This article was downloaded by:
On: *25 January 2011*
Access details: *Access Details: Free Access*
Publisher *Taylor & Francis*
Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK

Separation Science and Technology

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

Surfactant-Enhanced Carbon Regeneration in Liquid-Phase Application

Anawat Bhummasobhana[a]; John F. Scamehorn[b]; Somchai Osuwan[a]; Jeffrey H. Harwell[b]; Shooshat Baramee[a]
[a] THE PETROLEUM AND PETROCHEMICAL COLLEGE CHULALONGKORN UNIVERSITY, BANGKOK, THAILAND [b] UNIVERSITY OF OKLAHOMA, INSTITUTE FOR APPLIED SURFACTANT RESEARCH, NORMAN, OKLAHOMA, USA

To cite this Article Bhummasobhana, Anawat , Scamehorn, John F. , Osuwan, Somchai , Harwell, Jeffrey H. and Baramee, Shooshat(1996) 'Surfactant-Enhanced Carbon Regeneration in Liquid-Phase Application', Separation Science and Technology, 31: 5, 629 — 641

To link to this Article: DOI: 10.1080/01496399608000708

URL: http://dx.doi.org/10.1080/01496399608000708

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

SEPARATION SCIENCE AND TECHNOLOGY, **31**(5), pp. 629–641, 1996

Surfactant-Enhanced Carbon Regeneration in Liquid-Phase Application

ANAWAT BHUMMASOBHANA
THE PETROLEUM AND PETROCHEMICAL COLLEGE
CHULALONGKORN UNIVERSITY
BANGKOK, THAILAND

JOHN F. SCAMEHORN*
INSTITUTE FOR APPLIED SURFACTANT RESEARCH
UNIVERSITY OF OKLAHOMA
NORMAN, OKLAHOMA 73019, USA

SOMCHAI OSUWAN
THE PETROLEUM AND PETROCHEMICAL COLLEGE
CHULALONGKORN UNIVERSITY
BANGKOK, THAILAND

JEFFREY H. HARWELL
INSTITUTE FOR APPLIED SURFACTANT RESEARCH
UNIVERSITY OF OKLAHOMA
NORMAN, OKLAHOMA 73019, USA

SHOOSHAT BARAMEE
THE PETROLEUM AND PETROCHEMICAL COLLEGE
CHULALONGKORN UNIVERSITY
BANGKOK, THAILAND

ABSTRACT

In surfactant-enhanced carbon regeneration, a concentrated surfactant solution is passed over spent activated carbon to desorb the solutes in an in-situ process. The solutes solubilize into micelles in the regenerant solution. Residual adsorbed surfactant can be removed by a water flush. In this study, phenol is the solute

---

* To whom correspondence should be addressed.

and sodium dodecyl sulfate is the anionic surfactant used. Previous studies have measured the ability of the regenerant to remove the solute from the carbon. This study presents the breakthrough curves for subsequent adsorption cycles following the regeneration. The reduction of the effective carbon adsorption capacity is less if a higher regenerant concentration of surfactant, more pore volumes of regenerant, or more pore volumes of water flush solution are used. Effective adsorption capacities of over 50% of that on virgin carbon were observed on regenerated carbon.

INTRODUCTION

Activated carbon is the most widely used adsorbent today. It is usually categorized as a physical, nonpolar adsorbent. It is often used to remove less polar contaminants from polar bulk streams, usually water. A major advantage of activated carbon adsorption over some other separation techniques is the ability to remove organics at low concentrations (1–3).

In practice, the adsorption process proceeds until the solute concentration in the outlet reaches an unacceptable level, at which time the carbon needs to be regenerated. A common regeneration method is thermal reactivation which is carried out in a multihearth furnace or a rotary kiln at 870–980°C. However, sometimes thermal reactivation may not be feasible. This may be due to inorganic salts having deposited on activated carbon or adsorbates which may cause air pollution upon regeneration (e.g., PCBs), among other reasons (4). Of course, the solute is not recovered in thermal regeneration. A large fraction of the carbon may be burned in the furnace (5, 6).

Solvent regeneration involves the dissolution of the adsorbate into a volatile solvent (e.g., acetone) (7), followed by a steam or hot gas flush to remove residual solvent from the carbon (5, 6). In biological regeneration (8), bacteria are introduced into the bed to consume the adsorbed organic. However, the process is quite slow and complete regeneration may not be achieved, so thermal regeneration may be required periodically (5).

