

Emulsification of Alkyds for Industrial Coatings

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INTRODUCTION

During recent years, the interest in using waterborne alkyd paints has increased.^{1,2} Emulsified alkyds have been used primarily in the decorative and protective field. In most cases they are used as co-binders, especially in stains and primers for exterior use. Although alkyd emulsions cannot match the performance of a solvent-borne alkyd so far, they are an attractive alternative since they contain no volatile organic compounds. For health and environmental reasons, alkyd emulsions for industrial coatings have gained increasing attention. Alkyds for industrial coatings are often dissolved in toxic aromatic organic solvents such as xylene. The volume of organic solvents used in industrial paints is also considerably larger than the volume used in consumer paints.

In alkyd emulsions, in comparison to latex dispersions, the resin is post emulsified as fine droplets in water. A crucial factor for acquiring good colloidal stability is that small droplets are obtained in the emulsification process.³ The choice of surfactants here is critical. The surfactant should simplify the formation of the droplets during emulsification and afterwards stabilize them against flocculation and coalescence.^{4,5} The right surfactant should fulfill these requirements at low concentrations since high amounts of surfactants often give soft and water-sensitive paint films. In a previous study, the influence of surfactants on emulsification and shear stability of alkyd emulsions for consumer paints has been investigated.⁶⁻⁸

Alkyds for industrial coatings have a much higher viscosity than alkyds used in consumer paints, which demand a special emulsification technique to create finely dispersed emulsions. In the present work, the possibilities of emulsifying high viscosity alkyds by the inversion technique have been investigated. The influence of type and concentration of surfactant on the inversion properties and droplet size has been studied. An increased knowledge of the inversion technique, although it is applied on alkyds in this study, is also of interest for emulsification of other resins with high viscosity, for example, polyesters, wax, epoxy, and tackifiers.

EMULSIFICATION TECHNIQUES

In principle, emulsification can be made by two different techniques, direct emulsification or emulsification through inversion.

To date, alkyd emulsions have mostly been used in consumer paints. These are relatively easy to emulsify due to their low viscosity. Lately, alkyd emulsions for industrial paints have gained increasing attention. Alkyds for industrial paints have a much higher viscosity than alkyds for consumer paints which require other emulsification techniques. In this work an alkyd with 40% oil length has been emulsified by the inversion technique. Inversion emulsification accomplished by adding water to an alkyd/surfactant mixture at constant temperature affords emulsions with droplet sizes below 1 μm at a concentration of three percent on the alkyd phase. Small droplets are necessary for colloidal stability of the emulsion. The solubility of surfactant in the alkyd and the water phase determines at which water concentration the emulsion inverts. High molecular weight ethoxylated anionic surfactants are effective as emulsifiers. Using these surfactants, the emulsification becomes less dependent on emulsification temperature than when nonionic surfactants are used.

In direct emulsification the resin is dispersed directly into water by using high shear agitation or high pressure homogenization. The resulting droplet size depends to a large extent on the intensity of the agitation and on the self-emulsifying contribution from the surfactant. However, with increasing mixing, a more effective process is obtained, which is less dependent of the type of surfactant. To emulsify resins that are solid at room temperature by the direct method, the temperature must be increased to lower the viscosity. An alternative is to dilute the resin in a solvent. The solvent can be removed after emulsification to obtain a solvent-free emulsion.⁹

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Table 1—Surfactants Used for Emulsification

Surfactant	Symbol	HLB	Commercial Name	Supplier
Anionic				
Fatty alcohol ether sulphate, sodium salt	C ₁₂ EO5-S-Na	—	FES 32	Henkel KGaA Plastics & Coatings Technology
Fatty alcohol ether sulphate, sodium salt	C ₁₂ EO10-S-Na	—	FES 993	Henkel KGaA Plastics & Coatings Technology
Fatty alcohol ether sulphate, sodium salt	C ₁₂ EO30-S-Na	—	FES 77	Henkel KGaA Plastics & Coatings Technology
Fatty alcohol ether sulphate, sodium salt	C ₁₂ EO50-S-Na	—	FES 61	Henkel KGaA Plastics & Coatings Technology
Nonionic				
Fatty alcohol ethoxylate	C ₁₆ /C ₁₈ EO80	18.5	Berol 08	Akzo Nobel Surface Chemistry AB

