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Studies in Solvent Extraction Using Polyaphrons. I. Size Distribution, Stability, and Flotation of Polyaphrons

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ABSTRACT

Predispersed solvent extraction (PDSE) is a promising technique for the treatment of wastewater containing low solubility, hydrophobic contaminants. A stable predispersed organic solvent in the form of polyaphrons of very small diameter results in high surface areas with a minimum energy requirement for mass transfer of solutes from the aqueous phase to the organic solvent. PDSE should greatly improve the performance of a conventional extraction process. This paper focuses on the characterization and size distributions of polyaphrons. Polyaphrons were generated using different cationic, anionic, and nonionic surfactants in water and an oil-soluble nonionic surfactant. Kerosene was used as the organic solvent to form the polyaphrons. Size distributions were obtained using a particle size analyzer. The optimal instrument parameters (sample quantity, optical parameters,

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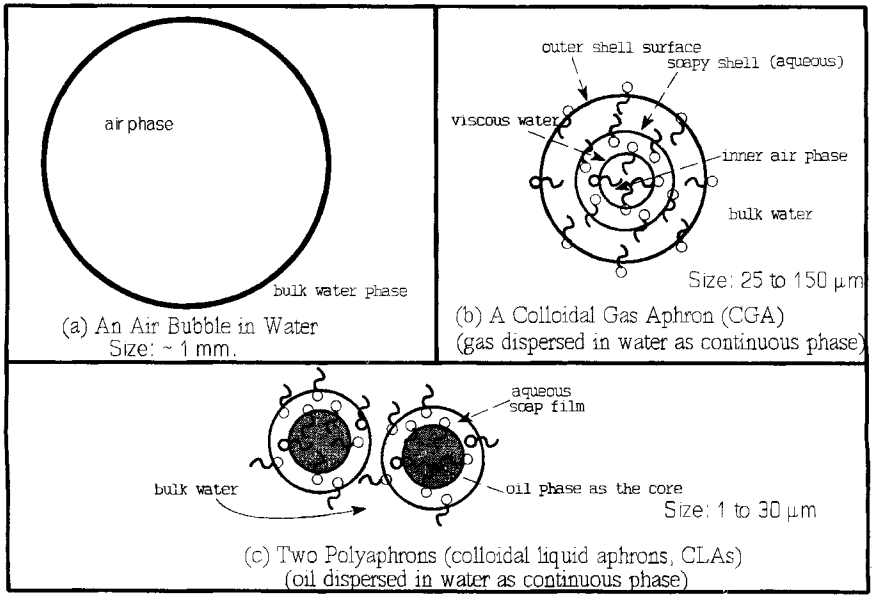
run time, etc.) were identified for these measurements. The size distribution based on volume fraction was found to show a bimodal behavior, with peak size maxima between 1–3 and 10–30 μm for all the polyaphrons. The effects of different surfactant types, surfactant concentrations, and storage times on the size distribution spectrum of polyaphrons were studied. The size distribution of different polyaphrons before and after flotation in an aqueous column using colloidal gas aphrons (CGAs) was also studied. Flotation was deduced to occur as a result of electrostatic forces between the CGAs and polyaphrons.

INTRODUCTION

The use of *polyaphrons* or *colloidal liquid aphrons* (CLAs) for solvent extraction was first proposed by Sebba (1). The separation process was termed *predispersed solvent extraction* (PDSE). The process involves precomminuting organic solvent into *polyaphrons*, which are used to scavenge solutes from the aqueous phase. The dispersed solvent is then separated by flotation using micro gas dispersions called *colloidal gas aphrons* (CGAs). The most striking feature of this technique as compared to a conventional solvent extraction is that the organic solvent can be predispersed in water in the form of polyaphrons. Therefore the need for a conventional mixer-settler stage is avoided. A large energy savings is accomplished by comminuting only one phase (the organic solvent) instead of both water and organic solvent. Moreover, the small sizes of the predispersed solvent affords high surface areas per unit solvent volume for solute transfer from the aqueous to the organic solvent phase, and high efficiencies can be realized using a very small volume of the organic solvent. The potential applications of PDSE for dilute solutions separations in wastewater treatment are obvious. In industrial wastewater treatment the high ratio of organic solvent volume to water volume required for satisfactory removals is a disadvantage for conventional solvent extraction. PDSE eliminates this drawback and should be favored for many compounds that are less volatile than water for which steam stripping or activated carbon are the current methods. When nonbiodegradable and/or toxic organic contaminants are concerned, PDSE may be a viable alternative.

