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POLYELECTROLYTE-ENHANCED ULTRAFILTRATION OF CHROMATE, SULFATE, AND NITRATE

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ABSTRACT

Polyelectrolyte-enhanced ultrafiltration (PEUF) involves addition of a polyelectrolyte of opposite charge to that of the multivalent ions to be removed from the contaminated water. In this study, a water-soluble polyelectrolyte, poly(diallyldimethyl ammonium chloride) or QUAT is added to the aqueous solutions containing divalent anions chromate or sulfate. Removal of monovalent anion nitrate is also studied to probe the effect of valence. The water is then passed through an ultrafiltration membrane with pore size small enough to reject the polyelectrolyte with the bound target ions. The rejection of anions increases with increasing concentration ratio of QUAT to anion. A high QUAT concentration in the retentate decreases relative flux due to accumulation of polyelectrolyte near the membrane surface (hydrodynamic boundary layer). Rejections of chromate and

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sulfate are similar and >98% at reasonable operating conditions. Rejection of nitrate is substantially below that of the divalent anions, but can be as high as 97% under feasible operating conditions. The gel concentration (where flux approaches zero) of the QUAT varied from 5.1 to 8.1 wt%.

INTRODUCTION

Polyelectrolyte-enhanced ultrafiltration (PEUF) (1–17), sometimes called polymer-assisted ultrafiltration or polymer filtration, is a specific colloid-enhanced ultrafiltration method (11) which is useful in removing multivalent ions from water. In PEUF, a water-soluble polymer of opposite charge to the target ion, is added to the contaminated water. For example, anionic polyelectrolyte has been used to treat water containing divalent cation copper (1,3). The water is then treated by an ultrafiltration membrane with pore sizes small enough to block the polymer with the bound target ion from passing through. The purified water passing through the membrane is called the permeate and that retained by the membrane is the retentate, as shown in Fig. 1. In a modification of PEUF, ligands can be attached to the polymer, giving selectivity on a basis other than charge to

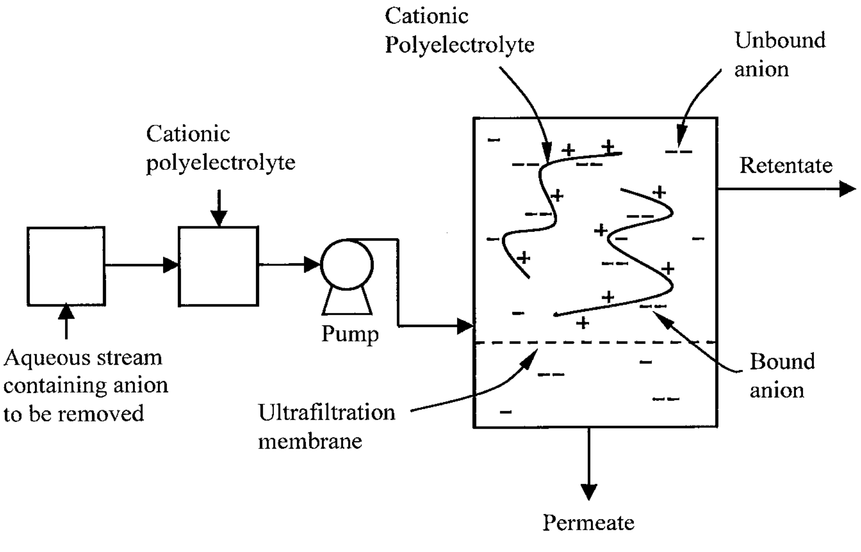

Figure 1. Schematic diagram of PEUF to remove anions from water.

the separation (e.g., Cu^{2+} vs. Ca^{2+}) in a method called ligand-modified PEUF (18,19).

A target ion of particular interest in this study is chromate (CrO_4^{2-}). Chromate contamination of water can come from a number of industrial sources, including electroplating. The cationic polymer used is poly(diallyldimethyl ammonium chloride) or QUAT. In a previous study of this system by our group, high rejections and a substantial reduction in rejection with added NaCl were observed (2). However, QUAT concentrations well below the gel concentration (at which flux approaches zero) were used (2). For high water recovery (permeate/feed ratio), the retentate must have a high QUAT concentration (approximately one-third to one-half of the gel concentration) and these conditions were studied here. Another reason to study chromate is that an efficient method has been developed to recover the QUAT for reuse involving precipitating the chromate from the retentate using barium (11–13).

