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Powdered Activated Carbon Separation from Water by Foam Flotation

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ABSTRACT

Powdered activated carbon was separated from dilute aqueous suspensions (200–1000 mg/L) by foam flotation using surfactants (anionic or cationic). The effects of surfactant type, pH value of the suspension, initial carbon and surfactant concentrations, flotation time, and air flow rate on the dispersed-air flotation of powdered activation carbon were investigated. In optimum conditions the powdered activated carbon separation was almost complete. The ζ-potential of powdered activated carbon was also measured in the presence and absence of surfactants. Finally, carbon flotation was examined after the carbon had adsorbed chromate ions from an acidic solution (pH 2). Almost complete separation of Cr(VI)-loaded carbon was obtained by using an anionic surfactant.

Key Words. Powdered activated carbon; Flotation; ζ-Potential; Chromate adsorption

INTRODUCTION

The increasingly important role of adsorption as a technology for removing toxic compounds from wastes or wastewaters has been well established for many years (1). Activated carbon is a nonspecific adsorbent with pores of widely differing sizes; its effective adsorptive properties

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have been studied for nearly a century (2). Activated carbon is employed as powdered activated carbon (denoted hereafter as PAC) with a particle diameter between 5 and 100 μm and as granulated activated carbon (denoted as GAC) with a particle diameter between 0.25 and 4 mm. The comparison between these two forms as applied for wastewater treatment has been reviewed (3).

Adsorption onto PAC has proved to be a very effective technique for the removal of low concentrations of organic compounds (4–6) and inorganic toxic species (7–9) from water supplies or from wastewaters. The use of PAC for the enhancement of activated sludge treatment (10, 11) and in treating water (12) has been reviewed.

PAC has the advantage of being less costly to manufacture, it offers more surface area for adsorption, and it distributes more easily and uniformly in water. PAC also offers the advantage that the dose and the reaction time can be adjusted according to the composition of the liquid streams to be treated, as is the case for color or odor removal. PAC requires minimal capital expenditure for feeding and contacting equipment. It can also be applied only when needed. Economic limitations have been placed on the use of PAC due to the difficulties encountered in separating it from suspension in order for it to be regenerated. Although the thermal reactivation of PAC is technically feasible, it is used mainly on a throwaway basis. For PAC to become more acceptable economically, better means for separation and regeneration are needed.

In the first part of this paper, foam flotation will be considered as an attractive method for PAC separation/recovery. Foam flotation is a technique in which gas bubbles are used as a means of separation. The constituents to be separated (ions, molecules, or particles) are attached to the surface of a bubble and rise to the air/liquid interface. The addition of a surfactant is usually necessary in order to render the constituents, which are not naturally hydrophobic, surface active. Past efforts to separate colloidal coal or PAC from dilute suspensions by coagulation and/or flotation have been limited (13, 14).

The use of flotation, followed by activated carbon adsorption as a final polishing step but applied as separate processes, has been reported (15, 16). It is also possible to contemplate a process involving both carbon adsorption and foam separation for the treatment of wastewaters as, for example, in the cases of phenol removal (17) or of Pb(II) and Cd(II) (18). In the second part of this work PAC was floated after having adsorbed chromate ions. The adsorption of Cr(VI) onto GAC, as well as subsequent GAC regeneration, has been examined (19–21). The use of PAC for the removal of Cr(VI), but in combination with an activated sludge process

(22) and the removal of Cr(VI) using fibrous carbon electrodes (23), has also been suggested.

EXPERIMENTAL

The PAC used in this study was a steam-activated carbon ex Norit (type SA-2). A typical analysis provided by the manufacturer is given at Table 1. The PAC was sieved and the −75 μm fraction was taken. It was dried for 2 hours at 125°C and subsequently kept in a desiccator. PAC (200 mg/L) was used in most flotation experiments (unless otherwise stated). A 500-mL PAC suspension was prepared in a stirred glass beaker. The suspension pH was adjusted using HCl or NaOH solutions, the appropriate surfactant quantity was added, and it was stirred at 200 rpm for a further 10 minutes. The surfactants used were cetyltrimethyl-ammonium bromide (CTMA-Br, cationic) or sodium dodecyl-sulfate (SDS, anionic), both strongly ionized. The suspension was then transferred to the flotation column.

