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Lei Jiang[a]; Senlin Tian[a]; Ping Ning[a]; Hong Mo[a]
[a] Faculty of Environmental Science and Engineering, Kunming University of Science and Technology, Kunming, Yunnan, China

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Solubilization Absorption of Toluene Vapor by Formation of Oil-in Water Microemulsions with Cation Surfactants

Lei Jiang, Senlin Tian, Ping Ning, and Hong Mo
Faculty of Environmental Science and Engineering, Kunming University of Science and Technology, Kunming, Yunnan, China

Absorption reactors to remove volatile organic compounds from gas streams can be retrofitted, as volatile organic compounds act as oils and form microemulsions. Using the region of microemulsion in the phase diagram as the indicator, the main influences on the solubilization capacity of various microemulsion systems were investigated. When the mass ratio of the surfactant (cetyltrimethylammonium bromide) to the cosurfactant (tetrabutylammonium bromide) was 1:1, the microemulsion was the best absorbent. Comparative absorption tests of microemulsion, distilled water, and surfactants were performed in a double-stirred reactor. The results indicate that the microemulsion has a relatively higher absorption rate and absorption capacity than the other absorbents.

Keywords absorption; microemulsion; phase diagram; solubilization; toluene

INTRODUCTION

Large volumes of exhaust gases contaminated by volatile organic compounds (VOCs) such as toluene, xylene, and acetone are discharged into the atmosphere by industrial facilities. VOCs are the source of serious environmental issues and their emission has been subjected to more and more stringent legislation (1–3). Commonly used technologies of VOC emission abatement, such as activated carbon adsorption, absorption in liquid, biofiltration, incineration, and catalytic oxidation, have many strengths, as well as considerable limitations (4–6).

To remove VOCs, absorption is a promising candidate as it is easy to implement, with low capital and running costs; however, the selection of a suitable absorbent is a significant problem. A suitable absorbent is generally considered by the following criteria—absorption capacity, selectivity with respect to other gases, low toxicity, and volatility (7–14). When hydrophobic VOCs, such as benzene, toluene, ethylbenzene, and xylenes (BTEX), are to be removed from industrial exhaust gases, an absorption reactor filled with pure water is not efficient due to the low solubility of hydrophobic VOCs in water. When fresh Tween 81 solution (0.3% w/v) is replaced every hour, it absorbs more than 90% of the toluene in an intel gas containing a maximum of 1000 ppmv ($\mu L\, L^{-1}$) toluene over 48 h (14). Certain combinations of water, oil, surfactant, and an alcohol-amine based cosurfactant produce clear, seemingly homogeneous solutions termed "microemulsions" (15). The oils are simple long-chain hydrocarbons and the surfactants, which have a hydrophilic head and a hydrophobic tail, can be dissolved in water to form a micelle. Hydrophilic groups attach to water groups while hydrophobic tails aggregate in the middle of the micelle. As hydrophobic VOCs are trapped in the hydrophobic tails, their solubility in the surfactant is much greater than in pure water. Microemulsions, which are composed of surfactants and cosurfactants, are soluble in water. The solubilization capacity of organic compounds in the oil-in-water (O/W) surfactants is about 5%, and in the O/W microemulsions (about 60%) is greater than in the surfactants (16–19). Hence, an absorption reactor filled with the microemulsion is more efficient than one with pure water or surfactant. When using a microemulsion for the absorption of VOCs, the VOCs can be recovered by breaking microemulsion-stabilized. The selection of a suitable microemulsion is generally made by considering surfactant types, cosurfactant types, mass ratios of surfactant to cosurfactant, temperature, and brine. In this study, ordinary cationic surfactants, such as myristyltrimethylammonium bromide (MTAB), cetyltrimethylammonium bromide (CTAB), and stearyl trimethyl ammoium bromide (STAB), were selected as surfactants, and alcohols, acids, and short-carbon chain quaternary ammonium salts act as cosurfactants. Using the region of the microemulsion in the phase diagram as the indicator, the main influences—cosurfactant types, mass ratios of surfactant to cosurfactant, temperature, and brine—of the solubilization capacity of the various systems were investigated. The

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Address correspondence to Senlin Tian, Faculty of Environmental Science and Engineering, Kunming University of Science and Technology, Kunming, Yunnan 650093, China. Tel.: 86-871-5170906; Fax: 86-871-5170906. E-mail: tiansenlin@yahoo.com.cn; tiansenlin@yahoo.com

absorption rate and the absorption capability of distilled water, CTAB solution, and microemulsion composed of CTAB and TBAB were investigated and compared.