In this study, divalent sulfate and monovalent nitrate were removed from water using PEUF under the same conditions as the chromate. Comparison of these systems indicates the effect of divalent anion structure and valence on the separation efficiency. Although not of as much interest as chromate, removal of sulfate and nitrate from water is of some concern. For example, a combined reverse osmosis/nano-filtration process has been investigated for nitrate removal from tap water (20). Biological desulfurization of wastewater has been considered (21).

EXPERIMENTAL

Materials

Poly(diallyldimethyl ammonium chloride) or QUAT having an average molecular weight of approximately 240,000 Da, was supplied by Calgon Corporation (Pittsburgh, PA) and has the trade name MERQUAT®. The repeating unit of the polymer is $(H_2C{=}CHCH_2)_2N(CH_3)_2Cl$. The polyelectrolyte was purified using a 10,000 molecular weight cut-off, spiral wound membrane in order to remove the lower molecular weight components to the point at which only trace amounts of polyelectrolyte was detected in the permeate. Sodium chromate was of analytical grade (purity of 99%) supplied by Reidel-deHaen (Seelze, Germany). Sodium sulfate (AR grade) and sodium nitrate (purity of 99.5%) were obtained from Carlo Erba (Milan, Italy). *p*-Hydroxybenzoic acid (99%) and *sym*-diphenylcarbazide were manufactured by Fluka (Buchs, Switzerland). Glacial acetic acid (99.9%) was obtained from J.T. Baker (Phillipsburg, NJ). All chemicals were used as received. Deionized and distilled water were used to prepare solutions.

Methods

Ultrafiltration experiments were carried out in a 400 mL stirred cell. Spectrum™ cellulose acetate (type C) ultrafiltration membranes from Spectrum Medical Company (Houston, TX) were used in these experiments with molecular weight cut-off equal to 10,000 Da. A 300 mL solution of polyelectrolyte, chromate, sulfate, or nitrate ions was placed in the stirred cell which was submerging into an acrylic plexiglas plastic box containing circulating water to control the solution temperature. The experiments were run at 30°C and a natural pH of approximately 8. The solution was stirred at a speed of 250 rpm with a pressure drop of 60 psi (414 kPa) across the membrane using nitrogen gas. Approximately 200 mL of the solution was filtered through the membrane as permeate to produce eight samples of 25 mL each. Fluxes were determined during the run by timing and weighing samples of permeate. The permeate samples were analyzed for concentrations of polyelectrolyte, chromate, sulfate, and nitrate. The rejection of chromate, sulfate, or nitrate was determined at the midpoint of the run where 100 mL of permeate has passed through the membrane. By knowing permeate concentrations during the run, the retentate concentrations at any point in the run were calculated from a material balance. In this work, initial ratios of QUAT to the anions were fixed. It is this ratio which is reported. Since rejection of the QUAT is essentially 100%, if rejection of the target anion is high (as in most experiments here), this ratio varies little throughout the experiments.

Analysis

The chromate concentrations were measured by using a UV/VIS spectrophotometer (Perkin–Elmer, Lamda 16, Uberlingen, Germany) at wavelength 541.2 nm after complexation with *sym*-diphenylcarbazide. *sym*-Diphenylcarbazide reagent was prepared by dissolving 0.1 g *sym*-diphenylcarbazide in 50 mL ethylalcohol and adjusted to 250 mL by adding the solution of 10% acetic acid in distilled water.

The concentrations of sulfate and nitrate ions in permeate and retentate solutions were analyzed by a Hewlett–Packard (Palo Alto, CA) Series 1050 ion chromatography with a Alltech (Deerfield, IL) Anion/R column ($1\,\mu$m, 150×4.6 mm) and conductivity detector (Alltech 350, Deerfield, IL). The mobile phase was 5 m*M* *p*-hydroxybenzoic acid and operated at a flow rate of 1.0 mL/min. Retention time of sulfate and nitrate were 10.7 and 7.0 min, respectively.