Surfactant-enhanced carbon regeneration (SECR) involves the flushing of concentrated surfactant solution (regenerant) through the spent carbon. Organic adsorbate desorbs and is solubilized into micelles (surfactant aggregates typically containing 50–100 molecules) in the regenerant solution. When the desorption process is complete, some residual adsorbed surfactant may be left on the activated carbon, while the regenerant stream contains concentrated solute and can be further treated to recover and recycle the surfactant (9, 10). A water flush is then used to remove residual surfactant from the carbon, leaving the carbon ready for reuse (for vapor

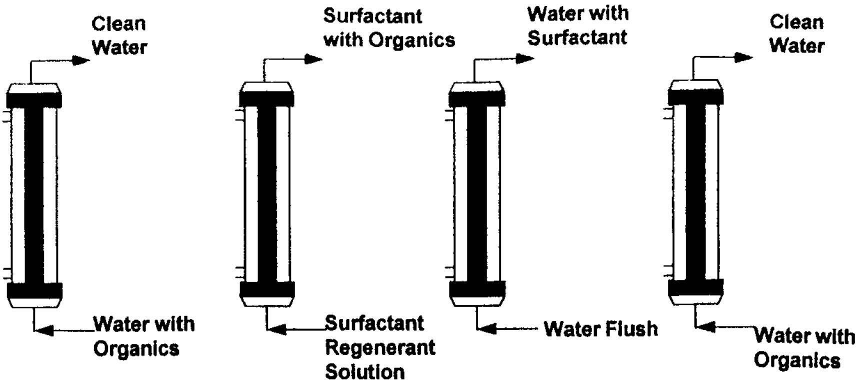

FIG. 1 Process strategy for surfactant-enhanced carbon regeneration.

phase applications, a drying step is needed). The process strategy is shown in Fig. 1.

Previous studies on SECR (11, 12) have demonstrated that the technique can give excellent removal of the adsorbate with a sharp breakthrough curve during regeneration. However, for liquid-phase applications, the residual surfactant on the carbon following the regeneration/water flush can substantially reduce the adsorption capacity of the bed on subsequent cycles. The purpose of this investigation is to determine the effect of regeneration and flushing conditions on the breakthrough of the solute in subsequent adsorption cycles, and hence, on effective adsorption capacity upon repeated use.

BACKGROUND

At low surfactant concentrations in water, all the surfactant exists as unaggregated molecules (monomer). At a specific concentration, the critical micelle concentration or CMC, surfactant aggregates called micelles first form. At concentrations above the CMC, the monomer concentration remains approximately constant while almost all additional surfactant added forms micelles. The surfactant used here (sodium dodecyl sulfate or SDS) has a CMC of 8.3×10^{-3} M and forms roughly spherical micelles containing about 70 surfactant molecules (13, 14). The micelles have a hydrophobic environment in the interior where alkyl chains of the surfactant form an oil-like liquid droplet. The charged groups of the SDS are on the surface of the micelles. Organic solutes in the solution tend to concentrate in the micellar interior, a phenomena called solubilization. The solubility of an organic can be many times higher in a surfactant solution above the CMC than in just water due to solubilization (14–16).

In SECR, the regenerant solution contains surfactant above the CMC. The adsorbed solute solubilizes into the micelles in the regenerant and is desorbed as a result. In previous work, Blakeburn and Scamehorn (11) found that the first approximately 80% of *tert*-butylphenol can be removed using a regenerant stream containing a cationic surfactant with a reasonable volume of regenerant, but a heel of adsorbed solute is very difficult to remove. The majority of the residual surfactant can be fairly easily removed by a water flush, but large volumes of water are needed for nearly complete removal of surfactant from the carbon. The effective adsorption capacity of the carbon upon subsequent cycles was found to be reduced by the regeneration technique, although detailed breakthrough curves were not obtained.

In a vapor-phase application, Roberts et al. (12) studied the application of SECR to regenerate carbon containing either toluene or amyl acetate. Using SDS, the regeneration step and water flushing to remove residual surfactant were effective. Unlike the aforementioned liquid-phase application, in this vapor-phase case, little reduction in adsorption capacity of the carbon was observed after regeneration, but detailed breakthrough curves were not generated. Roberts et al. (12) also found that the regeneration step was equilibrium limited for removal of toluene, but some mass transfer effects were present for the higher molecular weight amyl acetate. The water flush was found to be nearly equilibrium limited.

These previous studies have demonstrated the general feasibility of SECR in both liquid- and vapor-phase applications. The purpose of this study is to measure breakthrough curves for adsorption of a model solute (phenol) in an adsorption cycle following a regeneration/flush process under varying conditions to quantify the effect of the regeneration process on effective adsorption capacity on repeated usage.

