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# Flotation Separation Using Microgas Dispersions

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## Abstract

Microgas dispersions, agglomerations of micron-sized bubbles capable of being pumped, were studied for use in flotation operations. Methods for producing these dispersions and their use as a substitute for conventional flotation in the separation of an organic dye are described.

## INTRODUCTION

A Microgas dispersion (Fig. 1) is a collection of small spherical bubbles linked or touching each other and distributed in an aqueous medium. The bubbles are generally found to be from 1 to 50 $\mu$m in diameter and do not coalesce during the time needed for a flotation operation. It is possible to manufacture the dispersion at one location and pump it to a second point for utilization. A dispersion of these bubbles can contain up to 65% gas by volume.

Microgas dispersions (MGD) were first manufactured by Sebba (*1*, *2*) by utilizing a modified venturi device. These dispersions were later made by Shaler and McLean (*3*) using cyclones. Both the modified venturi and the cyclone provide a point of high velocity and low pressure in a moving stream. If a gas is introduced at this point into a rapidly moving stream of water containing a small quantity of surface-active agent, a dispersion is formed which contains a mixture of single bubbles and the characteristic MGD bubbles (Fig. 1). By repeated recirculation, bubbles that do not have the desired characteristics are then eliminated by coalescence. The

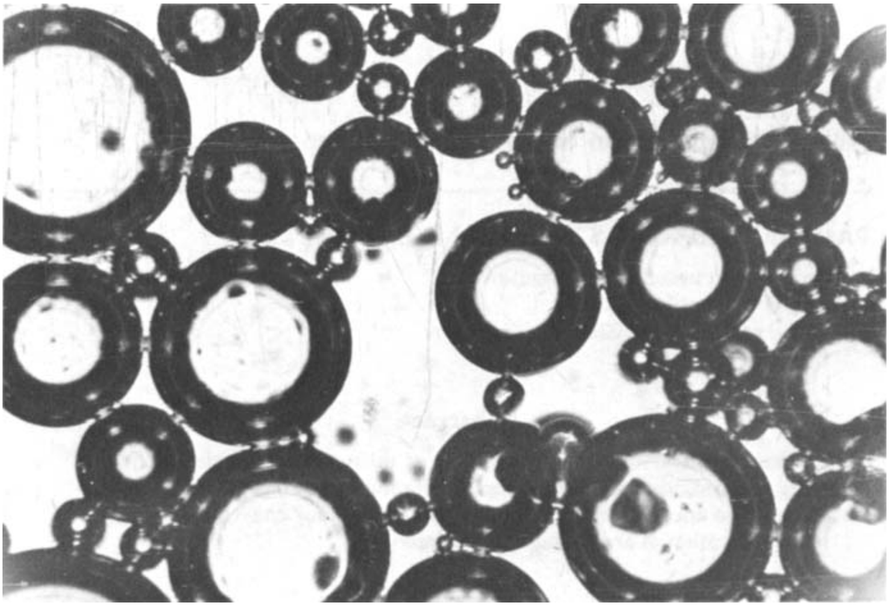

FIG. 1. Photomicrograph of a typical Microgas dispersion (1 cm = 35 μm).

micron-sized MGD bubbles retain their integrity, thus providing a large proportion of surface area per volume of bubbles charged to a flotation column. Since the bubbles do not coalesce but are linked together or touch each other, they can rise as quickly as a single larger bubble. This matrix of MGD bubbles removes contaminants in solution during its rise to the surface of the column. Unlike the conventional bubbles of flotation operations, which could be looked upon as holes in the liquid, whether they are generated through a fritted ceramic disk or by a release of pressure in dissolved air flotation, the MGD bubbles allow a more thorough contacting of the solution and strip the solution of its impurities in a shorter period of time than in conventional flotation because they are true bubbles (i.e., have a double interface, one with the water and one with the air).

Thus MGDs have three properties which suggest their use in flotation operations. First, a large surface area is available for adsorption; second, the mass of linked bubbles rises rapidly through a column of liquid; and third, these dispersions can be pumped from the generator to a column for use in a flotation operation.

