New Smopex® Ion Exchange Materials for the Removal of Selenium from Industrial Effluent Streams

Material characterisation, modelling and process implementation

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This article discusses new Smopex® ion exchange materials which have been developed by Johnson Matthey Water Technologies, and highlights their performance relative to other commercially available materials for the removal of selenocyanate, selenate and selenite ions from aqueous solutions. The ion exchange mechanisms by which these materials sorb these ions are also explained and modelled in order to highlight the additional benefits that these materials offer that non ion-exchange materials do not, such as the ability to achieve the full material exchange capacities at feed concentrations lower than 1 mg l\(^{-1}\) selenium. The unique characteristics of these fibrous type materials are also discussed, including fast sorption kinetics, facile regeneration and enhanced selectivity for selenium ions against competing sulfate ions. Finally, the performance of these materials in a continuous stirred tank reactor setup is demonstrated, showing that performance levels as high as in fixed bed processes can be achieved, due to the high selectivity and mass transfer kinetics of Smopex® materials.

1. Introduction

Selenium is a non-metal, trace element with crucial roles in animal and plant biology, although it becomes highly toxic at relatively low levels. The European Union suggests a maximum Se concentration of 10 \(\mu g\) l\(^{-1}\) in drinking water (1) while the guideline in USA was set at 50 \(\mu g\) l\(^{-1}\) (2). Selenium forms different water-soluble ions that can be found in aquatic environments from both natural and industrial origins. Fossil fuel and mining related activities, agriculture and glass manufacture constitute the most significant contributors to anthropogenic sources of selenium contamination (3).

Selenium remediation has been widely investigated during the past few decades, although today few technologies are being applied on a commercial scale (3). An efficient process must deal with the difficult task of removing selenium from large volumes at low concentrations (although above the regulated levels), the complex speciation of selenium, and the presence of high concentrations of sulfur. For example, wastewater from oil refineries can contain selenium in the order of few parts per million (ppm, mg l\(^{-1}\)), typically as a mixture of selenocyanate (SeCN\(^{-}\)) and selenate (SeO\(_4^{2-}\)) ions (4). Sulfate (SO\(_4^{2-}\)) would be found in the same feeds at more than 10 times that concentration (1). Treated effluents from flue-gas-desulphurisation (from coal combustion power plants) can contain ppm levels of selenium, most commonly as selenate, but as much as 1 g l\(^{-1}\) of sulfur in the form of sulfate (5).
Current technologies include chemical and biological methods. Amongst the first group it is easy to find processes based on the reduction of selenium ions using reagents such as zero-valent iron (6, 7) or sodium sulfide (8). Although relatively cost-effective techniques, they are extremely dependent on pH and temperature conditions and require vast quantities of non-reusable reactants. Biological methods still demand rigorous control of pH, salinity and temperature, although they consume smaller amounts of additional chemicals (3, 9). Anaerobic tank reactors or packed bed systems can be designed to accommodate bioreduction of selenium ions by specific bacteria strains. Treatment of large volumes of slurry waste is necessary to separate the solid elemental selenium.

Physical separation methods have also been explored for selenium remediation, but only reverse osmosis and nanofiltration seem to decrease the concentration of selenium ions below the acceptable discharge levels. Both technologies present high operating costs and require efficient pretreatment of feeds to avoid fouling and degradation of membranes (10).

Ion exchange technology is widely used for many different municipal and industrial wastewater treatments. All kinds of natural and synthetic sorbents have been investigated as potential materials for selenium remediation at bench scale, but there is no reference to full scale processes being implemented (3). Materials from varied groups such as resins, carbon-based adsorbents and metal oxides have been found to show potential activity to adsorb Se ions from water media. Amongst them, only a few synthetic materials were reported as having the necessary selenium capacity to be considered potential solutions (3). Unfortunately, most of them are unable to achieve good levels of selectivity for the removal of selenium in the presence of sulfur or other contaminants, limiting the prospects of any potential applications. Advanced selective ion exchange technology could help in mitigating these disadvantages.

In this article we discuss our investigations of the fundamentals of selective sorption of selenium ions from aqueous solutions using strong-base functionalised materials. Exploratory tests highlighted the potential of Johnson Matthey’s synthetic scavengers for the removal of inorganic selenium species. Strong-base type materials were identified as the best candidates (11), although weak-base type materials sometimes also show high affinities for inorganic anions such as phosphate (12) and arsenate (13). Strong-base functionalisation is generally based on fixed quaternary amines (ammonium cations), while weak-base type materials are based on alkyl and/or aromatic amines. The advantage of strong-base materials is that, thanks to having fixed positively-charged groups, they can act as anion exchangers in wider pH ranges than weak-base type materials. In this work we investigate in detail the performance of some materials within this class: the polymeric fibres Smopex®-103 and Smopex®-269, the silica-polymer composites WP11 and WP13, and two standard polystyrene resins from Dow Chemicals, Amberlite™ IRA 900 and Dowex™ 1x2 100–200. All these materials have fixed positively-charged groups and mobile chloride ions as the exchangeable anions. In particular, Smopex®-103 bears trimethylammonium groups (similar to Dow’s resins) while the functionality of Smopex®-269 is based on the aromatic base benzyl-pyridinium (Figure 1).

Smopex® (Figure 2) is a unique type of material where the binding functionality lies on side chains grafted onto 0.3 mm long polymeric fibres (olefin or natural) (14), in contrast with standard spherical porous resin beads used in common adsorption or ion exchange processes. This structure grants an efficient, fast recovery of the target species with very high loading capacity. On the other hand, Johnson Matthey’s silica-polymer composites could seem, in principle, a very different kind of material (Figure 2) (15), formed by a silica particle core coated with a polymer bound to the silica chemically. However, the active functionality is homogeneously distributed onto the surface of the coating polymer, allowing an excellent interaction with the liquid medium and thus enhancing properties such as kinetics and loading capacities, as in the case of Smopex®.

In the next sections we discuss experimental results and modelling work carried out in order to determine the principles governing the sorption of selenocyanate (SeCN⁻), selenite (SeO₃²⁻) and selenate (SeO₄²⁻) ions onto the different strong-base sorbents investigated. A special attention is paid to the removal of selenium ions in the presence of the competing ion sulfate (SO₄²⁻).
The effects caused by other potentially competing ions such as nitrate, phosphate or bicarbonate are not covered in this work.

