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REMOVAL OF METHYL ORANGE AND METHYLENE BLUE DYE FROM WATER USING COLLOIDAL GAS APHRON—EFFECT OF PROCESSES PARAMETERS

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**REMOVAL OF METHYL ORANGE AND
METHYLENE BLUE DYE FROM WATER
USING COLLOIDAL GAS
APHRON—EFFECT OF PROCESSES
PARAMETERS**

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ABSTRACT

Colloidal gas aphon (CGA) was used in a flotation column to remove methyl orange and methylene blue dye from water. The effect of process parameters, i.e., surfactant type and concentration, CGA flow rate, CGA diameter and gas holdup, pH, residence time, and salt concentration, were studied through measurements of percent dye removal. The overall percent removal of methyl orange and methylene blue was 95–98% for the range of experimental parameters studied. When the surfactant and dye had a similar charge, 40% removal of dye from the water was achieved. This result was unexpected because dye removal by CGA was thought to be an ion-flotation process. However, the oppositely charged dye and surfactant resulted in 98% removal of dye. Based on these observations, the mechanism for dye removal from water using CGA was elucidated. The removal of methyl orange was higher in alkaline condi-

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tions when CGA was generated from the cationic surfactant, hexadecyltrimethyl ammonium bromide. The removal of methyl orange is higher in alkaline condition when generated from the anionic surfactant, sodium dodecylbenzene sulfonate. The percent removal of methyl orange and methylene blue increased with increased flow rate, decreased CGA diameter, and increased gas holdup. The percent removal of dyes increased with increased residence time of CGA in the flotation column. No effects of surfactant concentration above the cmc or salt concentration on the percent removal of dye were observed within the range of experimental parameters.

Key Words: Colloidal gas aphon; Flotation; Dye removal from water; Surfactants

INTRODUCTION

Micro gas bubble or a colloidal gas aphon (CGA) is a gas bubble encapsulated in a thin, aqueous, soapy film. The use of CGA in separating chemicals and fine particulates from effluent water was first reported by Sebba and Barnett (1). The fundamental properties and the structure of CGA were discussed in detail by Sebba (2). Recently, many investigators have used CGA in various ways, e.g., for the removal of pentachlorophenol (3), heavy metal (4), and dye (5) from water; flotation of yeast cells (6); recovery of protein (7); removal of 2,4-dichlorophenoxy acetic acid (8), hazardous oily waste (9), and naphthalene (10) from soil; transport of bacteria through a soil matrix (11); and harvesting of microorganisms (12). Previous research has established that flotation through the use of CGA is an efficient and economically viable process.

Roy, Valsaraj, and Kottai (5), using CGA, studied the separation of organic dyes, e.g., methyl orange, methylene blue, cibacrome-4G, and cibacrome-6B, from wastewater. They used hexadecyltrimethyl ammonium bromide (HTAB) and sodiumdodecyl benzene sulfonate (SDBS) as surfactants for the generation of CGA. They showed that approximately 98% removal of the dye is possible through the use of CGA. They did not study in detail the effect of different process parameters on the removal of dye from water.

In this study, CGA was generated in the laboratory with a spinning disc generator. The CGA was used in a flotation column to remove methyl orange and methylene blue dye from water. The oppositely charged methyl orange and methylene blue were used to elucidate the mechanism of dye separation using CGA. The percent removal of dye from water was estimated through the use of a UV-spectrophotometer and the results were compared with data available in the



literature. The effect of different process parameters, i.e., pH, CGA flow rate and diameter, gas holdup, surfactant type and concentrations, salt concentration, and residence time on the removal of dye was studied.

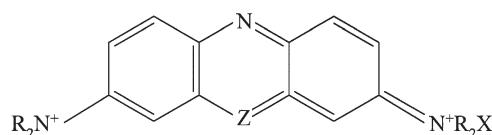
EXPERIMENTAL

Material

Two different surfactants were used in distilled water for the generation of CGA: SDBS (Sigma, India) and HTAB (Eastman Kodak). SDBS (M.W. = 348.5) is an anionic surfactant with a critical micelle concentration (cmc) value of 500 ppm. The structure of SDBS is $\text{CH}_3-(\text{CH}_2)_{10}-\text{CH}_2-\text{C}_6\text{H}_4-\text{SO}_3^{2-}-\text{H}^+\text{Na}^+$. HTAB (M.W. = 364.6) is a cationic surfactant with a cmc value of 350 ppm. The structure of HTAB is $\text{CH}_3-\text{[CH}_2]_{15}-\text{N}^+(\text{CH}_3)_3\text{Br}^-$. The dyes used for the separation process were methyl orange and methylene blue. Methyl orange is anionic with a sulfate group attached to a long carbon chain that contains an azo group:



Methylene blue is cationic with an amine group attached to polycyclic carbon atoms:



where Z is N, O, or S, and X is an anion. The pH was adjusted with analytical grade HCl and NaOH, and analytical grade NaCl was used to study the effect of salt added to the system.

Experimental Setup

The experimental setup used for CGA flotation for dye removal is shown in Fig. 1. The outlet of a CGA generator was connected with a flexible tube to the bottom of a flotation column. A peristaltic pump was fitted to the flexible tube. The 60-cm glass column was 4 cm in diameter and was used for flotation. Two sample taps in the column were at 20 and 40 cm from the bottom of the column. The glass column was bent at the top to collect the CGA foam.

Based on the method of CGA generation suggested by Sebba (2), a CGA generator was fabricated in the laboratory. A shaft was fitted to a 0.5-hp motor and a 50-mm diameter flat disk was attached to the end of the shaft. The flat disk



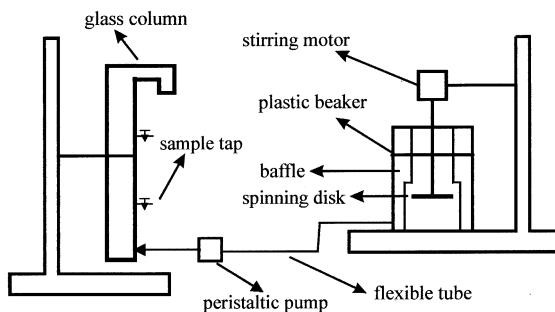


Figure 1. Schematic of experimental setup for CGA generation and flotation to remove dye from water.

worked as an impeller. A 5-L beaker containing surfactant solution was placed under the motor to generate CGA. Two baffles were placed in the beaker to assist in CGA formation.

METHOD

The beaker in the CGA generator was filled with a known quantity of distilled water and surfactants. The surfactant concentrations used were at or above the cmc. The level of the surfactant solution was initially kept 15 mm above the disk by adjusting the position of the beaker. The CGA-generator disc was rotated at 8000 rpm until a sufficient quantity of CGA was formed and stable bubble size was achieved. The speed of the disc was then adjusted to 5000 rpm to prevent creaming the CGA. The flotation column was filled with 50 ppm dye dissolved in water. The CGA was passed through the bottom of the flotation column using the peristaltic pump at the desired flow rate. The CGA floated to the top creating a foam layer. The dye was collected at the top of the aqueous phase trapped in the foam structure. The color of the dye was visually observed in the foam structure. A visual examination showed that the dye had not dispersed back into the aqueous phase. Finally, dye trapped in foam flowed out of the column and was collected in a measuring cylinder.

Samples were collected from the top and the bottom taps of the column after a known volume of CGA passed through the column. The samples were analyzed in a UV-spectrophotometer (Shimadzu, UV1201) to determine the amount of dye separated. The absorbency spectra analysis of methyl orange and methylene blue indicated that the maximum absorbency values for the dyes were 510 nm and 665 nm, respectively. The standard calibrating curve for concentration versus absorbency was plotted at the corresponding optical density. The collected sam-



bles were analyzed at these wavelengths and the concentration of dye was determined from the standard curve.

The absorbency spectra of dye showed that the color of the contributing species was not selectively removed. The difference in the dye concentrations collected from the top and bottom taps of the column was insignificant. This result is due to the vigorous mixing of the dye-aqueous phase solution during the passage of CGA. The dye trapped in the CGA foam structure was analyzed after the collected foam was broken to check the mass balance. During the passage of CGA, the dye-aqueous phase solution becomes diluted because of water present in CGA. The amount in CGA was estimated from the measured gas-holdup percentages (Table 1). The dilution effect of the dye-aqueous phase solution was taken into account when the percent removal of dye from water was estimated. The loss of aqueous phase in CGA foam formed at the air-aqueous phase interface of the column was also considered when we calculated the percent removal of dye (error 2–5%).