MATERIALS AND METHODS

Materials

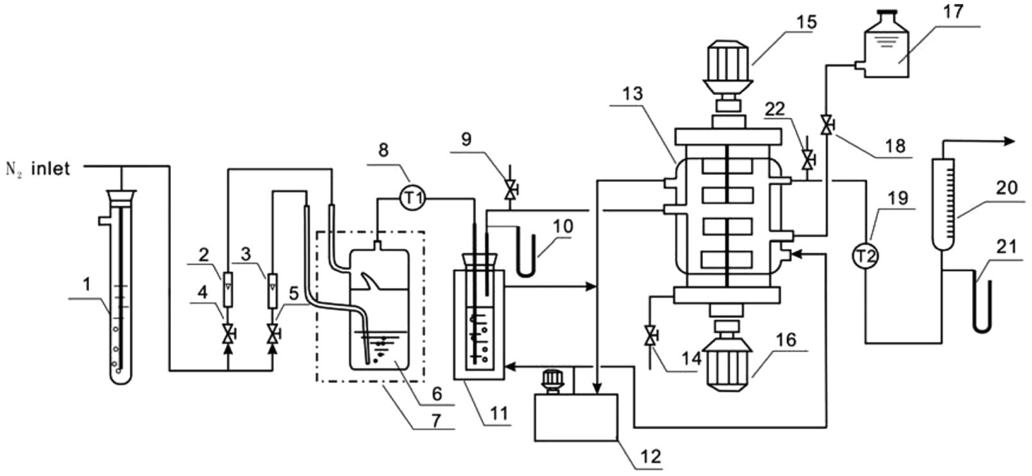
CTAB, n-butyric acid, n-hexylic acid, n-octanoic acid, NaCl, and toluene were purchased from Sinopharm Chemical Reagent Co. Ltd. MTAB, STAB, tetramethylammonium bromide (TMAB), tetraethylammonium bromide (TEAB), and tetrabutylammonium bromide (TBAB) were obtained from Aladdin Reagent Co. Ltd., and n-butanol and n-octanol were bought from Shanghai Chemical Reagent Co. Ltd. All reagents are analytical reagent grade and used as received.

Method of Determination of Pseudo-Ternary Component Phase Diagrams

CTAB, MTAB, STAB were chosen as the surfactants to form the microemulsion. The pseudo-ternary-component phase diagrams of the surfactant/cosurfactant/toluene/water (brine) systems were studied by the dilution method (20). The transparent regions of the microemulsion were determined by titration of toluene into a mixture of surfactant, cosurfactant, and water or brine with various concentrations, to a total of 2 g. The phase diagrams were developed methods previously used (20,21). On addition of a single drop of toluene, the altered weight of the system was recorded and the sample was shaken vigorously for approximately 30s. The phase behavior was affected by various factors, such as temperature (293 K, 303 K, 313 K, and 323 K), the chain length of the surfactant and cosurfactant, the type of cosurfactant, the ratio of surfactant to cosurfactant, and concentration of brine in the microemulsions.

Experiments on Toluene Absorption

Experiments on toluene absorption were carried out in a highly characterized double-stirred reactor with a planar gas–liquid interface. The double-stirred reactor had a gas–liquid interface that was planar to a close approximation (22,23). The reactor was 61 mm in diameter and equipped with four vertical baffles and two stirring blades. A water jacket was used to maintain the desired temperature by circulating water from a water bath at a constant temperature. The reactor had a continuous gas flow rate of $300\,\mathrm{mL \cdot min^{-1}}$. Toluene was diluted by carrier air, with the inlet concentration of toluene in this experiment $2000\,\mathrm{mg \cdot m^{-3}}$. All the experiments were conducted with a liquid phase stirring speed (N_{liquid}) of $100\ \mathrm{rev \cdot min^{-1}}$ and gas phase stirring speed (N_{gas}) of $250\ \mathrm{rev \cdot min^{-1}}$. As shown in Fig. 1, the experimental setup includes an organic compound generation system and a double-stirred reactor. The volume of the aqueous solution in the reactor was maintained at 250 mL. All the experiments were performed at