2. Experimental

All chemicals were purchased from Alfa Aesar and scavenging materials were used as supplied by Johnson Matthey, Finland. Sodium selenite (Na₂SeO₃·5H₂O), sodium selenate (Na₂SeO₄·10H₂O) and potassium selenocyanate (KSeCN) were used as selenium sources for the model solutions, while sodium sulfate (Na₂SO₄ anhydrous) was used for sulfur containing feeds. Sodium chloride (NaCl, 99% min) solutions were used as eluent. Solutions were prepared at room temperature using demineralised water. First, an adequate amount of the corresponding salt was dissolved in water in a beaker and then diluted to the final volume in a volumetric flask. Further dilutions were carried out in order to prepare the very dilute solutions. pH was adjusted using small volumes of HCl 1 M or NaOH 1 M when necessary.

Please note that concentrations expressed in w/v terms in this document are based on elemental concentration, not on molecular concentration. For example, a KSeCN solution with Se concentration of 200 mg l⁻¹ would actually contain 365 mg l⁻¹ of KSeCN.

2.1 Analytical

Selenium and sulfur concentrations in the samples were determined by elemental analysis by ICP-MS (Perkin Elmer Elan 6100 DRC) or ICP-OES (Thermo Scientific ICAP 7600 Radial). All samples were acidified with 0.1 ml of 69% HNO₃ prior to analysis. Selenocyanate-containing samples were kept at basic pH (addition of 0.1 ml of 1 M NaOH) to prevent decomposition until just before the analysis. Chloride levels were analysed by ion chromatography. Dilution steps were included, when necessary, to adjust sample concentration to calibration window in the equipment. Results were compared to certified external standards.

2.2 Materials

The Smopex® materials are polypropylene fibres, having a trilobal shape and a typical length of 300 μm and diameter 50 μm (see Figure 2). The polypropylene fibres are cut to this length during the manufacturing stage, prior to functionalisation. The bulk density of the dry Smopex® fibres is approximately 275 g l⁻¹. The dry content (mass of dry material per mass of the supplied material which includes moisture) of the supplied Smopex® is approximately 60%. The spherical silica polymer composite materials have a particle diameter range of 250–750 μm and bulk densities for the dry material of 500 to 600 g l⁻¹. The silica polymer composite materials are supplied dry. Properties of the Amberlite™ IRA-900 and Dowex™ 1x2 can be readily found online, however the dry contents of these materials as supplied were measured at 61% and 75%, respectively. Bulk densities of the dry material were measured at approximately 373 g l⁻¹ and 468 g l⁻¹, respectively. All of the materials were used in the experimental trials as supplied.

2.3 Adsorption Tests

Different procedures were followed to carry out tests, depending on the nature of the experiment. They are based on standardised procedures used by Johnson
Matthey Water Technologies. In this investigation all tests were done at room temperature.

2.3.1 Determination of Batch Kinetic Profiles
These tests were carried out at neutral pH and 25°C. Scavenger masses of 0.150 g (based on dry content) were put in contact with 15.0 ml of selenate, selenite or selenocyanate solutions with 1.0 mg l⁻¹ of Se (0.013 mmol l⁻¹). The mixtures were allowed to react for different lengths of time under gentle stirring (60 rpm), typically for 1, 2, 5, 10, 30, 60 and 120 minutes. Sorption tests with 30 seconds of contact time were also performed for Smopex® materials. Once the reaction time had passed, the solutions were filtered out from the suspensions and analysed by ICP-MS.

2.3.2 Determination of Adsorption Isotherms
The procedure requires using fixed concentration and volumes of solution and varying masses of scavenger material in parallel tubes in order to cover the expected range of loadings and equilibrium concentrations (Figure 3). These tests were carried out at room temperature and allowing enough reaction times to reach equilibrium conditions (generally overnight). The Se and S initial concentrations were kept fixed for a given isotherm trial but varied across different trials, and ranged from 10 to 500 mg l⁻¹ (Se: 0.13 to 6.33 mmol l⁻¹; S: 0.31 to 15.6 mmol l⁻¹). The required masses of the materials were calculated based on an ‘expected maximum loading’, i.e., a mass range of material was added such that the equilibrium concentrations at the end of the trial were expected to range from 10% to 90% of the initial concentration value. Competitive isotherms with sulfate were run using Se:S equimolar solutions with 150 mg l⁻¹ of Se and 60 mg l⁻¹ of S (1.9 mmol l⁻¹).

2.3.3 Continuous Flow Column Tests
The general procedure is based on passing a feed of known Se or Se/S concentration through a column (Figure 4) containing a known amount of scavenging material, generally between 1 and 2 g (dry content). The fixed-beds in these experiments had volumes between 3 and 7 ml. Flow rates were kept constant at 36 ml h⁻¹ while the lengths of the experiments were altered depending on the case. Initial Se concentration was adjusted to 1.0 g l⁻¹ (12.7 mmol l⁻¹) for Smopex® fibres and Dow resins and to 0.50 g l⁻¹ (6.3 mmol l⁻¹) for silica-polymer composites. Lower initial concentrations were used for the latter materials in order to optimise the length of the experiment, as they have generally lower maximum materials concentration than Smopex® and Dow materials.

For competitive tests with sulfate, a Se:S mass ratio of 1:12 was used (1:30 molar ratio) with initial Se concentrations of 0.50 and 0.25 g l⁻¹ (6.3 and 3.2 mmol l⁻¹) and S levels of 6.0 and 3.0 g l⁻¹ respectively (187 and 93.6 mmol l⁻¹). Desorption studies were carried out in a similar way, but passing NaCl solutions through the column at similar rates (36 ml h⁻¹), containing in this case about 1.0 g of scavenger pre-loaded with selenium ions. Eluents with different chloride concentration were used, ranging from 0.06 mol l⁻¹ (0.2 wt%) to 2.82 mol l⁻¹ (10 wt%).

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Fig. 3. Experimental apparatus (temperature controlled multi-vial carousel) for batch kinetics test and isotherms determination studies

Fig. 4. Experimental apparatus (column and fraction collector) for the continuous flow column tests
Samples were collected downstream at regular intervals using an automated fraction collector.