Some preliminary data have shown that PDSE can remove very fine oil droplets from wastewater (2) and some metals (3–5). Michelsen and coworkers tested the removal of an organic compound (*o*-dichlorobenzene) from water using decane as the solvent in a batch mode using PDSE (6) and showed that it was about 5 to 10 times more effective than straight solvent extraction using a similar quantity of the solvent. PDSE markedly lowered the solvent to feed ratio (1:500) and increased the rates of extraction.

The structure of an individual polyaphron was proposed by Sebba (2), a schematic of which is provided in Fig. 1(c). It is similar to a gas aphron (Fig. 1b), where the gas inside is replaced with an organic solvent and the outer surface is made up of a thin soapy shell. Both are quite different from a conventional air bubble (Fig. 1a). Polyaphrons, when dispersed in water, exhibits colloidal properties. The properties of polyaphrons have been reviewed by Sebba (2). The size of polyaphrons is a key property as far as their use in PDSE. In theory, a decrease in size will improve the extraction, but in practice, difficulties in separating the residual solvent will arise, and one will have to resort to subsequent flotation using air bubbles. The reported sizes of polyaphrons vary widely with ranges of submicrons to 100 microns or more. According to Sebba (2), a size of 10 μm was observed if the organic solvent contained about 1% of a nonionic polyoxyethylene surfactant that has three ethoxy groups per molecule, while the size was much smaller if the number of ethoxy groups was nine. With lauric acid as the surfactant, the aphron reached a diameter of 80



μm. Michelsen et al. (6) reported sizes of polyaphrons ranging from 10 to 20 μm in diameter.

It is important to note that all of the size measurements reported so far are based on microscopic examinations of polyaphron samples in a static mode placed inside a hollow slide. Obviously this technique is not always appropriate for size determination of polyaphrons in a dynamic environment such as existing in an extraction column where nonuniform size distribution patterns will exist in the aqueous phase. The shortcomings of the microscopic method in such a case can be overcome by using a particle size analyzer. This utilizes the phenomenon of low angle forward scattered light from a laser beam projected through a stream of particles. The amount and direction of light scattered by the particles is measured by an optical array detector and analyzed by a microcomputer, which calculates the size distribution of particles in a sample stream that is in a dynamic environment. The particle size analyzer has been used successfully previously in our laboratory to measure the size and stability of microdispersed gas bubbles (CGAs) (7). Though similar in structure, the polyaphrons exhibit different static and dynamic characteristics than CGAs.

This is the first paper in a series on the use of polyaphrons in a continuous countercurrent PDSE for the removal of several trace hydrophobic organics from wastewater. This paper reports the size distribution patterns of polyaphrons obtained from different water-soluble surfactant types (anionic, cationic, and nonionic), surfactant concentrations, and storage times. The subsequent steps after introduction of polyaphrons in the aqueous phase involve flotation using CGAs. Hence we also report here our study of the effects of flotation on the size distribution of polyaphrons as a basis for future designs and applications.

EXPERIMENTAL

Solvents and Surfactants

Kerosene (Curtin Matheson Scientific) was chosen as the organic solvent since it is inexpensive and has been previously investigated as a possible solvent for extraction of several organic compounds as part of an EPA project (8). In order to make polyaphrons, two types of surfactants, one oil-soluble (Tergitol 15-S-3 supplied by Sigma Chemical Co.) and four water-soluble surfactants were used. The properties of the four water-soluble surfactants [sodium dodecylbenzene sulfonate (SDBS), hexadecyltrimethyl ammonium bromide (HTAB), polyoxyethylene 23 lauryl ether (Brij 35) and polyoxyethylene sorbitan monooleate (Tween 80)] supplied by Sigma Chemical Co. are given in Table 1.

TABLE 1
Properties of Surfactants Used for Polyaphron Generation

Surfactant	Acronym	Molecular weight	CMC (mM)	Charge
Water-soluble:				
Sodium dodecylbenzene-sulfonate	SDBS	348.5	1.5	Anionic
Hexadecyltrimethyl-ammonium bromide	HTAB	364.5	0.9	Cationic
Polyoxyethylene 23 lauryl ether	Brij 35	~1200	0.06	Nonionic
Polyoxyethylene sorbitan monooleate	Tween 80	~1300	0.01	Nonionic
Oil-soluble:				
Polyoxyethylene–secondary alcohol	Tergitol 15-S-3	336	N/A	Nonionic