The photograph of CGA was taken from the entry point of the flotation column under a stereo-zoom microscope (Nikon Optiphot, HFX-DX microphotography arrangements). Photomicrographs were used to estimate the sauter mean diameter (d_{32}) of CGA. The brightened inner phase and the dark border are included in the diameter measurements of the aphon (Fig. 2). In each experiment, the size of 200 aaphrons was measured as a representative sample (5% error). We measured the liquid holdup by taking 100 mL of CGA in a measuring cylinder and finding

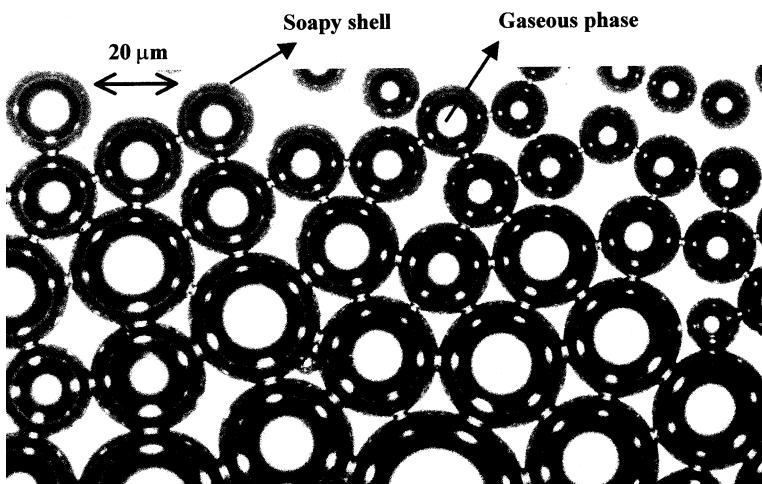


Figure 2. Photomicrograph of colloidal gas aaphrons generated from HTAB surfactant (350 ppm) at 8000 rpm.



Table 1. Physical Characteristics of CGA Used in the Flotation Column

Dye (concentration)	Surfactant	Concentration (ppm)	RPM	d_{32} (μm)	Gas Holdup (%)
Methyl orange (50 ppm)	HTAB	350	8000	22	67
		6000		36	64
		4000		75	45
	SDBS	500	8000	14.2	69
		500	8000	27	67
		6000		43	65
Methylene Blue (50 ppm)	SDBS	6000	4000	92	48
		700	8000	20.7	69.8

the volume of liquid in the measuring cylinder after all the CGA had disappeared. The detailed physical characteristics of CGA are given in Table 1.

RESULTS AND DISCUSSION

Physical Characteristics

Figure 2 shows the photomicrograph of CGA. The brightened inner phase is the gaseous phase and the dark border around the gaseous phase is the soapy shell. The dark border around the aphyrons is not a shadow, otherwise overlapping of shadows would have been observed. The photograph of CGA taken by Sebba (2) under a high-power microscope showed similar structures. The d_{32} and gas holdup for CGA at different HTAB and SDBS concentrations and rpm are given in Table 1. The d_{32} decreased and the gas holdup of CGA increased with the increase in rpm and surfactant concentrations. Similar results were reported by Chaphalkar, Valsaraj, and Roy (13), Jaugeri, Gilmour, and Varley (14), and Jaugeri, Mitchell, and Varley (15).

Selection of Surfactant

The selection of suitable surfactant for the treatment of the dye depends on the polarization of electrical charges of the dye and surfactant molecules. Methyl orange is negatively charged while methylene blue is positively charged. The CGA suspension produced from both cationic and anionic surfactant was used to elucidate the mechanism of dye removal from water through CGA. Figure 3 shows that the percent removal of positively charged methylene blue is 90% when treated with CGA produced from negatively charged SDBS surfactant. However,



the percent removal of positively charged methylene blue is only 40% when CGA produced from cationic HTAB was used. The different parameters used were a CGA flow rate of 40 mL/min, surfactants concentration at cmc, CGA diameter of 25 μm , and gas holdup of 67% for both HTAB and SDBS surfactants.

Methylene blue and SDBS are oppositely charged and hence they are electrostatically attracted to each other and form a loosely bonded ion-surfactant complex. The negatively charged polar head of the SDBS surfactant is pointed toward the water in the micellar structure. The separation of methylene blue occurs primarily through the formation of an ion-surfactant complex around the micelles present in the soapy shell of CGA.