2.3.4 Continuous Stirring Tank Reactor

For this test a baffled cylindrical glass reactor, fabricated in house, was used (Figure 5) which allowed continuous flow (at 100 ml h⁻¹) into the reactor of a SeCN⁻ feed having a concentration 450 mg l⁻¹ (5.7 mmol l⁻¹). The reactor liquid level was controlled using a glass dip tube. Both the inlet tube at the bottom of the reactor and dip tube were fitted with glass frits to prevent Smopex® exiting the reactor. 30 g (dry content) of Smopex® 103 was vigorously mixed with the reactor solution for the duration of the test. The operating volume of the reactor was 0.64 l. Samples were manually collected downstream at regular intervals (every hour). The experimental results from a single continuous stirring tank reactor (CSTR) are presented in Section 4.4. The model was validated for the single CSTR system and deemed to be suitable for accurately predicting performance in the two CSTR system, therefore only the predicted performance for a two CSTR in-series system are shown with no experimental results to verify these predictions.

3. Modelling

The modelling of the ion exchange mechanisms discussed in this paper is an important task as it not only enables the rigorous scale-up of material behaviour from lab to plant scale, but also further informs material researchers with additional information which would not usually be elucidated from simple charts and with which they can think up new interesting experiments. It must be emphasised that while modelling in this paper appears to be the representation of sorption mechanisms through mathematical equations, the bulk of the modelling exercise undertaken here is rather the understanding of the real sorption mechanisms taking place so that the correct mathematical expressions can then be applied. Special focus throughout the paper will thus be given to highlighting the mechanisms involved in the sorption processes and how they influence the choice of mathematical models.

Modelling the removal of ions from solution by ion exchange involves both mass transfer and equilibrium equations (16). Typical models used to represent sorption equilibrium are the Langmuir, Freundlich and mass action law models (17–18). The Langmuir and Freundlich models both consider a material as having adsorption sites with equal or different adsorption potentials for solution ions, and are often used to model the sorption isotherms of both adsorption and ion exchange systems as the fit to experimental data in both cases is often very good (19–21). Several authors (22–23) have already discussed the unsuitability of these models in accounting for all the mechanisms at play in ion exchange systems, leading to inaccurate and unreliable predictive models for scale-up and design of ion exchange processes. The effect of these mechanisms on the engineering of ion exchange processes is further discussed and featured in this study. The mass action law has been used to model the specific mechanisms of ion exchange systems, such as electroneutrality in the material, non-ideality of the solution and material phases as well as the effect of the ion released from the material during ion exchange (24). Equilibrium is represented in the mass action model by Equation (i):

\[ K_{i-j} = \frac{\gamma_{mi} q_i^{z_i} \gamma_{sj} C_j^{z_j}}{\gamma_{mj} q_j^{z_j} \gamma_{si} C_i^{z_i}} \]  

where \(i\) and \(j\) refer to two distinct ionic species in the system, \(K_{i-j}\) is the thermodynamic equilibrium constant, \(\gamma_{si}\) and \(\gamma_{sj}\) are the activity coefficients in the liquid phase, \(\gamma_{mi}\) and \(\gamma_{mj}\) are the activity coefficients on the material, \(q_i^{z_i}\) and \(q_j^{z_j}\) are the concentrations in the material phase at equilibrium (eq g⁻¹), \(C_i^{z_i}\) and \(C_j^{z_j}\) are the concentrations in the liquid phase at equilibrium (eq l⁻¹) and \(z_i\) and \(z_j\) are the absolute values of each species’ valency. The sum of the concentrations on the
material phase is equal to the real exchange capacity 
(REC) on the material, as given by Equation (ii):

\[ \text{REC} = q_1^* + q_2^* + \ldots + q_n^* \]  

Where \( n \) is the number of ionic species in a given system, including the species initially present and then released from the material and with which ion exchange can occur. The REC is determined experimentally and is usually lower than the theoretical exchange capacity (TEC) of a material which is measured by chemical analysis of the material (23).

In order to estimate activity coefficients for a given system, the Bromley method (25) has been used for estimating the liquid phase coefficients and the Wilson method (26) for the material phase coefficients. Estimating the values of additional parameters in the Wilson model and the values of the equilibrium constants in Equation (i) is achieved by using nonlinear optimisation methods to minimise predicted and experimental equilibrium data (for example sorption isotherms) from binary systems. A full description on the use of these methods has been well described by other authors (24) and will not be repeated here.

The equilibrium models are then combined with mass transfer models to predict the dynamic sorption behaviour of adsorption and ion exchange processes. As most commercially available ion exchange resins are typically macroporous polymeric resin beads with chemical functionality located both within the pores of the resin and on the surface (27), bulk mass transfer models are combined with film diffusion and intra-particle diffusion models to predict the material concentration profiles as a function of time (23). Furthermore as commercial processes usually consist of fixed-beds through which fresh feed is continuously introduced, concentration profiles which vary along the axis of the column must be predicted by the model as well as axial dispersion effects, ultimately resulting in a large set of partial differential and non-linear equations which must be solved simultaneously.

The system under study here is somewhat simplified as first of all, the Smopex® materials have all of their functionality located on the surface of the material and thus only bulk and film diffusion mass transfer equations need be considered. Secondly, the Smopex® behaviour has specifically been studied in a CSTR setup, where perfect mixing was assumed and the dynamic behaviour can be represented by ordinary differential equations. The film diffusion mass transfer equation is given by Equation (iii) (28):

\[ \frac{dq_i}{dt} = \frac{3}{r \rho} k_f (C_{bi} - C_{fi}^*) \]  

where \( r \) is the material particle radius (m), \( \rho \) the particle density (g l\(^{-1}\)), \( k_f \) the film mass transfer coefficient (m s\(^{-1}\)), \( C_{bi} \) the bulk solution concentration of species \( i \) (eq l\(^{-1}\)) and \( C_{fi}^* \) the concentration of species \( i \) (eq l\(^{-1}\)) at the surface of the material, at equilibrium with the ion concentration in the material \( q_i \). \( C_{fi}^* \) is equivalent to \( C_{fi} \) in Equation (i). The bulk mass transfer in the CSTR is given by (Equation (iv) (28)):

\[ \frac{dC_{bi}}{dt} = -\frac{m}{V} \frac{dq_i}{dt} + \frac{Q_i (C_{fi} - C_{ni})}{V} \]  

where \( m \) is the mass of material in the system (g), \( V \) is the volume of liquid (l), \( Q_i \) is the feed flow rate into the CSTR (l s\(^{-1}\)) and \( C_{ni} \) is the concentration of species \( i \) in the feed (eq l\(^{-1}\)).

