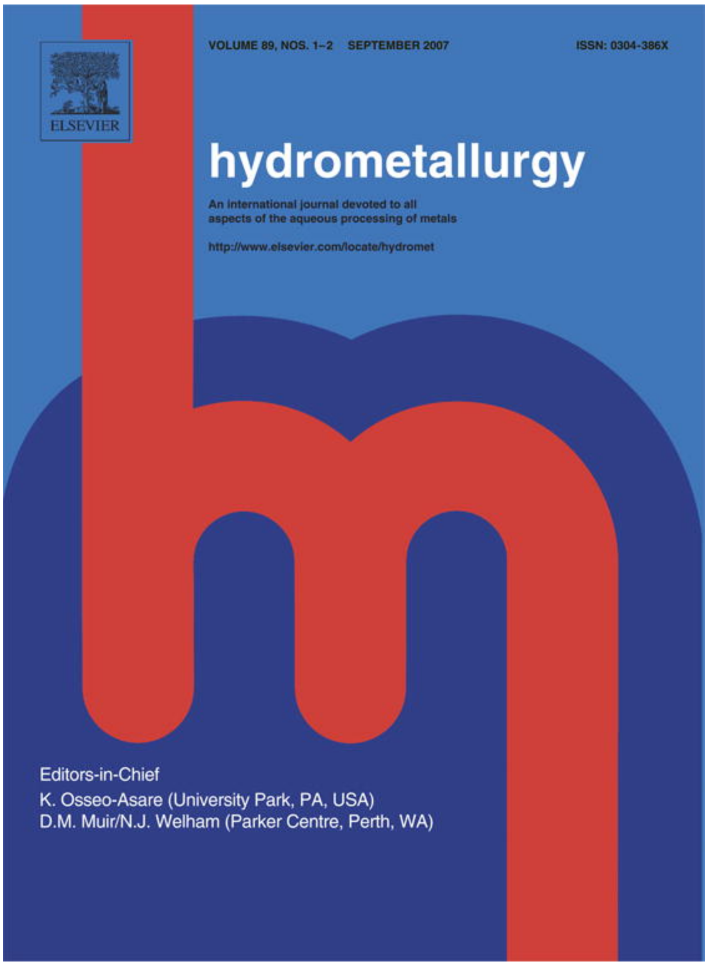
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Removal of entrained organic phase from zinc electrolyte: Pilot plant comparison of CoMatrix and carbon filtration

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Abstract

Zinc electrowinning is extremely sensitive to the presence of organic impurities in the electrolyte. In recent hydrometallurgical processes for zinc, where solvent extraction is employed for the purification of the leach liquor, it is essential to ensure that all trace organics are removed to levels of <1 ppm from the advance electrolyte.

This work compares the performance of activated carbon and CoMatrix filtration systems for the removal of organic entrainment (OE) from a zinc electrolyte. The CoMatrix pilot unit comprised a matrix packing in series with an anthracite/garnet dual media filtration system. The performance in the primary loading (lead) position of the CoMatrix combination was compared with a stand-alone carbon column. The performance of the CoMatrix system in series with a secondary (lag) carbon column was also assessed.

The organic-removal efficiency was dependent on the flowrate of electrolyte through the column for both CoMatrix and carbon in primary mode. The matrix packing alone provided very consistent organic removal to a concentration of 10 ppm, irrespective of the OE concentration of the feed solution, thereby acting as a reliable surge protector against periodic organic spikes in the system. The dual media performance was erratic, characterised by frequent breakthrough of the organic phase at all flowrates tested, and this packing contributed little to the overall organic removal.

In primary duty, a single carbon column was more reliable and produced better organic removal than the CoMatrix combination under appropriate operating conditions. The carbon column also demonstrated better ability to filter suspended solids from the electrolyte. The carbon column in secondary mode showed excellent lifetime, and the target output of <1 ppm OE was consistently produced under appropriate operating conditions.

