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Filtration Effects on Foam Inhibitors and Optically Detected Oil Cleanliness

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ABSTRACT

Foam inhibitors (FIs) are effective at reducing foam in lubricants but can be detected by optical particle counters as contaminants. Filtration can decrease the particle counts but will also adversely affect foam performance because the additive is filtered out of the fluid. In this research, we explore the opposing effects of filtration on optically detected particle counts due to foam inhibitors and foam performance. A custom-built test rig is used to filter fluid consisting of polydimethylsiloxane foam inhibitors in base oil and to measure particle counts per ISO 4406. Foam tests are then performed per ASTM D892 on samples taken during filtration. In order to meet both particle count and foam performance specifications, we find that the following three variables must be carefully optimized: FI treat rate, filter pore size, and number of filtration passes.

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Foam control additives; lubricant physical analysis; cleanliness

Introduction

Foam can be detrimental to lubricant performance because it is air incorporated in the lubricant and can prevent the lubricant from coming into contact with the solid surfaces where it is needed (Canter *(1)*). Foam performance is typically characterized in accordance with ASTM D892. ASTM D892 specifies that air is passed into an oil sample through a diffusing stone at a controlled temperature for 5 min (ASTM D892-13 *(2)*). Results of the process are quantified in terms of foam tendency, the volume of foam after 5 min, and foam stability, and the volume of foam remaining 10 min after the air flow stops. Foam inhibitors (FIs) are additives included in lubricant formulations to reduce foam tendency and stability (Friesen *(3)*; Denkov *(4)*; Centers *(5)*, *(6)*; Tamai, et al. *(7)*). Although multiple mechanisms have been proposed, it is commonly believed that FIs function by displacing the stabilizing surfactant from the surface of foam bubbles, thereby thinning the bubble walls to the point of rupture (Friesen *(3)*; Denkov *(4)*; Centers *(5)*). As such, effective FIs should have lower surface tension, be insoluble, and be dispersible with respect to the base oil. Silicones and acrylic copolymers are the most common foam inhibitors in petroleum-based fluids. Here we focus on polydimethylsiloxane (PDMS) compounds, which are known to perform well as foam inhibitors for a variety of applications.

Although FIs effectively control foam, they introduce oil cleanliness challenges. Fluid cleanliness, in general, is characterized by the number of solid particles per milliliter of fluid and particles are often counted using light-based automatic particle counters. In these particle counters, light is passed through a narrow stream of fluid onto a photoreceptor. Particles in the fluid disrupt the flow of light onto the receptor and result in voltage changes proportional to the size of the particles. FI droplets are insoluble and differ in index of refraction from the base oil and as such can also be reported as contaminants (Michael, et al. *(8)*; Michael and Wanke *(9)*; Sander, et al. *(10)*). Under ISO 4406, a standard commonly used to quantify fluid cleanliness (ISO 4406 *(11)*), counts of particles greater than or equal to 4, 6, and 14 μm are classified on a scale (ISO Code). The result is that FI droplets greater than or equal to 4 μm impede a fluid's ability to meet ISO 4406 cleanliness targets.

It is known in the industry that FI droplets are not real contaminants because they are intentionally added to the lubricant and will not cause abrasive wear damage. However, as other researchers have shown, most optically based particle counters cannot tell the difference between solid and liquid particles, so equipment users face a frustrating situation when trying to achieve both cleanliness and antifoam targets. One possible solution is to measure particulate contamination by a different technique, such as ASTM D7647 *(12)*, which is designed to eliminate the counting of water droplets and other soluble "soft" particles. However, this technique is not yet amenable to online, rapid analysis, so it is still worthwhile to pursue solutions that employ standard optically based particle counters.

The effect of FIs on optically detected particle counts was characterized in a study by Michael, et al. *(8)*. They tested several additive packages in either Group I or Group III base oil. It was found that the largest increase in particle counts came from PDMS FIs in a diesel engine additive package and that there was no observable difference between base oil groups. A later study confirmed those findings and further noted that non-silicone-based FIs can also cause a large increase in particle counts (Sander, et al. *(10)*).