In modelling the ion exchange sorption in the CSTR process (see Section 4.4), the following assumptions were made:

- The only resistance to mass transfer is film diffusion resistance
- Ion exchange at the liquid/solid interface, i.e. the material surface is instantaneous and the equilibrium between both phases can be represented by the mass action law
- The process occurs under isobaric and isothermal conditions
- Physical properties of the ion exchanger and liquid are constant
- As Smopex® materials are not spherical, the radius used in Equation (iii) corresponded to a sphere with equivalent surface area to a typical Smopex® particle (approximately 60 \( \mu \)m).

Equations (i)–(iv) were solved using the Athena visual studio (29) software to obtain the predicted liquid and material phase concentrations as a function of time of all ionic species involved in the ion exchange. Additional model parameters, such as the particle radius \( r \) and particle density \( \rho \), were obtained from experimental measurements or material characterisation techniques, while the film mass transfer coefficient \( k_f \) was estimated from the experimental bulk liquid concentration data.
4. Results and Discussion

4.1 Material Selectivity

In Figures 6, 7 and 8, the sorption isotherm results for selenocyanate, selenate and sulfate ions, respectively, are plotted for the four functionalised materials. The points represent experimental measurements while the dashed lines are fitted lines with no physical significance but which assist in interpretation of the material sorption behaviour.

In Figure 6, Smopex®-269 is the most ‘selective’ material for selenocyanate, since it has the highest material concentration at low liquid concentrations, while the highest material concentration is achieved on Smopex®-103, at approximately 1.7 mmol per gram of dry Smopex®-103. Interestingly, this concentration is approximately equal to the measured nitrogen content on Smopex®-103 of 1.8 mmol g⁻¹, i.e. the TEC, suggesting that nearly all of the functionality on the material is available for sorption. This is likely linked to design of the Smopex® material where all of the functionality is located on the surface of the polymeric fibres, in contrast to typical sorption materials where most of the functionality is located within the porous structure of the material. WP11 has the overall lowest material concentrations but appears to be more selective than WP13. The maximum material concentration of WP13 was not reached in these experiments but is likely to be similar to that of Smopex®-269 at approximately 1.2 mmol g⁻¹.

It must be pointed out that there is one critical omission from these results: the equilibrium liquid and material concentrations of chloride, which also change as selenocyanate concentrations change, due to ionic exchange of chloride and selenocyanate between the material and liquid solution. The sorption isotherms experimentally measured here are only valid for systems having equal levels of chloride initially present on the material. Thus a material having more or less functionality and hence differing initial concentrations of chloride on the material will lead to a different selenocyanate sorption isotherm. In the case of sorption by adsorption or chelation, there would be no exchanged ion from the material and so the isotherm itself would only depend on those ions initially in solution. Thus models such as the Langmuir or Freundlich isotherms are not applicable for modelling this system, even though they would probably fit the experimental data very well.

In Figure 7, Smopex®-103 is now the material with both the highest material concentration and selectivity for selenate. The maximum material concentration has approximately halved from 1.7 mmol g⁻¹ for selenocyanate to approximately 0.85 mmol g⁻¹ for selenate. This is because the selenate ion is divalent so two chloride ions must be exchanged in order to preserve electroneutrality in the material. The same decrease in capacity is observed for all four materials. This behaviour would not typically occur with materials where sorption occurs through chelation or adsorption, as there is no exchanged ion and thus valency is not important in determining material concentrations. The selectivity and maximum material concentrations of Smopex®-269 and WP13 are approximately equal for selenate. Contrasting Figures 6 and 7, it is clear that Smopex®-269 is no longer the material with highest

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Fig. 6. Selenocyanate sorption isotherm. ‘Equilibrium material concentration’ is the concentration of the sorbed species on the material, expressed in mmol of species per gram of dry material. The initial SeCN⁻ concentration was approximately 6 mmol l⁻¹ and volume of solution per sample was 200 ml.

Fig. 7. Selenate sorption isotherm. The initial SeO₄²⁻ concentration was approximately 3 mmol l⁻¹ and volume of solution per sample was 200 ml.
selectivity, as it was for selenocyanate. It is believed that the sorption sites of Smopex®-269 are hindered by bulkier molecular groups when compared to the other materials, which mitigates the sorption of the selenate ion, while the smaller and linear shaped selenocyanate ion is unaffected. Thus while Smopex®-269 still exhibits good recovery for divalent selenate ions, optimal use of its particular functionality is achieved when monovalent ions are recovered.

In Figure 8, WP13 has the highest selectivity but Smopex®-103 has again the highest overall maximum material concentration. The relative selectivity of both Smopex®-103 and Smopex®-269 for sulfate over selenocyanate has now decreased relative to the silica-polymer materials, with Smopex®-269 having the overall lowest selectivity for sulfate. Since sulfate recovery is generally undesired, this lower selectivity is a beneficial property of Smopex® materials, as selenium removal will be favoured in feeds containing both selenium and sulfate ions. Due to their design, the functionality of both Smopex® materials have a higher ionic character than those of the silica-polymer materials, which in turn leads to a stronger sorption interaction with the selenate, due to the selenate having a higher charge density on its oxygen atoms than sulfate. Note that the scale of the equilibrium liquid concentration axis is different in Figure 8 due to the lower molecular weight of sulfate over selenium and thus overall the selectivity of all materials for sulfate has decreased relative to selenium ions.