1. Regulator 2,3. Gas flowmeters; 4,5. Gas control valve; 6. Toluene vapor generator; 7. Water bath; 8. Inlet thermometer 9. Gas sampling point (inlet); 10. Manometer; 11. Water saturator; 12. Water bath; 13. Stirred cell reactor; 14. Absorbent sampling point 15,16. Driving motors; 17. Absorbent bottle; 18. Absorbent control valve; 19. Exit thermometer 20. Soap film flowmeter; 21. Manometer; 22. Gas sampling point (outlet).

FIG. 1. Experimental setup for toluene absorption.

303 K in a stirring reactor and operated in batch mode. The inlet and outlet gas concentrations of toluene were analyzed by gas chromatography (GC522, Shanghai Wufeng) with a flame ionization detector (FID). The column, injector, and detector temperatures were set at 363 K, 393 K, and 423 K, respectively. From the graphical integration of the data of the gas chromatograph peaks to quantify toluene in the gas phase at the inlet and outlet, the efficiency of removal of toluene was calculated. The sampled gas was injected through a six-way valve to the analyzers.

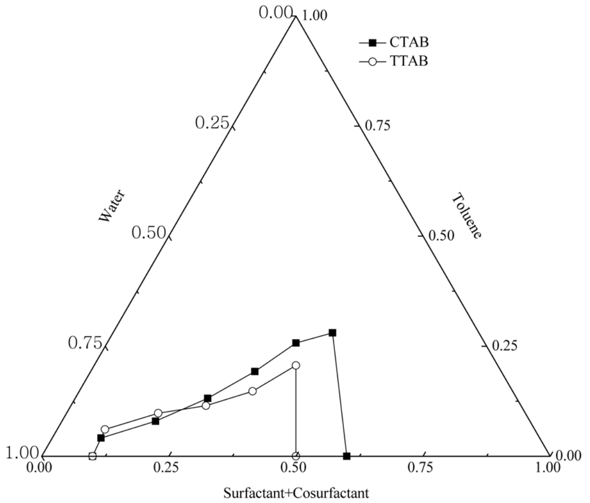
RESULTS AND DISCUSSION

Influencing Factors of the Phase Diagram

The solubilization behavior of the microemulsion can be illustrated with the phase diagram, and the solubilization capacity of microemulsion can be measured by the phase diagram. The lower part of the phase boundary represents a single-phase microemulsion region, whereas the upper part is a two-phase region.

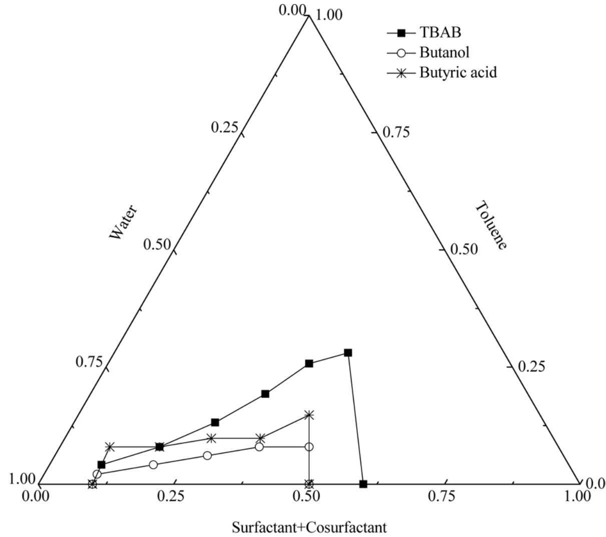
To better understand the role that the surfactant chain length plays in the solubilization capacity of the microemulsions, three kinds of cationic surfactants with various lengths of carbon chains were studied. CTAB is one of the most commonly used cationic surfactants, and CTAB, MTAB, and STAB have the same hydrophilic group, with the hydrocarbon chain length of MTAB, CTAB, and STAB 14, 16, and 18, respectively. MTAB, CTAB, and STAB were chosen as surfactants and TBAB was used as cosurfactant. The mass ratio of the surfactant to the cosurfactant was kept constant at 1:1 and the temperature at 303 ± 0.1 K. The phase diagrams are shown in Fig. 2. There is a conspicuous difference in the solubilization capacity of the microemulsions with an increase in the hydrocarbon chain length. But when STAB is used as the surfactant, microemulsions cannot form. The longer the hydrocarbon chain of the surfactant, the lower the tension of the interface is, so microemulsions based on surfactants with long hydrocarbon chains can form more easily, although a hydrocarbon chain which is too long would lead to the formation of a microemulsion of water-in-oil (W/O) type rather than oil-in-water (O/W) type. Because the region of the microemulsion was smaller when the surfactant had a smaller or greater hydrocarbon chain length, CTAB can be feasible as a candidate surfactant due to its appropriate hydrocarbon chain length. In this case, the greatest solubilization of toluene is as high as $280.42\,\mathrm{mg \cdot L^{-1}}$.