EXPERIMENTAL

Materials

The surfactant was sodium dodecyl sulfate (SDS) from Kao Industrial Co. with a purity of 95%. The solute used was phenol with a purity of 99.5% manufactured by Carlo Erba. The activated carbon was Filtrasorb 300 from Calgon Co. with a surface area of 950–1050 m^2/g.

Methods

The column used for adsorption studies was solvent-resistant with 2.5 cm ID and 120 cm long from Rainin. It was jacketed for temperature control at 25°C. An adjustable plunger was inserted to minimize the void volume existing in the bed. Liquid was pumped through the bed at a flow rate from 10 to 100 mL/min. A schematic diagram of the equipment used in this experiment is shown in Fig. 2.

The effluent concentration of phenol during all steps was detected by a UV spectrometer at a wavelength of 269 nm. The effluent SDS concentration was analyzed by a conductivity meter.

In order to desalt the carbon prior to use, 58 g carbon was boiled in deionized water to completely wet the pores. After the water was decanted, the carbon was flushed with approximately 10 L deionized water, after which it was packed into the column.

The phenol used for loading the carbon was prepared by dissolving 40 mg/L phenol in deionized water. It was fed into the column at a flow rate of 100 mL/min in an upflow direction until the effluent concentration reached the feed concentration. A mass balance on the breakthrough curve was done to determine the total amount of phenol adsorbed.

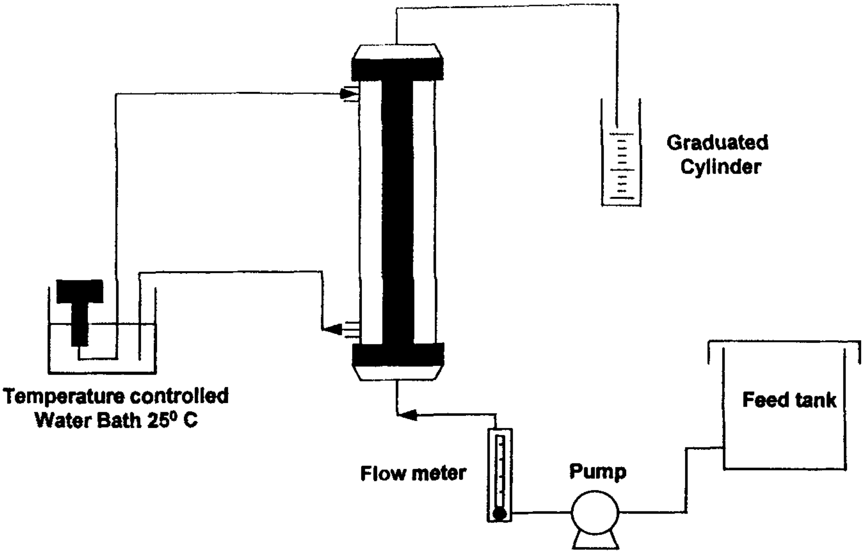

FIG. 2 Schematic diagram of experimental apparatus.

In the regeneration step, solutions of SDS were pumped into the adsorption bed at a flow rate of 20–40 mL/min in an upflow direction. Effluent samples were analyzed to determine both SDS and phenol concentrations.

In the flush step, deionized water was passed through the bed at a flow rate of 100 mL/min. After completion of the flush, the activated carbon was used for reloading the phenol to measure the breakthrough curve in this subsequent adsorption step.

RESULTS AND DISCUSSION

The breakthrough curve for the adsorption of phenol on unregenerated carbon is shown in Fig. 3. The pore volume which corresponds to the volume of liquid contained in an adsorber bed filled with water is 0.85 mL/g carbon. For a feed concentration of 40 mg/L phenol (case used to prepare bed for all regenerations), a flow rate of 100 mL/min, and a temperature of 25°C, saturation occurred at around 3000 per volumes.

