

Comparison of SIPERNAT[®] D 10 with in-situ hydrophobized fumed silica

Technical Information 1382



1 Introduction

Defoamer formulations often contain hydrophobic silica particles in order to boost their defoaming efficiency in numerous important industrial applications, for example, pulp production, water treatment, oil extraction, and waterborne paints and coatings, just to name a few. Defoamers are usually mixtures consisting of a carrier oil, emulsifiers, hydrophobic silica particles, and miscellaneous other compounds¹.

Table 1
Typical defoamer composition

Defoamer components	Typical Concentration	Functions
Carrier oil	≤ 95 wt.-%	Makes up the bulk phase of the formulation and should be more or less insoluble in the final application medium to be defoamed. Typical carrier oils have low surface tension, e. g. mineral oils, silicone oils, and polyethersiloxanes.
Emulsifier	≤ 10 wt.-%	Used to partly overcome the insolubility of the carrier oil in the foaming system.
Hydrophobic silica	≤ 10 wt.-%	To boost defoaming efficiency of the formulation via dewetting / bridging mechanism at the air-water interface.
Others		To adjust rheology of the formulation, as antisetling agents, and / or diluents

SIPERNAT® D 10 is a hydrophobic precipitated silica and as such very well known in the defoamer industry¹. Alternatively, hydrophobic silica can be generated “in-situ” during preparation of a defoamer formulation. In this process, hydrophilic silica particles are dispersed in the carrier oil and reacted with a hydrophobization agent (e. g. silicone oil) at elevated temperature in the presence of an alkaline catalyst. During the reaction the hydrophobization agent is grafted to the silica surface rendering it hydrophobic. The “in-situ” hydrophobization is quite commonly employed for fumed silica, too.

Content of this technical information is the experimental comparison of Evonik’s SIPERNAT® D 10 as defoamer silica with selected commercially available fumed silica grades, which were “in-situ” hydrophobized.

2 Experimental part

Four different model formulations were chosen according to defoamer formulations commonly used in technical applications. Two of them were based on a mineral oil carrier; one with and the other one without an emulsifier. The other two formulations were based on a silicone oil carrier, one of them emulsified in water.

Table 2
Model defoamer formulations used in this study

Formulation	Composition	In-situ Hydrophobization
1 Mineral oil formulation with emulsifier	92 wt.-% mineral oil ¹ , 5 wt.-% silica, 2 wt.-% PEG 600 mono/-dioleate 1/4; 1 wt.-% silicone oil ² ,	see text
2 Mineral oil formulation without emulsifier	93 wt.-% mineral oil ¹ , 5 wt.-% silica, 1 wt.-% silicone oil ² , 1 wt.-% catalyst ³	3 hours at 150 °C
3 Silicone oil dispersion	95 wt.-% silicone oil ² , 5 wt.-% silica, 1 wt.-% catalyst ³	3 hours at 200 °C
4 Aqueous silicone oil emulsion	71 wt.-% water, 19 wt.-% silicone oil ² , 1 wt.-% silica, 9 wt.-% TEGOPREN® 5863 ⁴	see text

¹ Shell, Ondina® 913,

² Dow Corning, 200° Fluid, 100 CST,

³ Merck, Ammonium-carbamate,

⁴ Evonik-Goldschmidt GmbH

The fumed silica grades had specific surface areas (BET) of 200 m²/g (one standard grade and one grade with increased thickening power in polyester resins (HV)), 300 m²/g, and 380 m²/g and were all produced by Evonik Industries².

In-situ hydrophobization of hydrophilic fumed silica was performed at elevated temperature. Mineral oil formulations 2 were heated for 3 hours at 150 °C and afterwards 2 wt.-% PEG 600 Mono/-Dioleate were added to form the formulations 1. Silicone oil formulations 3 were heated for 3 hours at 200 °C and subsequently diluted with water in order to create formulations 4.

Foam test medium and equipment

A 0.3 wt.-% solution of a synthetic powder detergent mixture (WfK Research Institute for Cleaning Technologies, D-47807 Krefeld (Germany), Formulation 1998, ISO 105-C08, without defoamer) in tap water was used as foam test medium. Test conditions were a pH value of 10 and a temperature of 60 °C.

