A New Approach to the Determination of Extracted Pro-foamants from Elastomeric Sealants

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ABSTRACT: Elastomeric seals are commonly used in the automotive mechanism. Over the last 20 years such sealants have been made relatively simple to apply using automated equipment. However, such elastomers may contain extractable pro-foamant components which can under certain circumstances adversely and seriously affect the performance of the mechanism. Pre-determination of the effect of sealants on oil and *vice versa* point to the need for a simple yet relevant test procedure to both study and ameliorate effects of both sealants and lubricants in generating pro-foamants. This paper presents further information gathered regarding the rate of extraction of profoamants using the simple Foamgen© Test apparatus and method discussed in an earlier paper.

INTRODUCTION

Importance of Control of Foaming Tendency

Any mechanical device required to contain and circulate a lubricant must have its various components sealed where necessary to prevent the lubricant from leaving the device and thus causing harm to the machine. In the case of the automotive mechanism and its components, loss or failure of the lubricant to perform its function can result in harm not only to the mechanism but to the operator and others.

From any point of view, then, tendency of a lubricating oil or hydraulic fluid to foam in operation is considered highly undesirable. As a consequence, much effort has gone into the development and application of anti-foaming additives in oil formulations to control oil foaming.

One of the more effective anti-foaming additives is made from relatively high molecular weights (MW) of polysiloxane that have limited solubility in mineral oils and some other organic liquids. However, lower MW polysiloxane homologs of this material, which have higher solubility, function as pro-foamants. Consequently, control of polysiloxane MW in the anti-foamant is important in controlling foaming tendency.

Measurement of the Foaming Tendency of an Oil

In 1946, ASTM Test Method D 892 was developed to meet the need to measure and predict the foaming tendency of oils and fluids. This method covered foaming tests at essentially room temperature and 94°C and has been frequently specified and applied to measure the foaming characteristics of lubricants over the years.

Much more recently, recognizing the higher operating temperatures of automotive mechanisms, another sequence for the method, D 6082, has been established for measurement of foaming tendency at 150°C.

The focus of these methods has been to evaluate only the foaming tendency of the oil or fluid without regard for other influences such as the seals of the mechanical devices.

Elastomeric Sealants and Foaming Tendency

Previous use of cork and other gasketing materials over many years gave few reasons for concern about the effect of different gasketing material on foaming tendency. However, with the introduction of elastomeric gasketing materials, that perception quickly changed when it became evident that such materials could be a source of pro-foamants extractable by the contacting oils and fluids.

Consequently, a number of different 'in-house', gasketing-contact foam tests were developed, none of which had common test procedures or equipment and most of which measured foaming tendency from different manufacturing and performance viewpoints.

Need for a Common Test of Oil/Elastomer Foaming Tendency

It seemed important that a simple, widely applicable, and relevant test of the foam producing relationship of the oil and the elastomer be developed. A recent paper [1] presented a test apparatus and procedure now called the Foamgen© Test.

The information presented in the latter paper showed that some factors preconceived to be important were not or only slightly so. Other factors were shown important and still others were shown to be temperature dependent in their influence.

However, it was stated in the previous paper that the laboratory work reported was not to study the details of foaming tendency but to separate influential factors from those that were not. Rather, the more detailed studies were left to following papers of which this is the first.

Equipment

Development of the Foamgen© Test

The Foamgen© Test was conceived and developed in response to a request for a relevant but simple test of foaming tendency involving the lubricant and the contacted elastomer. While authors were mutually involved in planning tests and analyzing the resulting data, design and operation of the Foamgen© Test was the responsibility of Savant Laboratories. The fundamental idea was that it would be best to generate the oil/elastomer interaction in a cell that could be used for test of foaming response after the exposure of the test oil to stress was completed.

The Tannas Foam Air-Bath (TFAB) used in ASTM Foam Test Methods D 892 and D 6082 was selected because of its designed capability to control liquid temperature in the test cells for long periods at 150°C. Six cells could be run simultaneously. The air bath was also desirable since it was necessary to change the bath temperature quickly for repeat foaming tests on the same oil. With the air bath this could be done in place whereas with liquid baths a cell must be transferred from one hot liquid bath to another.

Figure 1 - Air-heated foam test bath used for development of Foamgen© Test. Two cylinders at a time are rotated by carousel (Figure 2) in front of window for view of foaming behavior.

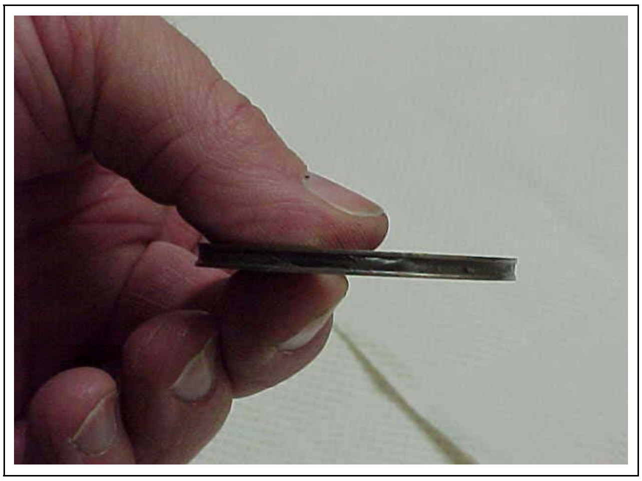

Figure 3 - Picture of a Foamgen©Test 'button sandwich' of elastomer gasketing material between two precisely spaced steel disks.