The dispersed-air flotation technique was used. More details of the experimental setup have been given previously (24). Air was pumped for 10 minutes at a flow rate of 200 cm^3/min. Carbon separating from the flotation column was collected in a funnel sitting on the top of the column. Kinetic experiments were also performed in batch experiments; that is, PAC suspensions treated under the same conditions were transferred to the flotation column and air was passed through for different time intervals. The foam produced acted as a carrying medium to separate physically the carbon particles from the flotation column. The foam collected was filtered through a G_5 sintered-glass filter (porosity 1–1.5 μm) and dried in an oven at 125°C to constant weight. The percentage PAC separation (Se%) was

TABLE 1
Typical Analysis of the PAC ex Norit Type SA-2

Apparent density	410 g/L
Ash content	7%
Phenol adsorption	4.5%
Methylene blue adsorption	14%
Iodine number	850 mg/g
Specific surface area (BET)	800 m^2/g
Particle size analysis	>10 μm, 77% >44 μm, 36% >74 μm, 18% >150 μm, 4%

calculated in the usual way. The amount of foam recorded was usually about 1–5% of the initial liquid volume.

Experiments for Cr(VI) removal were conducted in a similar manner. A stock solution of 1000 mg/L was prepared by weighing the appropriate quantity of potassium dichromate (99.9% analytical purity, ex BDH) and dissolving it in deionized water. PAC (0.5 g) and 500 mL of an aqueous solution containing 10 mg/L Cr(VI) were added to a stirred glass beaker. The suspension pH was adjusted to 2.0 and the mixture was stirred for a further 10 minutes at 200 rpm. Analytical determination of the remaining Cr(VI) was performed by applying the standard diphenyl-carbazide method (25), and the percentage Cr(VI) removal (Re%) was calculated as usual. At the end of the adsorption stage an anionic surfactant (SDS) was added, and the suspension was stirred for another 10 minutes. The suspension was transferred to the dispersed-air flotation column where it was treated as before.

In order to study the surface charge of the PAC and to explain the results observed, electrokinetic measurements were conducted by using 0.5 g/L PAC dispersions in the presence or absence of surfactants using the particle electrophoretic apparatus of Rank Brothers, U.K. (model Mark II), equipped with a flat cell. The results were expressed as ζ-potential values (mV).

RESULTS AND DISCUSSION

PAC Separation by Flotation

PAC separations with three different surfactant concentrations in a wide pH range are presented in Fig. 1. CTMA-Br proved to be a more effective surfactant than SDS at relatively low concentrations (100 mg/L). At higher concentrations (200 mg/L), separation was generally reduced. With SDS the separation curve reached a maximum with increasing SDS concentration and then it was reduced. SDS gave relatively better results in acidic pH values (for 100 or 200 mg/L concentrations) while CTMA-Br worked better in the alkaline pH region. It appeared that in a broad pH range, sufficient hydrophobicity of the surface of PAC particles was obtained and, hence, efficient separation by flotation could be achieved.

It is also worth noting that the addition of a surface-active agent was necessary for adequate separation of PAC from suspension because air flotation alone had very little effect on PAC separation. On the other hand, the presence of a surfactant should not pose a problem to PAC regeneration since the surfactant is volatilized at temperatures much below those used for regeneration. The variations observed for PAC sepa-