The concentrations of QUAT in the permeate and retentate solutions were determined by a Shimadzu (Kyoto, Japan) TOC-5000A total organic carbon (TOC) analyzer. This measurement was used to determine when pretreatment of the polymer was complete.

RESULTS AND DISCUSSION

Rejection of Anions

The removal efficiency of the chromate, sulfate, and nitrate are represented by the rejection, R in percent as defined by

$$R = 100[1 - (C_P/C_R)]$$

where C_P is the permeate concentration of anion and C_R is the retentate concentration of anion. The QUAT concentrations are based on the moles per liter of the repeating units and not on the total molecular weight. Since the repeating unit has a charge of + 1, the stoichiometric ratio of $[\mathrm{QUAT}]/[\mathrm{CrO}_4^{2-}]$ is two.

The anion rejection is shown in Figs. 2–4 as a function of retentate [QUAT] for chromate, sulfate, and nitrate, respectively. The low [QUAT] data from the previous PEUF work (2) have been combined with the high [QUAT] data from this work in Fig. 2 for chromate. For all three anions, as the initial [QUAT]/[anion] ratio increases, the rejection increases, since the increased polyelectrolyte in solution provides more binding sites for the target anion. When the initial [QUAT] or [anion] increases at constant [QUAT]/[anion], the rejection decreases. This trend is expected and at low initial [QUAT] or [anion], a modified Oosawa two-state binding model successfully described these results quantitatively (1,4,10). While this dilute solution model did not successfully model the high concentration data shown here, this trend at constant initial [QUAT]/[anion] is qualitatively predicted. For chromate and sulfate, rejections of greater than 98% are observed until high retentate [QUAT] levels are reached.

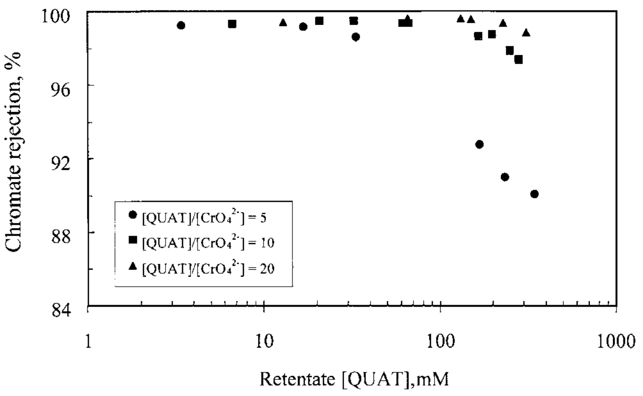

Figure 2. Effect of retentate [QUAT] on chromate rejection.

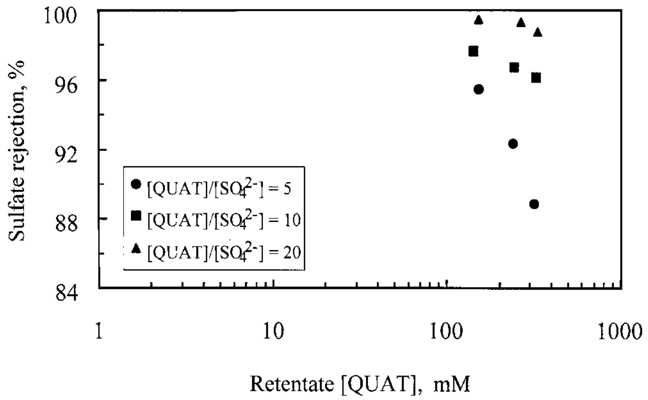

Figure 3. Effect of retentate [QUAT] on sulfate rejection.