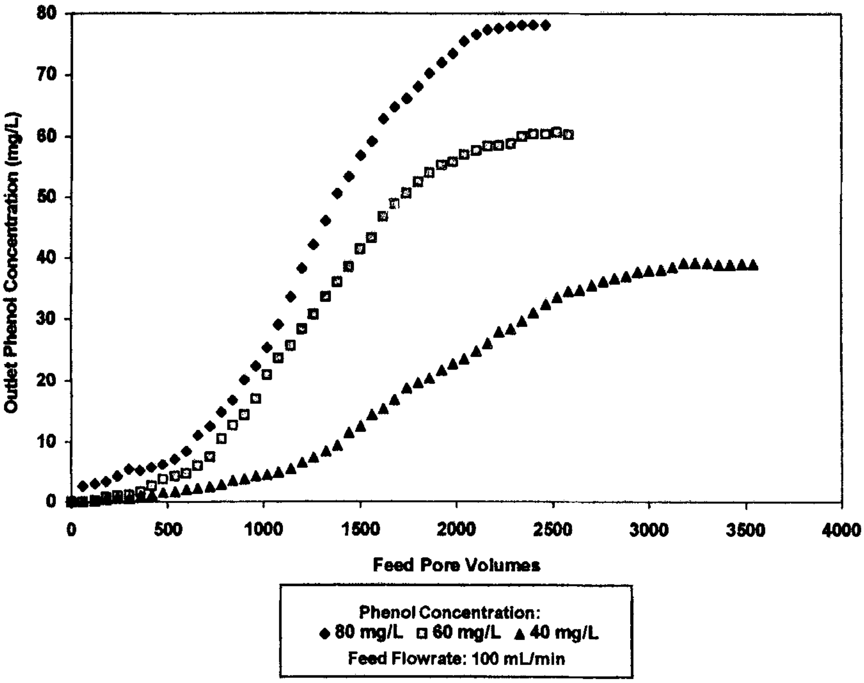

FIG. 3 Breakthrough curves at several phenol feed concentrations.

The equilibrium amount of phenol adsorbed was calculated from a mass balance on the breakthrough curve and was equal to 4.994 g or 85 mg/g carbon, which is in reasonable agreement with the measured loading on the same system of 105 mg/g carbon (17). The loading from the experiment reported in this paper may be less because it is a dynamic measurement, not a static experiment.

The breakthrough curves in Fig. 3 are not as sharp as would be anticipated in a commercial carbon bed because a high flow rate was used. Since the purpose of this study is measurement of the effect of regeneration conditions on breakthrough curves, the comparison is achieved under the conditions used.

The effect of regenerant solution surfactant concentration was examined at a flow rate of 40 mL/min as shown in Fig. 4. The percent phenol recovery results in Fig. 4 can also be presented as a function of SDS in micellar form which has passed through the column rather than pore volume of regenerant, as shown in Fig. 5. The SDS concentration in micellar form is equal to the total surfactant concentration minus the CMC. If the desorption/solubilization process during regeneration is equilibrium

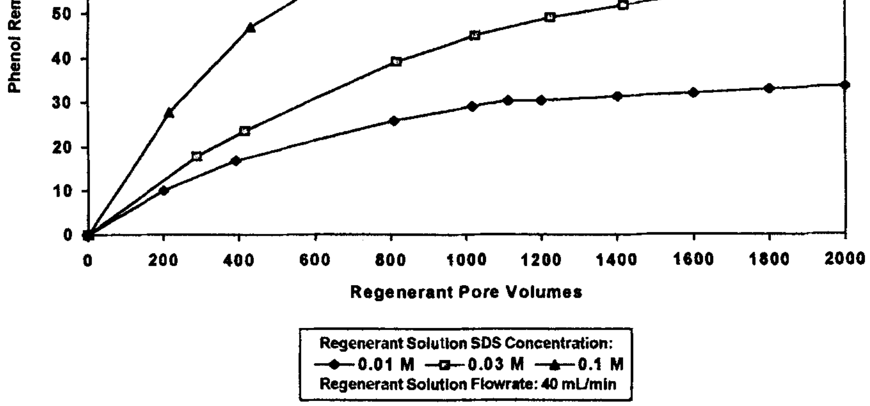

FIG. 4 Effect of SDS concentration in regenerant solution on phenol removal.