Preparation of Polyaphrons

The procedure employed was a modification of the one proposed by Sebba (9). First, a magnetic stirrer was used to attain a constant rate of mixing of an aqueous phase containing 4 g/L of the water-soluble surfactant to produce a stable foam. To 5 mL of this foaming solution, 95 mL of kerosene containing 0.6% of Tergitol 15-S-3 was added slowly to obtain 100 mL of polyaphrons. The relative volumes of the organic solvent and water used were chosen so that the phase volume ratio, PVR (volume of the dispersed phase to the total volume), was 19, which was reported to be ideal for the production of a stable polyaphron sample (2). The rate of kerosene addition was slow to begin with, and progressively increased. Slow addition is very important since the environment of the aphrons that have already formed should remain aqueous. Otherwise, the aphrons will congeal and no longer offer an interface for spreading, lowering the quality of the polyaphrons. The polyaphrons thus made were remarkably stable if stored properly. The gel-like polyaphrons made using SDBS (in water)/ Tergitol (in kerosene) remain unchanged, and no visible amount of kerosene was released even after 16 months of storage. A sample of the polyaphron was placed on a slide for a microscopic examination using Olympus System Microscope Model BHS with Olympus Photomicrographic System Model PM-10AD. Figure 2 is an example of the structure seen under these circumstances at a low PVR. The shapes were uniformly spherical, although some displayed a polyhedral shape. The core of one aphron filled

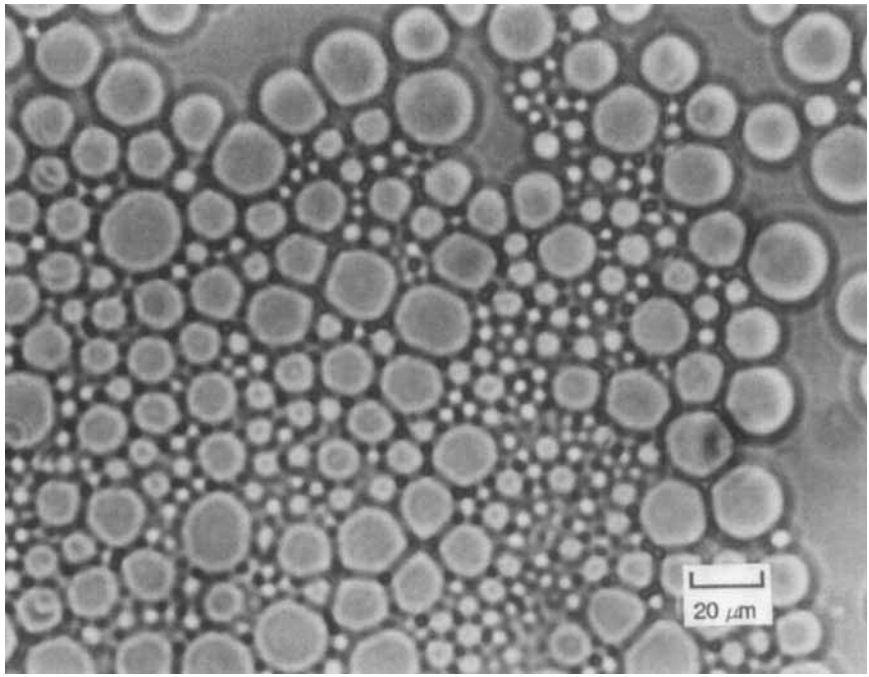


FIG. 2 The structure of an assemblage of polyaphrons generated from SDBS in water and Tergitol 15-S-3 in kerosene.

with oil remains separated from another one by a thin film of water as distinct oil droplets dispersed in a very small amount of continuous water phase. At high PVR we observed that the polyaphrons were nonspherical in shape with some distinct small droplets around each aphron. This was concluded to be due to free kerosene that was not encapsulated by the soapy film. It was observed that upon dilution with water the individual polyaphrons first separated and dispersed in the continuous phase (water), but reassembled to form a tight structure. This behavior is akin to crystallization via nucleation and has been discussed by Sebba (2).

Measurements of Size Distribution

The size measurements were conducted using a Microtrac Model 9210 Particle Size Analyzer (Leeds and Northrup Inc.) with a standard range of 0.7 to 704 μm. Polyaphrons were diluted with distilled deionized water

and added directly into a mixing chamber called a small volume recirculator (SVR). The SVR mixes the sample with recirculating water (approximately 275 mL) so that a stream of well-dispersed particles is pumped continuously through a transparent sample cell for analysis. Each sample was subject to measurements at intervals of 2 minutes, providing size distribution versus time data. For each measurement the instrument will give a summary of statistical data. Other intrumental parameters were optimized and are described below.

