

SURFACTANT PARAMETER EFFECTS ON CLEANING EFFICIENCY

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INTRODUCTION

In the search for chemicals to replace CFC-113 and trichloroethane (TCA) in metal degreasing and cleaning, many precision cleaning operations are pursuing the use of water and nonionic surfactants. This option is frequently considered in DASD (Direct Access Storage Device or disk drive) assembly operations. These operations typically require cleaning of all piece parts which are subsequently assembled in clean room environments.

The parts going into the cleanrooms have usually been cleaned previously by the original part manufacturer. This reduces the level of power required in a cleaning chemistry. Since the parts are most likely contaminated only with packaging debris or airborne contaminants present in warehouse environments, strong alkaline detergents are not required. The use of a surfactant in order to provide a material with a high wetting capability is much more relevant to the cleaning problem at hand. The nonionic surfactants in particular are of most interest to DASD operations, since any ionic residues, especially Cl^- , Br^- , Na^+ , etc., will cause part corrosion problems.

The members of the Research Triangle Institute (RTI) Surface Cleaning Technology Consortium (SCTC) have long been interested in understanding the fundamentals of aqueous cleaning processes. One of the areas of interest has been to identify the basic properties of nonionic surfactants that may affect cleaning processes. In particular, the properties of the linear ethoxylated-alcohol family of nonionics have been of interest.

The number of surfactants available for consideration can be daunting for those faced with selecting "just the right one." One reference¹, has over 192 pages of brand names of various types of surfactants. The SCTC members were interested in identifying the fundamental properties of a surfactant that would help in narrowing the selection.

The ideal surfactant would be infinitely soluble in all temperatures of water, have very good wetting and detergency at low concentrations and not corrode or otherwise affect the parts being cleaned. These characteristics are all controlled by the physical parameters of the surfactant molecule. The linear alcohol nonionic surfactants can be characterized by a number of physical parameters including the hydrophilic-lipophilic balance (HLB), ethoxylation level (EO), critical micelle concentration (CMC) and cloud point. These properties are well documented in the literature¹⁻³. The question of interest to the SCTC members was which of these properties matter in their applications and what values would identify potential candidates for use.

This article describes these properties and the affect they have on precision surface cleaning. Experimental design and results are presented, along with conclusions and application suggestions.

SURFACTANT PROPERTIES

The surfactant properties investigated in this study were the chain length, hydrophilic-lipophilic balance, ethoxylation level, cloud point and critical micelle concentration. The definition and effect of these properties are described in the following section.

Chain Length

Chain length refers to the number of carbon atoms contained in the longest direct line in the surfactant molecule. Some sources believe that cleaning performance is primarily a function of carbon chain length.⁴ Longer chain surfactants are usually more effective detergents, which reduce surface tension of water and help to emulsify oils.^{5,6} Surfactants with chain lengths of 10-14 carbons are commonly used for this purpose. However, the solubility of the surfactant in water decreases with increasing chain length³. These two properties must be balanced in order to choose the optimum molecule for each application.

In addition, the temperature to be used in the cleaning bath is important. Shorter chain length surfactants are more effective in cool temperature applications. In general, the higher the temperature, the longer the chain should be.⁷ Therefore, if you wish to reduce heating requirements and energy consumption, you should consider a shorter chain surfactant.

This is a good example of what happens when asking almost any question about the basic parameters involved with cleaning processes: Should we select a surfactant with a short chain length or a long chain length? - "It depends..."

In this medium temperature application, it was felt that high detergency and especially better wetting properties would be desirable. This lead to the selection of a longer chain length surfactant.

Hydrophilic-Lipophilic Balance

The hydrophilic-lipophilic balance (HLB) is an empirical number that describes the relationship between the hydrophilic (water-soluble) and lipophilic (oil-soluble) portions of a surfactant molecule. Oil-soluble surfactants have low HLB numbers and water-soluble surfactants have high numbers. This number is also related to the percent ethoxylation (EO) of the surfactant. Typical ranges and applications as given by Shell Chemical Company² are shown below. HLB numbers are so widely used that McCutcheon's provides a separate listing of surfactants in HLB number sequence.