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Filtering fluids can reduce particle counts. However, filtration has been shown to reduce the effectiveness of FIs. This effect was first shown in a study of tractor hydraulic fluids. It was found that tractor hydraulic fluid circulated through a 25-μm paper filter for up to 93 h successfully met foam specifications but that circulation through a 7-μm synthetic filter resulted in a sharp increase in foaming and failure to meet specifications after only 24 h of filtration (Friesen *(3)*). Subsequent tests with various filters showed that the loss of foam performance was accentuated by decreasing filter pore size and use of synthetic filters, and that the same trend was observed for three different fluids. These tests therefore suggested that the filters were removing the foam inhibitor from the fluid (Friesen *(3)*). The adverse effect of filtration on foam performance was confirmed in the study by Michael, et al. *(8)*, who further showed that the deterioration of foam performance with filtering was accompanied by a corresponding decrease in optically detected particle counts.

It is well established that FIs are effective at reducing foaming but can be detected by optical particle counters as contaminants. Filtration can reduce these particle counts but will also decrease the oil's foam performance as the additive is filtered from the fluid. Research to date indicates that the conflict between cleanliness and foaming can be affected by additive chemistry, additive treat rate, filter material, filter pore size, and the number of times the fluid passes through the filter. However, the interplay between these factors in determining the combined cleanliness and foam performance of a fluid is not fully characterized. In this research, we explore the effects of additive treat rate, filter pore size, and number of passes through the filter using a custom-built test rig for filtering the fluid and measuring particle counts per ISO 4406 to determine cleanliness and a foam tester that enables ASTM D892 characterization of foam performance.

Methods

Filtration and particle counts

Characterization of the effects of filtration on ISO 4406 cleanliness requires a way to circulate, filter, and perform particle counts on fluids. A test rig, shown in Fig. 1, was designed and built for this purpose. The rig consists of an 8.04-L fluid reservoir, a three-quarter horsepower, three-phase 208-V motor that powers a 3.44 cc/rev gear pump, a 20.3-cm aluminum filter housing, and an in-line particle counter. The rig is plumbed with 1.27-cm stainless steel tubing and three-way valves to allow the fluid to be circulated through the particle counter only, through the filter and the particle counter, or bypassing both filter and particle counter. There is also a port from which samples may be drawn during circulation.

Counts of particles greater than or equal to 4, 6, and 14 μm in size are characterized by codes described in ISO 4406. Each increase in code represents up to a doubling of the number of particles, with zero representing a particle count of between 0 and 0.01 particles per milliliter and $>$28 representing a particle count of more than 2,500,000 particles per milliliter. The numbers of particles of each size are reported as a sequence of three codes separated by slashes; that is, 4 μm ISO Code/6 μm ISO Code/14 μm ISO Code. When less than 20 particles are counted, the statistical significance of the count is diminished and the results are reported with the "less than" ($<$) symbol (ISO 4406 *(11)*). For the flow rate in our test rig, we determined that there should be a sufficient number of particles for the counts to be statistically significant for ISO 9 or above. However, usage has shown that there is significant noise in the data once the ISO Code falls below 10, so we report all codes of 9 or smaller as $<$10. It is also important to note that the accuracy of the particle counter is given as $\pm$0.5 ISO code.

Foam testing

Foam tests were performed following ASTM D892 *(2)*. The apparatus consist of a 1,000-mL graduated cylinder, two baths capable of maintaining 24 $\pm$ 0.5°C and 93.5 $\pm$ 0.5°C, a supply of clean dry air to a metal diffuser at 94 $\pm$ 5 mL/min, and a method for measuring the volume of air supplied over the 5-min test period. The test procedure is summarized as follows: Air is passed through a diffusing stone into a sample of oil for 5 min at controlled temperature. After 5 min have elapsed, the volume of foam is recorded in milliliters as the oil's foaming tendency. The oil is then allowed to rest for 10 min. As is common for oils with FIs, all samples tested in this study were found to have a foam stability of zero. The standard prescribes that this process be carried out three times or in three sequences. The first trial, Sequence I, is carried out at 24 $\pm$ 0.5°C. A

(a) (b)

Figure 1. (a) Schematic and (b) photo of the custom-built filtration test rig that enables circulation, filtration, and particle counting of fluid samples.

second sample, Sequence II, is tested at 93.5 ± 0.5°C. For the third test, Sequence III, the second sample is retested at 24 ± 0.5°C. In this work, we found that the qualitative effects of additive treat rate, filter pore size, and number of times through the filter were the same for all three sequences. Based on those findings, we performed Sequence I only for all subsequent cases. Each sample was tested at least twice, by two different technicians, with the average value being reported.