In inversion emulsification, the water phase is first emulsified into the resin forming a water-in-oil (W/O) emulsion. By changing the temperature or the water concentration, the W/O emulsion can invert into an oil-in-water (O/W) emulsion. The surfactant, and also to some degree the concentration of resin in water, determines which emulsion type is formed¹⁰⁻¹³ at a certain temperature. A hydrophobic surfactant, with a low hydrophilic lipophilic balance (HLB), is most soluble in the resin phase and gives a W/O emulsion according to Bancroft's rule.¹⁴ A hydrophilic surfactant (with a high HLB) is most soluble in the water phase and favors the formation of an O/W emulsion.

In principle there are two different techniques to induce inversion:

(1) In the phase inversion temperature (PIT) method (or thermal inversion), the oil, water, and a nonionic surfactant are mixed at a high temperature. The high temperature increases the hydrophobicity of a nonionic surfactant which results in a W/O emulsion. By decreasing the temperature, the surfactant becomes more hydrophilic and diffuses from the oil across the phase boundary over to the water phase. The diffusion causes an inversion to an O/W emulsion. The temperature at which the emulsion inverts is called the phase inversion temperature. This concept is well described by

Shinoda and co-workers^{10-12,15-17} and some others^{18,19} for oils of low viscosity. PIT is specific for every system and depends on type and concentration of surfactant, type of oil and additives, for example, salt. By proceeding through a microemulsion region (with low interfacial tension) at inversions, very finely dispersed emulsions can be obtained.²⁰⁻²²

(2) In the other technique, the emulsifier is first mixed with the resin. Water is then added to the mixture at constant temperature to obtain a W/O emulsion. At a certain ratio of water to resin, the emulsion inverts. This concentration is called the emulsion inversion point, EIP.²³⁻²⁶ This technique is often used in industrial applications.

The inversion technique is especially advantageous for oils of high viscosity since emulsification can be made in a simple laminar shear field. The emulsification is strongly enhanced by a high viscosity of the continuous phase, which is the resin before inversion. The droplet size, d , in a laminar shear depends on the viscosity ratio of the phases as:³

$$d \propto \eta_{\text{continuous phase}}^{-0.6} \text{ if } \frac{\eta_{\text{dispersed phase}}}{\eta_{\text{continuous phase}}} < 4 \quad (1)$$

In inversion emulsification, however, the choice of surfactant is more critical than in direct emulsification.

MATERIALS

The alkyd used is a chain-stopped alkyd, based on linoleic rich fatty acids. The oil length is 40%, the acid value is 10 mg KOH/g alkyd, and the hydroxyl number is 85 mg KOH/g alkyd. The viscosity is 18,300 mPas at 100°C. The surfactants used are listed in Table 1.

The fatty alcohol ether sulphates are supplied as solutions in water. The solutions were freeze dried and the surfactants were used as solids. The nonionic surfactant is solid.

The alkyd was neutralized with dimethylethanolamine or potassium hydroxide (in water solution) before emulsification.