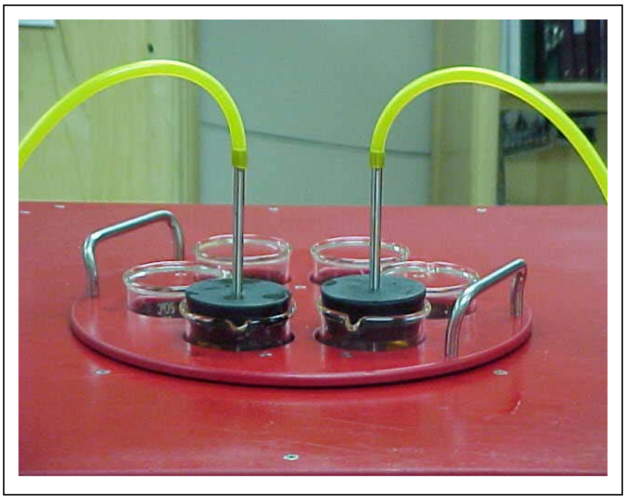

Figure 2 - Carousel of air-heated foam testing device in Figure 1 showing two cylinders of six containing oil under foaming test.

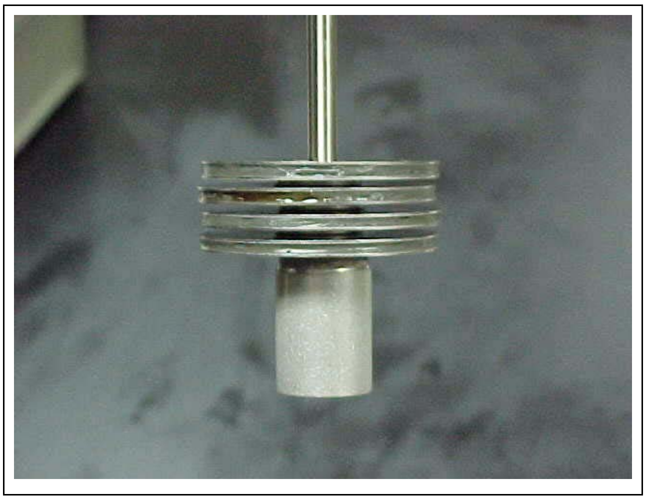

Figure 4 - Stack of four Foamgen©Test buttons of elastomeric sealant threaded on air-supply tube and resting on top of foam-head.

Figure 1 is a picture of the TFAB bath showing a foaming test in one of the six-cell carousel of Figure 2.

Elastomer Test Specimens

Design of the elastomer test specimen for the Foamgen© Test took into consideration the foam test cell and it was decided to make a steel-elastomer-steel sandwich 'buttons' of 1.50 mm elastomer thickness. Specially made punched and reverse-coined steel discs were separated by precision spacers to give a 1.50 mm gap.

When the elastomer was in the form of an extrudable mastic rather than a pre-cured slab, the mastic was applied carefully to avoid developing hidden holes in the mastic inside of the sandwich. One of the Foamgen©Test sandwich 'buttons' is shown in Figure 3.

To obtain sufficient area of exposure and to permit variation of this area, the Test sandwich buttons (which were made with holes in the center) were threaded on the steel air supply tube to the foam head for the cell. This is show in Figure 4 where four Test buttons are threaded above the foam head.

Also, the area of elastomer exposed to the oil in the Foamgen©Test could be changed by varying either or both the thickness of the elastomer in the button and the number of buttons.

One of the early questions concerned the amount of curing time required for the mastic after the Test button had been assembled and this was one of the first of several parameters studied.

Taguchi Studies

Although the earlier paper by the authors covered many introductory details, it was considered important to present some of that information again to help the reader of this paper grasp the significance of the new information developed.

Taguchi Method

The Taguchi method [2] was designed to obtain the maximum amount of information from the least number of experiments. Suspected influences (factors or variables) on a given property (such as foaming tendency) are selected at two considerably different but reasonable levels (e.g., a short and a long curing time). This selection of sufficiently different levels for each factor is important in establishing whether or not, and to what degree, the chosen factor is influential relative to the other factors.

In the course of these Taguchi experiments, several anticipated influences on foaming tendency were evaluated and some of these had surprisingly low influence. Interestingly, the influence of some factors varied with foaming test temperature.

Table 1 - Factors, Description and Levels in Seven-Factor Taguchi Study			
Factors	**Description**	**Level 1**	**Level 2**
A	Cure Time of Mastic	5 min.	72 hrs.
B	Surface Area Exposed	128 mm^2	958 mm^2
C	Naphthenicity of Oil	0%	50%
D	Additive Content	3%	12%
E	Oil Agitation	End of Test	Every 12 hrs.
F	Oil Temp.	Room Temp.	150° C
G	Exposure Time to Oil	24 hrs.	7 Days

Initial Study of Factors

Seven factors were selected for first studies to eliminate any of these that were not critically important. A simple blend of a naphthenic and a paraffinic base stocks was used. Table 1 lists these factors and their high and low levels important for running a discriminating Taguchi analysis.