It was concluded that the most suitable configuration for organic removal in this application was a matrix packing alone (acting as a surge protector), followed by activated carbon columns in both primary and secondary (lead and lag) positions.

Keywords: Solvent extraction; Zinc; Di(2-ethylhexyl)phosphoric acid; D2EHPA; Organic removal; Entrainment; Dual media; Activated carbon

1. Introduction

Some modern zinc hydrometallurgical processes purify the leach liquor by solvent extraction (SX), following which zinc is electrowon to produce special high

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grade (SHG) (>99.995% purity) zinc cathode (Fuls et al., 2005; Cole and Sole, 2002; Sole, 2001). The zinc electrowinning (EW) process is extremely sensitive to the presence of trace organics which may be carried through to the cell house in the advance electrolyte (AE). The specifications for organic removal from a zinc electrolyte ahead of electrowinning are far more stringent than those for copper SX-EW, as egress of trace organics into a zinc cell house can have potentially catastrophic effects. Detrimental effects may include a decrease in current efficiency, changes in orientation and morphology of cathode deposit, and the formation of so-called "sticky zinc", in which zinc adheres strongly to the cathode starter sheets and cannot be readily removed by automatic stripping machines. Furthermore, entrained organics can carry over base-metal impurities to the electrolyte which can cause problems with cathode purity or hydrogen evolution and the associated explosion and fire risk. It is therefore critical to reduce the organic entrainment of the electrolyte to the lowest possible value to avoid transfer of organic material to the EW circuit.

Several strategies, usually deployed in series, may be used to reduce the organic content of the AE leaving the SX circuit. The electrolyte typically reports to an after-settler, which allows additional settling time for phase separation. This could then be followed by flotation cells, coalescers, or both. For the final polishing stage, two options are available. Activated carbon is conventionally employed, both for its surface-active properties to remove dissolved organic material and as a coalescing agent to facilitate the removal of entrained organic material. An alternative is the so-called CoMatrix™ system, developed by Spintek Filtration, USA, for use in the copper industry.

At Skorpion Zinc, Namibia, the zinc AE originates from an SX circuit. A pilot-plant trial was carried out to compare the organic-removal efficiency from this electrolyte and assess the general performance of activated carbon and CoMatrix filtration systems in various filtration configurations. Critical operating parameters were evaluated and an optimised configuration for this particular application identified. The objective of the trial was to achieve a residual organic content from the secondary column of less than 1 ppm at a linear flowrate of the feed through the column of 12 m/h.

1.1. Definition of entrained organic phase

The SX organic phase comprised 40 vol.% D2EHPA (di(2-ethylhexyl)phosphoric acid) in Escaid 100 (a partially aromatic kerosene diluent). Organic phase can be carried over to the EW circuit by two main mechanisms. *Entrained organic* (OE) phase is physically entrained in the AE as a consequence of the mixing of the two phases in the SX circuit and subsequent incomplete settling. Since the entrainment of organic phase is a physical process, the mechanisms for its removal are also physical in nature.

Organic can also be carried over to the EW circuit by chemical means. Both the extractant and diluent have finite solubilities in the aqueous phase. These are dependent on the acidity, temperature, and ionic strength of the electrolyte, as well as on the nature of the organics themselves. This chemical carryover of organic phase is referred to as *dissolved organic* (DO). This component has a fixed value for given operational conditions, and can only be removed by chemical means. The DO concentration is only a very small fraction of the overall organic transfer.

The analytical technique employed in this study measures the total organic component of the aqueous phase, however, the DO component was demonstrated to be below the detection limit. The organic contamination in the electrolyte is therefore reported as OE values. A measurement of 1 ppm OE refers to one part per million of the organic phase as defined above (40 vol.% D2EHPA in Escaid 100). Although the different physical and chemical properties of the individual components of the organic phase imply that they will be transferred at differing rates through the circuit, for the purposes of this study, it was assumed that the organic phase is transferred without preferential loss or enhancement of one phase over the other. Quantification of the differential behaviour of the components of the organic phase is beyond the scope of this study.