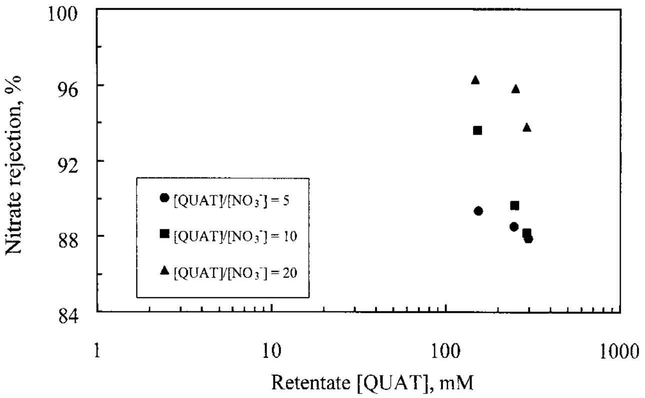

Figure 4. Effect of retentate [QUAT] on nitrate rejection.

However, even at the high retentate [QUAT] levels expected in a PEUF operation with high water recovery (ca. 0.3 M), if a high enough [QUAT]/[divalent anion] is used, high rejections are still attainable. For example, at a [QUAT]/[CrO$_4^{2-}$] of 20 and retentate [QUAT] = 300 mM, chromate rejection = 99.4%.

The anion rejection is shown in Figs. 5–7 at initial [QUAT]/[anion] ratios of 5, 10, and 20, respectively. Chromate and sulfate rejections are similar under all conditions. The rejection of nitrate is much lower than that of the chromate or sulfate. For example, at an initial [QUAT]/[anion] ratio of 20 and retentate

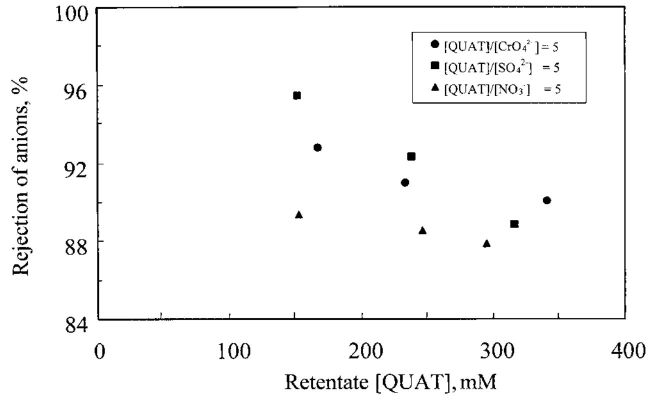

Figure 5. Effect of retentate [QUAT] on chromate, sulfate, and nitrate rejections at a [QUAT]/[anion] of 5.

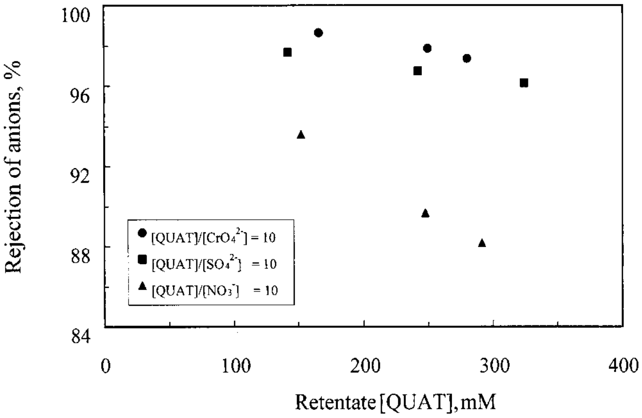

Figure 6. Effect of retentate [QUAT] on chromate, sulfate, and nitrate rejections at a [QUAT]/[anion] of 10.

[QUAT] = 200 m*M*, chromate and sulfate rejections are >99.5%, while nitrate rejection is 96.3%.

Anions commonly form species of different valancies in water and the concentration of each ion depends on pH (22). For chromium (VI) ions present here, the predominant species between pH 1.5 and 4.0 is $HCrO_4^-$. At pH 6.5, $HCrO_4^-$ and CrO_4^{2-} exist in equal amounts and at high pH, CrO_4^{2-} predominates.