Chaphalkar, Valsaraj, and Roy (3) and Roy, Valsaraj, and Kottai (5) proposed similar views on the mechanism of dye removal. They pointed out that the similarly charged surfactant and dye did not result in separation of dye from water. In the present investigation, 40% removal of methylene blue is possible using CGA prepared from HTAB. Methylene blue molecules move up with the buoyant action of myriads of small aaphrons and finally they are trapped during the collapse of CGA and the formation of foam at the aqueous phase-air interface. Methylene blue trapped in the foam structure overflows out of the flotation column. Chaphalkar, Valsaraj, and Roy (3) did not report on the formation of standing foam above the aqueous phase. In the present study, the formation of foam above

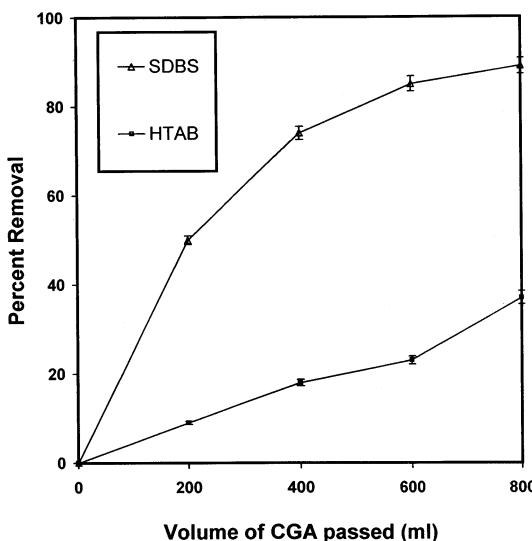


Figure 3. Effect of surfactant type on the removal on methylene blue. The concentration of SDBS and HTAB used were 500 ppm (cmc) and 350 ppm (cmc), respectively. The CGA diameter is approximately 25 μm and the CGA flow rate is 40 mL/min.



the aqueous phase was observed because the CGA had a high gas holdup percentage. Similarly, the percent removal of negatively charged methyl orange was higher when CGA prepared from cationic HTAB was used instead of that prepared from anionic SDBS.

Effect of pH

The plot for percent removal of methyl orange versus a volume of CGA passed at varying pH is shown in Fig. 4. The surfactant HTAB was used to generate CGA. Figure 4 shows that 93% removal of methyl orange was obtained in alkaline conditions while 87% removal was obtained under acidic conditions. NaOH was added to increase the pH of dye solution. Na^+ activates the sulfonate group ($-\text{SO}_3^{-2}$) of methyl orange, and thus, electrostatic attraction between HTAB and methyl orange molecules increases and results in a higher percent removal of dye. Chaphalkar, Valsaraj, and Roy (3) observed a similar trend with pH for the removal of pentachlorophenol (PCP) from water. When CGA generated from HTAB was used, the removal of PCP was higher in alkaline conditions than it was under acidic conditions.

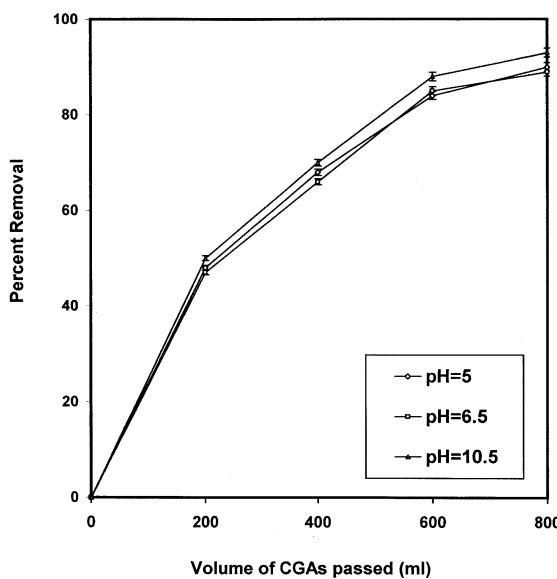


Figure 4. Effect of pH on the removal of methyl orange using CGA prepared from HTAB surfactant.