The cosurfactant acts as an "electronegative spacer" that minimizes repulsion between the positively charged surfactant heads (24), so that they could reduce the surface tension and increase the flexibility of the interface and lead to the spontaneous formation of microemulsions (25). The effect of the cosurfactant types on the phase behavior was investigated by testing the cosurfactants, including n-butanol, n-octanol, n-butyric acid, n-hexylic acid, n-octanoic acid, and TBAB. CTAB was chosen as a surfactant, with the mass ratio of the surfactant to the cosurfactant kept constant at 1:1 and the temperature at 303 ± 0.1 K (Fig. 3). The longer chain cosurfactant, such as n-octanol, n-hexylic acid, and n-octanoic acid, were unfavorable to O/W microemulsion formation. The region of O/W microemulsion of TBAB was larger than that of n-butyric acid and n-butanol. The main reason could be that since TBAB and butyric acid have an electronic charge, they could reduce the surface tension more effectively.



CTAB (n_s=16) / TBAB / toluene/ water system (■), TTAB (n_s=14) / TBAB / toluene / water system (○)

FIG. 2. Effect of chain length of the surfactants in the region of the microemulsion.



CTAB / TBAB / toluene/ water system (■), CTAB / Butanol / toluene / water system (○), CTAB / Butyric acid / toluene/ water system (✳)

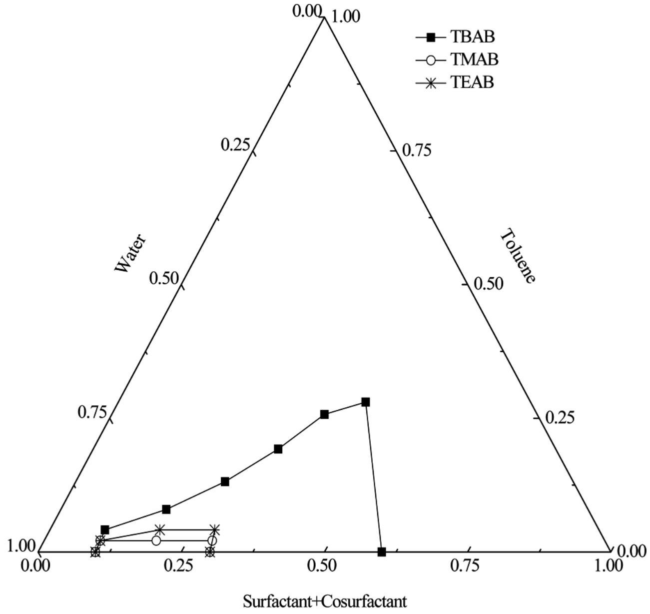
FIG. 3. Effect of cosurfactant types in the region of the microemulsion.

On the other hand, TBAB and CTAB have the same hydrophilic group, and the emulsification of TBAB was more efficient than for other compounds. Thus quaternary ammonium salts, such as TBAB, with shorter hydrocarbon chains are good candidates for use as cosurfactants.

