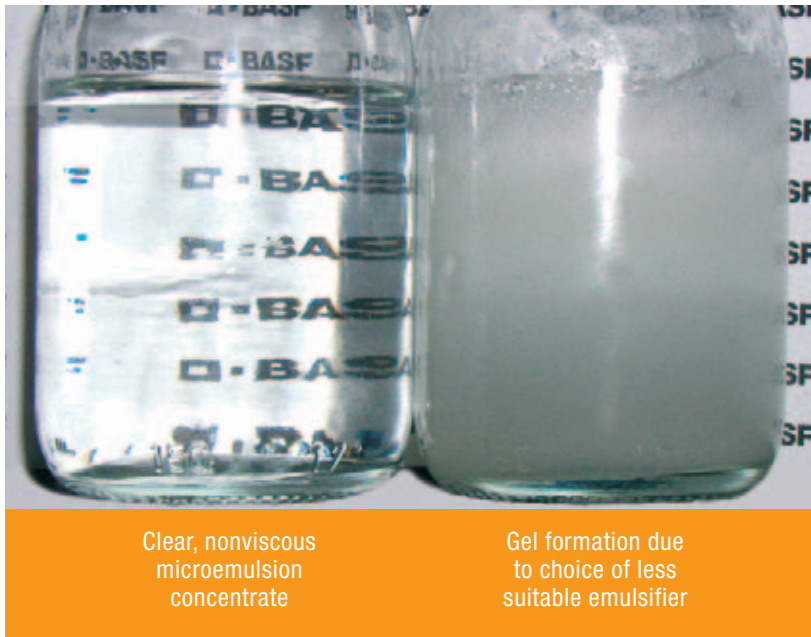
- mix the non-aqueous nonionic surfactant with the silicon oil and stir
- upon stirring add acetic acid to charge the amino oil
- upon vigorous stirring add water and emulsify

In both cases elevated temperatures help to overcome the formation of gels or gel particles.

Typical start values for the final microemulsion are:

25% silicon oil, 12% nonionic emulsifier (HLB = ca. 10–12), water to 100%, adjust pH to 4.5. Subsequently, optimize oil and emulsifier content plus HLB.

Ranking of the nonionics – microemulsion



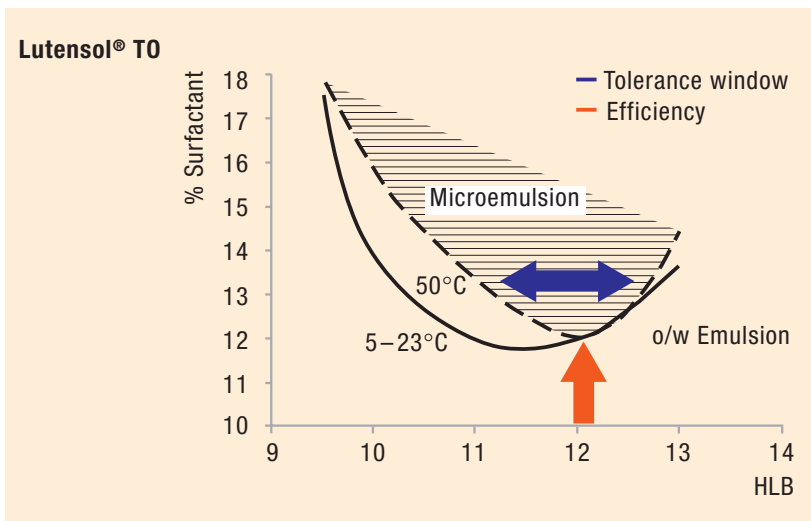
Definitions:

Efficiency is the required minimum amount of nonionic emulsifier to manufacture a microemulsion with phase stability between 5 and 50°C.

Tolerance window is the width of the HLB window of the nonionic emulsifier leading to a microemulsion with phase stability between 5 and 50°C.