Materials and procedures

This section describes the standard procedure performed for each test. First, the filter test rig reservoir is charged with 7.6 L of base oil. The base oil has the following typical properties: Viscosity at 40°C = 41.2 cSt, viscosity at 100°C = 6.4 cSt, viscosity index = 104, density = 0.865 g/mL, sulfur < 6 ppm. The base oil is allowed to circulate until all air is removed from the system. The flow rate is then set to 3.8 L/min and the particle counter is activated. The particle counter records data at approximately 10-s intervals throughout the remainder of the test. If the base oil has a 4 μm ISO Code greater than 17, it is filtered until the ISO code is 17 or less. With the fluid flowing, the particle counter active, and a base cleanliness reached, FI is injected into the reservoir and allowed to mix for 1 h. After 1 h of mixing, the first fluid sample is taken. First, a small amount (about 100 mL) of fluid is collected from the system in a sample bottle. The bottle is capped and shaken; the small amount of fluid is discarded and 500 mL of fluid is collected in the prepared bottle. Then, filtration is initiated at t_o and additional samples are taken after 2.5, 5, 7.5, and 10 filter passes (corresponding to time intervals of 5, 10, 15 and 20 min, respectively). Each filter test results in ISO code data for the entire process and produces five samples ready for foam testing.

This procedure was performed for several fluid samples consisting of a Group II base oil blended with a PDMS FI. The FI additive was prepared in dilute form for easier dispersion. The FI consisted of 3.0 wt% PDMS blended with 97.0 wt% aliphatic hydrocarbon solvent. The PDMS polymer has a typical viscosity of 1,000 cSt at 25°C and contains about 38 wt% elemental silicon. The diluted FI had the following properties: Flash point measured via ASTM D56 *(13)* = 65°C minimum; water content = 0.100 wt% maximum; relative density 20/20°C = 0.792 minimum to 0.820 maximum. The FI additive (diluted version) was used at three different treat rates: 0.14, 0.10, and 0.07 wt%, where 0.14 wt% is the standard commercial treat rate. Each fluid sample was tested using synthetic β1000 filters (glass fiber media with an epoxy-based resin system) rated at 4, 5, or 12 μm (subsequently referred to as the pore size) based on ISO 16889 *(14)*.

A representative particle count result from one test is shown in Fig. 2. Prior to addition of the FI, the particle counts are 16/13/<10, corresponding to 320–640 particles per milliliter larger than 4 μm and 40–80 particles per milliliter larger than 6 μm. The particle counts increase to >28/22/15 upon addition of the FI, which is significant because an increase of one ISO code corresponds to doubling the number of particles detected. Then, once filtration begins, the particle counts decrease rapidly. This general trend was observed in all cases tested.

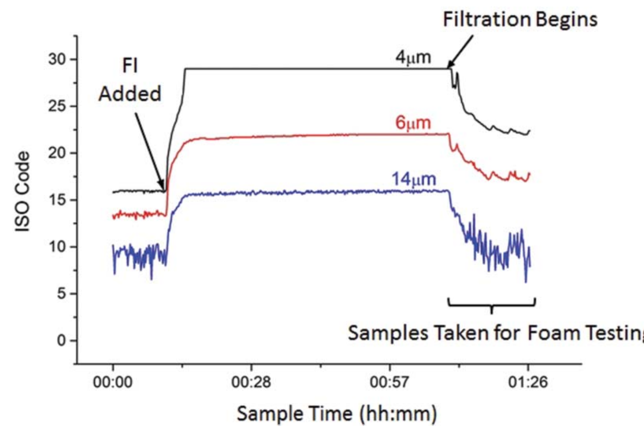

Figure 2. Representative particle count ISO Codes from the filtration test rig versus time. The increase in particle counts occurs when the FI is added to the system, and the particle count decreases after filtration.