Chain length compatibility of the surfactant, the cosurfactant, and the oil is a very important factor in the formation of microemulsions (26–28). The Bansal–Shah–O'Connell (BSO) equation shows that the maximum water solubility for ionic microemulsions occurs when $n_a + n_o = n_s$, where n_a is the length of the hydrocarbon chain in the cosurfactant (carbon number), n_o is the length of the hydrocarbon chain in the oils, and n_s is the length of the hydrocarbon chain in the surfactant (27). Shiao et al. reported that the BSO equation should be used with caution when studying systems with branched components (29). One of the limitations of the validity of this equation is that the oil molecules should have a straight chain structure (30). For our systems, CTAB was selected as the surfactant, and three kinds of cosurfactants were uses TMAB, TEAB, and TBAB. The mass ratio of the surfactant to the cosurfactant was kept constant at 1:1, and the temperature was $303 \pm 0.1\,\mathrm{K}$. The phase diagrams are shown in Fig. 4. When TMAB or TEAB were used as cosurfactants, there was little difference in the O/W microemulsion region, but when TBAB was chosen as a cosurfactant there was an obvious difference in the solubilization capacity of the microemulsions due to an increase in the hydrocarbon chain length. According to the BSO equation, when $n_s = 16$ for CTAB and $n_o = 1$ for toluene, the maximum solubilization should occur when n_a is 15. However, maximum solubilization was observed at $n_a = 4$ for TBAB. One possible explanation is that as the hydrocarbon chain length of the cosurfactant exceeds 4 it easily forms W/O rather than O/W microemulsions. The conclusion that could be drawn is that the BSO equation is not applicable to systems which only form O/W type microemulsions. Thus TBAB was the most suitable cosurfactant.

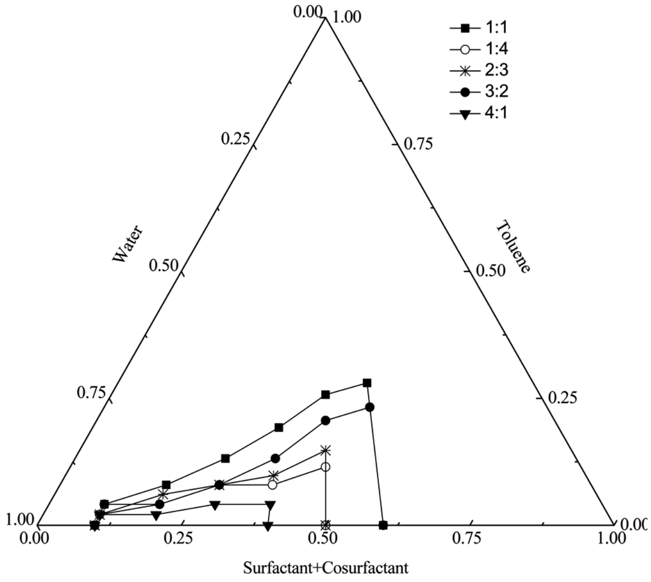
Although cosurfactants could reduce surface tension, the interface flexibility would be only slightly reduced if the mass ratio of the surfactant to the cosurfactant was not optimum. When the concentration of the cosurfactant exceeds the optimum value, the cosurfactant would be in the continuous phase (31). The effect of CTAB/TBAB/toluene/water systems on the phase diagrams was determined by the five mass ratios of the surfactant and the cosurfactant used: 1:4, 2:3, 1:1, 3:2, and 4:1 (Fig. 5). It was observed that the phase areas of the microemulsion were smaller when the mass ratio of the surfactant to the cosurfactant were 1:4, 2:3, 3:2, and 4:1. In summary, a system that includes a 1:1 mass ratio of the surfactant to the cosurfactant forms a better single-phase region than systems with other ratios. The optimum mass ratio of surfactant to cosurfactant of microemulsion systems was found to be 1:1.