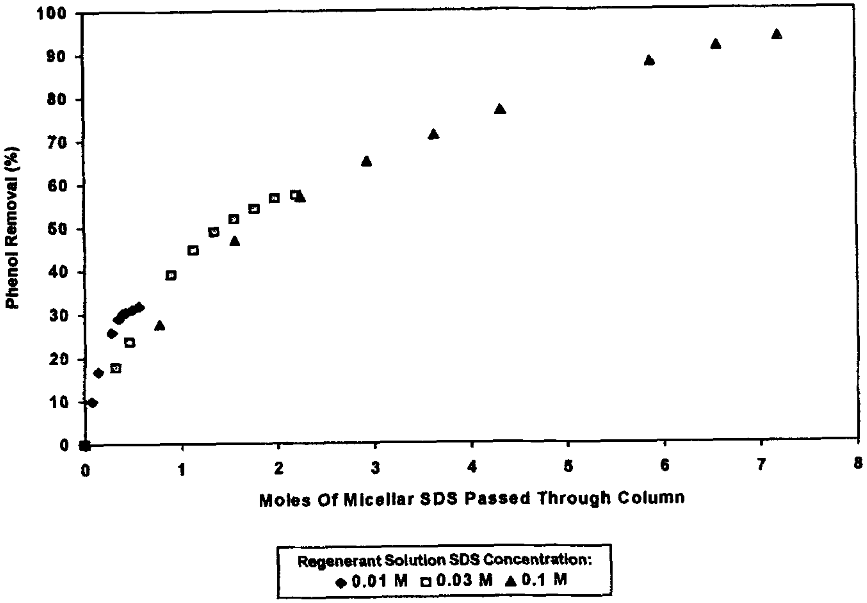

FIG. 5 Effect of amount of SDS in micellar form in regenerant solution on phenol removal.

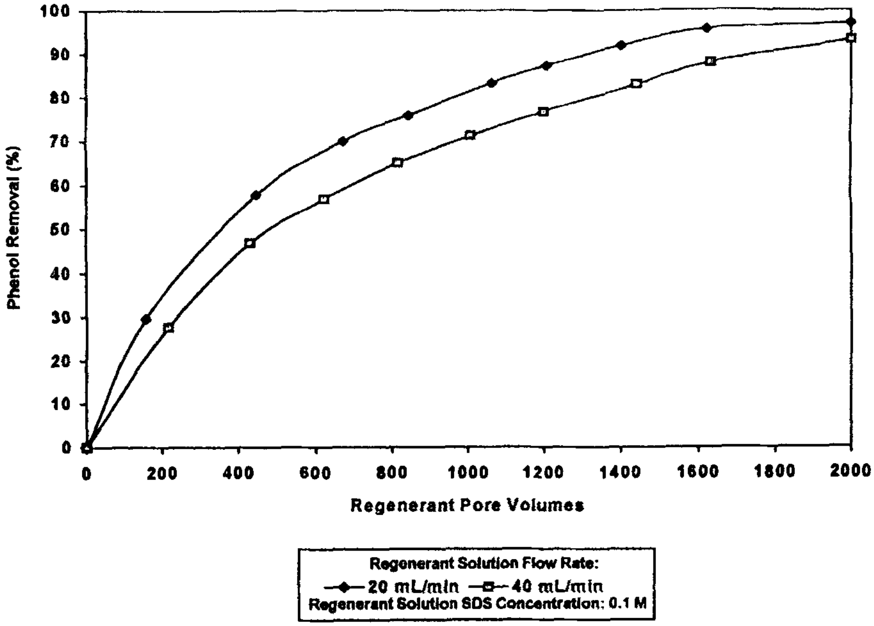

FIG. 6 Effect of regenerant solution flow rate on phenol removal.

limited, then the curves in Fig. 5 at different surfactant (micellar) concentrations should coincide. The small observed effect of surfactant concentration indicates that mass transfer effects are small during regeneration.

For a SDS concentration of 0.1 M, 50% removal could be achieved at 500 pore volumes of regenerant. At 2000 pore volumes, nearly 90% of the phenol could be washed off. At still greater regenerant volumes, the rate of removal was quite slow, probably due to the chemisorbed nature of the remaining phenol. This is consistent with the previous work of Blakeburn and Scamehorn (11), which also illustrated that removal of the last portion of the solute was hard to achieve due to chemisorption.

The effect of regenerant solution flow rate is shown in Fig. 6. More pore volumes are required to regenerate the bed to a specified percent recovery when using a higher flow rate. However, the effect was small at the flow rate used, indicating that regeneration is mainly equilibrium controlled and that mass transfer effects are secondary. This is in agreement with previous work done by Roberts et al. (12).