METHODS

Inversion Experiments

Inversion experiments were performed in a laboratory reactor (Parr Instrument Company, USA). A schematic of the reactor is shown in Figure 1. The reactor allows experiments above atmospheric pressure. The reactor volume is 300 ml and it is equipped with an anchor stirrer, a thermocouple, and inlet

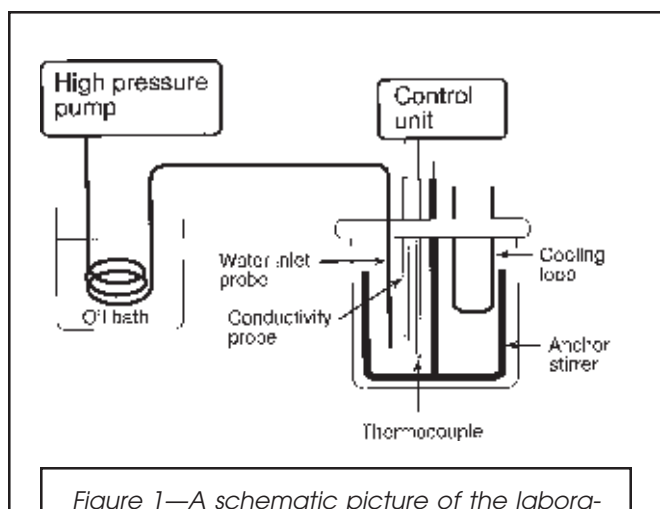


Figure 1—A schematic picture of the laboratory reactor for the inversion experiments. The reactor can operate above atmospheric pressure, over 100°C. The reactor is equipped with a thermocouple, an anchor stirrer, a water inlet probe, and a conductivity probe. The reactor is heated by an external heater. The water is pumped into the reactor by a high pressure pump.