The Microtrac software required four additional parameters to be defined (transparent, spherical, particle refractive index, and carrier refractive index value) before any sample run was initiated. Different settings of these parameters will influence the size distribution data since the mathematical treatment is based on these parameters in order to make optical compensation. Among these parameters the particle refractive index was set at 1.5 for all measurements. The carrier refractive index was set at 1.33 since water was used. For the selection of the other two parameters, a series of measurements were conducted on different polyaphron samples. We ascertained that the setting NO was appropriate for the "transparent" category, whereas at a setting of YES for this category a loss of distribution tail at the fine end was observed. The appropriate selection for the "spherical" category was YES.

Flotation of Polyaphrons

Flotation was carried out in a batch mode using a 1-m tall column with internal diameter of 0.08 m. The total aqueous phase volume used was 2.5 L. A 5-minute pulse of diluted polyaphrons (made from HTAB, SDBS, or Tween 80 as the water-soluble surfactant) at a flow rate of 10 mL·min^{-1} was introduced at the bottom of the column. A 5-minute pulse of CGAs produced from SDBS was also introduced at the bottom of the column at a flow rate of 40 mL/min. Samples (50 mL) of the aqueous phase were collected from the bottom of the column at different times after flotation and analyzed for the size distribution of polyaprhons using the particle size analyzer. The experiments were repeated four times under identical conditions, and the average results are presented.

RESULTS AND DISCUSSION

One of the parameters of importance that the particle size analyzer responds to is a dimensionless parameter called DV. This value is proportional to the amount of sample used. The relationship is linear up to a

certain value of DV, and care should be taken to make sure that the size determinations are made in this range. The original polyaphron sample was diluted 1:25 with distilled water, and 1 to 10 mL of the sample was used in the particle size analyzer. Precise measurements of sample quantity was not necessary within this range. If too little sample was used (DV ≤ 0.01) the system's noise was so high that no statistically significant data were obtained. The size distribution in the above range of sample volumes showed no effects on the sample volume itself.

In a previous study from our laboratory, DV was used as a parameter to determine the stability of CGA suspensions (7). We observed a significant decrease in DV with time at high surfactant concentrations. The CGA bubble volume was observed to decrease to one-third of the original volume in 6 minutes when HTAB was used as the surfactant, and 12 minutes when SDBS was the surfactant in the CGAs. The decrease in volume was considered to be due to the disappearance of extremely small bubbles due to their high Laplace pressure, and/or the disappearance of extremely large bubbles by buoyant forces. For polyaphrons dispersed in water, a change in DV with time was found to be insignificant for both ionic and nonionic surfactants. Figure 3 is a representative plot for polyaphrons generated from two water-soluble ionic (SDBS and HTAB) and two nonionic (Tween 80 and Brij 35) surfactants. This clearly indicates that polyaphrons dispersed in water are dynamically very stable. For polyaphrons produced from Brij 35 there was an initial sharp increase in DV which remained constant at 0.6 thereafter. The value of DV was largest for polyaphrons from Brij 35 and lowest for SDBS. The value was intermediate for HTAB and Tween 80. However, all of the polyaphrons studied had a stable DV for the duration of the measurements.

At the appropriate parameter settings discussed above, a bimodal distribution was observed for all the samples tested. One peak maximum was observed between 1 to 3 μm, while another was observed between 10 to 30 μm. Figure 4 shows the size distributions for the different types of polyaphrons generated in our laboratory. The measurements were made 55 days after preparation of the aphrons. Table 2 lists the average diameter and the standard deviation for the four types of polyaphrons. The maximum at the lower end of the spectrum appears to show no variation between the polyaphrons. However, for the other peak in the size distribution, larger values were observed for the ones made from Brij 35. The average size (Peak 2) of polyaphrons generated using the anionic surfactant SDBS was the smallest while that generated from the nonionic surfactant Brij 35 was the largest. Thus the nature of a water-soluble surfactant does effect the size of the polyaphrons that can be realized. The poly-