## EXPERIMENTAL

### Apparatus

In this study a double-cyclone MGD generator developed by Shaler and McLean was employed for the manufacture of the dispersions. Other means of manufacture are available (*2*). The generator, shown in Figs. 2 and 3, consists of a means for feeding surfactant solution, for pumping this solution through a continuous loop, and for releasing the MGD foam. Two hydrocyclones (*4*) are arranged in series at one point of this

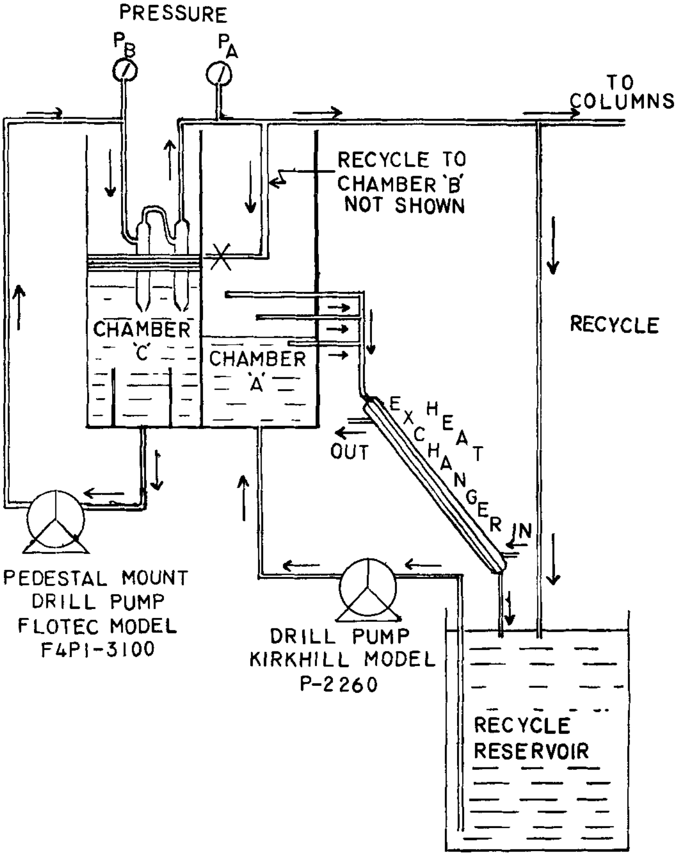

FIG. 2. Schematic of the Microgas dispersion generator.

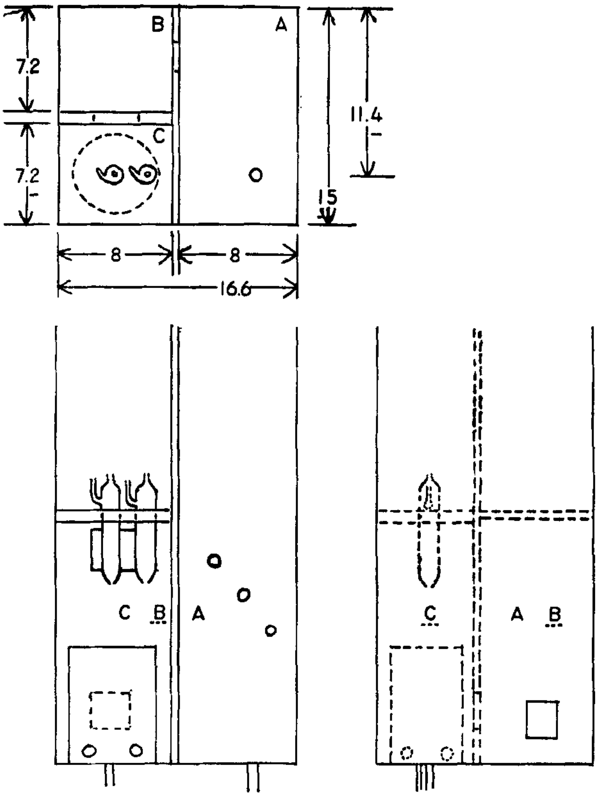

FIG. 3. Orthogonal projection of the Microgas dispersion generator. (All dimensions are in centimeters.)

loop. It is by this process of continuously recirculating the surfactant mixture through the cyclones that one is able to form a Microgas dispersion.