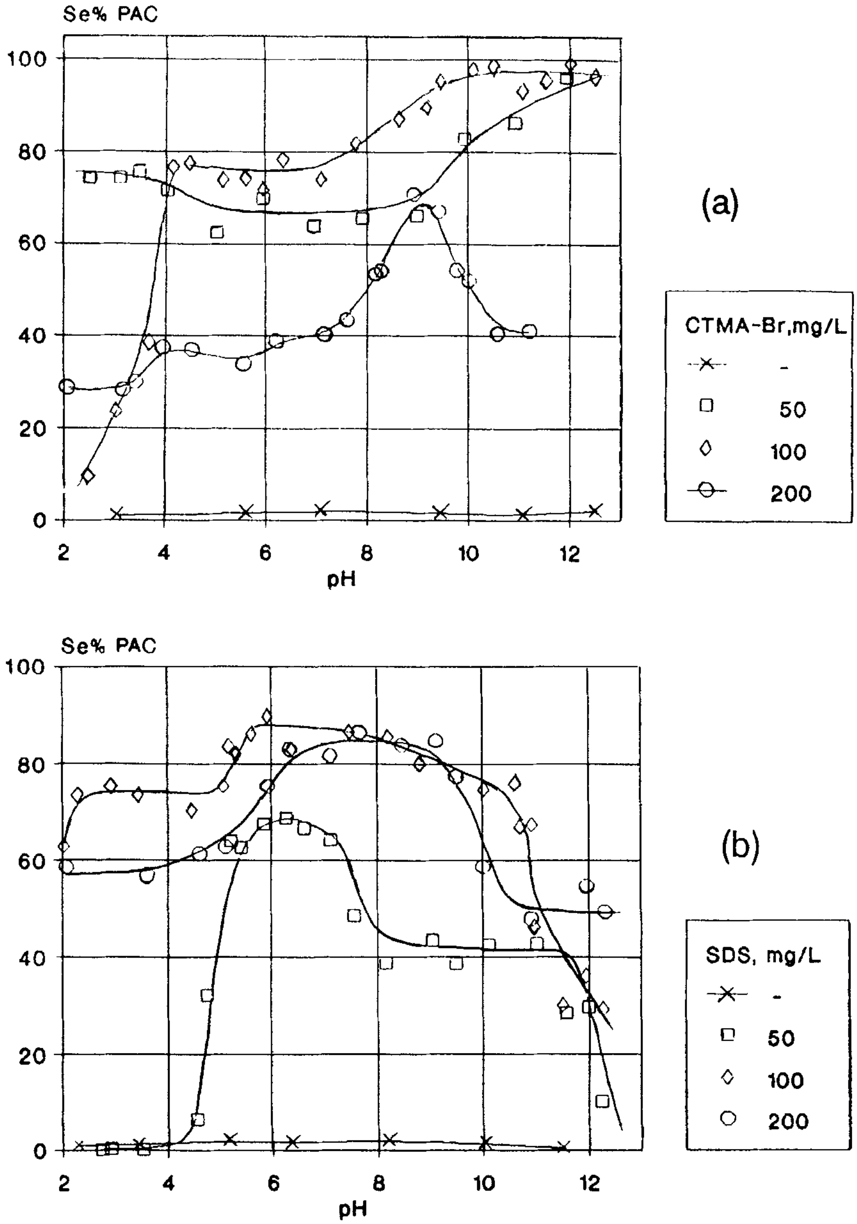

FIG. 1 Effect of pH on PAC separation, applying three different surfactant concentrations: (a) CTMA-Br and (b) SDS.

ration were probably due to the different ionic forms under which the surfactant existed in these suspensions or, perhaps, to the adsorption of protons onto PAC in the acidic pH range, which would hinder the surfactant from being adsorbed.

The influence of surfactant concentration on PAC separation for three different pH ranges was examined (Fig. 2). Maximum PAC separation again appeared for a CTMA-Br concentration between 100 and 150 mg/L. The separation was nearly 100% in the alkaline pH range; in the acidic or neutral pH range it was slightly reduced (Fig. 2a). In the case of SDS (Fig. 2b), higher PAC separation was obtained at pH 9 by using 100 mg/L SDS, but this was low in comparison with the respective separations using CTMA-Br. A lower PAC separation was observed at pH 2. An increase in surfactant dosage resulted in an increase of the amount of foam recorded. On the other hand, by increasing the foamate volume, the concentrations of PAC in the foam decreased, thereby making separation less effective.

It is generally believed that the initial adsorption of an ionic surfactant occurs with the polar groups orientated toward the solid surface. Excessive quantities of surfactant impaired flotation. This impairment has been discussed by Klassen and Mokrousov (26), who concluded that poor flotation at high surfactant concentration is caused by the formation on the air bubble surface of a stable, hydrated envelope of surfactant or, perhaps, by the formation of a hydrated micelle coating on the solid surface. As a result, the hydrophobicity of this surface was not satisfactory for flotation.

The flotation/separation behavior generally followed a more or less expected pattern. Most carbon surfaces are negatively charged, and it seemed logical that the cationic surfactant CTMA-Br would be a better foam separation agent in the neutral/basic pH range than the anionic SDS would be. Part of the advantage of CTMA-Br over SDS may also have been provided by its longer hydrocarbon chain (C_{16} compared to C_{12}). Therefore, a relatively small surfactant quantity is sufficient for PAC flotation provided that the conditions are such that it can be adsorbed onto PAC, giving a hydrophobic surface. The fact that both surfactants, which carry different charges (CTMA-Br is cationic and SDS is anionic), are capable to float PAC and to give high separation rates means that the PAC surface can adsorb molecules with positive or negative charges. It should be stressed that the extent of adsorption on the external carbon surface and the orientation of the surfactant are the factors that mainly control the flotation behavior.

The effect of pH on the ζ-potential of PAC dispersions was studied in an attempt to understand its flotation behavior. Three separate carbon