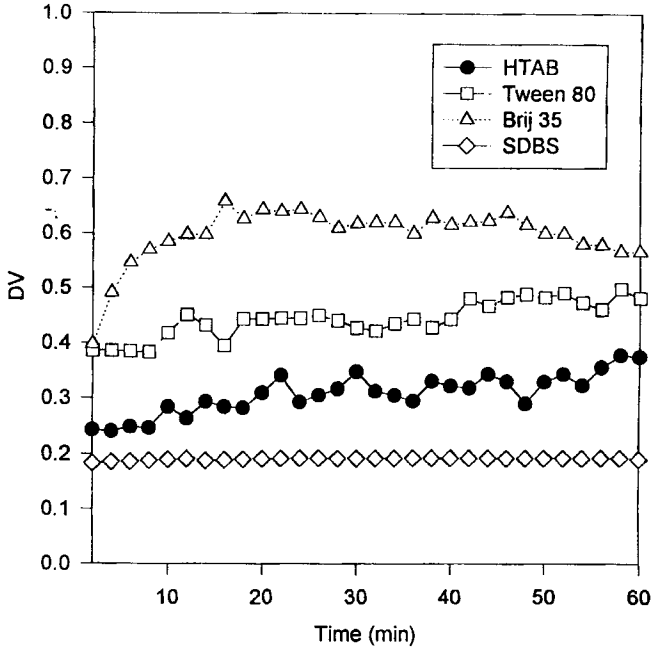


FIG. 3 Change in DV with time.

aphrons generated using the nonionic surfactant Tween 80 and the cationic surfactant HTAB gave sizes in between those of the other two. A large fraction of polyaphrons in every case appeared to be in the range between 10 and 30 μm. For both nonionic surfactants (Tween 80 and Brij 35) a higher volume fraction of aphrons was in the upper range of diameters. The average diameters observed for the polyaphrons are similar to those for conventional microemulsions of kerosene in water reported by others (10); the essential differences lie in the remarkable stability of polyaphrons, their negligible creaming rates, and the ease with which they can be produced. A conventional emulsion can be considered to be a two-liquid phase dispersion, whereas the aphrons are three-liquid phase dispersions (2).

Figure 5 shows the effects of storage time on the sizes of polyaphrons generated using SDBS. The size distributions were measured after 70 and 230 days of shelf storage of the polyaphrons. Although the smaller polyaphrons (1.5 to 4 μm) observed in the 70-day sample appeared to have

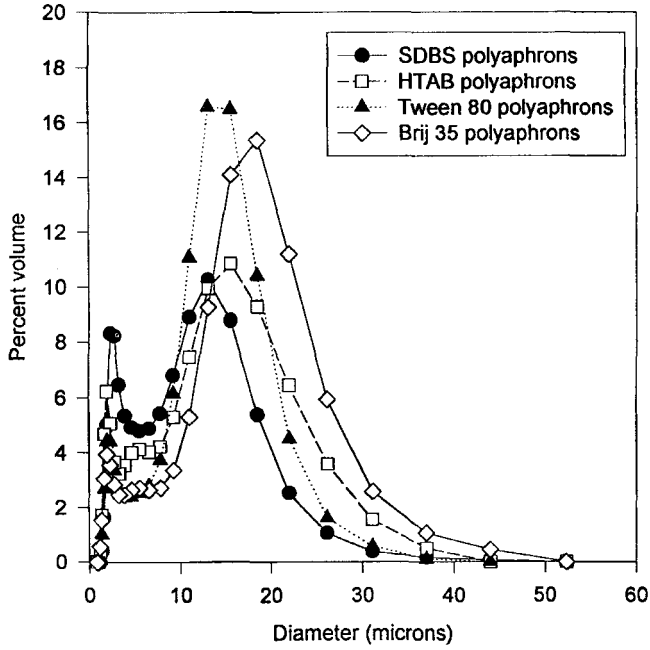


FIG. 4 The size distribution of different polyaphrons 55 days after preparation.

decreased in number, the majority of the aphrons are within the 10 to 30 μm range after 230 days storage. The maximum in peak size moved from 11 μm in the upper range to 18 μm. The remarkable stability of these polyaphrons is evident from this measurement. There appeared to be an oil floating at the top of the sample after prolonged storage in an open

TABLE 2
Average and Standard Error in Sizes for Different Polyaphrons[a]

Type of polyaphrons (water-soluble surfactant)	Peak 1, $d_{avg} \pm \sigma$	Peak 2, $d_{avg} \pm \sigma$
SDBS	2.9 ± 0.9	12.4 ± 5.3
HTAB	2.7 ± 1.0	14.6 ± 6.4
Brij 35	2.6 ± 1.0	17.0 ± 6.8
Tween 80	2.7 ± 1.0	14.0 ± 4.8

[a] Size in microns. The average and standard deviations calculated from percent volume fraction data. All size measurements were made 55 days after preparation of the polyaphrons.