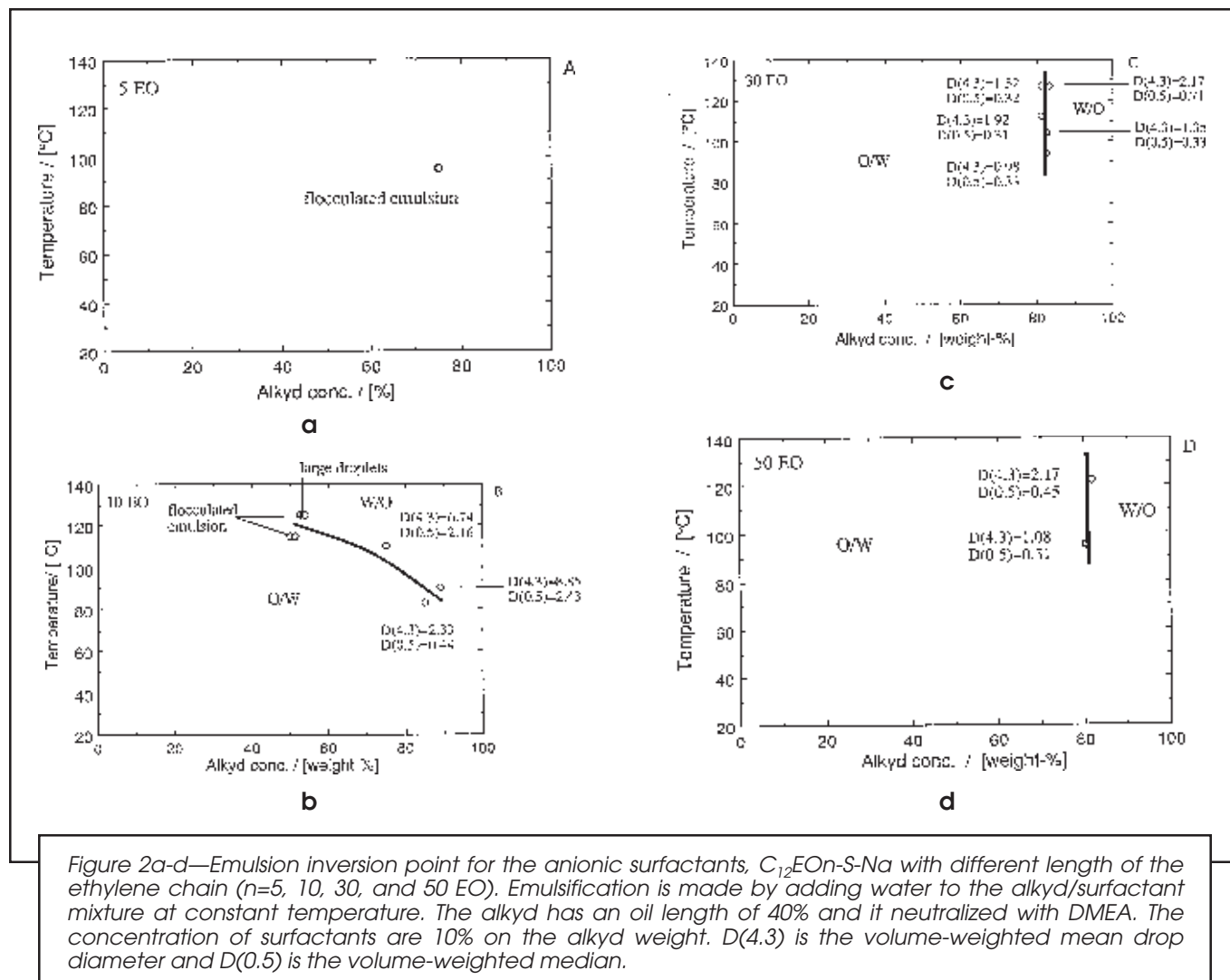


Figure 2a-d—Emulsion inversion point for the anionic surfactants, $C_{12}EO_n-S-Na$ with different length of the ethylene chain ($n=5, 10, 30,$ and 50 EO). Emulsification is made by adding water to the alkyd/surfactant mixture at constant temperature. The alkyd has an oil length of 40% and it neutralized with DMEA. The concentration of surfactants are 10% on the alkyd weight. $D(4.3)$ is the volume-weighted mean drop diameter and $D(0.5)$ is the volume-weighted median.

probe for adding the water, a cooling coil for a proper control of temperature, and a conductivity probe for detecting inversion. The water phase contains 1 mM sodium chloride to obtain conductivity. A distinct increase in conductivity is observed when the emulsion inverts from a W/O to an O/W emulsion. The reactor is heated by an external electrical heater connected to a control unit. Water is pumped into the reactor by a high pressure pump which allows pumping above 100°C. The water is preheated to the emulsification temperature by dipping the water tube into an oil bath before it enters the reactor.

Emulsification is made by the EIP technique. The alkyd and emulsifier is mixed and neutralized in the reactor. When the emulsification temperature is reached, the preheated water is pumped into the reactor at constant temperature.

After inversion the emulsions are diluted to 50% during cooling to room temperature.

Droplet Size

Droplet sizes were measured with a laser diffraction instrument, Mastersizer (Malvern Instruments, England). The instrument uses an approximation of the Mie scattering theory, which utilizes the refractive index of the dispersed phase and

its absorption. The relative refractive index $n_{\text{alkyd}}/n_{\text{water}} = 1.15$ for the alkyd used. The absorption value was estimated to be 0.1. The results are recorded as a volume distribution. The initial droplet size is expressed as $D(4.3)$ which is the volume-weighted mean diameter or as $D(0.5)$ which is the volume-weighted median. Surfactant layer thicknesses (see the following) are estimated from the $D(3.2)$ value which is the area weighted mean drop diameter. The emulsions were also examined by microscopy.

Calculations of an Apparent Surfactant Layer Thickness

If it is assumed that all surfactant is located at the surface of the alkyd droplets (which is an overestimation), an apparent surfactant layer thickness, Δ , can be calculated⁶ from the known volume fraction of surfactant with respect to the oil phase, $\phi_{\text{surfactant}}$, and from the area weighted drop radius, r . For small values of $\phi_{\text{surfactant}}$ the layer thickness becomes:

$$\Delta = \frac{\phi_{\text{surfactant}} \cdot r}{3} \quad (2)$$

If the calculated thickness is compared to the length of the extended surfactant molecule, one can estimate the surface