Table 2 shows the combination of the factors and levels that were used in each trial. The seven factors required eight trial analyses to form the the necessary orthogonal array [3]. As may be seen, each trial involved a different combination of the seven factors. These seven factors were chosen to include several that were potentially important as well as some that might be unsuspectedly effective.

Table 2 Taguchi Seven-Factor Orthogonal Array

Factors	A	B	C	D	E	F	G
Trial 1	1	1	1	1	1	1	1
Trial 2	1	1	1	2	2	2	2
Trial 3	1	2	2	1	1	2	2
Trial 4	1	2	2	2	2	1	1
Trial 5	2	1	2	1	2	1	2
Trial 6	2	1	2	2	1	2	1
Trial 7	2	2	1	1	2	2	1
Trial 8	2	2	1	2	1	1	2

Foaming Tendency Data - Foaming tendency was used as the test condition against which to measure the influence of the factors. This value is the height of foam above the top of the remaining liquid. measured in milliLiters in a 1,000 mL graduate, 60 mm in inner diameter.

To evaluate the influence of seven factors, eight trials or tests are required. Each test oil was re-foamed three timesin quick succession and the results averaged to obtain the trial result. Results are shown in Figure 5.

In the Taguchi evaluation, proper choice of ranges for each factor will result in good differentiation and, from this, good separation of the factors regarding their influence. The results shown in Figure 5 show both good differentiation and foaming repeatability.

Analysis of Influence Level - In order to determine the relative influence of the various factors, orthogonal matrix analysis techniques of the results shown in Figure 5 were necessary [Roy]. Figure 6 shows this information.

Three Positive (Level 2) Factors - Of the seven factors incorporated in the study, only four were found to have significant effects on foaming tendency at 150°C, that is,

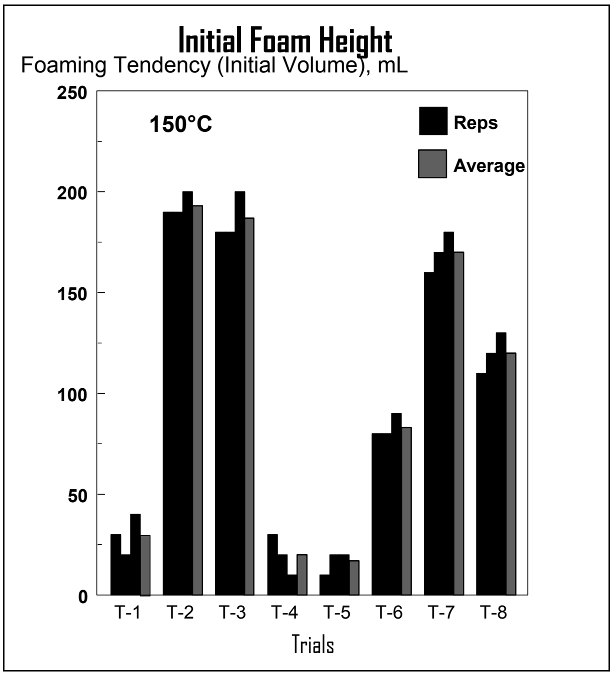

Figure 5 - Results of seven-factor Taguchi test matrix showing good differentiation and repeatability using foaming tendency as the test property or discriminator.

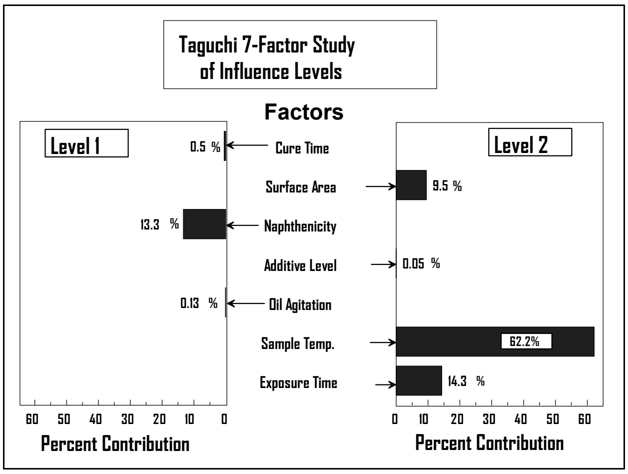

Figure 6 - Analysis of 7-variable Taguchi test matrix showing considerable differences in influence among factors.

associated with more than 5% of the influence on foaming tendency.

Those four factors, in order of importance and Level (*Level 1*, **Level 2**) were:

Sample Temperature - **62.2%**,

Exposure Time - **14.3%**

Naphthenicity - **13.3**%, and

Surface Area - **9.5%**

Of the three factors influencing foaming tendency at a positive, Level 2 activity, none were surprising. Sample temperature is associated with increased solubility and was by far the most important factor.

Both the factors Surface Area Exposed and Exposure Time would be reasonably expected to be inclined toward Level 2. Actually, the fact that exposure time does not appear to have a greater influence suggests that extraction of the pro-foamant may be an interactive effect with temperature of extraction.

Importance of Molecular Dimensions in Penetrating Elastomeric Mastic Structure - However, another factor associated with solubilization, Naphthenicity, showed a negative influence. The meaning of this observation seemed to be that the lower the amount of naphthenic base oil present and, therefore the more paraffinic base oil, the greater the extraction of pro-foamant material. It would appear that the bulkier naphthenic molecules have greater difficulty than paraffinic molecules in penetrating the elastomeric mastic and removing pro-foamant material .