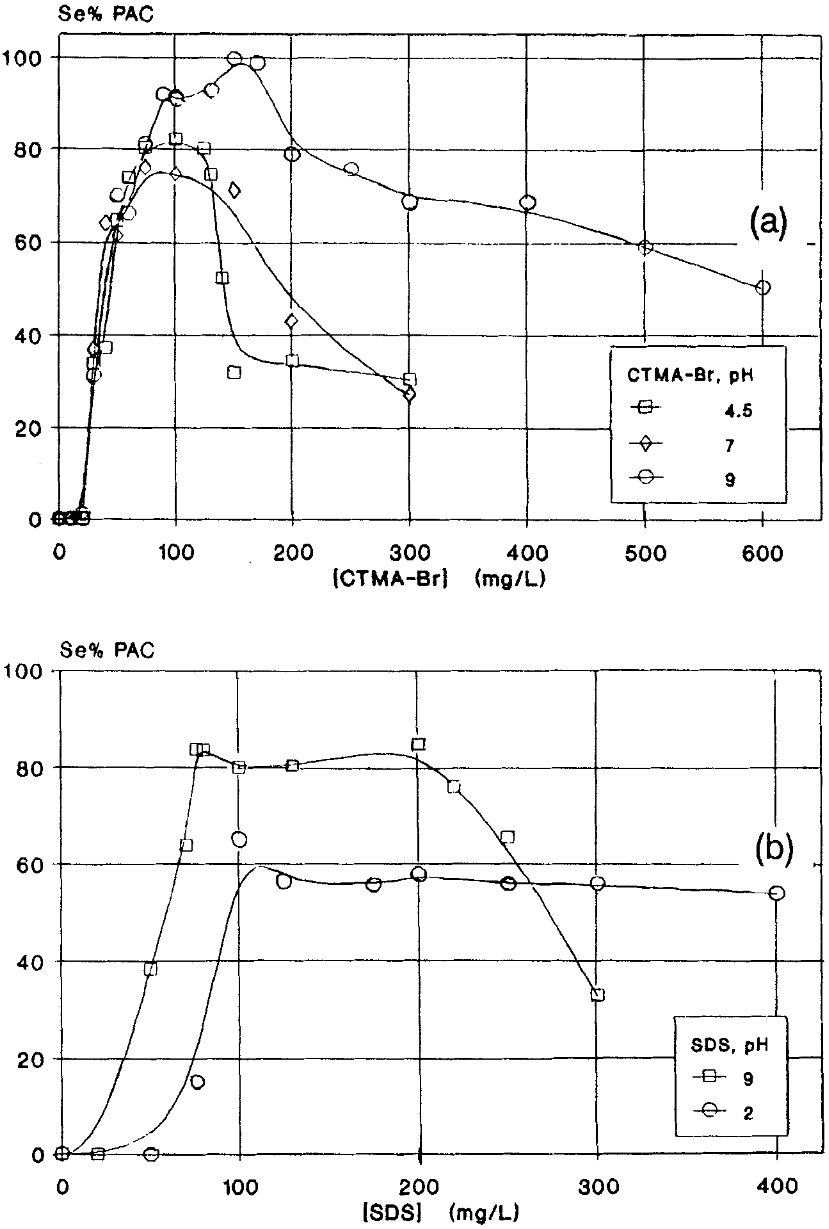

FIG. 2 Effect of surfactant concentration on PAC separation at different initial pH's of the PAC suspension; (a) CTMA-Br and (b) SDS.

suspensions were examined. These were suspensions of PAC alone and PAC in the presence of CTMA-Br or SDS; the results are summarized in Fig. 3. In the presence of SDS the carbon surface continued to be negatively charged in the whole pH range examined, but there was a shift at more negative ζ-potential values. The PAC ζ-potential remained negative in the whole range of pH values examined. CTMA-Br was effective in reducing the negative PAC ζ-potential, giving a positively charged surface. The greatest CTMA-Br adsorption was expected in the basic pH range where PAC attained a maximum negative charge, apparently due to adsorption of hydroxyl groups.

In the acidic pH range, neutralization of negative charges at the PAC surface with increasing hydrogen ion concentration enhanced surface adsorption, while in the basic pH range, the surfaces become more negatively charged due to increased hydroxide ion concentration. With CTMA-Br, this pattern was reversed. The specific chemical nature and the dissociation of each surfactant, as well as the fact that the surface of the PAC particles was originally negatively charged and that in acidic pH the surface charge can be modified by the adsorption of protons, should be considered in order to evaluate the observed results.

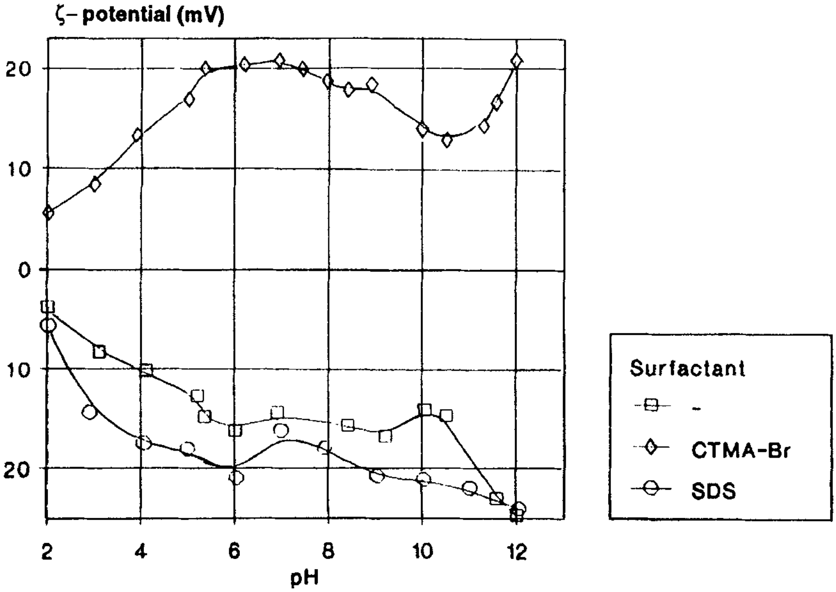

FIG. 3 Effect of pH on the ζ-potential in the absence or presence of surfactants used at 100 mg/L concentrations.