Compartment 'A,' shown in Figs. 2 and 3, is a constant level reservoir which retains a small quantity of surfactant solution (1 to 3 liters) and is kept filled from the main recycle reservoir (15 liters). A heat exchanger, located between the overflow ports and the recycle reservoir, is used to control the temperature of the surfactant solution. Chamber 'B,' closed at the top and connected to 'A' through a 4-cm$^2$ porthole, provides for the transition of surfactant solution from 'A' to Chamber 'C,' the cyclone compartment. Chambers 'B' and 'C' are connected by two 6-cm$^2$ portholes. The cyclone Chamber ('C') contains both cyclones located at the top of the compartment on a sliding platform. The classifying action of the hydrocyclones allows for the separation of the MGD bubbles from the ordinary bubbles. The underflow from the hydrocyclones is pumped directly back to the cyclones, and the separation process is continuously repeated. A circulating flow rate of approximately 14 liters per minute (based on water with the cyclones disconnected from Chamber 'C') is necessary to form a good dispersion. The product stream leaving the secondary cyclone can be recycled to Chamber 'B,' fed to a sampling device to measure dispersion quality (% gas, time to break), fed to a column for use in a flotation scheme, or returned to the recycle reservoir. The hydrocyclones are easily constructed by a glass blower while the rest of the device can be easily fabricated.

The flotation experiments were carried out in two glass cylindrical columns. One column was 5.0 cm i.d. and 90 cm in height and the other was 7.6 cm i.d. and 60 cm in height.

## Procedure

### *Microgas Dispersion Flotation Runs*

The column was filled with a saturated dye solution, approximately 0.000052 *M* (30 ppm) Remazol Golden Orange 3-G, to a height of about 50 cm. Dispersion additions were accomplished in single bursts fed into the column bottom. The dilution factor (D.F. = original column volume/final column volume) allowed for the calculation of the percent removal (%r).

$$\%r = (1 - (CF/((CI)(D.F.)))) \times 100$$

where CF = final concentration of impurity
CI = initial concentration of impurity

Surface area was calculated from an assumed bubble radius of 0.0025 cm (an estimate based on the photomicrographs taken at the time of each run) and the volume of air contained in the MGD feed.

### *Conventional Flotation Runs*

Conventional flotation runs were performed in a similar manner as the MGD runs. Metered, compressed air was passed through a fritted ceramic disk located at the bottom of the column. The dilution factor did not change since no liquid was added to the column with the gas bubbles.

Bubble diameter was estimated to be approximately 0.1 cm for surface area calculations. Surfactant (0.05 g/l, 50 ppm) was added at the beginning of each run by mixing the surfactant with the starting dye solution for 10 min prior to adding the solution to the column.

### Analytical

Dye concentration was measured at a wavelength of 475 nm with a spectrophotometer. Prepared calibration samples and unknowns were all adjusted to a pH of less than 3.5 for color stabilization before analysis.

## RESULTS AND DISCUSSION

The results obtained for several batch runs using the surfactant ethylhexadecyldimethylammonium bromide (EHDA-Br) for the manufacture of the Microgas dispersions are presented in Table 1. The results obtained for the several conventional flotation batch runs using the surfactant EHDA-Br in intimate contact with the dye solution are shown in Table 2.

Flotation with the Microgas dispersion enabled faster removal of the dye than conventional flotation due to the smaller bubble size in the MGD and the ability to inject these bubbles as part of a Microgas dispersion into the bottom of the column (i.e., their pumpability). Figure 4 shows an increase in removal as more and more dispersion is injected into the solution. However, it is also shown that dispersion quality (as measured by percent gas in the dispersion) plays an important role in dye removal. Three regressions are drawn through the points. The top line represents the removal obtained with excellent quality dispersions, those containing more than 42% air. The middle line represents the data obtained for those runs which had between 38 and 41% air, while the bottom line is drawn through points for poor dispersions (13% air). In most cases the generator would be operated such that high quality dispersions could always be used in separation operations. Dispersion quality is a critical variable which can significantly affect removal based on the amount of dispersion used in the separation. More importantly, because poorer dispersion quality is a result of less MGD bubbles contained in the dispersion, less surface area is fed to the column during a timed addition. Therefore we may expect less removal with a poor quality dispersion based on a consideration of the amount of surface area fed to the column.