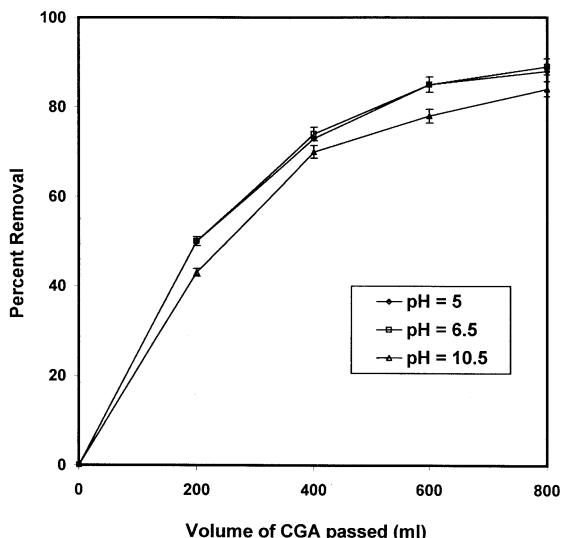


Figure 5. Effect of pH on percent removal of methylene blue using CGA prepared from SDBS surfactant.

The plot for percent removal of methylene blue versus volume of CGA passed at different pH conditions is shown in Fig. 5. SDBS was used to generate CGA. Figure 5 shows that the percent removal of methylene blue is 89% in acidic conditions compared to 81% at alkaline conditions. The 2 amine groups present in methylene blue is activated in the acidic conditions, resulting in higher electrostatic attraction between SDBS and methylene. Thus, the percent removal of methylene blue was higher in acidic conditions than it was in a basic environment.

Effect of Flow Rates

The plot for percent removal of methyl orange versus volume of CGA passed at different flow rates is shown in Fig. 6, while that of methylene blue at different flow rates is shown in Fig. 7. Figures 6 and 7 show that the percent removal of dye increases with the increase in flow rate (20 to 40 mL/min). The percent removal of dye increases with the increase in flow rate primarily because of the increase in flux of CGA bubbles passing through the cross-section of the column. The rise velocity of CGA is hindered at the higher flow rate of 40 mL/min compared to that at 20 mL/min because of the presence of large number of CGA bubbles. This leads to the increase in residence time of the bubbles, and hence, percent removal of dye increases. Amiri and Woodburn (16) studied the creaming



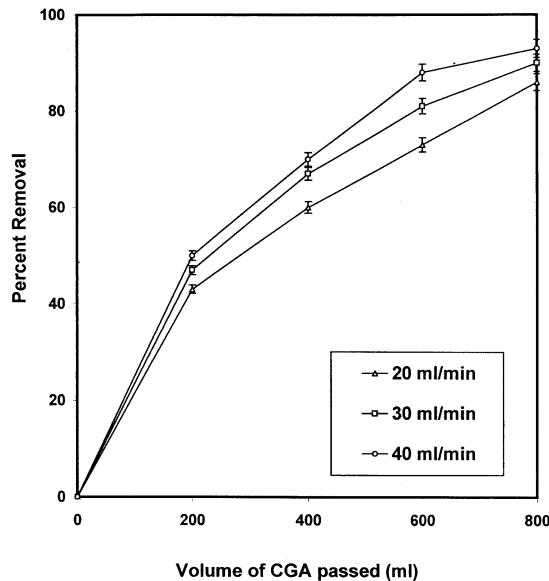


Figure 6. Effect of CGA flow rate on the percent removal of methyl orange. HTAB is the surfactant.

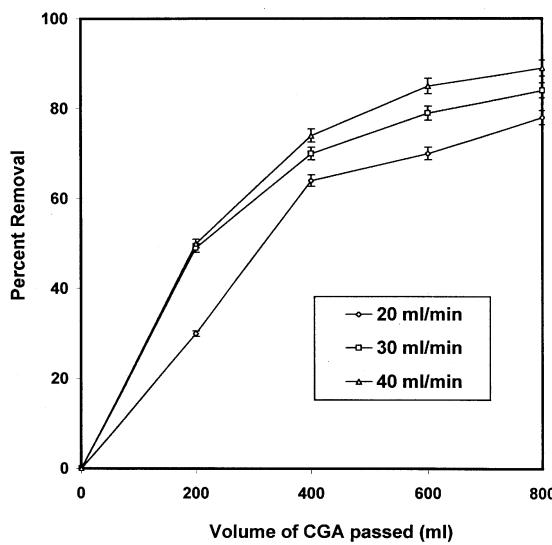


Figure 7. Effect of CGA flow rate on the percent removal of methylene blue. SDBS is the surfactant.



behavior of CGA, and they showed that the CGA velocity in a flotation column decreased with an increase in gas holdup of CGA. The CGA diameter was the same for the 2 different flow rates studied. In the present investigation, removal of methyl orange and methylene blue was above 90%, which is of the same order observed by Roy, Valsaraj, and Kottai (5), who used CGA, and Caballero, Cela, and Perez-Bustamente (17), who used a CGA-assisted solvent-sublation process.