Temperature is essential to the stability of microemulsions, as increasing temperature is known to increase solubility of cationic surfactants. The effect of temperature on the phase behavior of these systems was investigated



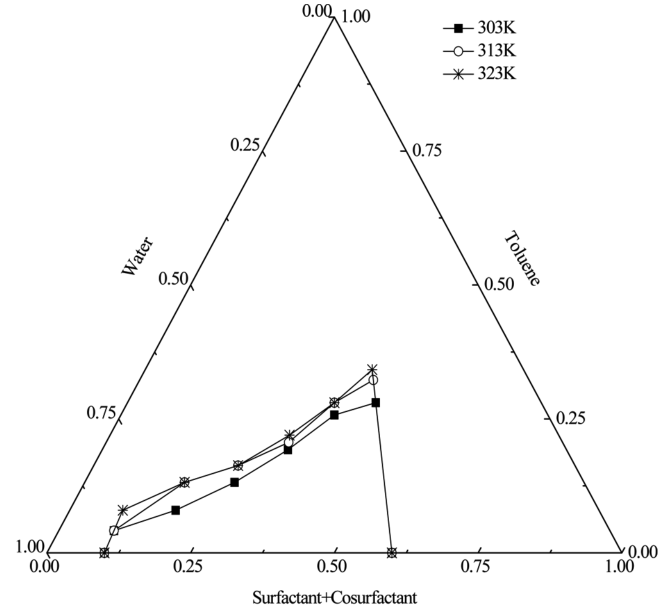
CTAB / TBAB (n_a=4) / toluene/ water system (■), CTAB / TMAB (n_a=1) / toluene / water system (○), CTAB / TEAB(n_a=2) / toluene/ water system (✳)

FIG. 4. Effect of chain length of the cosurfactant in the region of the microemulsion.



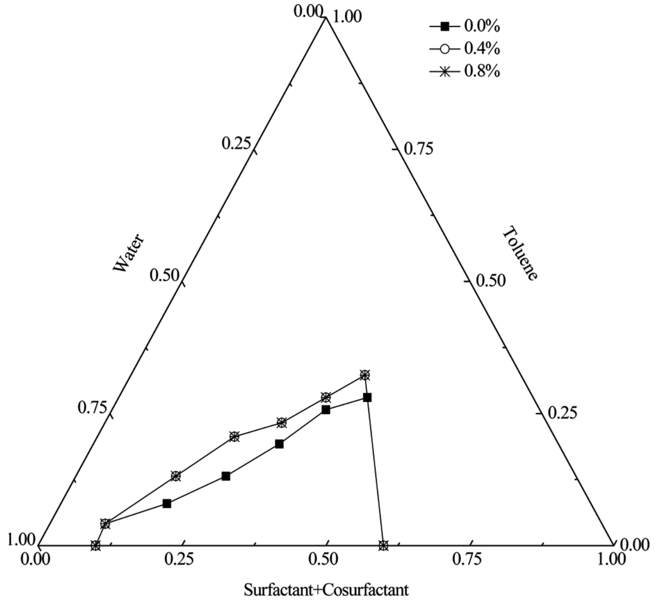
CTAB / TBAB (n_a=4) / toluene/ water system, the five surfactant/cosurfactant ratio in experimental conditions are 1:1 (■), 1:4 (○), 2:3 (✳), 3:2 (●), and 4:1 (▼)

FIG. 5. Effect of surfactant to cosurfactant ratio in the region of the microemulsion.



CTAB / TBAB / toluene/ water system, the three temperature in experimental conditions are: 303K (■), 313K (○), 323K (✳)

FIG. 6. Effect of temperature in the region of the microemulsion.



CTAB / TBAB / toluene/ water system, the three brine (solution of NaCl) with different concentration (m/v) in experimental conditions are: 0.0% (■), 0.4% (○), 0.8% (✳)

FIG. 7. Effect of concentration of brine in the region of the microemulsion.

(Fig. 6). The CTAB/TBAB/toluene/water system with mass ratio of surfactant to cosurfactant of 1:1 was investigated at 293 K, 303 K, 313 K, and 323 K. The microemulsion did not form at 293 K. According to Fig. 6, as the temperature increased, the solubilization capacity of the microemulsions increased. However, in this study, the microemulsion system does not absorb more toluene at higher temperatures. This may be because toluene vaporizes more with an increase in temperature. Making an allowance for the cost, 303 K was the most economical option for the absorption temperature.