Low Influence Factors; Elastomer Curing Time - Of the factors having little or no influence, Cure Time was the most surprising. It would normally be expected that the degree of curing the elastomer would influence the rate of penetration of the oil. This did not seem to be the case.

The lack of influence of the other two factors, Oil Agitation and Additive Content were not surprising. Oil agitation is likely to be completely superflous with the natural circulation of hot oil. The package additive was an automotive engine oil type.

Presence and Influence of Silicon in Seven-Factor Study

In the above analysis of the influence of various factors, foaming tendency was used as a discriminator -- a property reflecting the influence of a factor. However, any number of other properties can be used to see if they are affected by the conditions of testing. One of these -- important to the understanding of the pro-foamant extraction process is the silicon level found in the test oils. Accordingly, the test oils at the conclusion of the trials were analyzed for their silicon contents. Figure 7 shows the results.

Once again it is evident that there is good differentiation among the eight trials. However, the pattern is somewhat different than that shown by foaming tendency in Figure 5. More important, the high levels of silicon observed in some of the trials show how effective the extraction process can be.

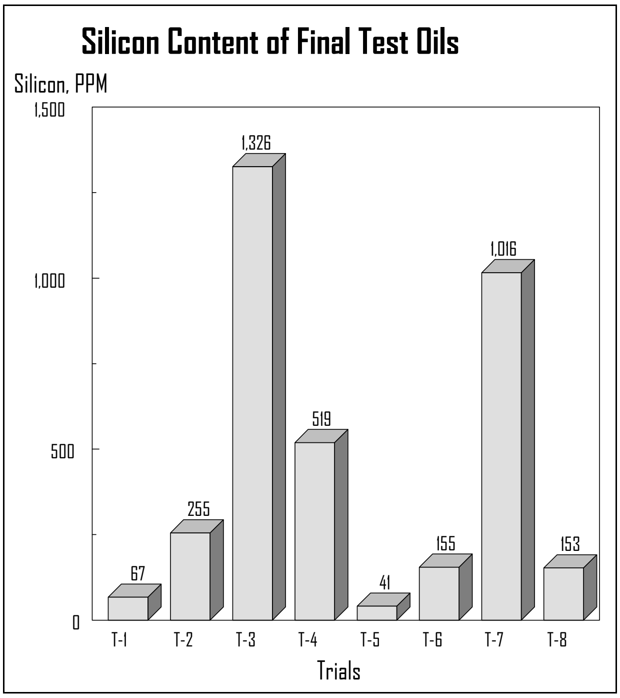

Figure 7 - Results of seven-factor Taguchi test matrix showing good differentiation and repeatability using silicon content as the test property or discriminator.

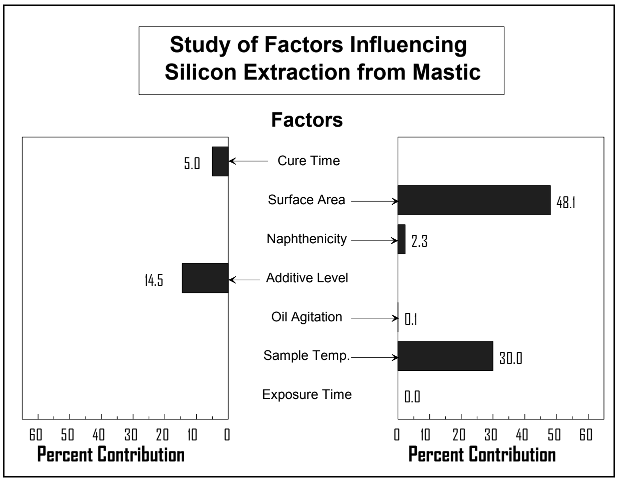

Figure 8 - Influences on silicon extraction from matic during seven-factor Taguchi analysis.

Matrix Analysis - Analysis of the data from the viewpoint of the level of influence of those seven factors evaluated is shown in Figure 8.

The apparent effect of surface area exposed is the dominant influence. This shows the direct relationship between either the area permitting extraction or the volume of elastomer from which to extract the silicon-carrying materials. (Distinction between area and volume effects is a challenging subject for later study.)

Sample temperature remains an important factor in regard to extraction of silicon-bearing components of the elastomeric mastic.

The strong negative influence of the additive used was surprising. Apparently, additives can inhibit the extraction of silicon-containing components of the elastomeric mastic.

Results of this silicon level study is interesting when contrasted to the associated level of foaming tendency.

Effect of Trial Conditions

High Silicon Extraction Levels - From Tables 1 and 2, the combination of variables giving the highest extraction of silicon are:

For Trial 3 - Cure Time: min.; Surface Area : max.; Naphthenic Base Oil Content: max.; Additive Content: min.; Oil Agitation: min.; Oil Temp.: max.; Exposure Time: max.

For Trial 7 - Cure Time: max.; Surface Area: max.; Naphthenic Base Oil Content: min.; Additive Content: min.; Oil Agitation: max.; Oil Temp.: max.; Exposure Time: min.

Of the seven factors only three major factors are common to both: surface area (at Level 2), additive content (at Level 1), and oil temperature (at Level 2).

Low Silicon Extraction Levels - Similarly, from Tables 1 and 2 the variables giving lowest extraction of silicon are:

For Trial 1 - Cure time: min.; surface area : min. naphthenic base oil content: min.; additive content: min.; oil agitation: max.; oil temp.: max.; exposure time: max.