Effect of Surfactant Concentrations

The plot for percent removal of methyl orange versus volume of CGA passed at 2 different HTAB concentrations is shown in Fig. 8, while that for methylene blue at 2 different SDBS concentrations is shown in Fig. 9. Figures 8 and 9 show no effect of surfactant concentrations on percent removal of dye above the cmc value. This is because the CGA diameter and the gas holdup do not vary appreciably with the increase in surfactant concentrations above the cmc value at 8000 rpm (Table 1). The effect of CGA diameter and gas holdup on percent removal is subsequently discussed. The CGA is not stable below the cmc value of the surfactants, and hence, it is not used for flotation. Chaphalkar, Valsaraj, and Roy (3) showed that the percent removal of PCP increased when the surfactant concentration was increased from below the cmc to its cmc value.

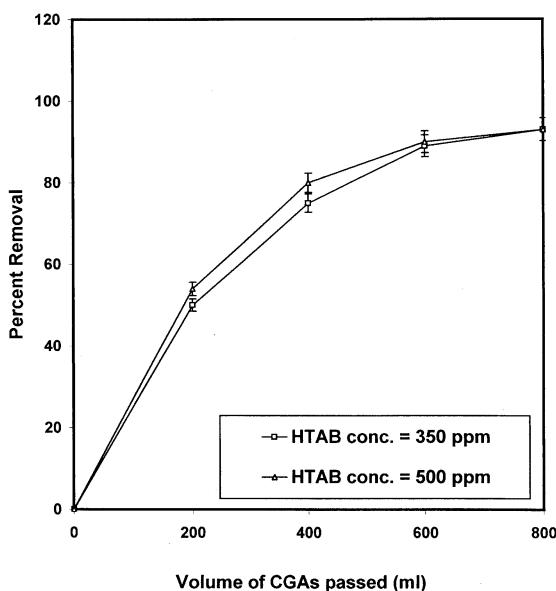


Figure 8. Effect of HTAB concentration on the percent removal of methyl orange.



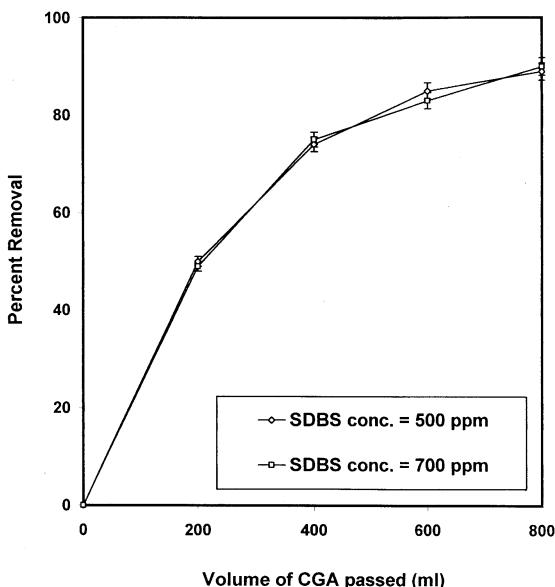


Figure 9. Effect of SDBS concentration on percent removal of methylene blue.

Effect of CGA Diameter and Gas Holdup

The diameter and gas holdup of CGA varied when the rpm of the spinning disc generator was changed. The variations of d_{32} and gas holdup percentages for 2 different surfactants (i.e., SDBS and HTAB) at different rpm values are shown in Table 1. The d_{32} decreased and gas holdup increased with the increase in rpm.