Figure 5 shows the correlation between available bubble surface area and removal of dye solution. All of the initial dye concentrations were

TABLE 1

Dye Removal Using Microgas Dispersions

| Run no. | Initial dye concentration (ppm) | MGD surfactant concentration (ppm) | Final surfactant concentration (ppm) | Initial volume (ml) | Final volume (ml) | Dilution factor | Percent dye removal | Percent air MGD | Volume of air (ml) | Bubble surface area ($m^2$) | Time (sec) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 31.1 | 250 | 25 | 1030 | 1130 | .912 | 72.6 | 52 | 110 | 13.2 | 30 |
| 2 | 30.3 | 250 | 25 | 830 | 930 | .892 | 80.4 | 54 | 120 | 14.4 | 30 |
| 3 | 31.7 | 250 | 34 | 1030 | 1165 | .884 | 70.7 | 50 | 135 | 16.2 | 30 |
| 4 | 24.9 | 287 | 52 | 1030 | 1210 | .851 | 71.8 | 49 | 170 | 20.4 | 60 |
| 5 | 30.3 | 250 | 36 | 830 | 975 | .851 | 84.6 | 56 | 185 | 22.2 | 60 |
| 6 | 31.7 | 250 | 50 | 1030 | 1230 | .837 | 86.5 | 50 | 200 | 24.0 | 45 |
| 7 | 29.7 | 250 | 71 | 1005 | 1290 | .779 | 90.0 | 44 | 225 | 27.0 | 60 |
| 8 | 32.2 | 250 | 72 | 1030 | 1320 | .780 | 96.5 | 49 | 280 | 33.6 | 60 |
| 9 | 28.9 | 250 | 37 | 830 | 980 | .847 | 65.1 | 49 | 145 | 17.4 | 30 |
| 10 | 29.7 | 250 | 47 | 905 | 1095 | .826 | 93.2 | 44 | 150 | 18.0 | 60 |
| 11 | 24.9 | 287 | 42 | 975 | 1120 | .871 | 65.2 | 48 | 135 | 16.2 | 60 |
| 12 | 29.6 | 250 | 32 | 830 | 960 | .865 | 58.2 | 38 | 80 | 9.6 | 30 |
| 13 | 29.7 | 250 | 29 | 830 | 945 | .878 | 55.8 | 41 | 80 | 9.6 | 30 |
| 14 | 29.7 | 250 | 35 | 830 | 970 | .856 | 67.5 | 41 | 95 | 11.4 | 45 |
| 15 | 29.7 | 250 | 47 | 830 | 1020 | .814 | 72.4 | 41 | 130 | 15.6 | 60 |
| 16 | 30.1 | 250 | 60 | 830 | 1070 | .776 | 67.2 | 38 | 145 | 17.4 | 75 |
| 17 | 30.1 | 250 | 76 | 830 | 1135 | .731 | 73.8 | 38 | 185 | 22.2 | 90 |
| 18 | 30.1 | 250 | 89 | 830 | 1185 | .700 | 75.6 | 41 | 245 | 29.4 | 105 |
| 19 | 30.1 | 250 | 99 | 830 | 1225 | .678 | 80.1 | 41 | 275 | 33.0 | 120 |
| 20 | 31.0 | 248 | 16 | 830 | 895 | .927 | 20.9 | 13 | 10 | 1.2 | 15 |
| 21 | 31.0 | 248 | 25 | 830 | 930 | .892 | 34.0 | 13 | 15 | 1.8 | 30 |
| 22 | 31.0 | 248 | 42 | 830 | 1000 | .830 | 31.7 | 13 | 25 | 3.0 | 45 |
| 23 | 27.8 | 248 | 16 | 830 | 895 | .927 | 17.0 | 13 | 10 | 1.2 | 15 |
| 24 | 27.8 | 248 | 29 | 830 | 945 | .878 | 25.1 | 13 | 15 | 1.8 | 30 |
| 25 | 27.8 | 248 | 42 | 830 | 1000 | .830 | 33.6 | 13 | 25 | 3.0 | 45 |
| 26 | 31.0 | 248 | 27 | 1865 | 1975 | .944 | 22.4 | 13 | 15 | 1.8 | 34 |
| 27 | 31.0 | 248 | 42 | 1865 | 2035 | .916 | 35.2 | 13 | 25 | 3.0 | 68 |
| 28 | 31.1 | 248 | 76 | 1865 | 2170 | .859 | 32.9 | 13 | 45 | 5.4 | 101 |