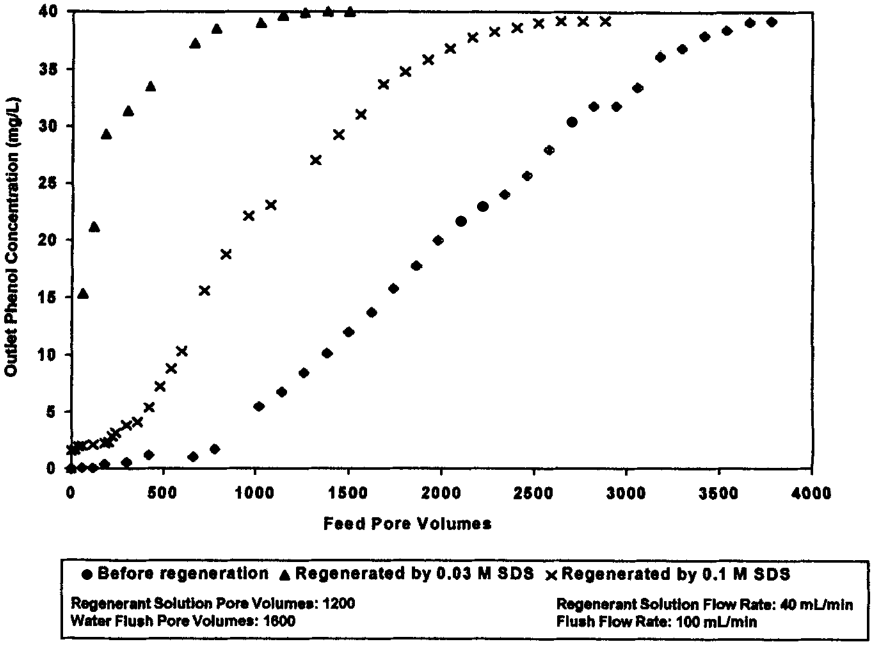

FIG. 7 Effect of SDS concentration in regenerant solution on subsequent breakthrough curve of phenol.

The effect of surfactant concentration during regeneration on the subsequent solute adsorption step is shown in Fig. 7 and of the number of pore volumes of regenerant used in Fig. 8.

The higher the regenerant surfactant concentration or the more pore volumes of regenerant solution used, the better cleaned the carbon is and the more pore volumes of fresh feed which can be treated until a specified level of contaminant in the effluent is detected. The 1200 pore volumes of regenerant used correspond to 78% of the phenol being removed during regeneration from Fig. 4, and 1600 pore volumes correspond to 89% removal. As an example of the effect, if the acceptable level of phenol in the effluent were set to be 5 mg/L (for a feed containing 40 mg/L of phenol), 40% of the fresh feed volume could be treated after regeneration compared to using fresh unregenerated carbon if 1200 pore volumes of regenerate were used. If 1600 pore volumes of regenerant were used, this fraction of fresh feed treated would be 55%.

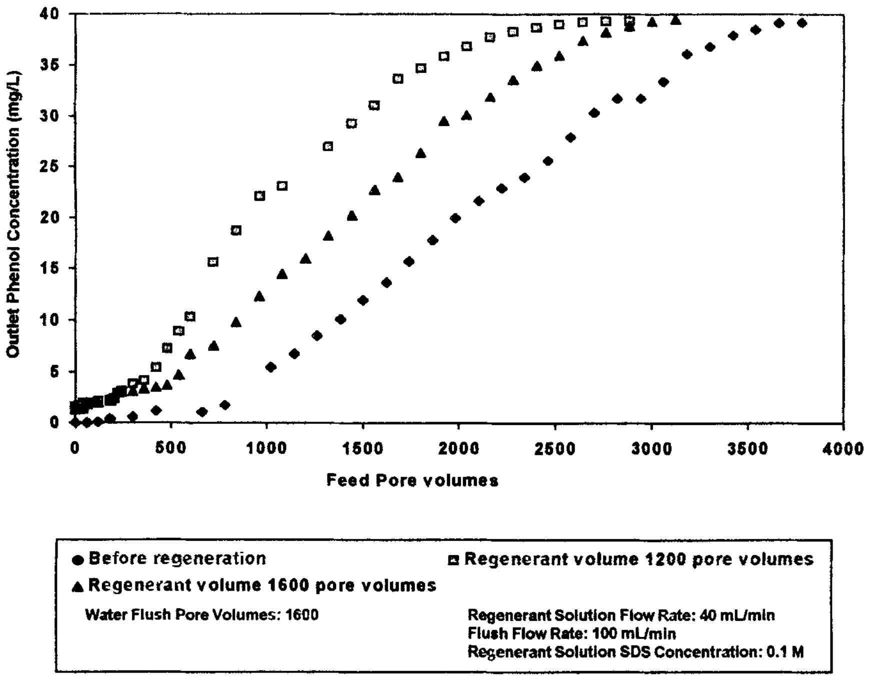

FIG. 8 Effect of volume of regenerant solution used on subsequent breakthrough curve for phenol.