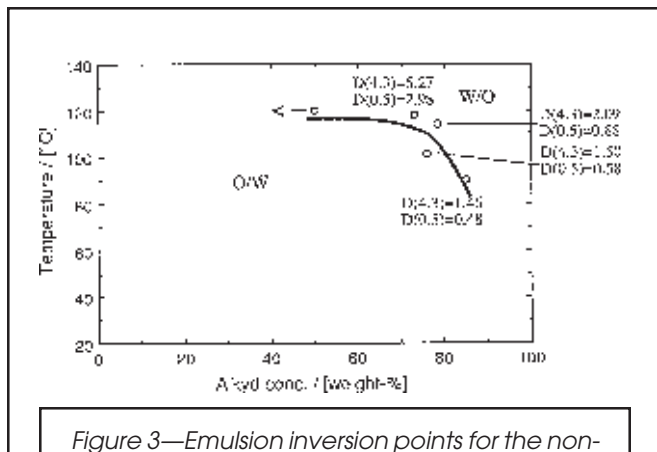


Figure 3—Emulsion inversion points for the non-ionic surfactant, C₁₆/C₁₈EO80. Emulsification is made by adding water to the alkyd/surfactant mixture at constant temperature. The arrow indicates that no inversion is obtained down to the measured concentration. The alkyd has an oil length of 40% and is neutralized with sodium hydroxide. The concentration of surfactants are 10% on the alkyd weight. D(4.3) is the volume-weighted mean drop diameter and D(0.5) is the volume-weighted median.

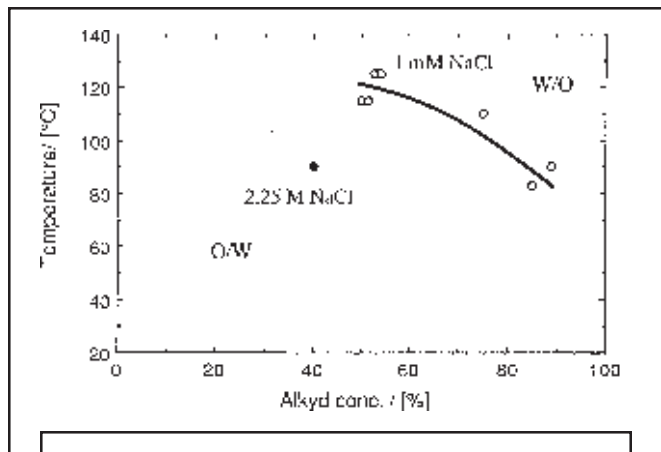


Figure 4—The change of emulsion inversion point for emulsions of alkyd with 40% oil length and C₁₂EO10-S-Na when salt (2.25 M NaCl) is added to the water phase. The emulsifier becomes more hydrophobic when the salt is added which favors the w/o emulsion and inversion at higher water concentrations.

coverage. In the calculation the densities were estimated to 1.0 for both the alkyd and the surfactants.

Cloud Point

Cloud point was measured by dissolving one percent surfactant in sodium chloride. The solutions were introduced in glass ampoules that were sealed. The ampoules were placed in an oil bath at different temperatures and cloud points determined visually.

RESULTS AND DISCUSSION

Influence of Surfactant on the Emulsion Inversion Point

The results from the inversion emulsification using the anionic surfactants are presented in Figures 2a-d. The results are presented in phase inversion diagrams. Using the EIP technique, we are going from the right side in the diagram at 100% alkyd, at constant temperature, to the left when water is added. The lines (only one point in Figure 2a) indicate when inversion is obtained. Above the inversion line, the emul-

sions are W/O and below O/W. Droplet sizes of the emulsions after inversion are included in the diagrams.

With the anionic surfactant with 5 EO at 95°C (Figure 2a), inversion was obtained but the emulsion was crude and flocculated. The anionic surfactant with 10 EO (Figure 2b), gave better emulsions. Especially at lower temperatures relatively small droplets were obtained. At temperatures above 110°C, the emulsions were crude and flocculated immediately after inversion. It can be seen that emulsion inversion point (EIP) changes with temperature using this surfactant. The higher the emulsification temperature, the more water was required before the emulsion inverts. For the more hydrophilic surfactant with longer EO chains, 30 and 50 EO, the EIP is independent of the temperature. Also, for these emulsions the droplet size of the emulsions is independent of the emulsification temperature.