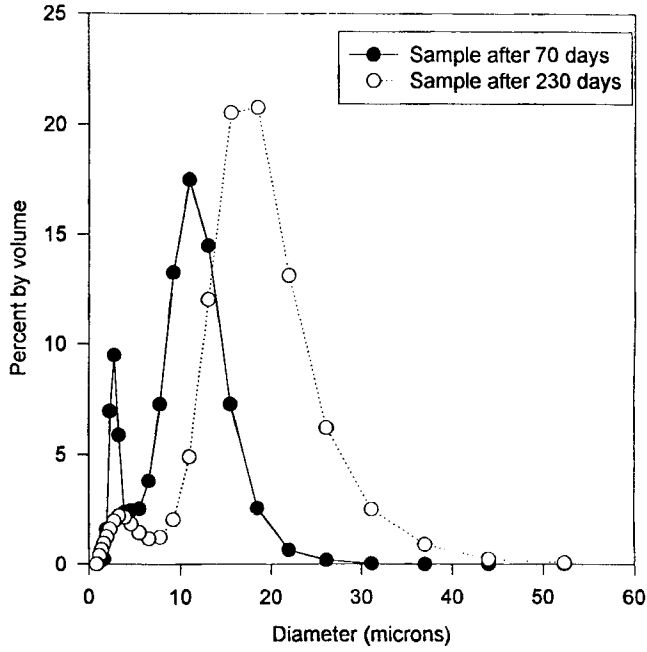


FIG. 5 The effect of storage time on polyaphrons generated using SDBS as the water-soluble surfactant and Tergitol 15-S-3 in kerosene.

bottle when HTAB was used as the water-soluble surfactant. This is due to slow creaming of the polyaphron, which, of course, occurs at a negligible rate when compared to ordinary emulsions. This was of no consequence since upon shaking the sample the oil phase disappeared and the aphron size was not affected at all in subsequent measurements. The polyaphron made using SDBS as the water-soluble surfactant was also tested for stability with respect to temperature. The sample was frozen in the laboratory refrigerator, and upon thawing almost all of the kerosene was released. This should be expected since freezing of water in the encapsulating soapy shell around the oil droplet will destroy the integrity of the shell and the polyaphron structure can be expected to break down. The addition of a melting point lowering substance in water would be required if the polyaphrons are to be utilized for low temperature applications. The density of the polyaphron made from SDBS in the aqueous phase at ambient temperature (298 K) was estimated to be 0.82 $g \cdot mL^{-1}$, which was in between that of water and kerosene (0.79 $g \cdot mL^{-1}$).

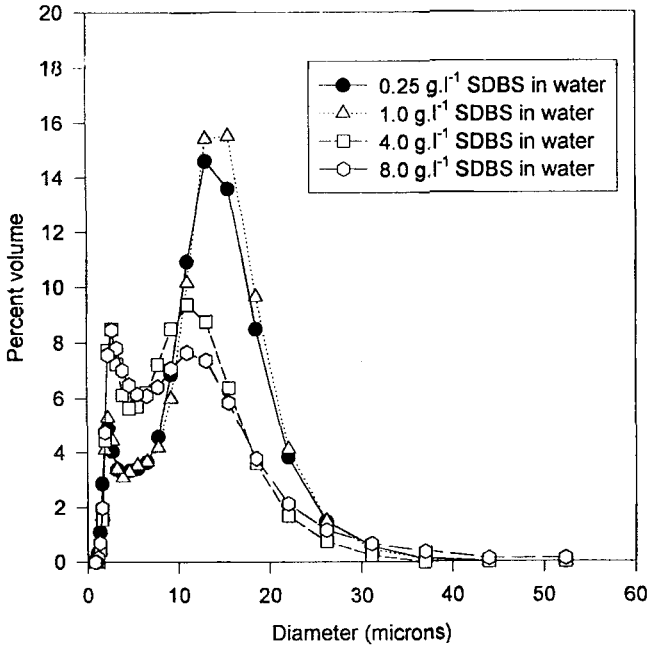


FIG. 6 The effect of SDBS concentration in water on polyaphrons generated from it.