The ζ-potential is an appropriate index for surface electrostatic potential because it can be measured experimentally. The surface electrostatic potential of PAC is believed to be influenced by the surface functional group of activated carbons, such as carboxyl or phenolic hydroxyl groups (27). Depending on the raw material used and on the production process, PAC may contain 0–15% of inorganic constituents remaining as ash after heating in air. Furthermore, it contains chemically bound hetero-atoms, such as H, O, and N and sometimes S. In general, the carbon surface is nonpolar, but surface oxides incorporated during production can give some parts of the surface a polar character.

With respect to the air flow rate (Fig. 4), it was observed that a low flow rate (50 cm^3/min) separated all the suspended PAC but at a relatively longer flotation time (around 100 minutes) than the higher applied flow rate (200 cm^3/minutes) which needed only 20 minutes. Nevertheless, the use of higher flow rate, although capable of separating PAC in a much shorter time, would result in a greater amount of foam, which should be further treated.

Kinetic experiments using CTMA-Br or SDS (100 mg/L), but at different suspension pHs, are presented in Fig. 5. These results show that CTMA-

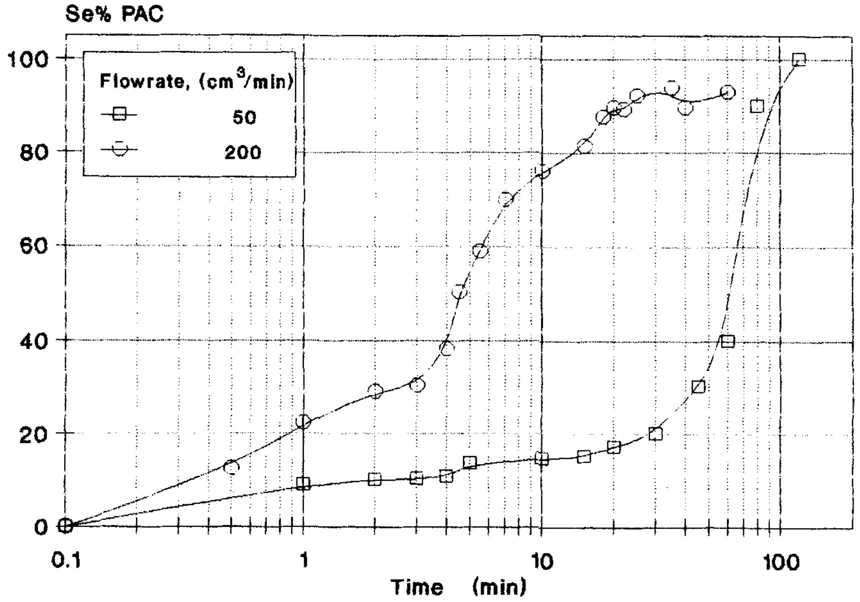

FIG. 4 Effect of flotation time and of air flow rate on PAC separation; SDS concentration 100 mg/L, pH 2.

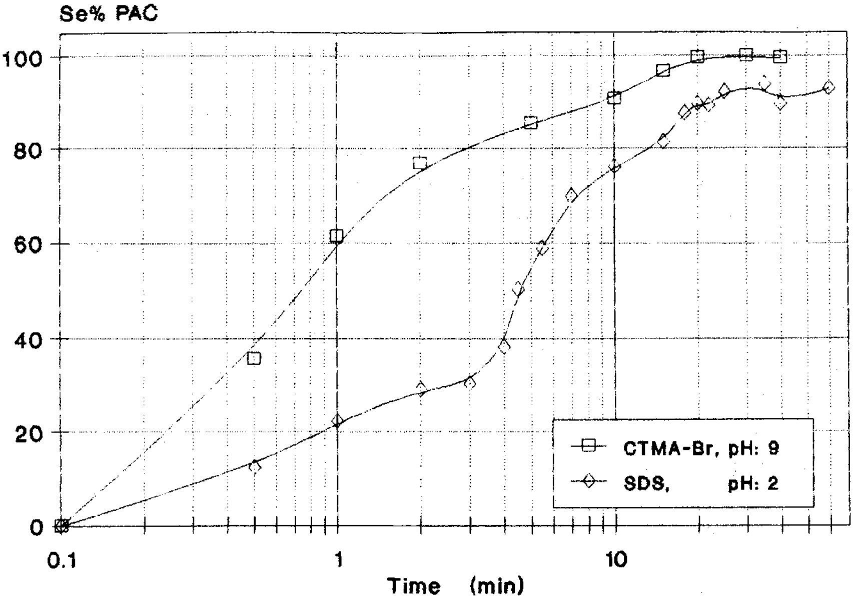

FIG. 5 Effect of flotation time on PAC separation using a 100 mg/L surfactant concentration.