TABLE 2

Dye Removal Using Conventional Flotation

| Run no. | Initial dye concentration (ppm) | Surfactant concentration (ppm) | Volume (ml) | Dilution factor | Percent dye removal | Volume of air (ml/min) | Bubble surface area ($m^2$) | Time (min) |
|---|---|---|---|---|---|---|---|---|
| 1 | 25.7 | 50 | 945 | 1.0 | 43.8 | 150 | 4.5 | 5 |
| 2 | 25.6 | 50 | 945 | 1.0 | 60.9 | 150 | 9.0 | 10 |
| 3 | 26.5 | 50 | 945 | 1.0 | 84.6 | 150 | 13.5 | 15 |
| 4 | 24.3 | 50 | 945 | 1.0 | 91.9 | 150 | 18.0 | 20 |
| 5 | 27.2 | 50 | 945 | 1.0 | 99.0 | 150 | 22.5 | 25 |

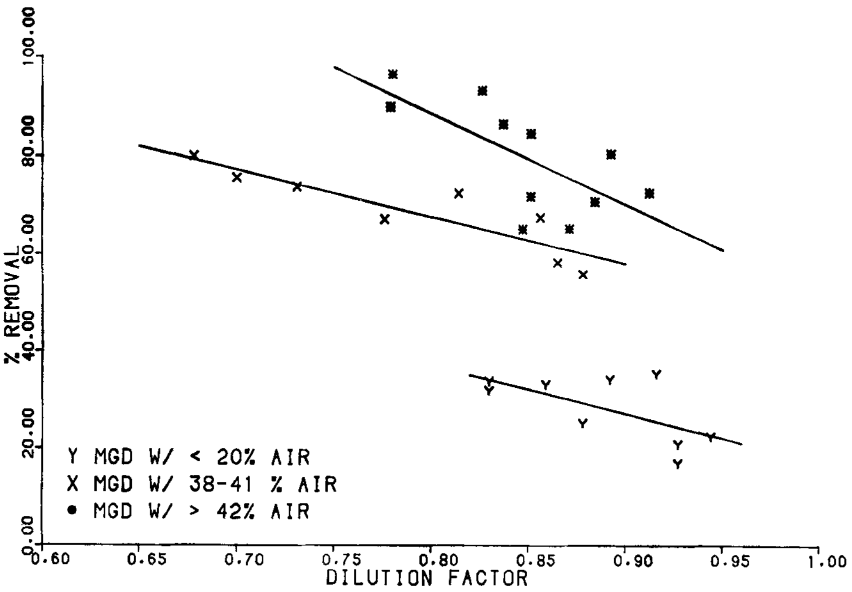

FIG. 4. Percent removal vs dilution factor for the Microgas dispersion runs.