For Trial 5 - Cure time: max.; surface area : min. naphthenic base oil content: max; additive content: min.; oil agitation: max.; oil temp.: max.; exposure time: max.

Surface area, additive content, and oil temperature are all at Level 2. As in high silicon extraction,these are the three major factors in common between the two trials.

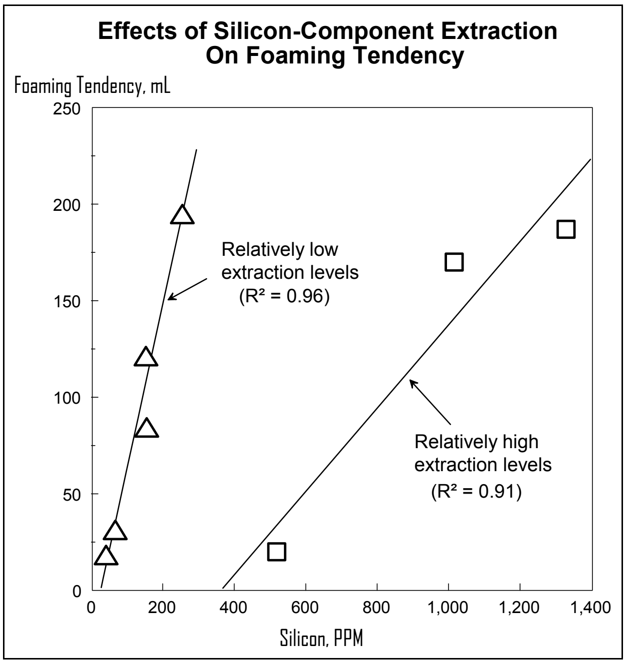

Figure 9 - Analysis of the cross-correlation of silicon-carrying materials and the associated foaming tendency. (Information taken from the seven-factor Taguchi test matrix.)

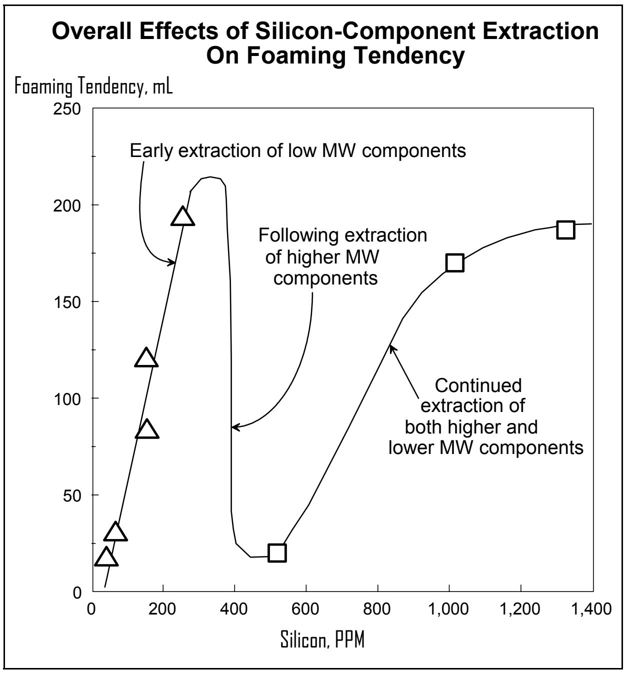

Figure 10 - Re-analysis of Figure 9 showing the cross-correlation of the progressive influence of higher and lower MW polysiloxane.

Correlation of Foaming Tendency and Silicon Content

Perhaps the most important consideration in studies of silicon extraction from elastomers is the degree of correlation between foaming tendency and silicon extraction. That is, it is of interest to know whether higher levels of silicon imply higher foaming tendency and *vice versa*.

The data generated in the seven-factor Taguchi study are useful. The end-of-test samples provide an interesting opportunity to evaluate the relationship between silicon level and degree of foaming tendency. A cross-plot of silicon levels versus foaming tendency is shown in Figure 9 and seems to contain several pieces of information.

First of all, there seem to be two zones of relationships, a highly correlated zone at lower silicon concentrations and another, more loosely correlated set at higher silicon concentrations.

First Zone - In the absence of addition of anti-foamant to any of the blends used in this Taguchi study, the first zone may be expressing a direct and close relationship between foaming tendency and extracted amounts of the pro-foamant, silicon-containing material from the elastomeric mastic. If so, the relationship is very close (Coefficient of Determination $R^2 = 0.96$) with an intercept reasonably close to zero as shown.

This indicates an immediate and highly effective adverse influence of the more readily extractable low MW polysiloxanes on foaming of the oil.

Second Zone - The second zone shows that at a comparatively moderate level of silicon (~500 PPM), the foaming tendency drops considerably to a relatively low level (20 mL). This implies that, at a certain level of silicon extraction, the polysiloxane extracted becomes more of an anti-foamant.

A reasonable conjecture is that as silicon extraction continues, higher MW polysiloxane anti-foamants are leached from the elastomeric mastic. As more and more silicon-containing material is extracted, the amount of both lower and higher MW polysiloxane would increase to the point of limited effectiveness of the higher MW polysiloxane because of solubility. At this point the lower MW materials would again exert more influence on foaming but in the presence of limiting amounts of the higher MW material.