Br is capable of PAC separation in a shorter time. However, the kinetic data of Fig. 5 could not be fitted to a simple rate equation. The literature on flotation kinetics contains a great deal of information on proposed mechanisms, generally considering the rate to be first order (28).

The effects of variations in initial PAC concentration are presented in Fig. 6. It is possible through a proper combination of flotation time, of surfactant concentration, and of pH values to have nearly 100% PAC separation, even at relatively higher PAC concentrations. The quantity of PAC added to the waste stream must be carefully controlled in order to ensure that the proper surfactant concentration is acting simultaneously as a frothing agent.

Adsorption of Cr(VI) onto PAC and Separation of PAC by Flotation

The removal of Cr(VI) from solution by adsorption onto PAC occurs mainly through the following interfacial reactions: (a) the direct adsorption of Cr(VI) onto the carbon surface, and (b) the reduction of Cr(VI) species to Cr(III) by the activated carbon on the surface (19). The sorptive pro-

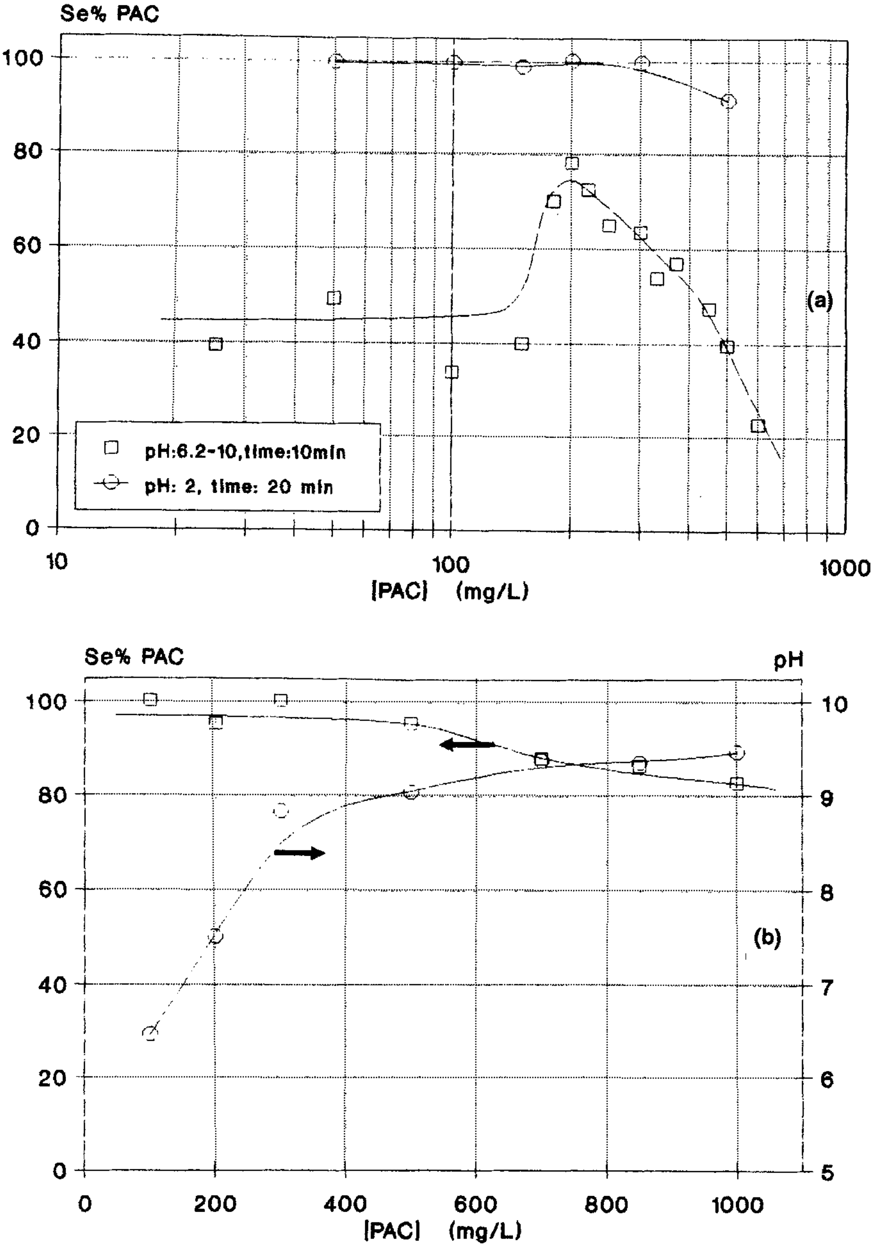

FIG. 6 Effect of initial PAC concentration on PAC separation using SDS: (a) at different initial pH's and flotation times for a SDS concentration of 100 mg/L; and (b) at equilibrated pH for a SDS concentration of 300 mg/L and a flotation time of 20 minutes.