Brine could increase the concentration of ions and, consequently, the conductivity of the solution to lead to a spontaneously formed microemulsion. The CTAB/TBAB/toluene/water system at a temperature of 303 ± 0.1 K with a mass ratio of the surfactant to the cosurfactant of 1:1 with different concentrations of brine was investigated. The results showed that, given the experimental constraints, the concentration of brine had a little effect on the phase behavior (Fig. 7). For concentrations of NaCl of 0.4% and 0.8%, the regions of the microemulsions were the same. This can be ascribed to the compression of an electric double layer and the decreasing of water permeability resulting from the elevated concentration of the salt ions. Consequently, the drops of microemulsion would assemble and the amount of microemulsion drops in a unit volume would increase. Correspondingly, the region of microemulsion of the O/W type is enlarged and that of liquid crystal is reduced (32).

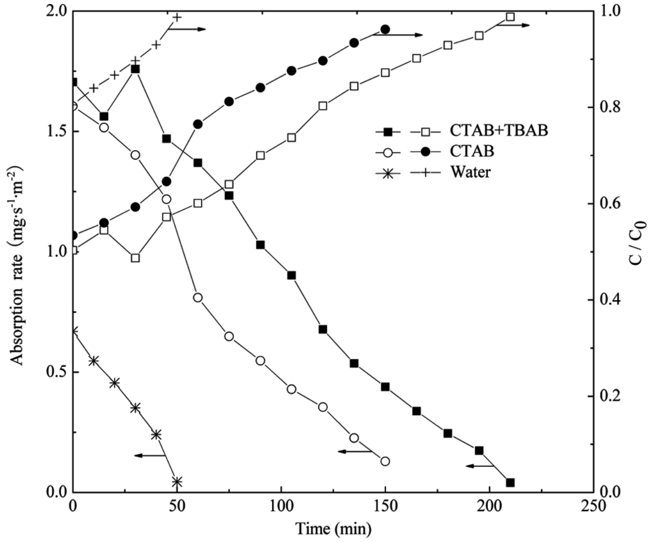
Comparison of Toluene Absorption Rate and Ratio of Inlet to Outlet Concentration by Various Absorbents

To search for evidence of absorption of toluene by a microemulsion, using the absorption rate and the ratio of the inlet to the outlet concentration as an indicator, distilled water, a solution of CTAB, and the microemulsion formed by CTAB and TBAB were tested in the double-stirred reactor. The absorption rate of toluene can be expressed as:

$$N_{\mathrm{A}} = (C_0 - C) \cdot Q/A$$

where N_{A} is the absorption rate ($\mathrm{mg \cdot s^{-1} \cdot m^{-2}}$), C_0 is the inlet concentration of toluene ($\mathrm{mg \cdot m^{-3}}$), C is the outlet concentration of toluene ($\mathrm{mg \cdot m^{-3}}$); Q is the inlet flow rate ($\mathrm{m^3 \cdot s^{-1}}$) and A is the mass transfer of the gas-liquid interface ($2.926 \times 10^{-3}\,\mathrm{m^2}$) (33). The ratio of the inlet to the outlet concentration can be expressed as C/C_0.

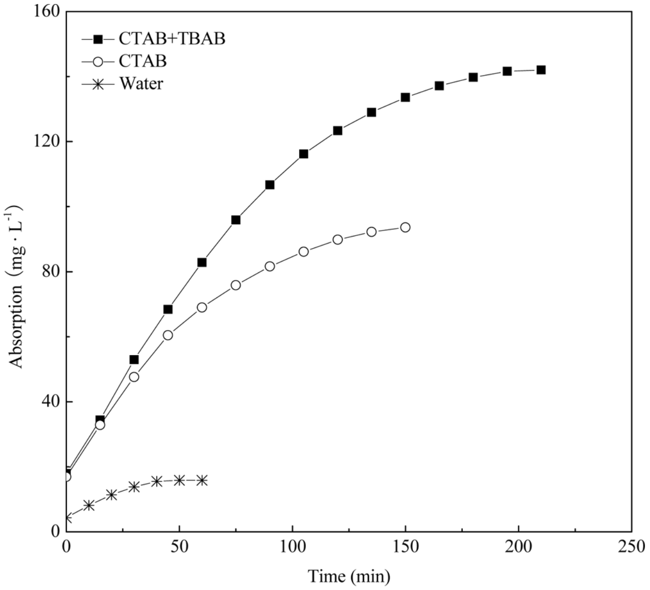
The results of the comparison of the absorption rate and the ratio of the inlet to the outlet concentration of toluene are summarized in Fig. 8. The data from the double-stirred reactor indicates that the microemulsion formed by CTAB and TBAB has the highest absorption rate in experimental conditions, with the order of toluene absorption rate being the greatest for microemulsion, followed by that for CTAB and then H_2O. When only the CTAB solution was used to absorb toluene, only micelles form, and the absorption of toluene was comparable to physical absorption. Thus, in the CTAB solution, the micelle absorption capacity of