<u>HLB NUMBER</u>	<u>EO CONTENT, % WT.</u>	<u>APPLICATION</u>
4 - 6	20 - 30	Water in oil emulsifier
7 - 15	35 - 75	Wetting agent
8 - 18	40 - 90	Oil in water emulsifier
10 - 15	50 - 75	Detergent
10 - 18	50 - 90	Solubilizer

One of the greatest concerns of the SCTC members was the rinsability of the surfactant. Any residues left on a part by the cleaning process could be disastrous to product performance. Since the processes of interest were all aqueous based, it would follow that the higher HLB numbers (the more water-soluble surfactants) would result in less residue after rinsing. Private conversations with D. Smith of Vista Chemicals indicated that better rinsability would also be achieved with surfactants with higher EO levels. This favored the use of a high HLB number surfactant.

Ethoxylation Level

The ethylene oxide or ethylene oxide/propylene oxide chain is the water-soluble portion of the surfactant molecule. Higher EO gives higher water-solubility. Again, this will indirectly drive the HLB number because EO levels are directly related to HLB numbers.

Product literature also indicates that, at the low concentrations typical in this application, the surface tension of a solution using a surfactant decreases as the EO level increases.⁸ This is another factor in favor of higher EO levels, and hence higher HLB numbers.

The effects of EO levels may not be linear. Farella et al. indicate that cleaning efficiency increases with increasing EO to a point, then decreases again.⁸ This lack of linearity is important in process optimization studies.

Cloud Point

The cloud point of a nonionic surfactant is the temperature above which the surfactant becomes insoluble in the solution. A number of references point out that cleaning processes should operate just below the cloud point for optimal detergency.^{2,5,6} It follows that the cloud point of the surfactant should be considered when choosing the operating temperature of a cleaning bath. There is a standard ASTM test method (ASTM D2024) for determining cloud point. Cloud point is usually determined at a 1% (w/w) concentration. What effect do concentration changes have on cloud point, and do they affect cleaning efficiency?

As EO level increases, the cloud point of the solution also increases. This implies that if we select a high EO surfactant for its surface tension and solubility properties, we may have to operate at a higher temperature. This higher temperature may not be beneficial for the particular process under consideration.

Since there is a relationship between EO and cloud point, and EO effects may not be linear, does this imply that there are interactions between cloud point and EO that may also not be linear? These questions were considered when designing the experiments in this study.

Critical Micelle Concentration

A micelle is a colloidal aggregate of surfactant molecules. The critical micelle concentration (CMC) of a surfactant is the concentration at which micelles begin to form. At this point, several properties of the solution change abruptly, including surface tension and solubilization. The surface tension decreases with increasing surfactant concentration until the CMC is reached, at which point it levels off. Solubilization remains fairly constant with increasing concentration of surfactant until the CMC is reached. Then it increases rapidly. Detergency also increases with increase in concentration of surfactant but not as much as solubilization.⁵ The procedure for measuring CMC is described in ASTM D1331-89.

The question of concern to the SCTC members was what effect CMC had on cleaning efficiency. Since low surface tension and high solubilization are desirable for effective surface cleaning, the surfactant should be used at concentrations greater than the CMC. Shell literature also mentions that a surfactant may be ineffective at concentrations below CMC.

In summary, the above information led us to hypothesize that the most efficient cleaning in our application would be achieved if HLB values were high (better solubility), chain lengths were long (better detergency and wetting at the expense of cold temperature performance), cloud point was low (less energy consumption), EO levels were high (lower surface tensions), and concentrations were above the CMC (lower surface tension and higher solubilization).

OBJECTIVES

The objectives of the experiment were to identify the effects of and interactions between the surfactant properties defined above: hydrophilic-lipophilic balance, ethoxylation level, chain length, cloud point and critical micelle concentration. In addition, the experiment was designed to identify any nonlinearity in the effects. This required at least a 3-level experiment. Testing 5 factors at 3 levels with analysis of all interactions required 3^5 experiments for a full-factorial experiment (assuming only one repetition per setting)⁹. Fractional-factorial and other designs are available that decrease the number of experiments required, at some loss of resolution.

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The surfactants listed below were chosen to cover a reasonable range in each of the physical properties to be tested, as opposed to changing one property at a time. This selection meant that 3 factors at 3 levels (3^3) experiments in a full-factorial could be tested with approximately the same results as the larger test. In addition, 3 repetitions of each setting could be run.