A CONTIFOAM circulation device (F. Raab Datentechnik, Austria) was used to determine the efficiency of all defoamer formulations. During circulation of the detergent containing water, foam is developed. The foam height in the device (see Figure 1) as a function of added defoamer and time was automatically measured and recorded resulting in a diagram as shown in Figure 2.



Figure 1
CONTIFOAM circulation test device

Typical test results (example)

Figure 2 shows the development of the foam height over time after addition of four different defoamer quantities between 30 and 100 µl. Right after the defoamer is added, the foam breaks down very fast (so called “knock-down”) from the initial foam height of about 380 mm. Subsequently, the foam level rises again as the defoamer becomes more and more used up. The “hold-down” time is used within this brochure as the period of time from the minimum foam level until the foam level rises again to a height of 250 mm. The “hold-down” time was determined for all defoamer formulations in order to compare different silica grades in the respective defoamer formulations.

Figure 2

Influence of the defoamer quantity on the “hold-down” time in a typical CONTIFOAM circulation test (added amounts as indicated in Table 3)

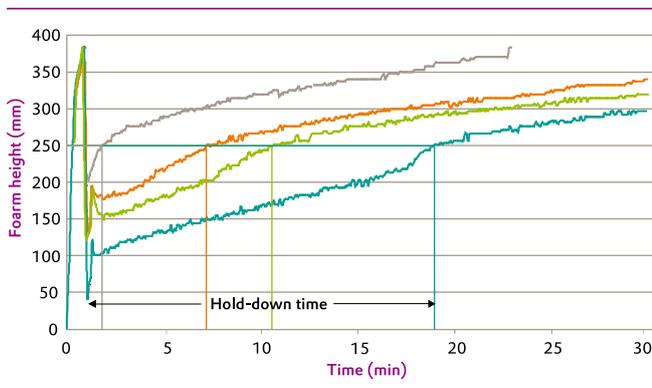


Table 3

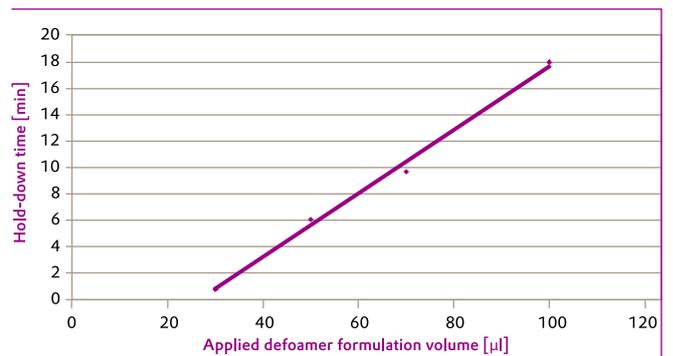
Influence of the defoamer quantity on the “hold-down” time in the CONTIFOAM test with defoamer formulation 1 containing 5 weight-% SIPERNAT® D 10

Defoamer quantity applied (µl)	Corresponding SIPERNAT® D 10 content (mg)	Hold-down time (min)
30	1.5	0.8
50	2.5	6.1
70	3.5	9.7
100	5.0	18.0

According to Figure 3 which is based on the results summarized in Table 3, the “hold-down” time shows an almost linear dependency on the defoamer quantity applied and the corresponding SIPERNAT® D 10 quantity. Similar correlations were found for all other defoamer formulations.

Figure 3

Correlation between the defoamer quantity used and the “hold-down” time in the CONTIFOAM test with defoamer formulation 1 containing 5 weight-% SIPERNAT® D 10



3 Comparison of the defoaming efficiency of SIPERNAT® D10 and in-situ hydrophobized fumed silica

In order to compare the defoaming efficiency of SIPERNAT® D 10 with in-situ hydrophobized fumed silica, the “hold-down” times of all silica products in defoamer formulations 1 to 4 were determined in the CONTIFOAM circulation test described in the previous chapter. Data presented below are the mean value of at least two test runs and normalized to the same silica concentration with the “hold-down” time achieved with SIPERNAT® D 10 set to 100%. The percentages given for the different silica types therefore reflect their relative “hold-down” times in comparison to SIPERNAT® D 10 at the same addition level.