In Figure 9 the effect of equilibrium pH on the sorption performance of selenite on Smopex®-103 is depicted. The solution pH of each experimental trial was adjusted prior to the addition of material and then measured at equilibrium, where they were found to have not significantly varied. The results found matched expectations and ion exchange theory. Sorption performances of selenocyanate and selenate were not affected by changes in pH within the range 3–10 (acidic conditions for selenocyanate were not investigated as the ion decomposes under those conditions). On the other hand, the performance for selenite was found to be highly influenced by the pH of the solution. The strong-base functionality on Smopex®-103 remains unaltered in a broad pH range, and neither the monovalent selenocyanate nor the divalent selenate ion change their protonation states in the pH range investigated (conjugated bases of very strong acids: pK<sub>a</sub>(HSeCN) < 1, pK<sub>a</sub>(H<sub>2</sub>SeO<sub>3</sub>) = –3.0, pK<sub>a</sub>(HSeO<sub>4</sub><sup>–</sup>) = 1.7). On the other hand, selenous acid is a considerably weaker acid than the other species (pK<sub>a</sub>(H<sub>2</sub>SeO<sub>3</sub>) = 2.6, pK<sub>a</sub>(HSeO<sub>3</sub><sup>–</sup>) = 8.1). Thus, the optimum performance for the adsorption of selenite ions takes place at pH 10, where almost all selenium is in the form of the fully deprotonated SeO<sub>3</sub><sup>2–</sup> (ca. 99% of the total selenium content). A selenite concentration on the material of 0.7 mmol g<sup>–1</sup> was achieved at pH 10. Under neutral conditions, ca. 93% of the selenium present is HSeO<sub>3</sub><sup>–</sup> with the rest being in the form of SeO<sub>4</sub><sup>2–</sup> (ca. 7%). Thus, a significant decrease of the selectivity is observed at pH 7, as reflected in the shift to higher equilibrium liquid concentrations for the exchange isotherm, although maximum material concentration remains similar to that at pH 10. A major decline in performance is observed under acidic conditions, with a further shift to higher equilibrium liquid concentrations and a significant reduction in material concentrations. At pH 3, the major Se species is HSeO<sub>3</sub><sup>–</sup> (ca. 70%), with significant...
presence of fully protonated, selenous acid (\(\text{H}_2\text{SeO}_3^-\), ca. 30\%) and nothing of the fully deprotonated \(\text{SeO}_2^{2-}\). In these conditions, a moderate loading of 0.3 mmol g\(^{-1}\) was measured, most likely originated by the uptake of available monovalent \(\text{HSeO}_3^-\) ions.

These results indicate that the Smopex\(^{®}\)-103 material presents a greater selectivity for divalent \(\text{SeO}_2^{2-}\) than for monovalent \(\text{HSeO}_3^-\), and that it is incapable of scavenging the neutral species \(\text{H}_2\text{SeO}_3\), consistent with the ion exchange nature of the process. Different sorption mechanisms are required to efficiently remove selenous acid under acidic conditions, as demonstrated by Awual \textit{et al.} using silica based material functionalised with chromogenic Schiff bases (30, 31).

In Figures 10 and 11, the effect of initial selenium concentration in solution on the sorption isotherms is shown. Again, in the case of adsorption or chelation, a fixed isotherm would be expected with changing initial concentration. In the case of ion exchange, a varying isotherm can be observed depending on the relative ionic valency of the ions exchanged. In Figure 10, for the case of selenocyanate and chloride exchange, sorption isotherms at both initial selenium concentrations overlap, as the valency of each ionic species is one and hence the isotherm does not depend on initial concentration. In Figure 11, the sorption isotherm of the divalent selenate ion changes as a function of initial selenate concentration, with ‘apparent selectivity’ increasing with decreasing initial concentration. These effects are attributed to a phenomenon known as the Donnan potential (18). The material initially has a higher chloride anion concentration than the surrounding solution which results in the chloride ions moving out of the material into solution by diffusion. However, since electroneutrality with the fixed cations in the material must be preserved, the chloride ions are pulled back into the material by an electric potential difference, the strength of which increases as the relative electric potential between material and solution increases (for example as solution dilution increases). For a given electric potential difference, the force with which it acts on an ion also increases with ionic charge. Divalent selenate ions are thus more strongly attracted by this electrical potential than the monovalent chloride ions and hence favoured by the material. As dilution increases and the electric potential force increases, this relative preference of the selenate over chloride ions increases further, resulting in the observed increase in ‘apparent selectivity’. The only suitable model for predicting this behaviour must thus include as parameters the selenium and chloride concentrations as well as the valency of the respective ions and activity coefficients to account for the non-ideal sorption behaviour due to the Donnan potential effects.

Figures 12 and 13 show the selenium sorption isotherms for solutions having both selenocyanate and sulfate initially present in solution, for Smopex\(^{®}\)-269 and WP11 respectively. Only the results for these materials are shown as Smopex\(^{®}\)-269 was observed (Figure 7) to have the highest overall selectivity for selenocyanate and lowest for sulfate, while WP11 had the lowest selectivity for selenocyanate. In Figure 12 the selenocyanate concentration on Smopex\(^{®}\)-269 is very selective over sulfate, with sulfate desorbing from the material as liquid concentrations increase. A maximum 5:1 ratio of equilibrium concentrations for
selenocyanate:sulfate is achieved. A maximum 3:1 ratio was achieved with Smopex®-103. By contrast, in Figure 13, there is no discernible difference in selectivity for selenocyanate versus sulfate on the WP11 material. The strong base Smopex® materials are thus well suited to selectively removing selenocyanate from solution in process streams containing both selenocyanate and sulfate.

Figures 14 and 15 show the selenium sorption isotherms in solutions having both selenate and sulfate initially present in equimolar concentrations, for Smopex®-103 and WP11. In this case, there is still a significant selectivity of selenate over sulfate on Smopex®-103 (an approximate ratio of 1.5:1), which is interesting considering the fundamental similarities between these ions. WP11 again exhibits no difference in selectivity for both ions. Smopex®-269 achieved an approximate 2:1 ratio, greater than Smopex®-103, however the overall maximum material concentration is still greater for Smopex®-103. As mentioned previously, the higher ionic character of both Smopex® materials likely leads to a stronger interaction with the selenate.

### 4.2 Material Kinetics

In Figure 16 the sorption of selenate kinetics for the different materials is shown. For all materials, approximately 90% of the selenate concentration is transferred to the materials within ten minutes. For the two Smopex® materials, this material concentration is reached within one minute. Typical ion exchange materials would not exhibit such fast kinetics, as they are porous resins with most of the functionality located
within the material pores. The ions in solution must thus diffuse slowly through the resins in order to reach the functionality. In contrast, all of the functionality on these silica-polymer resins and the Smopex® materials is located on the particle surface and hence is much easier for the liquid ions to access. Only diffusion through the stagnant film surrounding the material particles needs to be considered in kinetic modelling of sorption for these materials. The Smopex® sorption kinetics are believed to be so fast relative to the silica-polymer materials, due to the rod shaped geometry of Smopex® fibres as well as their small particle size, resulting in a generally higher surface area per volume of material than the larger silica-polymer resins. Similar trends in the rates of sorption were observed for selenocyanate and selenite.