The fraction of surfactant that could be removed during the flushing step is shown in Fig. 9 with a water flush flow rate of 100 mL/min. The regenerant concentration of SDS in the previous regeneration step was 0.1 M. The effect of the severity of the water flush step following regeneration on the subsequent solute breakthrough curve is illustrated in Fig. 10. The more pore volumes of flush solution used and the more surfactant removed from the carbon, the more pore volumes of fresh feed which can be treated until a specified level of contaminant in the effluent is detected. The 1600 pore volumes of water flush used correspond to removal of 75% of the SDS during regeneration from Fig. 9, and 2000 pore volumes correspond to 80% removal. As an example of the effect, if the acceptable level of phenol in the effluent were set to be 5 mg/L, 55% of the fresh feed volume could be treated after regeneration compared to using fresh unregenerated carbon if 1600 pore volumes of flush solution were used. If 2000 pore volumes of flush solution were used, this fraction of fresh feed treated would be 75%.

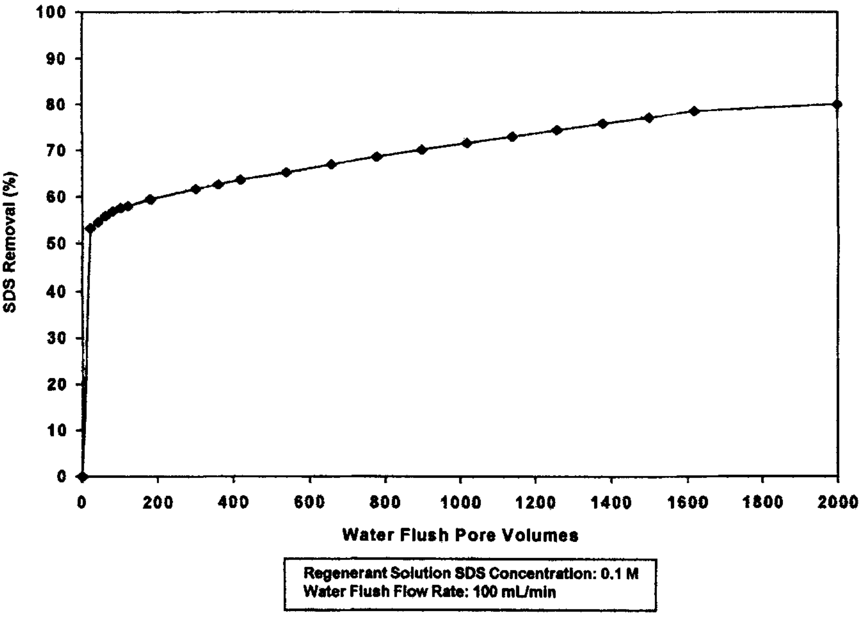

FIG. 9 Effect of volume of water flush on SDS removal.

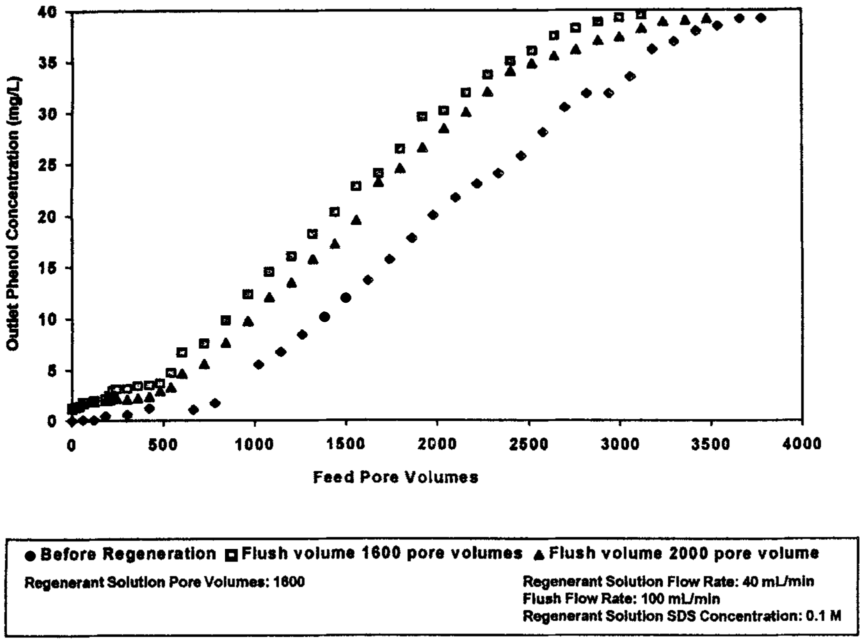

FIG. 10 Effect of volume of water flush on subsequent breakthrough curve of phenol.