	SURFACTANT A (810-60)	SURFACTANT B (91-6)	SURFACTANT C (25-12)
Chain Length	C8 - C10	C9 - 11	C12 - C15
Ethoxylation Level ^a	5	6	12
HLB	12	12.5	14.4
CMC (% vol.)	0.007%	0.02%	0.0009%

^aEO groups/alcohol, mole/mole, average

In order to test the effects of cloud point and concentration, we first needed to know if there was a relationship between the two parameters. All standard values for cloud point were given at a 1.0% (w/w) concentration. What happens at concentrations of 0.1% or less? A preliminary set of experiments was run in order to identify this relationship, and is shown below in Figure 1.

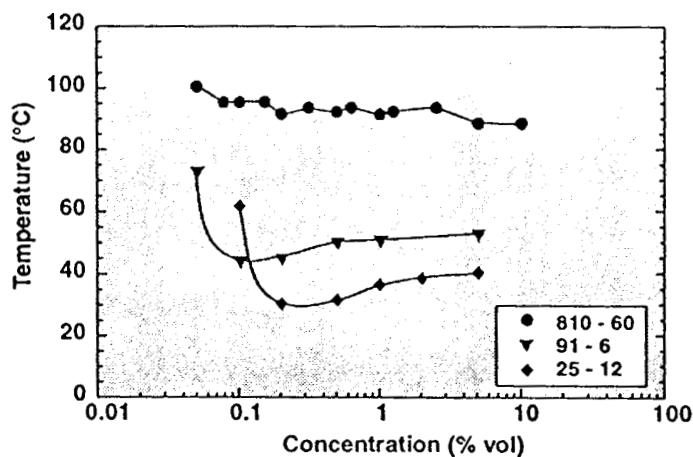


Figure 1. Cloud Point vs. Concentration

The relation between the two values changed markedly at low concentrations for two of the surfactants. These curves were plotted for a number of different surfactants prior to selecting the three shown above. The curves allowed us to define the experimental settings in terms of temperature (cloud point) and concentration. The SCTC members refer to these curves as surfactant operating (OC) curves.

The final experimental parameters and settings were:

SURFACTANT TYPE - A, B, C

TEMPERATURE - Cloud Point-7 °C, Cloud Point, Cloud Point+7 °C
(as determined by the OC)

CONCENTRATION - 0.2%, 1.0%, 5.0% (vol.)

MEASUREMENT METHOD

Previous experiments for the members of the RTI Surface Cleaning Technology Consortium led to development of a contamination and measurement method used for cleaning efficiency tests.¹⁰

The process begins with the use of a standard test part (See Figure 2). This part is a small (1.2" x 1.2" x 1.2") aluminum cube. Two of the sides of the part can be removed. These removable side plates have had various size tapped holes machined into them when they were bolted to the body of the block. This fabrication method allows the disassembly of the part for subsequent contamination or examination of the inside of the tapped holes. In addition to the tapped holes, numerous other mechanical features, such as tapered slots and countersunk holes have been machined into the part. This allows testing of cleaning efficiency for many common machining features with one part.