4.3 Continuous Flow Through Fixed Bed Columns

In Figure 17 the column outlet concentrations of selenocyanate, selenate and selenite are shown as a function of the mass of selenium fed to each fixed bed column of Smopex®-103. For all selenium species the column outlet concentration is 0 mmol l\(^{-1}\) then increases sharply to the feed selenium concentration value of approximately 12 mmol l\(^{-1}\). Data from fixed bed experiments is typically represented in this manner, however an alternative format has been adopted in this study for ease of performance comparison between different material types, and will be used in all subsequent column results figures.

In Figures 18 and 19 the performance of Smopex®, silica-polymer resins and two commercially available polystyrene ion exchange resins, Dowex™ 1x2 100–200 and Amberlite™ IRA 900 is plotted using the alternative format. The maximum concentrations achieved for the Smopex® and silica-polymer resins are also in agreement with the values seen in the sorption isotherms (Figure 6). An important factor in the design of fixed bed ion exchange systems is the material utilisation factor, defined here as the fraction of the maximum material concentration which has been reached at the point where a detectable concentration of the targeted ion for removal from solution (for example selenocyanate) appears in the
column outlet solution. For full-scale ion exchange processes, the drive is often to minimise column size while maintaining high (greater than 0.9) material utilisation factors, as column size dictates material (i.e. capital) costs. In Figure 18 the material utilisation factors are approximately 0.75, 0.85 and 0.5 for the Smopex®, silica-polymer and Dowex™ resin, and Amberlite™ materials respectively. Also considering the maximum material concentrations shown in Figure 19, the Smopex®-103 is reaching the overall highest material concentrations before selenocyanate starts to appear in the column outlet. The WP11 material achieves the lowest material concentration. The Amberlite™ material presents the highest material concentration overall, but as mentioned above it also has the lowest utilisation factor. This reflects the low selectivity and slow sorption kinetics this material likely has for the selenocyanate species, resulting in a larger ‘mass transfer zone’ in the fixed bed, i.e. the length of the bed over which exchange of selenocyanate ions from liquid to the material occurs. As Amberlite™ is also a standard polystyrene type resin, most of the functionality is likely located within the particle and thus the sorption rate decreases significantly as the material concentration increases since more time is required for the selenium to diffuse into the particle to reach the available functionality.

The Dowex™ material exhibits a similar maximum material concentration to Smopex®-269, with a higher utilisation factor. In the case of Dowex™ this is believed to be predominantly due to its low particle size of 100 to 200 μm which increases material surface area in contact with the solution and decreases the internal particle distance for porous diffusion, both likely to result in faster sorption kinetics and a smaller mass transfer zone. The silica-polymer materials exhibit similar utilisation factors to Dowex™ while having a much larger average particle size of 500 μm, reaffirming the idea that having the functionality on the surface of the material and hence exposed to the solution is very beneficial to material performance in fixed beds. Larger particle sizes are also important for limiting the pressure drop across fixed beds in full-scale applications with high feed flow rates.

In Figures 20 and 21 the fixed bed performance charts are shown for a feed solution containing selenate ions. Again the Smopex® materials achieve the highest utilisation factors, approximately 0.9 for both materials, again with maximum values consistent with the sorption isotherms in Figure 7. In this case WP11 has the lowest material concentrations and utilisation factor. The Amberlite™ material achieves the highest material concentrations but again a low utilisation factor.

In Figures 22 and 23 the fixed bed performance for a feed containing both selenocyanate and sulfate, both present at a molar concentration ratio of 1 to 30, respectively is shown. This low selenocyanate:sulfate ratio was selected in order to study sorption performance under realistic conditions, similar to those in industrial wastewaters having a highly competitive environment for selenium removal. Considering this, low material concentrations of selenocyanate would be expected due to the competitive sorption
of sulfate on the materials. The maximum material concentrations of selenocyanate on Smopex®-103 and Smopex®-269 are indeed lower than in the case of a feed with selenocyanate only, but for Smopex®-103 the maximum material concentration has only decreased to approximately 1.2 mmol g⁻¹ from the maximum observed value of 1.8 mmol g⁻¹ with only selenocyanate present in solution, while for Smopex®-269 the maximum material concentration is approximately 1.05 mmol g⁻¹, down from the maximum of 1.2 mmol g⁻¹. Furthermore, Smopex®-269 was shown in the sorption isotherms to have the highest relative selectivity for selenocyanate over sulfate, while it is also the material with the lowest decrease in capacity, approximately 12%, when sulfate is introduced to the feed. The Dowex™, Amberlite™ and Smopex®-103 materials all show an approximate 30% decrease in maximum material concentration when sulfate is present in the feed. Overall, considering the absolute maximum material concentrations and utilisation factors, Smopex®-103 is the best material for selenocyanate removal in this competitive feed solution as it has the highest material concentration at the point when selenium appears in the column outlet. At even higher sulfate to selenocyanate ratios however, it is likely that the Smopex®-269 would perform better than Smopex®-103 due to its higher relative selectivity of selenocyanate over sulfate.

Another important factor to consider is the absolute values of the selenocyanate and sulfate concentrations in the feed, where these experiments were carried out with selenocyanate and sulfate feed concentrations of 6 and 200 mmol l⁻¹ respectively, for the Smopex® and Dow materials and 3.2 mmol l⁻¹ and 93.6 mmol l⁻¹ respectively, for the silica polymer composite materials.
0.37 mmol l\(^{-1}\), respectively (equivalent to 1 mg l\(^{-1}\) of selenium and 12 mg l\(^{-1}\) of sulfate). These results are shown in Figures 24 and 25. Due to the laborious nature of these tests (more than 14 weeks of continuous flow being required to reach maximum material concentrations on only 1 g of material in the column), only results for Smopex\(^®\)-103 were obtained.

Contrasting Figures 25 and 23, maximum material concentration of selenocyanate on Smopex\(^®\)-103 for the feed with selenocyanate to sulfate concentration ratio of 1:30 has decreased from approximately 1.2 mmol g\(^{-1}\) to 0.35 mmol g\(^{-1}\). This is due to the increased ‘apparent selectivity’ of the sulfate at these lower feed concentrations. During the experiment, once this maximum material concentration for selenocyanate was reached, the feed selenocyanate concentration was increased such that the selenocyanate to sulfate concentration ratio was decreased to 1:15. Maximum material concentration for selenocyanate subsequently increased to approximately 0.45 mmol g\(^{-1}\). The feed selenocyanate concentration was further increased to lower the concentration ratio to 1:2.5. Maximum material concentration subsequently increased to approximately 1.2 mmol g\(^{-1}\). Selenocyanate sorption versus sulfate has thus clearly been affected by the absolute feed concentrations of each ion in the feed.