Gelling tendency is the degree of formation of gel lumps during microemulsion manufacture, which are difficult to dissolve.

High oil content suitability is the possibility to form micro-emulsions with both high oil content and low viscosity.



Examples

Microemulsions with different types of amino oils are checked in a temperature interval of 5 to 50°C to determine both the efficiency and the tolerance window of the nonionic emulsifiers.

Recipe:

25% Silicon Oil 1.0 Pas/
0.6 mmol/gN
x% Lutensol® TO
75-x% Water
pH = 4.2

Please note that the microemulsion phase diagram is highly dependent on the oil type and needs to be optimized for every formulation.

High oil content suitability

Microemulsions with high oil content have been optimized with respect to both emulsifier content and HLB value and their viscosity have been determined. Systems with low viscosity offer the possibility to process viscous amino oils or manufacture micro-emulsions of high oil concentration.

Experimental conditions:
40% oil, ca. 20% surfactant,
HLB ca. 10. Brookfield Spindle 4–6,
30 rpm, 23°C.

	Silicon oil A: 1.0 Pa-s, 0.3 mmol N/g	Silicon oil B: 1.0 Pa-s, 0.6 mmol N/g
NPEO	solid/gel	solid/gel
Lutensol® TO	ca. 40,000 mPa-s	ca. 9,000 mPa-s
Lutensol® XL	ca. 6,000 mPa-s	ca. 5,000 mPa-s
Lutensol® XP	ca. 2,000 mPa-s	ca. 3,000 mPa-s



Conclusion: Ranking of the nonionic surfactants with respect to microemulsion formation

	Low gelling tendency*	High oil content suitability	Efficiency & tolerance window
Lutensol® T0	++	0	+
Lutensol® XL	++	+	+
Lutensol® XP	++	++	+
Lutensol® ON	+	++	++

Please note that the table exhibits the average trend. From oil to oil slight variations may occur.

For comparison:

Nonyl phenol ethoxilate	0	--	+
Fatty alcohol ethoxilate	0	-	-

* Microemulsions are checked for the degree of intermediate formation of gel particles and their dissolution kinetics.

Ranking of the nonionics – diluted microemulsion under application conditions

Application

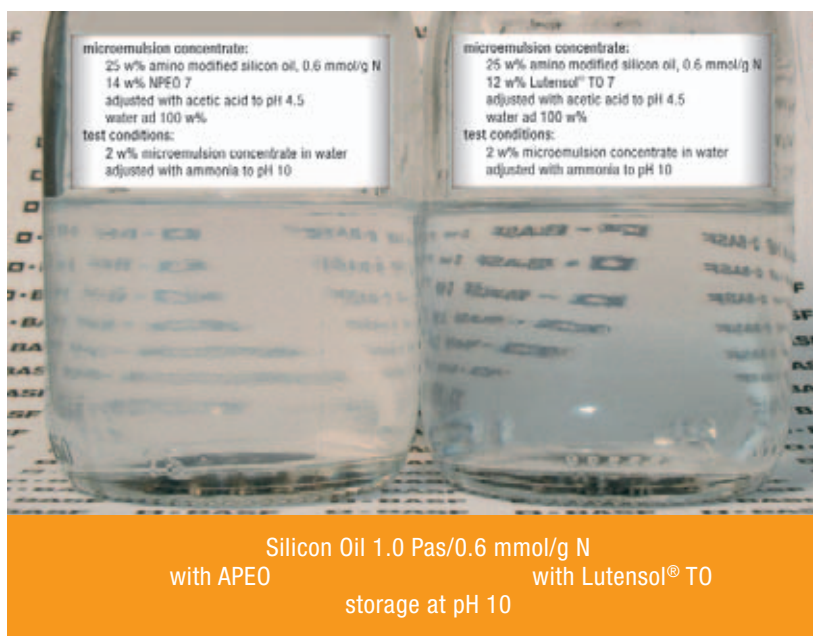
The microemulsion concentrate is diluted to ca. 5 g/l silicon oil to be applied on the fabric. Upon heating, addition of salts, bases or shear stress this diluted emulsion must be stable, otherwise staining of the fabric or silicon residues in the machinery are possible. Emulsifiers with additional high-speed wetting powder help to deaerate the fabric during the impregnation step.