The plot for percent removal of methyl orange versus volume of CGA passed for 3 different CGA diameters and corresponding gas holdup percentages is shown in Fig. 10, and the plot of methylene blue for 3 different CGA diameters and corresponding gas holdup percentages is shown in Fig. 11. The HTAB and SDBS concentration was maintained at 350 ppm (cmc) and 500 ppm (cmc), respectively. The percent removal of methyl orange and methylene blue increased with the decrease in CGA diameter and increase in gas holdup. The CGA generated from HTAB with 22 μm bubble diameter and gas holdup of 67% yielded 93% separation of methyl orange compared to 81% separation from CGA with 75 μm bubble diameter and gas holdup of 45% (Fig. 10). Similarly, CGA generated from SDBS with 27 μm bubble diameter and gas holdup of 67% yielded 89% separation of methylene blue compared to 79% separation from CGA with 92 μm of bubble diameter and gas holdup of 48% (Fig. 11). The smaller diameter of CGA and higher gas holdup gave rise to an increased number of aaphrons for a specific



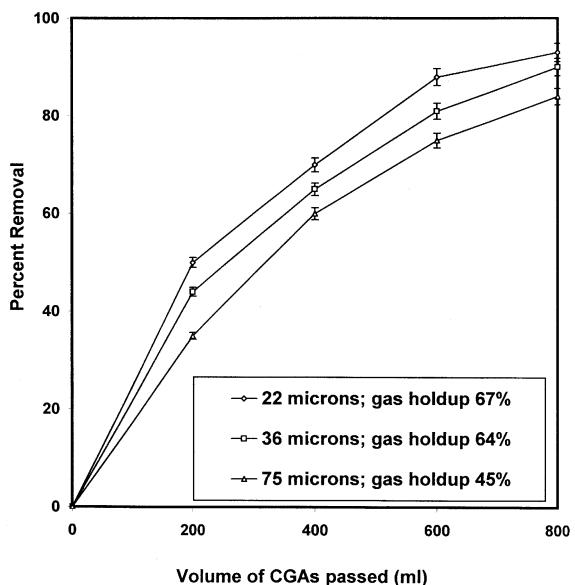


Figure 10. Effect of CGA diameter and gas holdup on the percent removal of methyl orange.

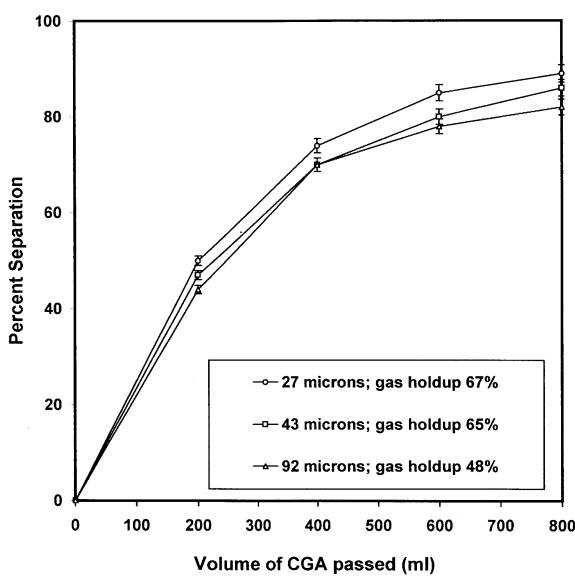


Figure 11. Effect of CGA diameter and gas holdup on percent removal of methylene blue.



volume of CGA. The percent removal of dye increased because of the increase in the number of CGA, which provides larger interfacial area to form surfactant-dye complexes through which the dye molecule can penetrate into the soapy shell of CGA.

Residence Time

The percent removal of dye from water strongly depends on the residence time of CGA in the flotation column. The residence time is a function of diameter and flow rate of the CGA and the height of the aqueous phase that contains dye in the column. The amount of the aqueous phase was varied while the CGA diameter and flow rate were kept constant. The 2 samples were taken 22 and 40 cm from the bottom of the column and are equivalent to 400 and 700 mL of aqueous phase that contains dye, respectively.