At the higher absolute feed concentrations, the Smopex\(^®\)-103 was the overall best choice of material for selenocyanate sorption, however at the lower absolute feed concentrations, the Smopex\(^®\)-269 material is likely to perform better than the Smopex\(^®\)-103 due to its greater relative selectivity for selenocyanate over sulfate. Choosing the best material for selenocyanate sorption is thus a complex choice as it depends on many factors, hence the correct choice of model to predict material performance at differing absolute feed concentrations is critical.

Also shown in Figures 24 and 25 is the column performance for a feed containing only selenocyanate, but at the lower feed concentration of 0.013 mmol l\(^{-1}\), compared to 6 mmol l\(^{-1}\) used previously (Figure 22). A maximum material concentration of approximately 1.7 mmol g\(^{-1}\) was reached, very close to the observed maximum in the selenocyanate sorption isotherm (Figure 6).

Considering the frequently employed adsorption models, for example Langmuir or Freundlich, the reader might be surprised that the approximate total exchange capacity of the material was reached with such a low selenocyanate concentration in the feed. According to these sorption models, for a given feed concentration the corresponding material concentration, as given by the sorption isotherm, is also the maximum material concentration that should be attainable experimentally, since equilibrium between the material and solution is reached at these conditions and no further ions can be loaded without an increase in feed concentration. In the case of ion exchange however, the situation is different as there is also an ion from the material which is exchanged and released into solution. In the case of these particular experiments, the chloride released from

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**Fig. 24.** Column material concentration versus column outlet concentration profile for a mixed selenocyanate and sulfate feed solution at low concentrations (0.013 mmol l\(^{-1}\) SeCN\(^{-}\) and 0.37 mmol l\(^{-1}\) SO\(_4^{2-}\)). Results are also shown for a feed solution containing only selenocyanate at low concentration (0.013 mmol l\(^{-1}\))

**Fig. 25.** Column material concentrations versus selenium fed to the columns, for a mixed selenocyanate and sulfate feed solution at low concentrations (0.013 mmol l\(^{-1}\) SeCN\(^{-}\) and 0.37 mmol l\(^{-1}\) SO\(_4^{2-}\)). Results are also shown for a feed solution containing only selenocyanate at low concentration...
the material is also subsequently washed out of the fixed bed due to the feed flow through the fixed bed. Thus chloride concentrations inside the fixed bed are always decreasing and equilibrium is continuously pushed in favour of more chloride releasing into solution, which in turn requires selenocyanate to be exchanged into the material. In summary, provided there is no chloride in the feed itself then in a fixed bed the selenocyanate material concentration should always reach the highest maximum material concentration observed experimentally in sorption isotherms, regardless of the absolute feed selenocyanate concentration.

This phenomenon also highlights another point, that if only chloride ions were present in the feed, regardless of their concentration, complete desorption of loaded selenocyanate from the material should be achievable, even though the selenocyanate ion has a much greater selectivity for the material than chloride ion. The results of fixed bed trials carried out to investigate this are shown in Figures 26 and 27.

Figures 26 and 27 both show that significant selenocyanate desorption is achievable regardless of the feed chloride concentration. While not all the experiments were continued until a maximum selenocyanate desorption was achieved, in all experiments the selenocyanate desorption trend is still increasing when flow to the fixed bed was stopped and heading towards an approximate desorption value of 1.6 mmol g\(^{-1}\) selenocyanate. It was previously mentioned that the total material capacity of Smopex\textsuperscript{®} 103 is approximately 1.8 mmol g\(^{-1}\) of dry material, however this is only valid when the material is initially concentrated with chloride ions. When concentrated with selenocyanate ions (having higher molecular weight than chloride ions) the mass of the material is subsequently increased which reduces the maximum material concentration per gram of dry material to 1.6 mmol g\(^{-1}\). This value is in agreement with the maximum achieved selenocyanate desorption for the experiment having feed chloride concentration of 2.82 mol l\(^{-1}\), indicating complete desorption of the selenocyanate from the material. Even for the experiment having fifty times less chloride in the feed, i.e. 0.06 mol l\(^{-1}\), a significant desorption of approximately 1.0 mmol g\(^{-1}\) selenocyanate was achieved after 30 bed volumes of flow through the fixed bed.

Considering adsorption theory, such a significant desorption of selenocyanate using a low concentration feed of a competitive ion such as chloride would not be expected. The material concentration of chloride ions on the material would quickly reach equilibrium with the low feed concentration, mitigating the exchange of further chloride ions into the material. In Figure 27, it can also be seen that the low concentration chloride feed is also the most effective at desorbing selenocyanate, per mass of chloride that has flowed through the fixed bed, with efficiency decreasing as feed chloride concentration increases. This is believed to be predominantly due to increases in the viscosity of the feed solution as chloride concentration increases, mitigating the diffusion rate of chloride ions to the surface of the Smopex\textsuperscript{®} material, rather than any ion

![Fig. 26. Selenocyanate desorption versus material bed volumes of chloride solution flowed through the fixed bed, for varying concentrations of chloride in the feed solution. A bed volume is defined as the volume flowed through the column divided by the resin bed volume.](image-url-1)

![Fig. 27. Selenocyanate desorption versus mass of chloride flowed through the fixed bed, for varying concentrations of chloride in the feed solution.](image-url-2)
exchange related phenomena. A 2.82 mol l\(^{-1}\) chloride solution has a viscosity of approximately 0.002 Pa s at 25°C, compared to approximately 0.001 Pa s for water at 25°C, a two-fold increase. Finally, while the low concentration chloride feed was the most efficient for selenocynate desorption, average selenocyanate concentrations in the outlet stream are also lowest for that feed. If a concentrated stream of selenocyanate is desired from the desorption process, then the higher chloride feed concentrations are required.