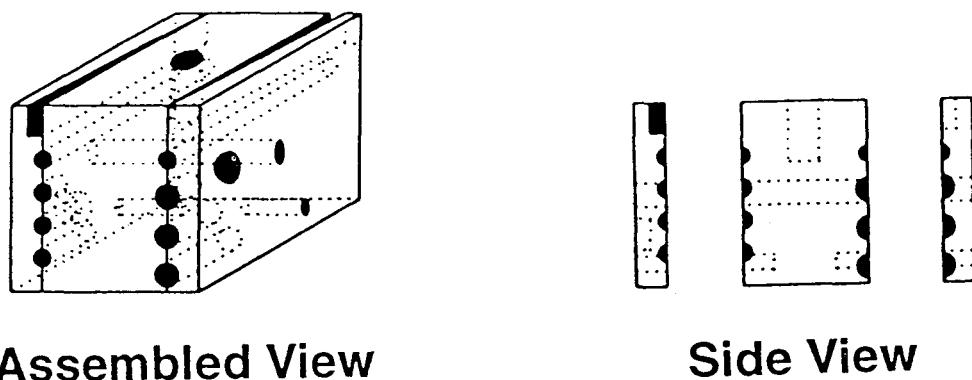


Figure 2. SCTC Standard Test Part

Typically, a number of test parts will be contaminated with fluorescent particles and oils. These contaminants were chosen based on their fluorescent properties and solubilities. There is very little overlap between the fluorescent excitation and emission spectra of these materials (See Figure 3). The contaminants may be extracted from the parts with an appropriate solvent and quantified with a fluorometer. Extracting the fluorescent material from a part that has been contaminated, but not cleaned provides a baseline 100% "dirty" numerical value. Extracting the contaminants from other parts after cleaning steps gives a result in milligrams of contaminant remaining that can be compared to the "dirty" value. This allows calculation of percent removal efficiency for the method under test. This method has been successfully used in tests involving the comparison and manipulation of such cleaning factors as process temperature, cleaning chemical concentration, and cleaning solution pH.

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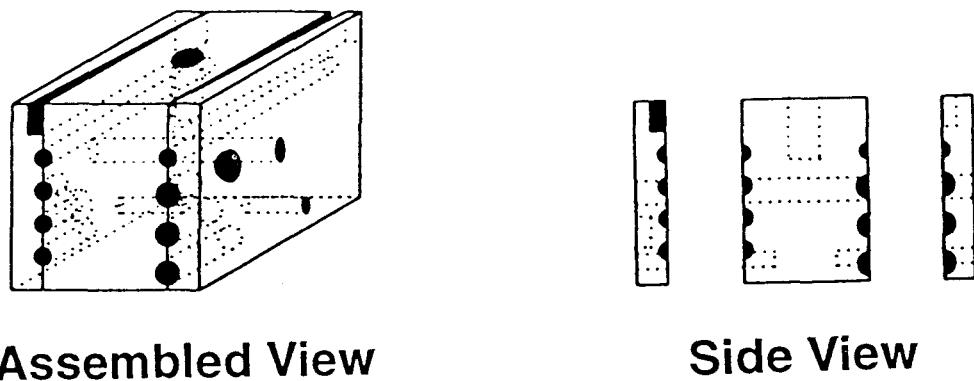


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**Excitation and Emission Fluorescence Spectra
Polystyrene Spheres (PS) and Motor Oil**

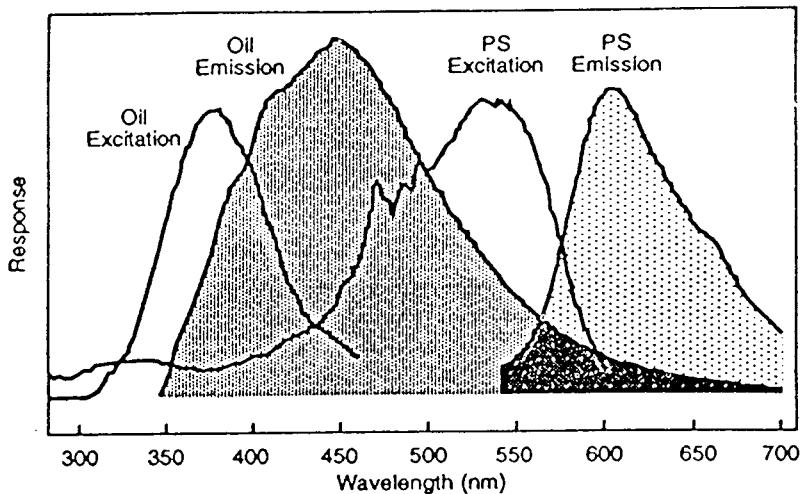


Figure 3 - Fluorescence Spectra

EXPERIMENTAL METHOD

Three standard test blocks were contaminated by applying 5 μl of the fluorescent particles (approx. 5 μm in diameter) to the bottom of each tapped hole in a side plate. The side plates and particles were then dried by placing them in a vacuum oven at 50 $^{\circ}\text{C}$ and 15" Hg for 15 minutes. The plates were removed and assembled onto the block. Each of the tapped holes in the block was contaminated with 3 μl of fluorescent oil. The parts were then cleaned using the appropriate cleaning solution. The cleaning cycle consisted of a 5 minute ultrasonic immersion followed by a 1 minute spray rinse with 18 Mohm deionized (DI) water. After rinsing, each part was disassembled and placed in its own extraction apparatus. The parts were extracted and the resulting solution was measured in the fluorometer. Results were recorded using fluorometer units (fl) and converted to cleaning efficiency numbers.