Absorption rate (-■-, -○-, - * -) and C/C_0(-□-, -●-, - + -)

N_{liquid}=100 r/min, N_{gas}=250 r/min, C_0=2000 mg·m^{-3}, T=303K

FIG. 8. Comparison of absorption rate and the ratio of inlet to outlet concentration for various absorbents.



N_{liquid}=100 r/min, N_{gas}=250 r/min, C_0=2000 mg·m^{-3}, T=303K

FIG. 9. Comparison of toluene absorption capability for various absorbents.

toluene decreased quickly, as did the toluene removal efficiency. After 150 min, all the CTAB micelles were saturated with toluene, and the removal efficiency of toluene decreased to zero. However, the solution containing CTAB and TBAB could form a microemulsion which had a solubilization capacity greater than that of the micelle. The lower surface tension of the microemulsion (at 303 K, the surface tension is 29.88 mN · m^{-1} and of CTAB solution is 44.00 mN · m^{-1}) allows for greater mass transfer at the gas–liquid interface. Therefore a more stable and reliable process could be achieved. The final removal time was up to 225 min under these operating conditions. When $C/C_0 = 1$, the absorbent was saturated with toluene.

Comparison of Toluene Absorption Capacity by Various Chemical Adsorbents

In order to investigate the absorption abilities of chemical reagents used in the experiments, comparison tests were performed in the same reactor as those using distilled water, CTAB, and microemulsion formed from CTAB and TBAB (Fig. 9).

In the first 50 min, the absorption capacity of CTAB and the microemulsion were similar, but after 50 min the absorption capacity of the microemulsion was greater than for CTAB. The microemulsion which formed from CTAB and TBAB has a higher absorption capacity in experimental conditions, and the absorption capacity of toluene is greatest for the microemulsion, followed by CTAB then H_2O. At the initial stage of absorption, the solubilization capacity of the system to toluene primarily derive from micelles composed of CTAB and TBAB, equal approximately to the solubilization capacity of the CTAB solution. With the progress of the absorption process, toluene adsorbed into solution would participate in the formation of microemulsion as the oil phase. As a result, the solubilization capacity of the microemulsion is greater than that of the surfactant solution; microemulsion solubilization absorption of toluene is the greatest of the three, but the absorption capacity of the microemulsion (142.00 mg · L^{-1}) is far less than the highest solubilization (280.42 mg · L^{-1}). The reason is that the mass transfer resistance of the gas–liquid interface and the equilibrium of toluene in the gas and liquid phases have a large influence on absorption process.

SUMMARY

This investigation focused on optimizing the absorption of the microemulsion system to solubilize toluene. Three cationic surfactants were investigated to select the absorbent which recovers toluene from a gas stream most efficiently. CTAB was determined to be the more suitable at 303 K with the cosurfactant TBAB with an optimum surfactant to cosurfactant ratio of 1:1.

The experiments showed that microemulsions formed from CTAB and TBAB had the optimal toluene absorption rate and capacity, followed by CTAB and distilled water.

We concluded that cationic surfactant microemulsions are easy to prepare from a variety of cosurfactants, and form microemulsions with different solubilization capacities—CTAB/TBAB/toluene/water microemulsions had significant ability in the solubilization of toluene. Such systems successfully absorbed toluene for 225 min where the concentration of toluene was 2000 $\mathrm{mg \cdot m^{-3}}$ or less. Therefore there is great potential to develop microemulsions which can remove VOCs (especially toluene) by absorption.

ACKNOWLEDGEMENTS

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