Selectivity of selenate over sulfate was also investigated in continuous flow experiments for Smopex\(^\circledast\)-103, the silica polymer composite WP13 and the Dow resins. Under the conditions tested, with a selenium:sulfur molar ratio of 1:30 and initial feed selenate concentration of 6.3 mol l\(^{-1}\), maximum material concentrations did not exceed 0.12 mmol g\(^{-1}\) (1 wt% Se) for any of the materials. Even though these results may seem very poor, a viable commercial process could be designed by implementing frequent regeneration cycles of the material once this low maximum concentration is reached (32).

4.4 Process Implementation of Smopex\(^\circledast\)

Smopex\(^\circledast\) materials have been shown to outperform all other tested materials in terms of kinetics, selectivity and maximum material concentrations. However due to the structure of Smopex\(^\circledast\), where functionalised polymeric chains are attached to a relatively small backbone polymer, a fixed bed of Smopex\(^\circledast\) can be easily compressed and at high feed flow rates (for example greater than 0.001 m s\(^{-1}\) superficial velocity) the pressure drop across a fixed bed can be significantly higher than if a silica or polystyrene ion exchange were used (often in excess of 5 bar per metre of fixed bed length). For such applications, an alternative process configuration is required. In this study, the CSTR has been explored as a viable process configuration for Smopex\(^\circledast\) materials. In contrast to fixed bed reactors, material utilisation factors in CSTR are generally lower, since concentration gradients between the bulk liquid and the material surface are lower than in fixed beds. Many CSTR connected in series are often required to achieve comparable material utilisation factors to fixed beds.

However, as will be shown from the trials carried out here, comparable material utilisation factors to fixed beds can be achieved using Smopex\(^\circledast\) and only two CSTR in series, due to the high selectivity and exchange kinetics of Smopex\(^\circledast\) materials.

In Figures 28 and 29 the experimentally measured and predicted reactor liquid and material concentrations are shown over the duration of the experiment using a single CSTR. The chloride liquid concentrations were only measured for the first 50 hours, in order to validate the predicted values. The experimental and model values are in agreement, likely due to the use of ion exchange models. Adsorption models would not have
included the effects of the chloride ion in this system and thus would not give an accurate prediction.

Comparing these results to Figure 22, a much lower material utilisation of 0.25 has been reached, with a material concentration of only 0.5 mmol g\(^{-1}\) at the point in time when a significant concentration of selenocyanate (< 0.1 mmol l\(^{-1}\)) is present in the reactor liquid solution (and hence the reactor outlet stream). As expected, the maximum material concentration of the Smopex\(^{®}\)-103 (i.e. 1.8 mmol g\(^{-1}\)) was eventually reached, as the exchanged chloride ions are again continuously flowed out of the reactor, driving the equilibrium towards further selenocyanate exchange.

Figures 30 and 31 show the predicted performance for the same system as Figures 28 and 29, but with the inclusion of a second reactor in series with the same solution volume and mass of Smopex\(^{®}\) as the first reactor. In this system, the outlet concentration from the second reactor would exceed 0.1 mmol g\(^{-1}\) at approximately 125 hours. By this time, the material concentration of Smopex\(^{®}\)-103 within the first reactor would reach approximately 1.8 mmol g\(^{-1}\). A material utilisation factor of approximately 1.0 could thus be reached for the Smopex\(^{®}\) in the first reactor, with only a single additional reactor required. Even for commercial fixed bed systems, two fixed beds in series (i.e. lead-lag operation) are commonly required and utilised to achieve such high utilisation factors. Thus predicted performance in this CSTR system is comparable to fixed bed performance.

For the case of selenate or selenite, and competitive removal of these ions versus sulfate, the ion exchange models can similarly be employed to engineer process solutions for their removal, either in fixed beds or CSTR process configurations, depending on the feed flow rates.

5. Conclusions

In this study it has been shown that Smopex\(^{®}\) materials, i.e. functionalised polymeric chains attached to polypropylene fibre backbones, are very effective for the removal of selenocyanate, selenate and selenite ions from solution. Their particular structure has been shown to give very fast rates of ion exchange when compared to resin bead ion exchangers (Dowex\(^{TM}\) 1x2 100–200 and Amberlite\(^{TM}\) IRA 900 were also tested in fixed bed trials), comparable with resin beads in the spherical diameter range of 100 to 200 µm. Furthermore, since all of the material functionality is readily exposed to solution and not contained within restricted pore spaces (as is the case for typical ion exchange resin products), the material utilisation factors achieved in fixed bed trials regularly exceeded 0.9. In competitive selenocyanate/sulfate feeds, the Smopex\(^{®}\) materials were shown to outperform all other materials.

The sorption mechanisms by which these ions are removed from solution were also studied. It was shown that ion exchange clearly dominates the sorption process. Knowledge of this mechanism was shown to be very important in modelling and understanding how changes in initial ion concentrations and ionic species can affect the sorption performance. It was shown for example that, in contrast to typically employed models such as Langmuir, the relative selectivity of two ions such as selenocyanate and sulfate can change as a function of their initial concentrations and that sorption
performance cannot be accurately predicted without considering the chloride ion exchanged from the material. It was also shown that maximum material concentrations (for example 1.8 mmol g\(^{-1}\) for the case of Smopex\(^{®}\)-103 and selenocyanate) can be reached for feeds having selenium concentrations as low as 1 mg l\(^{-1}\) (0.013 mmol l\(^{-1}\)), whereas such performance would rarely be seen when adsorption or chelation is the sorption mechanism.

The ion exchange models were then employed to predict the performance of Smopex\(^{®}\) materials in one and two in-series continuous stirred tank reactors. The single reactor results were validated experimentally. It was shown that due to their excellent selectivity and kinetics, material utilisation factors approaching unity are achievable using only two reactors in series, i.e. full capacity of the material can be reached before outlet concentrations from the reactor system rise significantly. This level of performance is comparable to fixed bed performance, where typically two fixed beds in series (lead-lag operation) are employed in order to achieve such high material utilisation factors in commercial setups.

Smopex\(^{®}\) materials are currently undergoing redevelopment so that their unique performance benefits are available without the pressure drop limitations attributed to their current morphology. Additionally, other reactor configurations are also under investigation to bring together the benefits of the Smopex\(^{®}\) materials with the ease of use and performance efficiency of fixed bed setups.

Acknowledgements

The authors want to thank S. Colley, P. Kauppinen and J. Stevens for their support and useful comments and M. Lincoln for his assistance in some experiments. Special thanks also to D. Scott and J. Clarke for provision of crucial analytical services.

References


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