CONCLUSIONS

This study has shown that activated carbon regenerated by the SECR process can be effective in subsequent adsorptions. As the volume of the regenerant solution, the regenerant surfactant concentration, and the volume of the flush solution are increased, the effective capacity of the regenerated carbon increases. The optimum degree of regeneration is system-dependent and would also be affected by the surfactant recovery scheme used. An economic optimization study is needed to evaluate how hard the regeneration should be pushed and how the resulting process compares to competitive techniques for specific contaminant removal.

ACKNOWLEDGMENTS

Financial support for this work was provided by National Science Foundation Grant CBT 8814147, an Applied Research Grant from the Oklahoma Center for the Advancement of Science and Technology, the Center for Waste Reduction Technologies of the American Institute of Chemical Engineers, Agreement No. N12-N10, the TAPPI Foundation, the National Research Council of Thailand, Rayong Refinery Co., Ltd., and U.S. AID (University Development Linkages Project). In addition, support was received from the industrial sponsors of the Institute for Applied Surfactant Research including Akzo Nobel, Amway, Amoco, Aqualon, Calgon-Vestal, Colgate-Palmolive, Dow, Dowelanco, DuPont, Henkel, ICI, Kerr-McGee, Reckitt and Colman, Lubrizol, Phillips Petroleum, Pilot Chemical, Shell, Sun, and Witco. Surfactant was provided by Kao Industries Co. Ltd. and the carbon by Calgon Co.

REFERENCES

1. I. H. Suffet and M. J. McGuire, *Activated Carbon Adsorption*, Vol. 1, Ann Arbor Science, Ann Arbor, Michigan, 1980.
2. R. A. Hutchins, in *Handbook of Separation Process Techniques for Chemical Engineers* (P. A. Schweitzer, Ed.), McGraw-Hill, New York, 1979, Section 1.13.
3. G. E. Keller, R. A. Anderson, and C. M. Yon, in *Handbook of Separation Process Technology* (R. W. Rousseau, Ed.), Wiley Interscience, New York, 1987, Chap. 12.
4. L. Hemphill, *Thermal Regeneration of Activated Carbon*, Environmental Protection Agency, Office of Research and Development, Springfield, Ohio, 1978.
5. N. P. Cheremisinoff, *Carbon Adsorption for Pollution Control*, Prentice-Hall, Englewood, New Jersey, 1993.
6. A. Yehoskel, *Activated Carbon: Manufacture and Regeneration*, Noyes Data, Park Ridge, New Jersey, 1978.
7. T. Sutikno and K. J. Himmelstein, *Ind. Eng. Chem., Fundam., 22*, 420 (1983).
8. W. A. Chudyk and V. L. Snoeylnk, *Environ. Sci. Technol., 18*, 1 (1984).
9. Y. Yin, J. F. Scamehorn, and S. D. Christian, *ACS Symp. Ser., 594*, 231 (1995).
10. L. W. Brant, K. L. Stellner, and J. F. Scamehorn, in *Surfactant-Based Separation Processes* (J. F. Scamehorn and J. H. Harwell, Eds.), Dekker, New York, 1989, Chap. 12.
11. D. L. Blakeburn and J. F. Scamehorn, in *Surfactant-Based Separation Processes* (J. F. Scamehorn and J. H. Harwell, Eds.), Dekker, New York, 1989, Chap. 9.
12. B. L. Roberts, J. F. Scamehorn, and J. H. Harwell, in *New Directions in Sorption Technology* (G. E. Keller II and R. T. Yang, Eds.), Butterworths, Boston, 1989, Chap. 8.
13. P. Mukerjee and K. J. Mysels, *Critical Micelle Concentrations & Aqueous Surfactant Systems* (NSRDA-NBS-36), US Government Printing Office, Washington D.C., 1971.
14. S. D. Christian and J. F. Scamehorn (Eds.), *Solubilization in Surfactant Aggregates*, Dekker, New York, 1995.
15. M. J. Rosen, *Surfactants and Interfacial Phenomena*, Wiley, New York, 1978, Chap. 4.
16. Th. F. Tadros, *Surfactants*, Academic Press, London, 1984.
17. R. A. Pobb and J. M. Cohen, *Carbon Adsorption Isotherms for Toxic Organics*, Environmental Protection Agency, Washington D.C., 1980.

Received by editor July 5, 1995