If this reasoning is correct, the resultant overall relationship might appear as in Figure 10.

Foaming Tendency, Base Oil Composition and Temperature of Foaming

Another variable critical to the appraisal of foaming tendency is the temperature at which the foaming evaluation is conducted.

Taguchi studies of three gear oils, two of which had a mineral oil basestock and the third having a synthetic basestock gave opportunity to evaluate the effect of several parameters on foaming tendency.

Apparent Effect of Base Oil Composition - Two gear oils of high VI having equivalent kinematic viscosities at 100°C were evaluated for the influence of composition, area and time of exposure. The viscosity of the mineral oil based gear oil was 25.82 cSt while the viscosity of the synthetic oil based gear oil was 25.34 cSt. The elastomer exposure temperature in the Taguchi trials was 150°C for 120 hours using 200 mL of oil. Analysis of the Taguchi study results of this three-factor comparison (base oil composition, elastomeric mastic surface area exposed and length of time of exposure) gave the results in Figures 11 at 150°C and Figure 12 at 22°C.

Composition - As shown in Figure 11, with foaming tests conducted at 150°C, the dominant effect seems to be the presence of the synthetic oil with little evidence of either surface area or length of time of exposure effects. On the basis of these results, it would be reasonable to say that the synthetic oil was singularly effective in producing the resulting foaming tendency.

However, there are other influences which must be considered as well. One of these is temperature and another is viscosity. Both are co-dependent. Separation of properties in order to obtain relevant Taguchi analysis is very important in the formation of conclusions as will be shown.

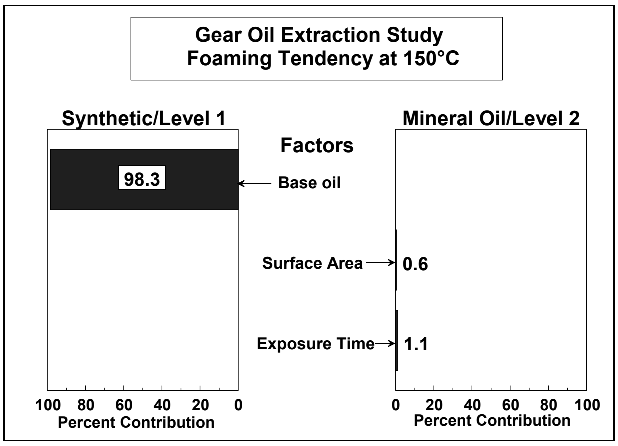

Figure 11 - Analysis of the influence of mineral versus synthetic oil basestock on foaming tendency. Extraction of pro-foamants tests conducted at 150°C. Foaming tests conducted at 150°C as well.

Temperature of Foaming Tests - When the same end-of-test samples from the extraction and foam-testing at 150°C were re-foamed at room temperature (~22°C), new information became available from significant differences the previous Taguchi 150°C analysis results shown in Figure 11. Taguchi analysis results from re-foaming at room temperature are shown in Figure 12.

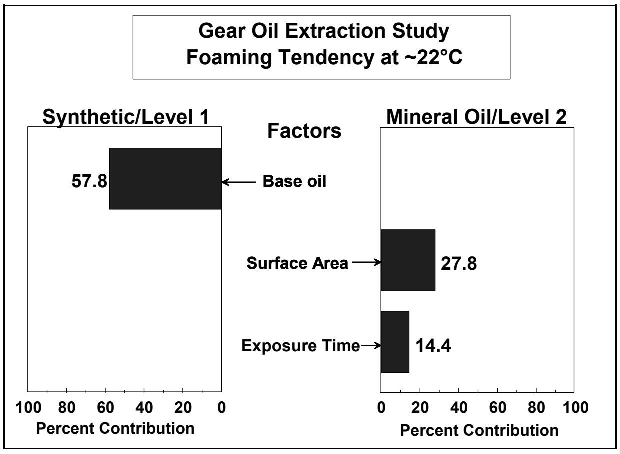

Figure 12 - Analysis of the influence of mineral versus synthetic oil basestock on foaming tendency. Extraction of pro-foamants tests were conducted at 150°C. Foaming tests were conducted at 150°C as well.

Conducting the foaming tests at room temperature reveals that not only the factor of synthetic oil is highly influential but the factors of surface area and exposure time are also significantly effective.

Why the influence of these factors are more evident when the samples are foam-tested at room temperature is relevant to understanding not only the effects of pro-foamant extraction from the elastomeric mastic but also other factors which influence foaming tendency.

Viscosity and Related Effects - An important factor whose influence has not been shown to this point is the viscosity of the oil.

Viscosity, normally considered a measure of the bulk flow rate of an oil, is also related to the surface viscosity of a foam bubble and thus plays a role in the formation and durability of foam.

Viscosity effects were evaluated in a further gear oil study by replacing the synthetic-based gear oil in the previous study with another mineral oil of about half the viscosity at 100°C (14.15 cSt versus 25.34 cSt) and using viscosity as one of the three factors rather than the base oil type.

Results obtained from foaming tendency analysis at 150°C are shown in Figure 13. In this comparison, low oil viscosity is essentially the only factor of evident influence. This result is consistent with the previous finding in the seven-factor analysis in which it was indicated that less bulky molecules can penetrate the elastomer matrix more easily. Presumably, this is also the case when the lower molecular weight molecules associated with lower viscosity are compared to a higher viscosity oil.