The emulsification results when a pure nonionic surfactant is used (see Figure 3). Also with this surfactant, the emulsion inversion point depends on the emulsification temperature and the droplets are smaller at lower temperatures.

It has been found that the phase inversion lines are different for different oils using the same type of surfactants. For a non-polar paraffin oil, the phase inversion temperature is completely independent of the oil-in-water concentration (i.e., the inversion line is horizontal) when ethoxylated nonylphenol was used. With a more polar oil, xylene, the

Table 2—Critical Surfactant Concentration and Layer Thicknesses

Emulsifier	Critical ^a Surfactant Concentration (weight-% on alkyd)	Droplet Size D(3,2) ^b (µm)	Critical Surfactant Layer Thickness ^c (Å)	Calculated Length of the Emulsifier ^d Molecule (Å)	Ratio ^e
C ₁₂ EO10-S-Na	3	0.90	44	56	0.78
C ₁₂ EO30-S-Na	3	0.41	20	128	0.16
C ₁₆ /C ₁₈ EO80	3	0.48	56	315	0.18

(a) Lowest concentration when the droplet size D(0.5) are below 1µm.
 (b) Area weighted mean diameter.
 (c) Critical surfactant layer thickness is taken from Figure 7.
 (d) Calculated from the bond lengths for an extended molecule, C-C 1,54 Å, C-O 1,44 Å, O-H 0,97 Å. All bond angles.
 (e) Critical surfactant layer thickness divided by calculated molecular length.

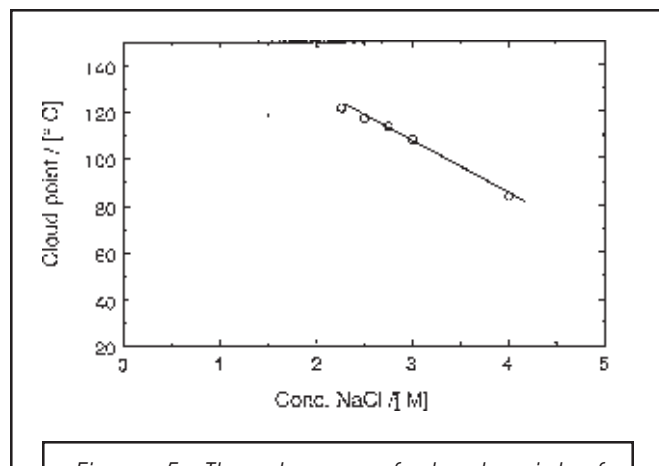


Figure 5—The change of cloud point of $C_{12}EO_{10}\text{-S-Na}$ with salt concentration. Below 2.25 M NaCl there is no cloud point under 150°C.

phase inversion temperature varies with temperature.¹⁷ The solubility of the surfactant changes more gradually in a polar oil compared to a non-polar oil. This indicates that it is the temperature dependent solubility of the surfactant in the oil and water phase respectively that determines the emulsion inversion point, i.e., the slope of the inversion line.

The different temperature dependency seen in our experiments can also be explained by the solubility of the emulsifier in the alkyd and the water phase. The solubility properties of nonionic surfactant display a different temperature dependence compared to anionic surfactants. Ethoxylated nonionic surfactants become more hydrophobic with increasing temperature due to the water structure around the ethylene oxide chain. Anionic surfactants do not display such a temperature dependency. The anionic surfactants used in this study, however, contain ethylene oxide chains of different length which gives them a somewhat similar character to a nonionic surfactant.