Results and discussion

Test results for the largest and smallest treat rates (0.14 and 0.07 wt%) and the two larger filter pore sizes (12 and 5 μm) are summarized in Table 1. First, we analyze the measurements taken before filtration, which are identified as zero filter passes in Table 1. Comparing the two treat rates, we observe that more FI additive corresponds to higher particle counts: particle counts before filtration with the 0.14 wt% treat rate are >28/22/15, and those with the 0.07 wt% treat rate are 26–27/20/14. Recalling that a decrease of one ISO Code corresponds to half the number of particles, this difference is significant. We also observe that the additive is very effective at minimizing foam at either treat rate before filtration. The prefiltration foam tendency ranges from 0 to 20 mL. This difference can be considered negligible in the context of the ASTM standard that reports the expected repeatability for a Sequence I foam tendency of 20 mL to be ±22 mL (ASTM D892-13 *(2)*).

We next analyze the effect of filtration. Consistent with findings in previous studies, filtration significantly decreases the counts of all particle sizes. Further, we observe that this decrease occurs primarily during the first five passes through the filter, after which further filtration has a very small or no effect on the ISO Codes. For the cases shown in Table 1, filtration has no statistically significant effect on Sequence I foam tendency, although there is a suggestion of decreased foam performance with increasing filtration for the 0.07 wt% treat rate.

Table 1. Particle counts and foam tendency results from 5- and 12-μm filter tests.[a]

TR	0.14 wt.% 12 μm		0.07 wt.% 12 μm		0.07 wt.% 5 μm	
Filter Pass	ISO Code	Foam	ISO Code	Foam	ISO Code	Foam
0	>28/22/15	20	26/20/14	0	27/20/14	0
2.5	24/19/11	14	21/16/<10	5	21/15/<10	12
5	22/17/<10	20	21/15/<10	10	20/14/<10	20
7.5	22/17/<10	17	20/15/<10	10	20/14/<10	25
10	22/17/<10	13	20/16/<10	15	20/14/<10	30

[a]Data are shown for two treat rates (TR) and two filter pore sizes as a function of number of passes through the filter. The ISO Codes correspond to 4-, 6-, and 14-μm particles. The Sequence I foam tendency is given in milliliters as specified by ASTM D892 *(2)*.

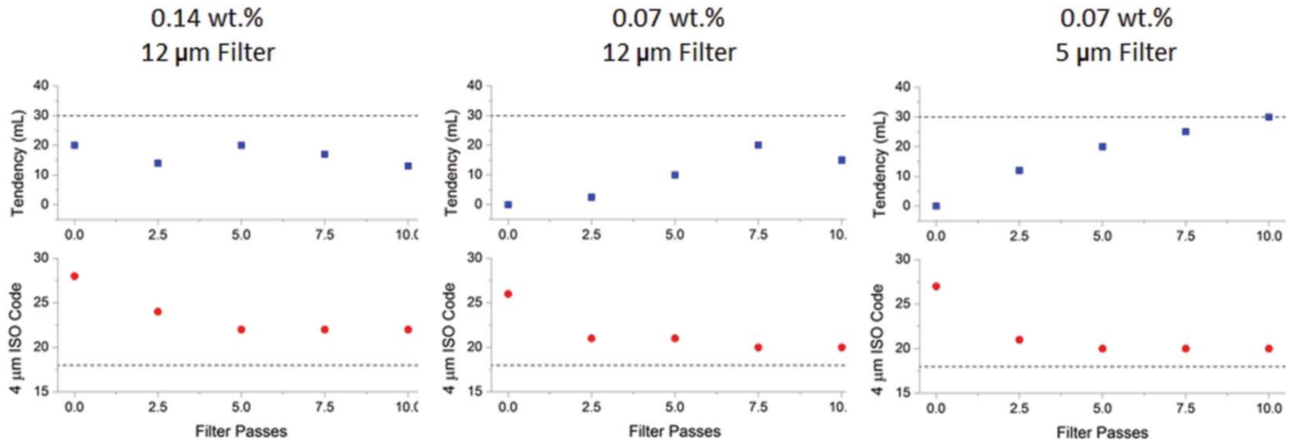

Figure 3. Foam tendency (top, squares) and 4 μm ISO Codes (bottom, circles) for the 0.14 and 0.07 wt% treat rate additives filtered through 12- and 5-μm filters. The dashed line indicates the maximum limits selected for this study and the color of the symbols indicates whether the spec was met (blue) or not (red). The results show that none of the cases tested here meet both particle count and foam targets.