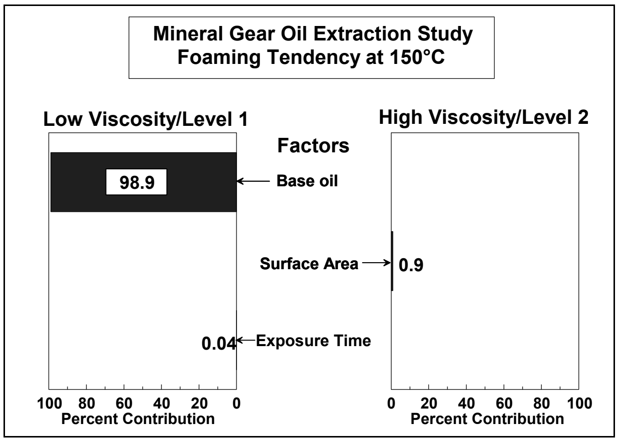

Figure 13 - Analysis of the influence of viscosity on foaming tendency. Extraction of pro-foamants tests conducted at 150°C. Foaming tests conducted at 150°C as well.

Discussion

Four Factors Affecting Generation of Pro-Foamants

Overall, using the Taguchi technique of analysis and the Foamgen© Test method and air-bath equipment, four factors have been identified which markedly affect the extraction of pro-foamants from elastomeric mastics by oils and fluids. These are:

1. Temperature of exposure
2. Area of exposure
3. Time period of exposure
4. Viscosity/molecular weight and size

These four factors are all associated with transport dynamics. This is expected since the extraction of pro-foamants involves the penetration of an elastomeric matrix, dissolution or isolation of pro-foamant polymeric molecules of various molecular weight followed by transport to the bulk of the oil bathing the elastomer.

Several other factors were either found of secondary importance (such as mastic curing time) or have not yet been adequately explored (such as the effect of additives).

Further Analysis of the Taguchi Data

One of the strengths of the Taguchi technique is the ability to dissect the individual trials for more information.

Temperature of Extraction - The two temperature at which the present Foamgen©Tests were carried out give some additional information.

While it would be expected that any Taguchi Trial in which the temperature of extraction is room temperature would have low levels of pro-foamant extraction, this is not entirely the case.

Inspection of the four Trials in the Seven-Factor study (see Tables 1 and 2, and Figure 5) shows that of the four Trials conducted at room temperature (Trials 1, 4, 5, and 8), only three have low levels of foaming tendency (Trials 1, 4, and 5 in Figure 5). Trial 8 shows the fourth highest foaming tendency despite room temperature exposure to the elastomeric mastic.

To reconsider the factors involved in Trial 8, Cure Time, Additive Content, and Oil Agitation can be eliminated as being of minor influence on foaming tendency as shown in Table 3. If Level 2 Paraffinicity is substituted for Level 1 Naphthenicity, then Paraffinicity, Surface Area and Exposure Time are all at Level 2 in Trial 8. In contrast, only one of these three factors are at Level 2 in Trials 1, 4, and 5. From this it appears that given enough solvency, area of exposure and time to penetrate the mastic, significant pro-foamant can be generated even at lower temperatures.

Table 3 Closer Analysis of Taguchi Seven-Factor Orthogonal Array

Factors	A	Area Exposed	Paraffin icity	D	E	Bath Temp	Time Exposed
Trial 1	Not Significant	**1**	**2**	Not Significant	Not Significant	**1**	**1**
Trial 2		1	2			2	2
Trial 3		2	1			2	2
Trial 4		**2**	**1**			**1**	**1**
Trial 5		**1**	**1**			**1**	**2**
Trial 6		1	1			2	1
Trial 7		2	2			2	1
Trial 8		**2**	**2**			**1**	**2**

Recognizing the considerable difference in solution dynamics between room temperature and 150°C, two possibilities come to mind to explain the foaming tendency level of Trial 8:

The first possibility is that not much time at higher temperatures is really required to release considerable pro-foamant from the mastic. That is, 120 hours at 150°C may be much in excess of the amount of time necessary to release the available pro-foamant. The second (a corollary) is that -- given sufficient surface area of mastic, enough pro-foamant can be released at room temperature to have major consequences on foaming tendency.

This line of reasoning tends to be supported by the silicon concentration study: the left-most, highly correlative curve of Figure 9 was generated from the four room temperature Trials plus Trial 6 in which, although the oil temperature was at 150°C, the mastic surface area (and volume), and exposure time were at Level 1 and could be expected to either curtail the time required to extract the silicon-bearing pro-foamant or reduce the amount of pro-foamant available from the Foamgen© Test buttons.

From the foregoing observations and reasoning, it would appear that restriction of solvent access to the elastomeric mastic by either test temperature, time of access, or amount of mastic, still produces enough pro-foamant to give evident foaming tendency.

Silicon Extraction - From the foregoing, more detailed analysis of the Taguchi study, it would appear that the extraction process first generates lower MW polysiloxane homologs and that this happens rapidly at higher temperatures but is effective even at lower temperatures if sufficient mastic is available.

At higher temperature and longer exposure times, however, the extraction process apparently begins to bring higher MW polysiloxane homologs out of the elastomeric matrix and some of these are believed to function as anti-foamants.