When the emulsification temperature is increased with the anionic surfactant, the 10 EO (in Figure 2b), and with the nonionic surfactant (in Figure 3), they become more hydrophobic. This makes them good stabilizers for the W/O emulsions so more water has to be added before inversion occurs. The amount of water needed to obtain inversion is likely determined by the amount of surfactant necessary to stabilize the water droplets. Because of the hydrophobic character of these emulsifiers at higher temperatures, they are poor stabilizers for the O/W emulsions, and crude emulsions are obtained after inversion. At lower temperatures the emulsifiers are more hydrophilic and dissolve in the water phase, which favors an O/W emulsion. This results in an earlier inversion and smaller droplets. An experiment supporting this theory is that the emulsion inversion point is changed from 85–89% alkyd in water to about 40% when the salt concentration was increased in the water phase (see Figure 4). Addition of salt decreases the cloud point and the water solubility of the ethoxylated surfactant, according to Figure 5.

The emulsifiers with 30 and 50 EO, however, seem to retain their hydrophilicity within this temperature interval resulting in an early inversion and finely dispersed emulsions at all temperatures.

Similar changes of inversion lines for an oil of low viscosity has also been reported when an ethoxylated nonionic surfactant was gradually replaced by a pure anionic surfactant.²⁷ The viscosity has also been reported to influence the inversion line²⁸ which indicates that diffusion of the surfactant is an important factor.

Influence of Surfactant Concentration on the Droplet Size

In the previous section, we could see that hydrophilic anionic surfactants with long EO chains gave emulsions with the smallest droplets. The influence of concentrations of surfactants on the droplet sizes is shown in Figure 6. The results for both the anionic surfactants with 10 and 30 EO and for the nonionic surfactant are included. Emulsification was made at 90°C. The sample at one percent concentration of nonionic surfactant did not invert after addition of 50% water but inverted during cooling. The emulsion flocculated directly after inversion. At 0.5% concentration of the anionic surfactant with 30 EO, the emulsion also flocculated directly after inversion.

From the results with the weight concentration in Figure 6, it seems as if the nonionic surfactant should be less effective than the anionic ones. The nonionic surfactant, however, has a much higher molecular weight than the anionic ones so if molar concentrations are considered instead, the nonionic surfactant is as effective as the anionics.

For a further understanding of the difference between the effectiveness of the surfactants, a more detailed discussion of the surfactant layer around the droplets is needed.

If it is assumed that all of the surfactant is located at the surface of the droplets (which is an overestimation since it is also soluble in the alkyd and in the water phase), an apparent thickness of the surfactant layer can be estimated using equation (2). If this thickness is compared to the length of the surfactant molecule, an estimation of the packing density of the surfactant on the droplet surface is obtained. The calcula-

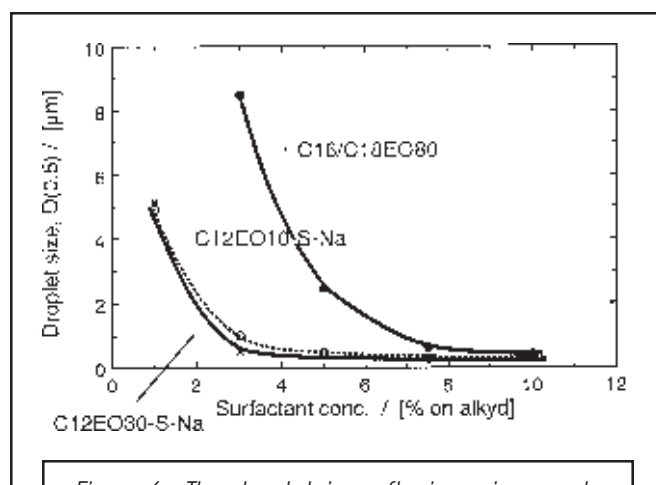


Figure 6—The droplet sizes after inversion emulsification at different surfactant concentrations. $D(0.5)$ is the volume-weighted median of the drop diameter. The data from both the anionic surfactants and for the nonionic surfactants are included in the figure. The emulsions are neutralized with sodium hydroxide.