RESULTS

The results were analyzed using standard ANOVA (Analysis of Variance) calculations and graphic response charts. The standard ANOVA table for the particle contamination is shown below.

FACTOR	SS	DF	V	F	P
Surfactant (SF)	309.80	2	154.9	75.19*	42.78
Cloud Point (CP)	5.85	2	2.93	1.42	0.24
Concentration (C)	86.26	2	43.13	20.94*	11.5
SF x CP	3.45	4	0.86	0.42	0
SF x C	64.15	4	16.04	7.79*	7.82
CP x C	48.8	4	12.22	5.93*	5.69
SF x CP x C	84.99	8	10.62	5.16*	9.59
Error	111.17	54	2.06	22.38	
Total	714.54	80			

*Significant at 99% confidence limits

Table 1. ANOVA of Particle Cleaning Efficiency

The surfactant type obviously has the largest effect on cleaning efficiency. The graphs of the effects shown later help point out which of the surfactant properties might be responsible for the effect. The surfactant concentration has the second largest effect on efficiency. The effect of varying the solution temperature around the surfactant cloud point had very little effect, when considered by itself. There are significant 2-way and 3-way interactions present in the system. This is not a relationship that one likes to find in industrial experiments.¹¹

The standard ANOVA table for the oil contaminant is shown below. Once again the surfactant type has the largest effect on the system. There are also significant 2-way and 3-way interactions.

FACTOR	SS	DF	V	F	P
Surfactant (SF)	44.6	2	22.34	46.54*	37.61
Cloud Point (CP)	2.08	2	1.04	2.17	0.96
Concentration (C)	2.15	2	1.08	2.25*	1.02
SF x CP	3.29	4	0.82	1.71	1.18
SF x C	4.39	4	1.10	2.29*	2.13
CP x C	18.09	4	4.52	9.42*	13.91
SF x CP x C	15.71	8	1.96	4.08*	10.21
Error	25.83	54	0.48	32.98	
Total	116.21	80			

*Significant at 99% confidence limits

Table 2. ANOVA of Oil Cleaning Efficiency

Graphic analysis of the results helps show the relationships between the various effects (Figure 4). The graphs for the main effects show that they are nonlinear, for both particle and oil removal. In addition, the effects are not the same for the two contaminants. It would be preferable for the effects to be the same between contaminant type so that the most efficient surfactant for oil removal would also be the most efficient for particle removal.

The best particle removal was achieved when the surfactant used (25-12) had the lowest CMC of the three. This surfactant also had the highest HLB, EO and chain length of the three. The least efficient surfactant for oil removal was the one with the lowest CMC level. The other main factor settings do not appear to be significant, as already indicated by the ANOVA table.