cesses occurring were brought about by van der Waals forces on the planar surfaces of the microcrystallite structural groups constituting the carbon and by coulombic forces due to the presence of oxygen complexes on the sides of the microcrystallites. The ash content might also be expected to alter markedly the nature of the active carbon surface if the inorganic species were distributed over the pores.

Bichromate ions ($HCrO_4^-$) are the major species removed by formation of oxo functional groups on the carbon surface, according to the following schematic reaction equations:

$$C_xO + HCrO_4^- + H_2O \Leftrightarrow C_xOHCrO_3^+ + 2OH^-$$

or

$$C_xO_2 + HCrO_4^- + H_2O \Leftrightarrow C_xO_2HCrO_3^+ + 2OH^-$$

where C_xO and C_xO_2 represent surface functional groups formed during heating and activation of raw coke materials. Preliminary experiments in which the contact time between PAC and Cr(VI) was varied showed that there was little difference in Cr(VI) removal between 10 and 60 minutes (5–7% higher) for the range of PAC concentrations tested.

It is known from the literature that the adsorbed quantity of chromium was higher at an acidic pH of 2.0 and at relatively low Cr(VI) concentrations (around 10 mg/L) (19). This is also clearly observed in this study (Fig. 7) where results are presented for the removal of chromate ions. At acidic pH values the quantity of Cr(VI) removed increased with increasing amounts of PAC, but in the neutral/basic pH range, where Cr(VI) removal was small, the PAC concentration had nearly no effect. Therefore, the initial pH suspension was adjusted for subsequent experiments to a value of 2.0.

The molar distribution of Cr(VI) species is a function of pH and Cr(VI) concentration. Following the formation of polynuclear chromium complexes (for example, $Cr_2O_7^{2-}$), Cr(VI) removal efficiency decreases due to increasing pH values or the initial Cr(VI) concentration. Dichromate ions ($Cr_2O_7^{2-}$), which are predominant in the neutral to acidic pH range, apparently have very little affinity for the carbon surface. When the Cr(VI) concentration increased (about 10^{-3} M or 52 mg/L), the percentage of Cr(VI) removal decreased to the same extent as the concentration of dichromate simultaneously increased (29). At the chromate concentration used for these experiments (10 mg/L), Cr(VI) mainly exists as $HCrO_4^-$ or CrO_4^{2-}, depending on the solution pH.

Adsorption experiments of Cr(VI) onto PAC are presented in Fig. 8. Although the stirring time in this case was only 10 minutes, i.e., not sufficient for equilibrium to be reached, these results could be satisfactorily

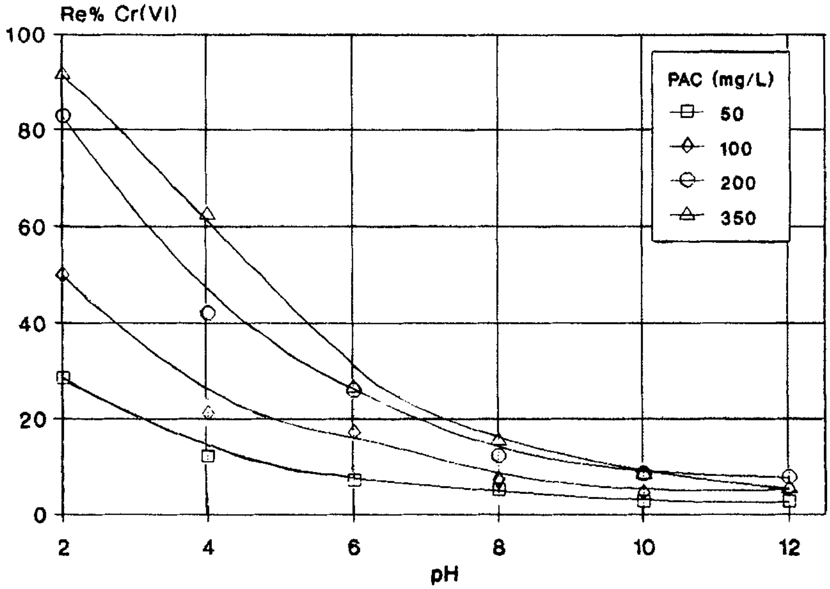

FIG. 7 Adsorption of Cr(VI) onto PAC; effect of solution pH on Cr(VI) removal using different PAC concentrations.