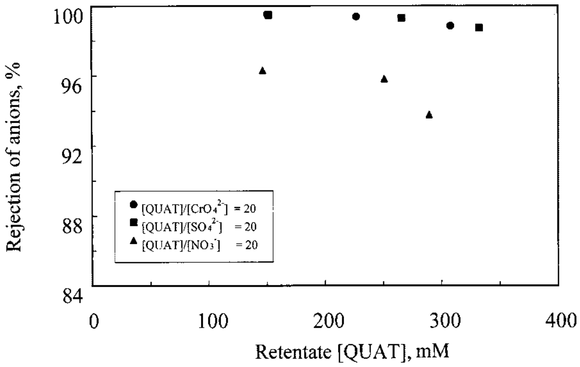

Figure 7. Effect of retentate [QUAT] on chromate, sulfate, and nitrate rejections at a [QUAT]/[anion] of 20.

For sulfate, SO_4^{2-} is present at pH level above 3.0. The sulfate and bisulfate ions exist in about equal amounts at pH 2.0 and the bisulfate ion predominates at pH 1.0. Nitrate ions from most metal nitrate salts or strong nitric acid are in NO_3^- form at both low and high pH. The pH condition at which the PEUF of these anions was operated was about 8.0. Therefore, the ion species mainly present would be CrO_4^{2-}, SO_4^{2-}, and NO_3^-. The divalent chromate and sulfate are removed with approximately the same efficiency, confirming that valence is the predominant variable affecting the removal of anion. In a previous study of micellar-enhanced ultrafiltration (similar to PEUF except that charged micelles were used instead of polyelectrolyte), a similar conclusion was reached for divalent cations removed by anionic micelles (23).

Flux

Figures 8–16 show the relative flux (flux/flux of pure water) as a function of the logarithm of retentate [QUAT]. As seen in previous studies, this semilogarithmic flux plot is linear at high concentrations. When extrapolated to zero flux, the [QUAT] is called the gel concentration, which are tabulated in Table 1 for these systems. These vary between 559 and 885 m*M*. A higher [QUAT]/[anion] ratio results in a lower gel concentration for all the three target anions. This might be due to the lower bound counterion/repeating polymer unit, causing the polymer to be less coiled, forming a more entangled network of polymer chains in the gel layer, and thus reducing flux. Bound monovalent anion may correspond to a less coiled configuration than divalent chromate and sulfate,

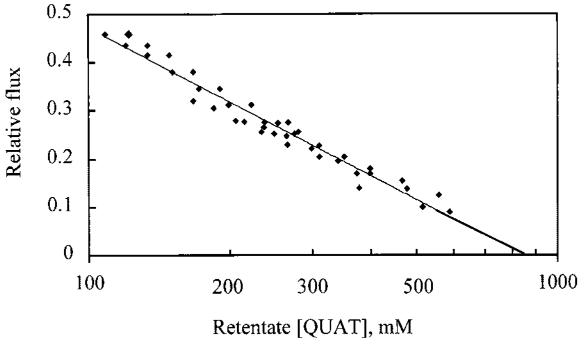

Figure 8. Relative flux of QUAT/chromate system in PEUF at a [QUAT]/[chromate] of 5.

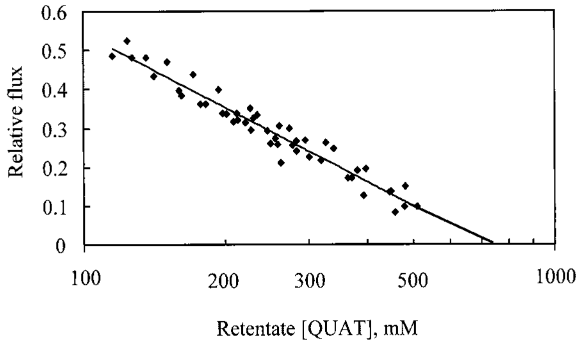

Figure 9. Relative flux of QUAT/chromate system in PEUF at a [QUAT]/[chromate] of 10.

accounting for the lower gel concentration for the nitrate systems. These explanations are speculative but consistent with the data. The gel concentration is obtained by extrapolation to zero flux on a log scale, so there is substantial opportunity for inaccuracy. Therefore, Table 1 contains the range of gel concentrations corresponding to a 95% confidence level from a statistical analysis of the curve fit, as well as the best fit value.