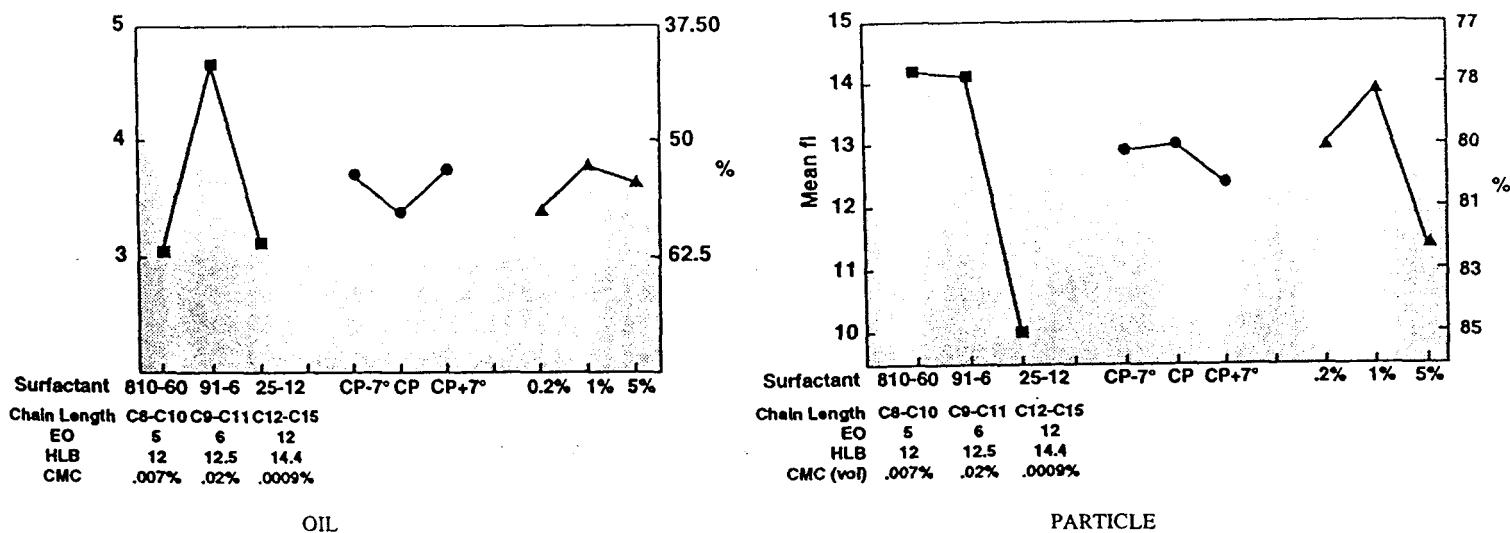


Figure 4. Main Effects

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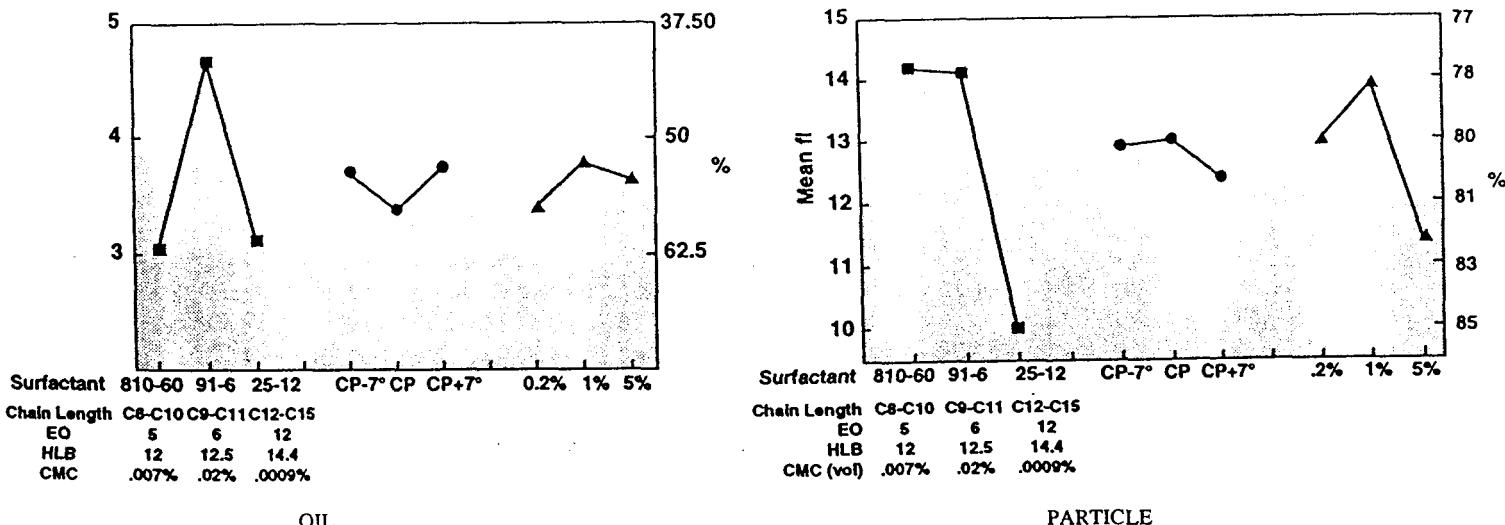