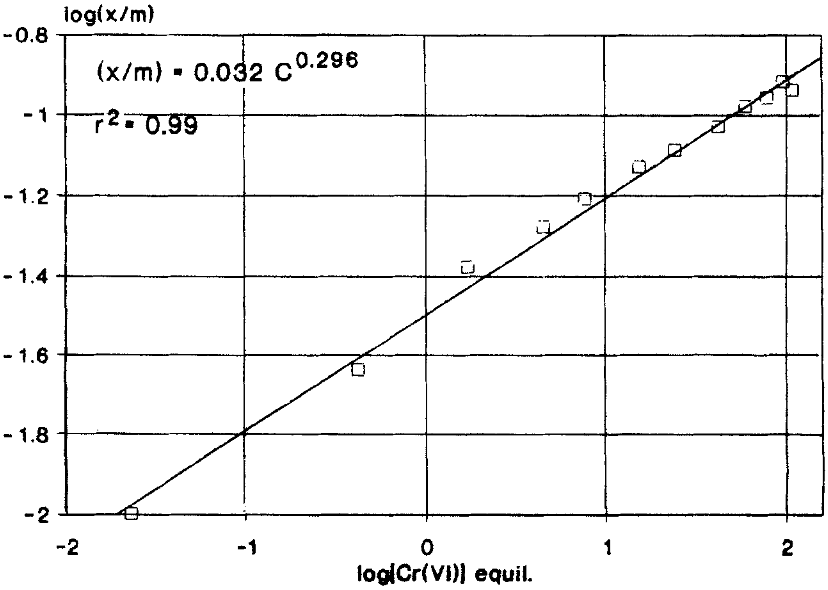

FIG. 8 Adsorption of Cr(VI) onto PAC according to the Freundlich equation; PAC concentration of 200 mg/L and Cr(VI) concentrations of 2–140 mg/L.

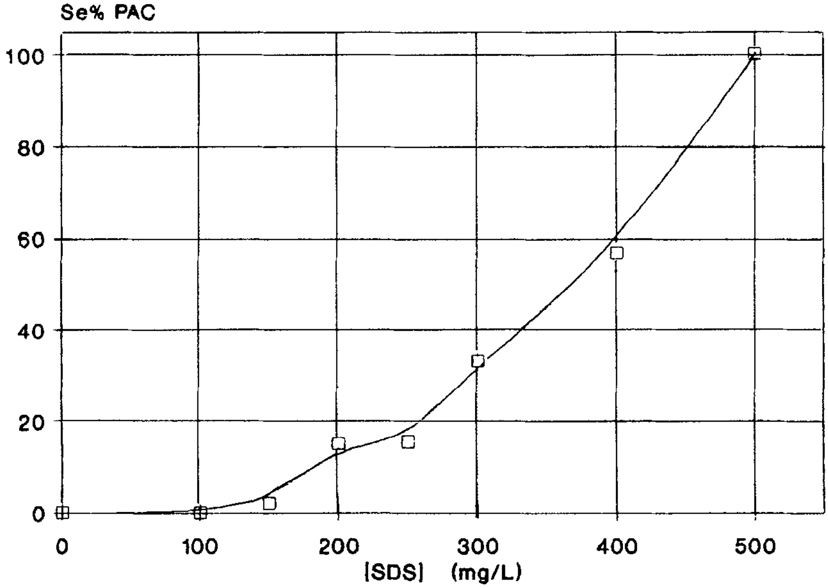

FIG. 9 Effect of SDS concentration on the separation of Cr(VI) loaded-PAC using an initial PAC concentration of 500 mg/L.

represented by a Freundlich-type isotherm. The adsorption capacity (x/m) was almost 0.11 mg Cr(VI)/mg PAC.

The influence of SDS concentration on PAC separation by flotation, where Cr(VI) has been previously totally adsorbed, is presented in Fig. 9. PAC was quantitatively separated by applying an equivalent concentration of surfactant (500 mg/L).

CONCLUSIONS

Dispersed-air flotation was proved to be a relatively quick and simple separation method for PAC from dilute aqueous dispersions. Under the optimum determined conditions, nearly 100% separation could be achieved. It was found that foam volume increased and foam carbon concentrations decreased with increasing aeration rate and surfactant dosage, but an overdosage of surfactant or of PAC could impair the foam flotation/separation process. Preliminary Cr(VI) adsorption onto PAC was also tried, the conditions for complete Cr(VI) removal were defined, and Cr(VI)-loaded PAC separation by flotation was successfully applied.

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