Nevertheless, all samples exhibit good foam performance, both before and after filtration.

To further explore the particle count measurements, we analyze the distribution of particle sizes present in the fluid during testing. For the 0.07 wt% treat rate additive before filtration, the ISO Codes are 27, 20, 14, and 12, corresponding to 4-, 6-, 14-, and 21-μm particles (the 21 μm code is not required by the standard but is reported by the particle counter). Using the known base oil and additive volumes from the test and the minimum number of particles per milliliter associated with these ISO Codes from the standard, we assume that the additive droplets are spherical and calculate that 97% of the additive in the fluid is composed of droplets between 4 and 6 μm in diameter (or smaller, which we do not measure). Analysis of the other cases yields similar results. This suggests that we focus subsequent analyses on the 4 μm ISO Code.

To facilitate the analysis of particle counts and foam tendency, we introduce "cleanliness" and foam performance targets. The former is selected to be a 4 μm ISO Code of 18 or below and the latter as a Sequence I foam tendency of 30 mL or below. These values are representative of the in-service lubricant specifications of major manufacturers of construction machinery. For example, a cleanliness level equal or better than ISO 18/16/13 is recommended for in-service wind turbine gearboxes (ANSI/AGMA/AWEA 6006-A03 *(15)*). Also a major commercial and military specification for axle lubricants requires a maximum foam tendency of 20, 50, and 20 mL for Sequences I, II, and III, respectively (SAE J2360 *(16)*).

Plots of foam tendency and 4 μm ISO Code vs. number of filter passes for the 0.14 and 0.07 wt% treat rate additive filtered through 12- and 5-μm filters (corresponding to data in Table 1) are shown in Fig. 3. Our target specification limits are identified by dashed lines. The data points are shown as symbols, where the color of the symbol reflects whether the sample met cleanliness and foam specs (blue) or not (red). These plots show clearly that the particle counts decrease with filtration, but neither sample meets the cleanliness target after 10 filter passes and, in all cases, the foam performance is below the maximum. These results, and the analysis of particle count distributions that showed that the majority of FI droplets are likely small, suggest the use of a smaller filter pore size.

Results from tests with 4-μm filters are reported in Table 2. We observe a much more significant ISO Code decrease with this smaller filter pore size (compared to the 5- and 12-μm filter results). In addition, in contrast to the larger filter pore size results, here we observe that, as the filtration proceeds, the foam tendency increases nearly monotonically. These results clearly illustrate the opposing effects of filtration on FI-induced particle counts and foam performance.

The 4-μm filter test results are compared to the target particle count and foam tendency values in Fig. 4. At the highest treat rate tested (0.14 wt%), the 4 μm ISO Code decreases with filtration, approaching but not reaching the cleanliness target after filtration. The foam tendency for this treat rate is below the maximum for all samples except the one taken after 10 filter passes. This suggests the use of a lower treat rate. However, reducing the standard treat by half (0.07 wt%) yields samples that do not meet the particle count target before filtering and then do not meet the foam tendency target after filtering.

A moderate treat rate (0.10 wt%) ultimately enabled both targets to be met. As shown in the center panel of Fig. 4, both the particle count and foam tendency measurements were at or below the maximum target values by 7.5 filter passes. These results show that, for this PDMS additive, there is a combination of treat rate and filter pore size that enables the fluid to

Table 2. Particle counts and foam tendency results for 4-μm filter test.[a]

TR Filter Pass	0.14 wt.% 4 μm		0.10 wt.% 4 μm		0.07 wt.% 4 μm	
	ISO Code	Foam	ISO Code	Foam	ISO Code	Foam
0	>28/22/15	20	28/20/14	0	27/20/14	5
2.5	20/11/<10	20	19/12/<10	5	18/<10/<10	38
5	19/11/<10	27	19/10/<10	15	17/<10/<10	93
7.5	19/11/<10	20	18/<10/<10	15	17/<10/<10	125
10	19/11/<10	35	18/<10/<10	20	17/<10/<10	100

[a]Data are shown for three treat rates (TR) as a function of number of passes through the 4-μm filter. The ISO Codes correspond to 4-, 6-, and 14-μm particles. The Sequence I foam tendency is given in milliliters as specified by ASTM D892 *(2)*.