The 559–885 m*M* gel concentration observed here compares reasonably with the gel concentrations of 550 m*M* obtained in a previous study of this system at a [QUAT]/[chromate] of 10 (2), 1000 m*M* for anionic polyelectrolyte (3), 708 m*M* for anionic surfactant (23), and 530 m*M* for cationic surfactant (24). These fluxes limit

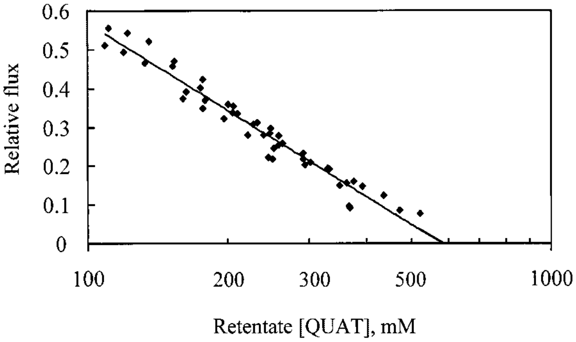

Figure 10. Relative flux of QUAT/chromate system in PEUF at a [QUAT]/[chromate] of 20.

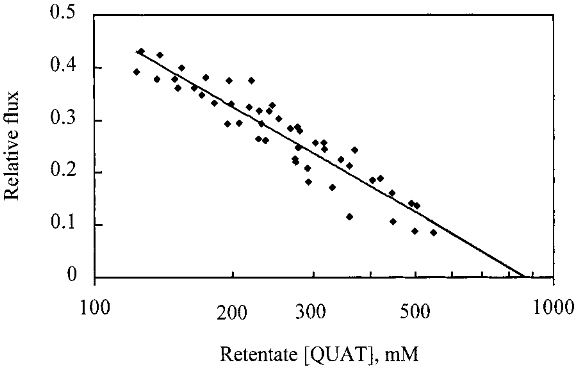

Figure 11. Relative flux of QUAT/sulfate system in PEUF at a [QUAT]/[sulfate] of 5.

the maximum concentration of the polymer practically attainable in the retentate since unacceptably low fluxes make an operation uneconomical. For example, the arbitrary level of 300 m*M* as the final retentate concentration used to discuss rejections before, relative fluxes vary from 0.208 to 0.288 from Figs. 8–16. However, this retentate polymer concentration is quite high and indicate that high water recovery levels are attainable in PEUF with high rejections.

As an example to summarize the efficiency of this process, if a feed water has a chromate concentration of 1.0 m*M*, a [QUAT]/[chromate] feed ratio of 20 is used and, the final retentate [QUAT] = 300 m*M*, the initial permeate [CrO_4^{2-}] will be 0.022 m*M* and the final will be 0.084 m*M* (compared to 1.0 m*M* in the

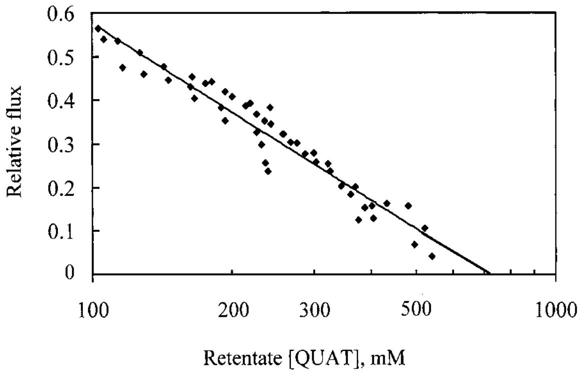

Figure 12. Relative flux of QUAT/sulfate system in PEUF at a [QUAT]/[sulfate] of 10.