1.2. Organic removal by CoMatrix filtration

CoMatrix filtration is a concept developed by SpinTek Filtration (Greene, 2002; SpinTek, 2005). The upper part of a CoMatrix column comprises the so-called matrix packing, which is manufactured of polypropylene. The pore size of this spongy structure is designed to remove all large droplets of organic phase from the incoming (downflow) electrolyte. The lower part of the column comprises a dual media arrangement. The top portion is a layer of anthracite that acts to remove entrained organic material still present in the electrolyte, while the lower portion is a layer of garnet, which acts primarily as a physical filtration medium for the removal of suspended solids from the electrolyte. The matrix packing does not require regular backwashing, and the coalesced organic removed is regularly tapped off the top of the packing material to be returned to the circuit. The dual media filtration unit is backwashed in the normal manner, and the backwash liquor treated for organic recovery.

CoMatrix packings have been used in several applications for organic removal ahead of electrowinning in the copper industry. Advantages claimed for their performance relative to carbon include (SpinTek, 2005):

- Capability to process up to five times higher flowrates;
- Up to 65% reduction in cost as well as lower installation cost.
- Up to 70% reduction in volume of backwash liquor required;
- Solids' removal to less than 10 μm;
- Organic removal to less than 2 ppm.

1.3. Organic removal by activated carbon

Activated carbon is widely used throughout the water-treatment industry for the removal of organic material from aqueous streams. Activated carbon can have surface areas up to 100 m^2/g, and so provides a very high surface area per unit volume. There are two mechanisms involved in the removal of organic. New or newly regenerated carbon has a large number of active sites in the pores and on the surface of the carbon for the adsorption of organic material. The removal mechanism relies on van der Waals' forces, and is chemical in nature. This allows the removal of both dissolved and entrained organic material from an aqueous stream. Once all of the active sites are saturated, then the carbon acts as a coalescing material only, and the removal mechanism is physical in nature.

2. Equipment and procedures

2.1. Pilot plant

The pilot trial was carried out on site at Skorpion Zinc, Namibia, over a period of 5 weeks. The pilot plant comprised four 0.3-m diameter columns, labelled as G, H, I, and J. A schematic arrangement of the flow configuration is shown in Fig. 1. This allowed for comparison of the CoMatrix system (Columns G and H) with activated carbon (Column J), both in primary (lead) mode, and for assessment of the CoMatrix in primary mode (Columns G and H) followed by carbon in secondary (lag) mode (Column I). (Although the term 'CoMatrix' is used to describe the combined filtration system, in this work a distinction is made between the individual performances of the matrix packing and the dual media system.) The columns and all piping were constructed of polyvinylchloride (PVC). Column G contained 0.5 m depth of matrix packing. Column H contained 0.6 m of garnet at the base, with a 1.1 m layer of anthracite above this. Columns I and J contained 2 m depth of Jacobi activated carbon (AquaSorb 2000). Sampling ports (S) were fitted on the outlet of each column.

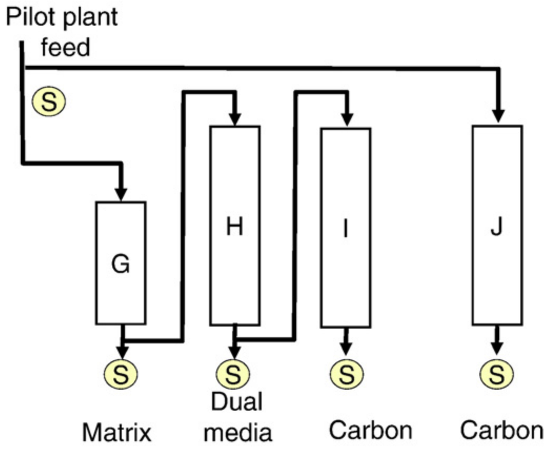

Fig. 1. Schematic of pilot-plant flow configuration.