Figure 6 shows the effect of different concentrations (0.25 to 8 $g \cdot L^{-1}$ in water) of water-soluble surfactant (SDBS) used in generating polyaphrons on the size distribution patterns. It shows a decrease in the mean bubble size and a broader distribution pattern at higher surfactant concentrations. This is in agreement with the fact that there is an inverse relationship between the surface area and the radius of the aphrons. If, for example, V mL of oil were to be converted into N polyaphrons each of radius r, then the total area per aphron is given by $3V/r$. The total quantity of water-soluble surfactant needed will depend on the total available surface area of all the aphrons, and hence when the radius is made smaller, the surfactant concentration in water has to be increased proportionally. It was also observed that polyaphrons made at the low surfactant concentration ($0.25\ g \cdot L^{-1}$) were not quite stable, since a lot of kerosene was released from the aphron within a month of storage. This clearly indicates the role of the water-soluble surfactant concentration in stabilizing the polyaphrons. Polyaphrons are not stable when made using an insufficient amount of water-soluble surfactant.

The introduction of polyaphrons in the water column for extraction of toxic organics invariably results in the presence of organic solvents. Since, as noted earlier, the polyaprhons are less dense than water, they should be expected to rise to the water surface under the influence of gravity. However, their rise velocity in the water column is very small due to their minute size. For example, a 2-μm polyaphron is expected to have a rise velocity of only 0.655 $cm \cdot h^{-1}$, and would therefore take 3.2 days to rise through a 0.5-m aqueous column. This poses a secondary pollution problem that necessitates a subsequent treatment process. The method proposed involves flotation of the polyaphrons using CGAs that have higher rise velocities due to their larger size range (30 to 150 μm) (7). CGAs are suitable since they can be created with the appropriate surface charges to attach to polyaphrons through coulombic forces, thereby aiding bouyancy. Any residual surfactant is also floated in the process and removed as foam at the top of the aqueous phase. In considering applications for the extraction of organics, we investigated the effects of flotation on the size distribution of polyaphrons in the aqueous phase.

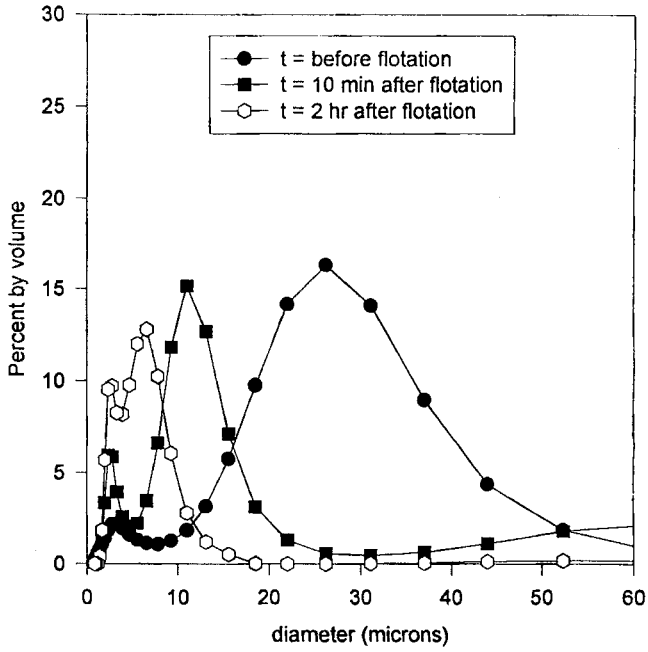


FIG. 7 The size distribution change with time after flotation for polyaphrons generated from HTAB as the water-soluble surfactant.

Figure 7 shows the results of flotation of polyaphrons made from HTAB as the water-soluble surfactant using CGAs made from SDBS. The size distribution of polyaphrons at different hold-up times after the flotation step are presented. The majority of polyaphrons were in the size range 10 to 30 μm before flotation with a very small fraction in the range 1 to