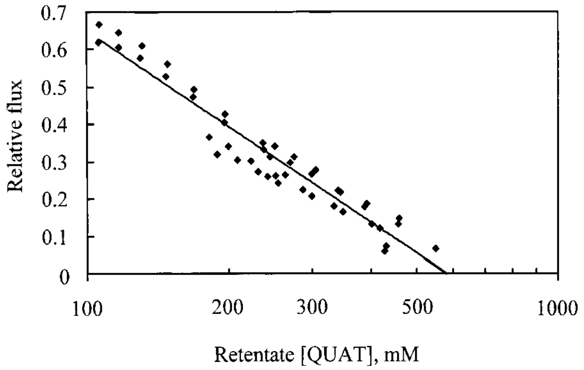

Figure 13. Relative flux of QUAT/sulfate system in PEUF at a [QUAT]/[sulfate] of 20.

feed). The initial relative flux will be 0.893 and the final relative flux will be 0.208 and water recovery will be 93.3%. If the processes were continuous, the relative flux and permeate [CrO_4^{2-}] will be between these extremes (an integrated average). However, this example shows that high purification with high water recovery and reasonable flux is attainable using PEUF. A higher final retentate [QUAT] yields a higher water recovery, lower average flux (more membrane area required), a slightly higher [CrO_4^{2-}] in the composite permeate, and a higher [CrO_4^{2-}] in the final retentate. A higher [QUAT]/[CrO_4^{2-}] feed ratio results in a purer composite permeate, lower average flux, lower water recovery, and lower

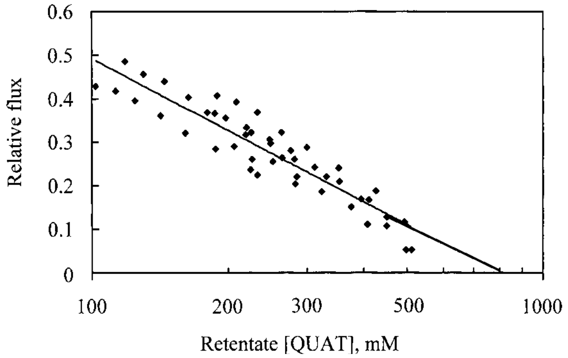

Figure 14. Relative flux of QUAT/nitrate system in PEUF at a [QUAT]/[nitrate] of 5.

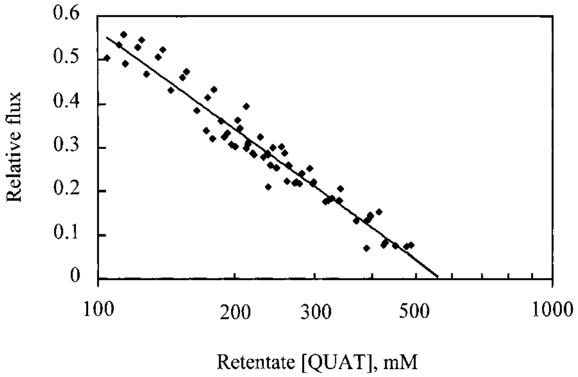

Figure 15. Relative flux of QUAT/nitrate system in PEUF at a [QUAT]/[nitrate] of 10.

Table 1. Gel Concentrations of QUAT (m*M*), Best Fit, and Range of Values Within 95% Confidence Level

Initial [QUAT]/[ANION]	5	10	20
Chromate	838 (778–913)	718 (670–776)	581 (549–621)
Sulfate	885 (780–1035)	719 (663–790)	582 (549–621)
Nitrate	797 (701–938)	574 (547–604)	559 (536–586)

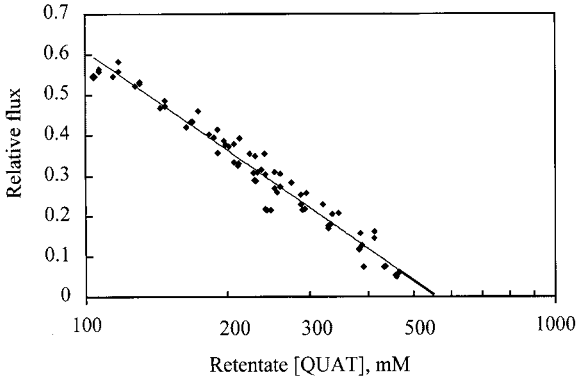

Figure 16. Relative flux of QUAT/nitrate system in PEUF at a [QUAT]/[nitrate] of 20.

$[CrO_4^{2-}]$ in the final retentate. So, the final retentate [QUAT] and the feed $[QUAT]/[CrO_4^{2-}]$ are optimization variables.

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