Figure 12 shows the plot for percent removal versus volume of CGA passed for 2 different aqueous phase amounts containing methyl orange, 400 and 700 mL, respectively. The percent removal is 84% for 400 mL of dye when treated with 400 mL of CGA. Whereas, the percent removal is 96% for 700 mL of dye when treated with 700 mL of CGA. The plot for percent removal versus

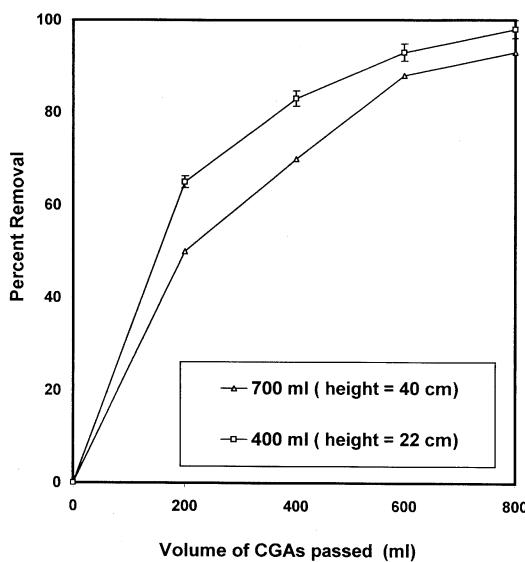


Figure 12. Effect of residence time of CGA on percent removal of methyl orange.



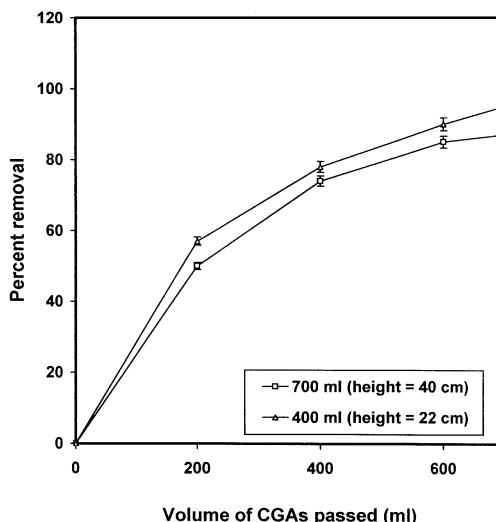


Figure 13. Effect of residence time of CGA on removal of methylene blue.

volume of CGA passed for 2 different amounts of aqueous phase containing methylene blue, sampled at 400 and 700 mL, is shown in Fig. 13. Similar to the results with methyl orange experiments, the aqueous phase containing methylene blue of 700 mL, when treated with 700 mL of CGA, resulted in higher percent removal than that from the aqueous phase containing methylene blue of 400 mL, when treated with 400 mL of CGA. The residence time of CGA is larger for 700 mL dye (40 cm height) sample taken at 40 cm than that for 400 mL dye (22 cm height). The volume ratio of dye to be treated and the CGA passed was kept at 1:1.

Effect of Salt

The removal of methyl orange and methylene blue was not significantly changed with the addition of NaCl in the presence of surfactant at the cmc value. Chaphalkar, Valsaraj, and Roy (13) and Jauregi, Gilmour, and Varley (14) found that the gas holdup slightly increases as the soapy film is compressed in the presence of NaCl due to the shielding of the surfactant charge. Thus, the percent removal of dye from water is not affected because the gas holdup and aphon size do not change significantly in the presence of NaCl.



CONCLUSIONS

CGA suspension generated using cationic surfactant, HTAB, and anionic surfactant, SDBS, is capable of removing 95% of methyl orange and 92% of methylene blue from water. The percent removal is higher when the surfactant used for the generation of CGA is opposite in charge to that of the dye molecule. The effects of various parameters are listed below.

1. The percent removal of methyl orange is increased under alkaline conditions when HTAB is used for the generation of CGA. Whereas, removal of methylene blue is higher at acidic conditions when SDBS is used for the generation of CGA.
2. The percent removal of methyl orange and methylene blue is increased with the increase in CGA flow rate in the column.
3. The change in surfactant concentration above the cmc does not affect the percent removal of methyl orange or methylene blue.
4. The percent of removal of methyl orange and methylene blue is increased with the decrease in CGA diameter and increase in gas holdup. This effect is achieved by increasing the rpm of the spinning disc generator.
5. The percent removal of methyl orange and methylene blue is increased with the increase in CGA residence time in the flotation column.
6. The presence of NaCl does not affect the percent removal of methyl orange or methylene blue.

The effluent water from the chemical industries may contain mixtures of various polarities of dye. In this study, pure dye was used to study the effect of various parameters on the removal process. A mixture of different polarities of dye may be able to be removed from water by using CGA generated from a mixture of cationic and anionic surfactants. However, this hypothesis needs to be verified.

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