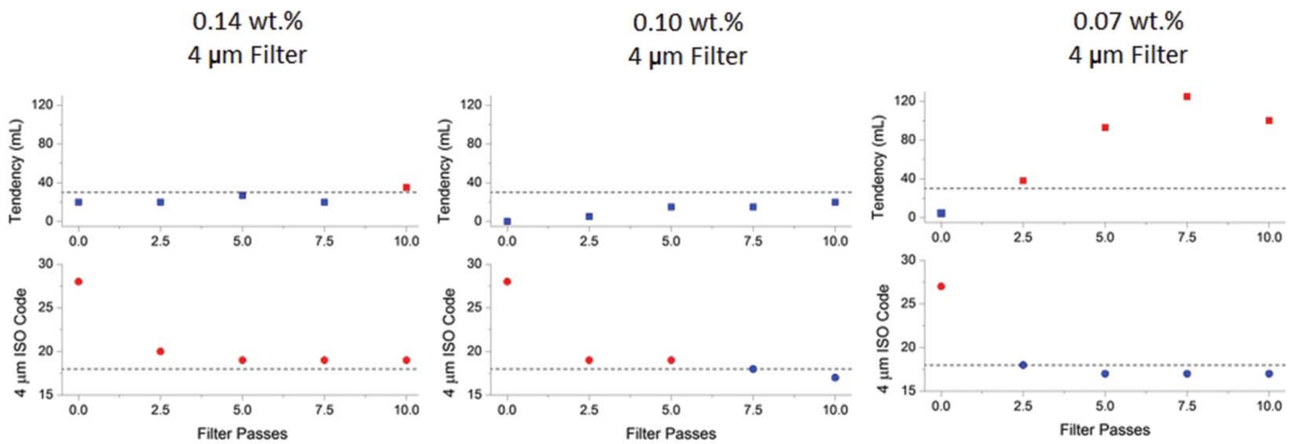

Figure 4. Foam tendency (top, squares) and 4 μm ISO Codes (bottom, circles) for different treat rate additives filtered through the 4-μm filter. Colors and dashed lines have the same meaning as in Fig. 3. With the 0.10 wt% treat rate (center) we observe that both particle count and foam targets are met after ∼5–10 passes through the filter.

exhibit good foam performance while still being considered clean when measured by optical particle counters. However, we also note that the foam tendency is increasing throughout the 10 filter pass test, suggesting that the fluid will no longer meet foam targets after additional filtration. Although we do not have data for more than 10 filter passes here, the trend within the first 10 passes suggests that the fluid should be allowed to pass through the filter between 7 and 10 times to meet both foam and particle count targets.

Conclusions

This study analyzed the effects of PDMS additive treat rate, filter pore size, and amount of filtration on oil foam performance and optically detected particle counts. We showed that, before filtration, the additive controls foam well but, for all cases tested here, the additive is likely to be detected by optical particle counters as contaminants. We were able to reduce the particle counts through filtration and found that the most significant decrease occurred during the first few passes through the filter. This suggests that, if filtration is used to address the issue of FI-induced particle counts, it is not necessary to filter for an extensive period. The decrease in particle counts with filtration was more significant with smaller filter pore sizes. However, filtration can also result in deterioration of the FIs ability to control foam, and this was particularly evident in tests with the fewest particles remaining after filtration. Analysis of the distribution of droplet sizes detected by the particle counter suggested that the smallest FI droplets detected (4–6 μm) make up the majority of the population. Based on this, to quantitatively evaluate the relationship between particle counts and foam performance, we introduced target values for the 4 μm ISO Code and the Sequence I tendency. The analysis highlighted the observation that filtration has opposite effects on particle counts and foam performance. For the specification values identified here, we were able to identify one combination of treat rate, filter pore size, and number of filter passes where the sample met both particle count and foam targets.

The work reported here represents a first step in understanding the effects of additive treat rate, filter pore size, and number of filter passes on both particle counts and foam performance. Future plans include more experiments to optimize the interaction between the aforementioned variables. In addition, future studies will examine different FI chemistries, either alone or in mixtures, to determine whether there are other conditions, under which we can meet both cleanliness and foam targets.

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