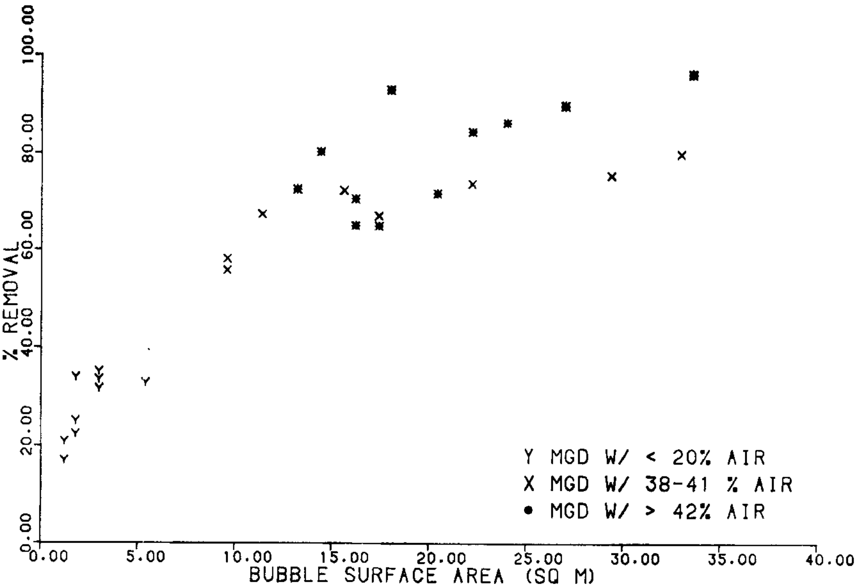

FIG. 5. Percent removal vs bubble surface area for the Microgas dispersion runs.

approximately equal. It is observed that removal is relatively linear with respect to bubble surface area until high removals are obtained. However, even with the better quality dispersions with their very high surface area per unit volume of dispersion, removal of the dye remains high due to the excellent contacting of the dye solution and the MGD. Since the bubbles are attached to each other in the dispersion, a somewhat smaller surface area is available because these points of attachment cannot accommodate dye molecules. This reduction in surface area was not considered here.

The amount of surfactant used in each experiment is indicated in Table 1. Here we note that no surfactant is present in the column at the start of the run. Surfactant is added with the MGD at approximately 250 ppm. The total amount of liquid MGD added to the column then dictates the final concentration of surfactant in the column. Although this is not necessarily dispersed throughout the column, but more likely located at the column surface, it does serve as a comparison with the total amount of surfactant used in the conventional runs.

Experiments have been performed in an attempt to concentrate the surfactant in the MGD formed from a 250-ppm surfactant solution, but they have been unsuccessful. This indicates that the dispersion made from such a solution actually contains 250 ppm surfactant. If we consider this surfactant to be concentrated in a monolayer on the bubble surface, this has the potential of removing an equivalent amount of dye from solution (i.e., on a mole to mole basis):

$$\text{ppm surfactant/M.W. surfactant} = \text{ppm dye/M.W. dye}$$

In all but two cases, more surfactant was added than required for total removal. In the two cases (Runs 20 and 23), enough surfactant was added for approximately 80% removal, far above that obtained. It is understood that more than the theoretical amount of surfactant is required, but it is also expected that total dye–surfactant contact on the bubble surface cannot be expected in the extremely fast removal procedure.

The data in Table 2 for conventional flotation of the Remazol dye indicate that an equal removal of dye is possible once the amount of surface area fed to the column becomes approximately equal. Here, approximately twice the necessary surfactant for 100% theoretical removal was added before the start of each run. Thus surfactant–dye complexing should not have influenced the separation results. A concentration of 50 ppm was chosen to simulate the surfactant concentration encountered in the MGD runs. It is evident that the times required for removal were much different due to the nature of the bubble in use. The MGD system was able to make available more surface area for a given amount of time than the conventional flotation.

The removal of the dye from solution is attributed to an ionic coupling of the dye's sulfate group with the surfactant's ammonium ion. The quaternary ammonium ion either coats the surface of the bubble and forms a complex on the bubble's surface or forms a complex in solution and later adsorbs to the bubble's surface. Flotations performed with an anionic surfactant (sodium lauryl sulfate), a nonionic surfactant (Triton X-100, an octylphenoxy polyethoxy ethanol), or no surfactant would not remove dye from solution. Flotation with EHDA-Br formed an insoluble scum at the surface of the column which is indicative of ion flotation (5).

## CONCLUSION

An organic dye was easily removed from dilute solution in a time an order of magnitude faster than that capable of being attained using conventionally generated bubbles for flotation operations. The time advantage is primarily due to the increased surface area provided by the Microgas dispersion. Flotation operations using a Microgas disperion, capable of being pumped from one location to another, are feasible. A method of producing these dispersions has been outlined.

## Acknowledgment

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