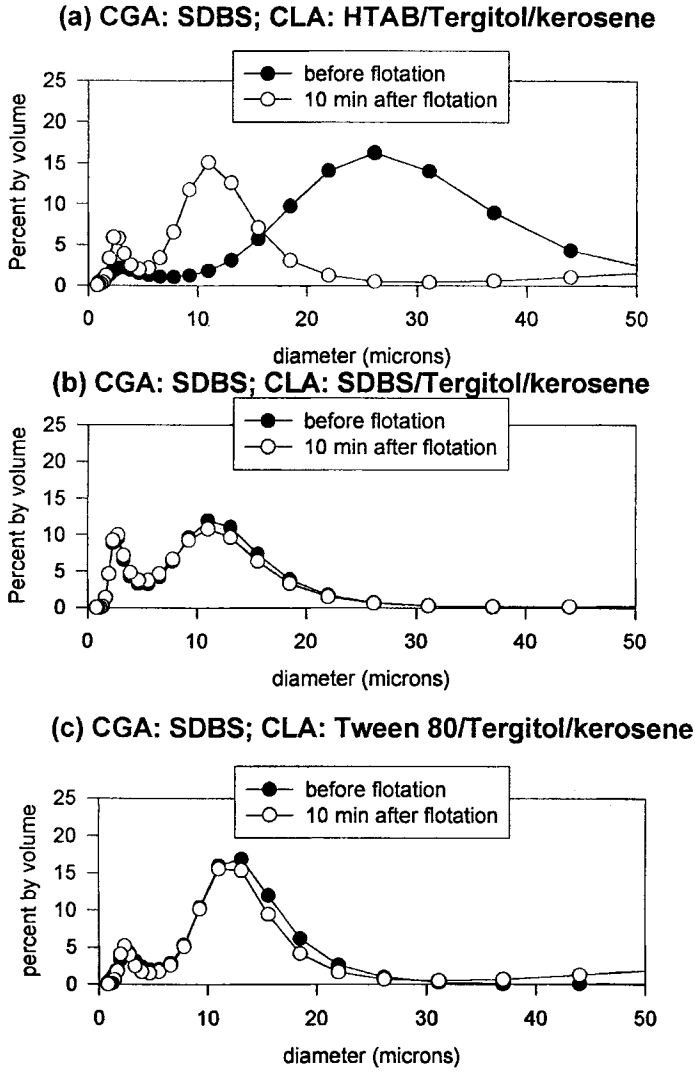


FIG. 8 The size distribution before and 10 minutes after flotation for polyaphrons generated from three different water-soluble surfactants: (a) HTAB, (b) SDBS and (c) Tween 80.

10 μm. Ten minutes after flotation the size distribution was observed to move toward smaller aphrons with a distinct bimodal distribution; a large fraction now falls in the range 6 to 18 μm. With more and more hold-up time the distribution moves progressively toward smaller aphrons, indicating that more and more of the larger aphrons are being removed from the aqueous phase. After 2 hours of hold-up time the size distribution is a bimodal one with an equal fraction in the ranges 1 to 4 μm and 6 to 12 μm. Thus the final distribution is dominated by small aphrons. Flotation using CGAs provides a method of clarifying the aqueous phase after PDSE.

Figure 8 compares the size distribution before and after flotation of different polyaphrons using a single type of CGA. The flotation conditions were identical in each case and the measurements were taken 10 minutes after flotation. The CGA was made from the anionic surfactant SDBS. As outlined earlier, the polyaphrons were prepared using the oil-soluble surfactant Tergitol 15-S-3 in kerosene, whereas three different water-soluble surfactants were used (SDBS, HTAB, and Tween 80). It is clear that polyaphrons generated from neutral and anionic surfactants are not affected by CGA flotation. Only the cationic polyaphron (HTAB) was effectively removed by the anionic CGAs. Flotation occurs by the conventional mechanism of physical interception of the smaller colligend species (polyaphrons) by the large collector species (CGAs). Furthermore, it is apparent that the mechanism of removal of polyaphrons involves a coulombic force between the oppositely charged collector (CGAs) and colligend (polyaphrons) species as is evident from Fig. 8(c). Thus, if the polyaphron is made using a cationic surfactant for the soapy film encapsulating the oil core, then the CGA should be made using an oppositely charged (anionic) surfactant. If the concentration of surfactant for preparing the CGA is carefully chosen so that the stoichiometry is taken into account, then the foam formation at the top of the aqueous column can be eliminated as well.

CONCLUSIONS

The polyaphrons studied showed a bimodal size distribution. They were in the range 1 to 3 and 10 to 30 μm in diameter. Polyaphrons generated using the nonionic Brij 35 in water showed the largest average diameter while those generated from the anionic SDBS had the smallest average diameter. Increasing the surfactant concentration of the water-soluble surfactant led to smaller polyaphrons being generated, but had a broader size distribution pattern. The polyaphrons were observed to show no distinct deterioration in quality even after storage for 230 days in a quiescent environment. The polyaphrons dispersed in water were removed from the

aqueous phase by flotation using CGAs; the mechanism of removal was one of coulombic attraction between the collector (CGAs) and the colligend (polyaphrons).

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