The AE feed to the pilot plant (typically comprising 100 g/l Zn and 110 g/l H_2SO_4) was a bleed taken from the live process stream ahead of the existing organic-removal carbon columns, and fed from the main plant *via* a PVC feed line to the pilot plant using a Bredel pump. Columns H and J were routinely backwashed according to a prescribed backwash cycle. The backwash liquor used was the filtered electrolyte exiting from Column I. The electrolyte from Column J was discarded to the sump, along with the spent backwash liquor. The inlet and outlet pressures and flowrates through each column were monitored hourly. Samples were taken from the outlet of each column on a four-hourly basis and analysed for OE content.

2.2. Analytical method

Samples were collected from sampling ports into glass reagent bottles for analysis. The samples were analysed for OE content using a Horiba oil analyser, model OCA 310. Although laborious, this is the only routine analytical method capable of attaining the low detection limits required for this application (0.1 ppm OE). In this method, the electrolyte sample (approximately 1000 ml) was contacted by manual shaking for 3 min with a measured volume of a high-purity fluorochlorinated hydrocarbon solvent known as S-316. The entrained organic partitions from the aqueous phase into the freon phase, which is then separated off for analysis. The analytical method is based on the infrared detection of C–H bonds present in the entrained organic phase. The instruments were calibrated daily using standards of zero and 120 ppm organic (40% D2EHPA in Escaid 100), and checked every ten samples using a 20 ppm control standard.

Although physical entrainment is the predominant cause of organic material reporting to the electrolyte, a small component is carried across by chemical means. Organic solubility is retarded under conditions of high acid strength, so this contribution is expected to be smaller in the case of the advance electrolyte than, for example, in the SX raffinate. An attempt was made to quantify the degree of organic carryover by chemical means. A series of ten duplicate samples was taken. One of each pair of samples was analysed in the normal manner for OE content; the other was first passed through fine hardened ashless filter paper (Whatman 542) before analysis in the conventional manner. Filtration through a hydrophilic medium allows removal of physically entrained organic material by the filter paper. In all ten samples, the DO values were found to be below the detection limit of the analytical technique (<0.1 ppm). It can therefore be assumed that all measurements reported represent physically entrained organic phase.

3. Results and discussion

3.1. Reproducibility of sampling procedures and analytical methods

Because of the non-homogeneity of OE samples and the well-known difficulties of achieving reproducible results in these types of measurements, considerable efforts were expended to ensure that rigorous sampling and analytical procedures were followed. Fig. 2 shows the typical reproducibility of replicates of five feed samples that were taken at the same time. Fig. 3 shows the short-term variability of the OE concentration of a secondary column outlet, taken at half-hourly intervals over five hours with no change of operating conditions during this period. The samples were all collected in identical manner by the same operator, and were analysed by the same analyst using the same instrument.

There is considerable scatter in the results for apparently ‘identical’ samples, as well as significant variation in OE column outputs under constant flow conditions. The data shown in Figs. 2 and 3 indicate typical ranges of OE values that could be measured for a given set of operating conditions. It is accepted that considerable variability exists both in the sampling process and in the analytical method itself. Within these constraints, the data presented in this work are self consistent and within acceptable QC limits. The results presented in the following sections should be interpreted with consideration of this degree of variability.

3.2. Loading performance of CoMatrix system in series with activated carbon

The pilot plant was started using a linear flowrate of 12 m/h (corresponding to a volumetric flowrate of 200 l/h). This was the benchmark under which the performance of the CoMatrix system (matrix packing combined with dual media filtration) was compared with a 2-m single column of new carbon (primary mode). To assess the impact that the expected improved performance of the CoMatrix system would have on the output of a secondary column, the filtrate from the CoMatrix was then passed through a single column of new carbon. The target OE content of the CoMatrix system (primary column) in series with carbon (secondary column) was less than 1 ppm: the target maximum permitted OE output for the primary column alone was 5 ppm.