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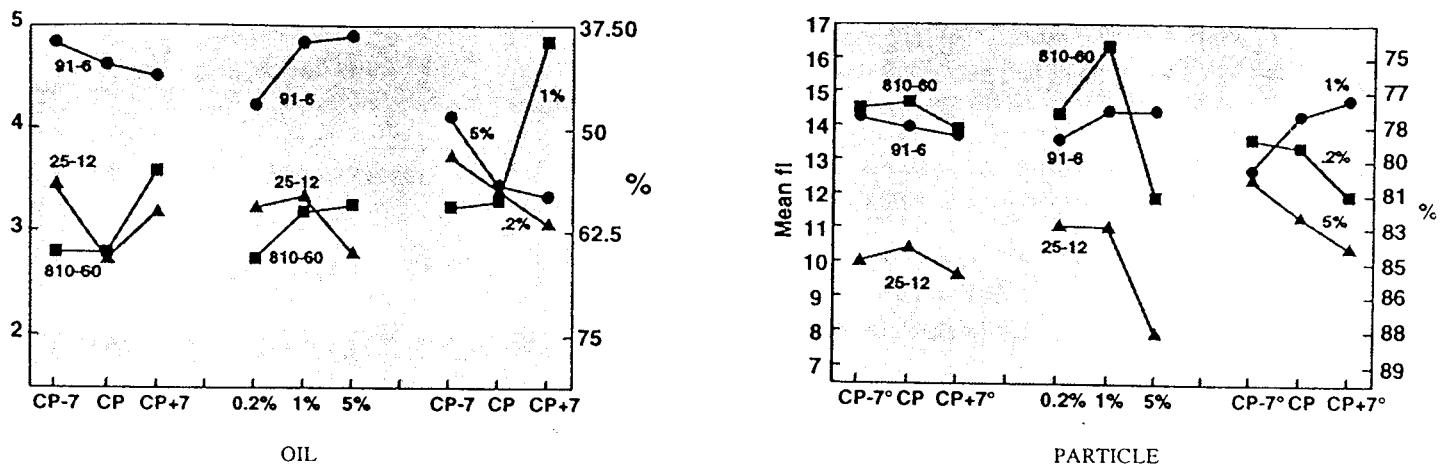


Figure 5. 2-Way Interactions

The two-way interactions (Figure 5) begin to show some of the complexity of this system. As previously shown in the ANOVA for the particle removal, the interactions between concentration and both surfactant type and operating temperature are significant. They are also nonlinear. At this time, we are not sure why the efficiency of the process decreases at 1.0% concentration and increased temperature. This effect holds true for the oil removal efficiency as well.

You will note that the oil removal effects are different from the particle removal effects. The three-way interactions serve to indicate the nonlinearity of the system and different responses between the two types of contaminants (Figure 6).