Stability against high temperatures:

The diluted microemulsion is subjected to high temperatures and stored or sheared. Instable emulsions lead to fogging, creaming or coalescence.

Stability against high pH:

The diluted microemulsion is subjected to high pH and stored or sheared. Instable emulsions lead to fogging, creaming or coalescence.



	Stability of the diluted emulsion		
	high temperature	MgCl ₂	high pH
Lutensol® T0	++	++	++
Lutensol® XL	+	+	+
Lutensol® ON	++	0	0
Lutensol® XP	++	-	-

For comparison:

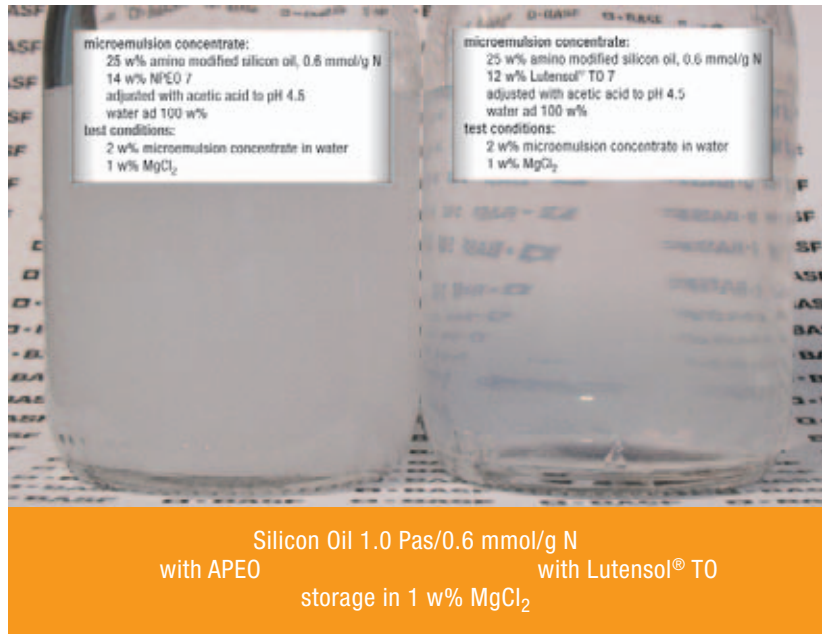
Nonyl phenol ethoxilate	+	+ to ++	+ to ++
Fatty alcohol ethoxilate	++	++	++

Please note that the table exhibits the average trend. From oil to oil slight variations may occur.

++ = excellent + = good 0 = average -- = poor --- = very poor

Stability against MgCl₂:

The diluted microemulsion is subjected to high pH and stored or sheared. Instable emulsions lead to fogging, creaming or coalescence.



Conclusion: Amino modified silicon oil microemulsions

For the manufacture of amino modified silicon oil microemulsions for textile softening we recommend Lutensol® TO and XL types. Both offer easy formation of the microemulsion. Lutensol® TO additionally offers optimum robustness in the application plus good wetting and penetration. Lutensol® XL offers high-speed wetting and thus very uniform penetration, the formulation of microemulsions of low viscosity plus good robustness under application conditions.

	Microemulsion	Robustness during application	Wetting, deaeration
	Lutensol® TO	++	+
	Lutensol® XL	++	++
	Lutensol® ON	++	+
	Lutensol® XP	++	++

For comparison:

	Nonyl phenol ethoxilate	0	+ to ++	0
	Fatty alcohol ethoxilate	-	++	0

Please note that the table exhibits the average trend. From oil to oil slight variations may occur.