Our test results with four different defoamer formulations proofed very clearly that SIPERNAT® D 10 is the superior booster silica in comparison to “in-situ” hydrophobized fumed silica. While in the mineral oil based formulations, between five and ten times longer “hold-down” times can be achieved with SIPERNAT® D 10, it is still twice as efficient in the silicon oil based formulations.

For further information please ask your local Evonik representative or application technologist.

Figure 4

Relative “hold-down” times in % of SIPERNAT® D 10 in comparison to in-situ hydrophobized fumed silica (FS) in **mineral oil defoamer formulations**:

(a) **with emulsifier** (formulation 1),

(b) **without emulsifier** (formulation 2).

In these formulations, SIPERNAT® D 10 increases the “hold-down” time by a factor of up to ten in comparison to the same amount of in-situ hydrophobized fumed silica.

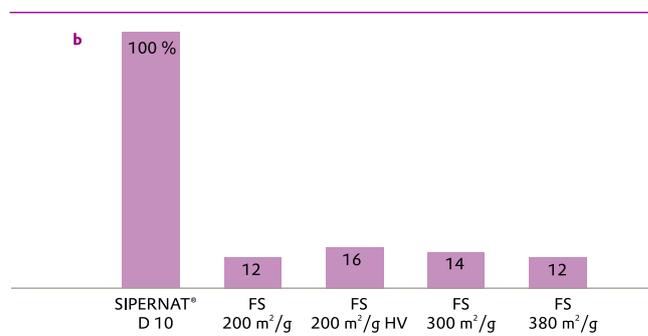
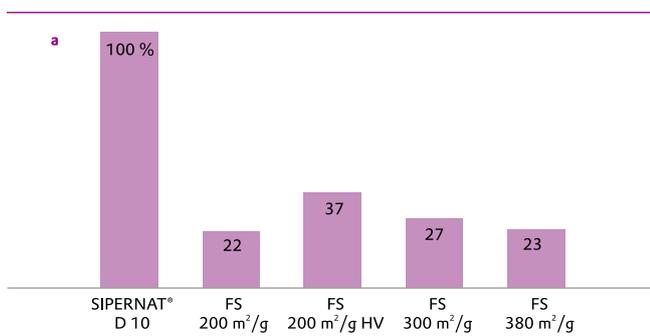


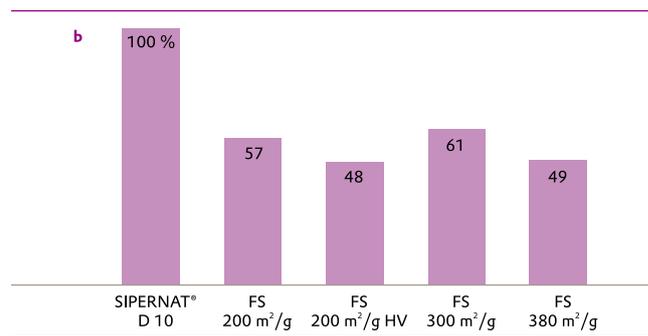
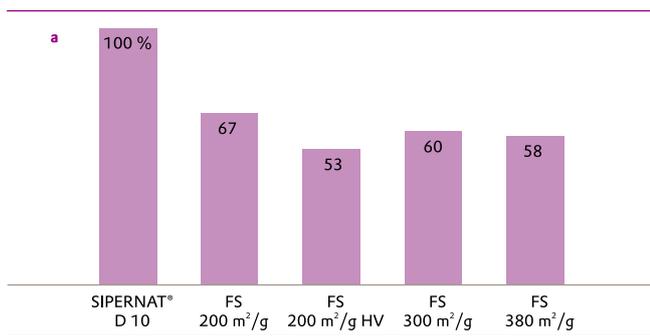
Figure 5

Relative “hold-down” time in % of SIPERNAT® D 10 in comparison to in-situ hydrophobized fumed silica (FS) in **silicon oil defoamer formulations**:

(a) **Silicone oil defoamer** (formulation 3),

(b) **Aqueous silicone oil defoamer** (formulation 4).

In these formulations SIPERNAT® D 10 increases the “hold-down” time by a factor of up to two in comparison to the same amount of in-situ hydrophobized fumed silica, showing that it is the more efficient defoamer silica even in silicon oil.



4 Literature

1 Technical Information TI 1313, SIPERNAT® and AEROSIL® for Defoamer

2 “AEROSIL® - Invented to Improve. Product overview”

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