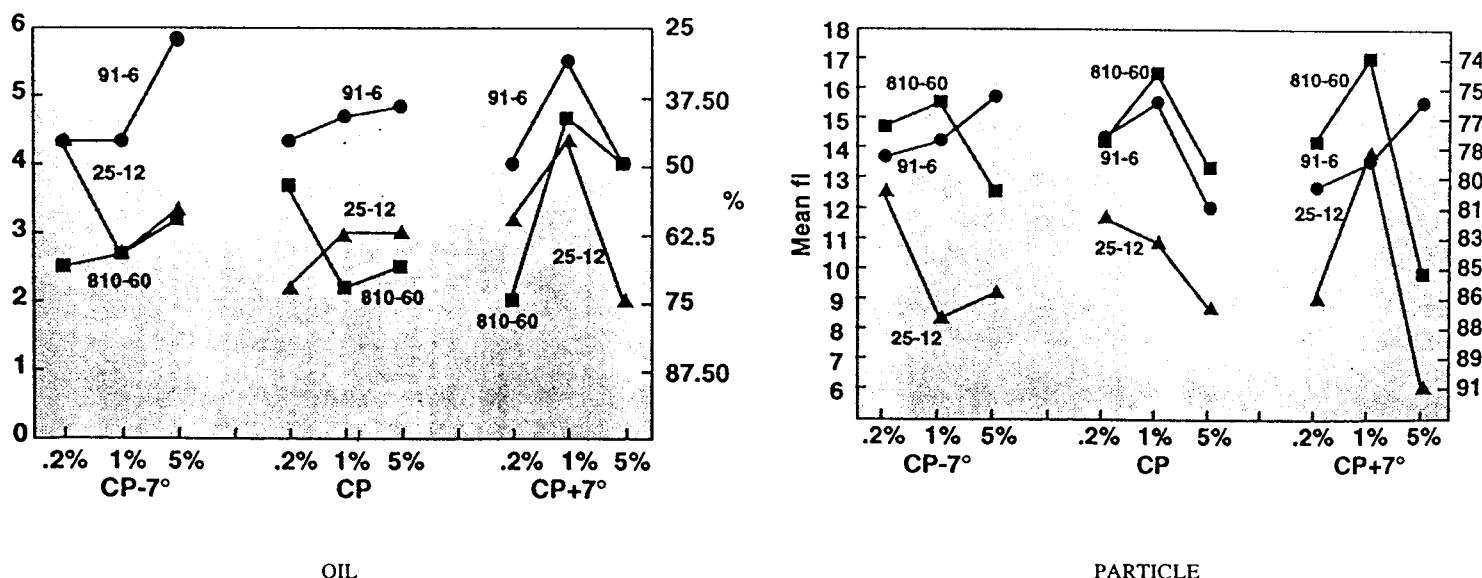


Figure 6. 3-Way Interactions

In summary, for both the contaminants, the experiment shows that:

The best overall cleaning efficiency was achieved with surfactant C, the surfactant with the highest HLB of those tested. This surfactant also had the longest chain length and highest EO level of those tested.

The best cleaning concentration was the 5% level. This provided for the highest multiple of the CMC value if using surfactant C, i.e. surfactant C had the lowest CMC of the materials tested.

The most effective temperature setting was found to be above the cloud point setting. Surfactant C did possess the lowest cloud point value, at any given concentration.

CONCLUSIONS

This experiment demonstrates the complexity that can be encountered when evaluating cleaning systems. The effects of the parameters studied are nonlinear and have complex 2-way and 3-way interactions. The preliminary hypotheses that were developed, (high HLB, long chain length, high EO, and low cloud point for higher cleaning efficiency) were borne out by the experimental data. One general method for surfactant selection could be defined as follows:

- Select surfactant(s) with the highest HLB, longest carbon chain and highest EO levels that are practical.
- Determine the cloud point vs. concentration (OC) curves for the materials selected.
- Select the material with the lowest OC curve, and operate at the highest concentration (within rinsibility limits) on that curve.
- Set the process temperature above the cloud point of the concentration previously selected.
- To increase precise control and understanding of the particular process, develop a detailed surface response model of the system. In addition, never change any single parameter unless the effect it will have on the various interactions present in the system has been determined.

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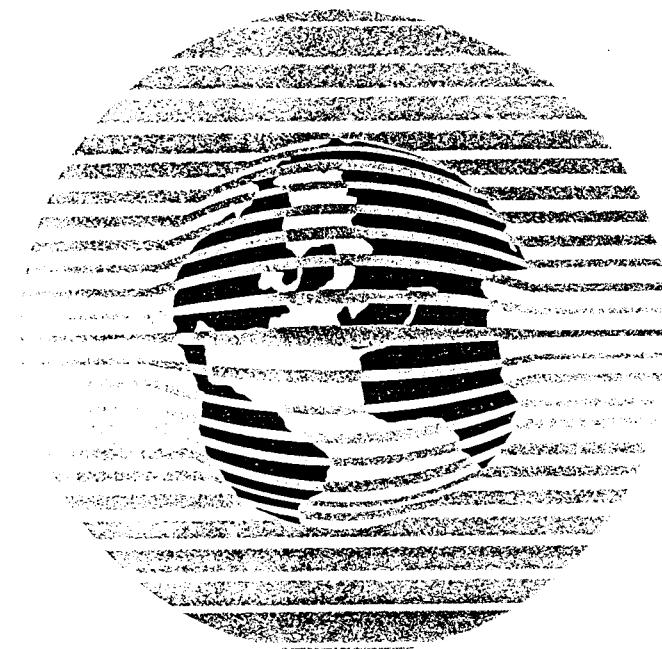
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