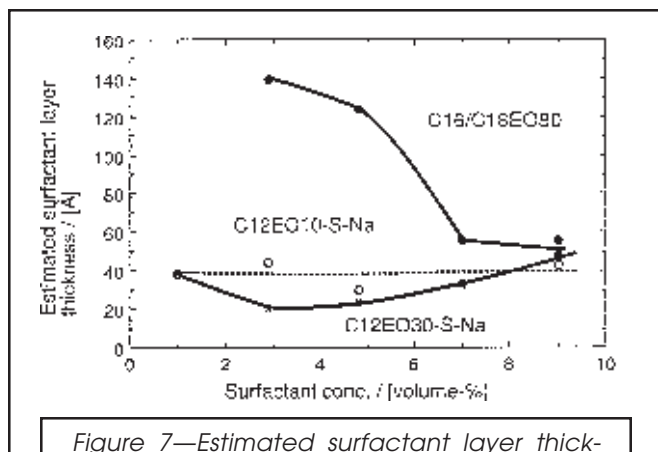


Figure 7—Estimated surfactant layer thicknesses on the emulsion droplet surfaces at different surfactant concentrations.

tions of the surfactant layer thicknesses are shown in Figure 7. From Figure 6 it can be seen that the critical concentrations for both the anionic surfactants are about three percent. Below this concentration the droplet size starts to increase over $1\mu\text{m}$. Since the surfactant layer thicknesses remain constant below three percent (see Figure 7), it can be concluded that it is the supply of surfactants that determine the droplet sizes obtained during emulsification. For the nonionic surfactant, the layer thickness increases below the critical concentration, 7.5%, indicating that these surfactants act differently than the anionics. Below the critical concentration effects, others than the supply of surfactants seem to determine the droplet size.

If the surfactant layer thicknesses are compared to the length of the molecules (see Table 2), it can be seen that both the nonionic surfactant and the more hydrophilic anionic surfactant with 30 EOs give stable emulsions at very thin surfactant layers around the droplets. The ratio between the critical layer thicknesses and the calculated molecular length are 0.16 and 0.18, indicating a very low packing density of surfactants at the droplet surfaces. The low packing density proposes a non-extended configuration of the surfactant molecule on the droplet surfaces. For the anionic surfactant with 10 EOs the ratio is higher, 0.78, showing a higher packing density.

If these results are compared to emulsification of alkyds by high pressure homogenization,⁶ it seems as if inversion emulsification is a more effective emulsification method, at least when the high molecular weight surfactants are used. In high pressure homogenization, the ratio between the critical layer thickness and the calculated molecular length were 0.37–0.63 for some nonionic surfactants showing a somewhat higher packing density. With a pure anionic surfactant, however, extremely low packing density were obtained in high pressure homogenization.

CONCLUSION

Alkyds of high viscosity can be emulsified by the inversion technique using simple hydrophilic nonionic and anionic surfactants. Fine dispersed, stable emulsions are obtained by using the EIP technique, i.e., by adding water to the alkyd/emulsifier mixture at constant temperature. There is no need

to make the inversion by going through a microemulsion region as proposed in the PIT technique to obtain small droplets.

The emulsion inversion point and the droplet size seem to depend on the solubility of the surfactant in alkyd and the water phases.

By using hydrophilic anionic surfactants with long ethylene oxide chains, the droplet sizes become less dependent on the emulsification temperature than when nonionic and more hydrophobic anionic surfactants are used.

ACKNOWLEDGMENT

The authors thank the Swedish Work Environmental Fund and the following industries: Jotun A/S, Akzo Nobel Surface Chemistry AB, Tikkurila Oy, DSM Resins bv, Akzo Nobel Industrial Coatings AB, Worlée Chemie GmbH, Bergvik Kemi AB, and Seppic for making this work possible by their financial support. Tommy Sebring is thanked for skillful and devoted help with the experimental work.

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