Viscosity and Synthetic Fluid Effects - Both viscosity and synthetic basestocks play important roles in the extraction of pro-foamant material from elastomeric mastic. On the basis of the data in hand, it appears that the lower the viscosity and the simpler and less bulky the molecules, the more effectively pro-foamants can be extracted.

No studies have yet been done on comparing various MW of synthetic oils to see if their relationship is the same as appears for mineral oils in regard to viscosity. Certainly, it might be anticipated that synthetic oils of certain chemistries might be more or less aggressive regarding the solubilization of polysiloxanes. Aggressiveness might be turned to advantage if the higher MW, anti-foamant polysiloxanes were more quickly extracted from the elastomeric mastic.

Foaming Temperature Effects

Very significant differences were shown in the same oil when foamed at 150°C in contrast to room temperature. The differences were great enough to suggest caution in analyzing the foaming tendency at only one temperature, particularly for mechanical devices used over a range of temperatures.

Foaming Tendency Control

There are obviously two primary materials critical to controlling foaming tendency. The first is the elastomer and the second is the oil or fluid and its content of additive. If the elastomer has little available pro-foamants either primarily available by early extraction or secondarily by later breakdown and extraction, foaming tendency will be considerably limited. Secondly, if the oil or fluid is formulated to block extraction of pro-foamants, foaming tendency will, likewise, be curtailed.

As previously mentioned, there is a strong need to have a common method and equipment to study the various factors involved in generation and control of foaming tendency.

Test Method

The Foamgen© Test approach reported in this and the previous paper has shown considerable flexibility in studies of pro-foamant extraction from elastomeric mastics. A principle advantage is the ability to perform foaming tendency tests in the same cell in which pro-foamant extraction takes place without removing either the cell or the oil being tested.

Use of an air bath with several test cells is particularly advantageous since it makes possible quick change of bath temperature. Such temperature is advantageous, particularly when simulating the foaming tendency of an oil at different temperatures of operation in the associated mechanism. The foaming tendency of a given oil is not restricted to one temperature but may occur at different temperatures depending on the mechanism.

As a consequence of these and other studies the Foamgen© Test protocol has been developed for the use of those interested and a summary of the test method is given as Appendix 1.

Conclusions

A relevant method of repeatably and simply determining the response of oils and elastomers to one another in regard to the generation of foaming tendencies has been proposed.

In initial work, this method, called the Foamgen© Test, discriminated among several factors when used in conjunction with Taguchi techniques.

Exposure temperature and time, and mastic area or volume were found to be important factors affecting the foaming tendency of an oil.

In addition, viscosity level was shown to be important apparently because of its relation to MW an ease of molecular penetration of the elastomeric matrix.

Similarly, synthetic base oils may be more or less aggressive in regard to extraction and the authors recommend studies of these materials to learn more about their individual and homologous performances.

Foaming temperature has highly significant effects on the value of foaming tendency. It is recommended that in appraising foaming tendency of an oil/elastomer pair that foaming be carried out at two widely different temperatures corresponding to the temperature use range of the mechanical device.

The relationship between the foaming tendency of the oil and the extraction level of silicon may be more complex than anticipated. Results shown in this paper suggest that two regimes exist, one of which is related to the early release of low MW polysiloxane pro-foamants and the second which may be affected and tempered by the generation of some anti-foamants from release higher MW polysiloxanes. Further work in this area is considered important in establishing this relationship.

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Bibliography

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Appendix 1

Test Method

Test Parameters Established by Present and Previous Studies

In the initial work reported on this technique [1], the studies led to a proposed test method. The parameters of the test method are shown in Appendix Table 1.

While the interested reader can obtain the full method from the authors, the essence of the test method is:

1. The steel components of each button (two end pieces and one precision spacer) are weighed, the elastomer applied, and the button assembled. Excess elastomer is wiped off carefully after several minutes.
2. After the elastomer has cured for a few minutes and developed a cohesive surface or 'skinned over', all excess elastomer is removed from the surface of the buttons and the buttons reweighed to determine the amount of elastomer contained. Weight of the elastomer in the buttons should not vary by more than ±0.1 g. (More variation than this indicates that there may be unseen voids in the elastomer.)
3. The desired number of buttons are threaded on the air-supply tube for each of the test cells to be used but <u>not</u> inserted into the test cells at this point.
4. Two hundred mL of the test oils are put into the chosen test cells and brought to 24°C.
5. A foaming test is run at 24°C using the techniques of ASTM D-982 and the values recorded.
6. The bath temperature is raised to 150°C and, when thermocouple measurements of the test oils indicate that they are at temperature, the foaming test is repeated and values recorded.
7. The foam-head air-supply tube and buttons are now placed in the cells and the 100-hr., 150°C extraction portion of the test begun.
8. If desired, every 24 hours, 1-mL samples of the oils in the cells may be taken to follow the progress of extraction by analysis for the presence of silicon or other component from the elastomer. Also, foam tests may be run periodically for the same purpose without upsetting the progress of the test.
9. After 100 hours of extraction, a final foaming test is made at 150°C. The air bath is then immediately cooled to 24°C and another foam test performed.
10. The residual test oil is retained, if desired, for further tests.

Appendix Table 1 - Test Parameters

Parameter	Value
Test time	100 hours
Test temperature	150°C
Test button dia.	510 mm
Button mastic thickness	1.5 mm
Number of buttons	4
Test cell dimensions	ASTM 892
Volume of test oil	200 mL (initial)