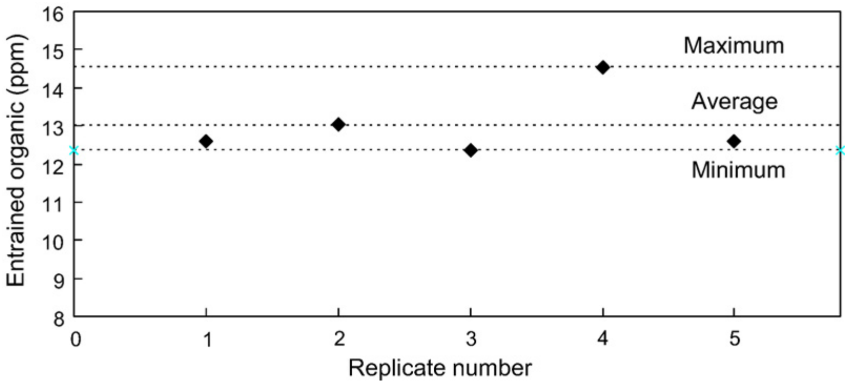

Fig. 2. Typical reproducibility of five “identical” feed samples.

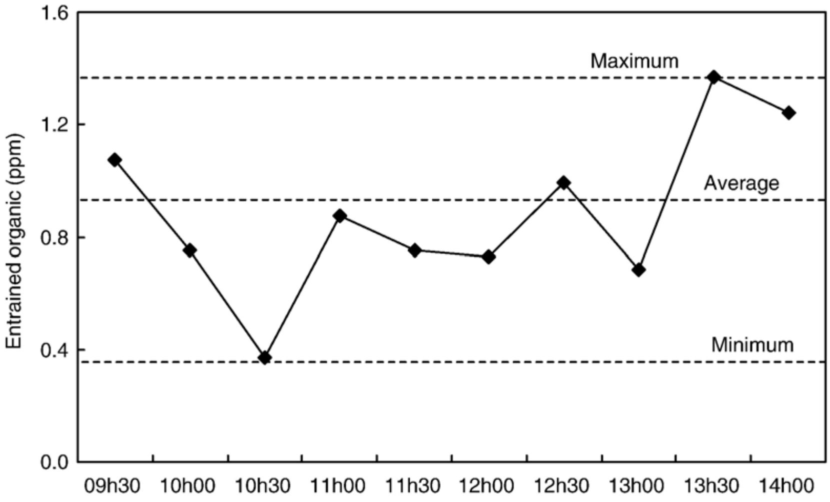

Fig. 3. Variability of the measured OE concentration of the outlet of a secondary column over a 5-hour period under constant operating conditions.

Fig. 4 compares the OE values of the pilot-plant feed with those of the streams exiting the matrix packing (Column G), the dual media filtration (Column H), and the secondary carbon bed (Column I) during the first ten days of operation at a linear flowrate of 12 m/h. The solid line shows the status of the dual media column (Column H), where level 2 indicates forward flow in secondary mode (relative to the matrix packing in primary mode), level 1 is forward flow in primary mode, and level 0 indicates a backwash (BW) cycle. The backwash frequency was varied to determine the effect of this variable on the system performance. The backwash flowrate employed throughout on the pilot campaign was 24 m/h (equivalent to 400 l/h).

As shown in Fig. 4, the feed concentration typically varied between 11 and 16 ppm OE. On passing through the matrix packing, the filtrate consistently analysed ~10 ppm OE. The subsequent performance of the dual media bed was disappointing. Although this generally did reduce the OE further, the behaviour of this column was characterised by a significant number of spikes of OE and there were evidently slugs of organic coming through the system on a regular basis. It should be noted that Column H (dual media) was the only column of the three that was backwashed. From the data of Fig. 4, it is evident that increasing the backwash frequency had little effect on the organic-removal efficiency of the dual medium filter. Despite the erratic behaviour of the

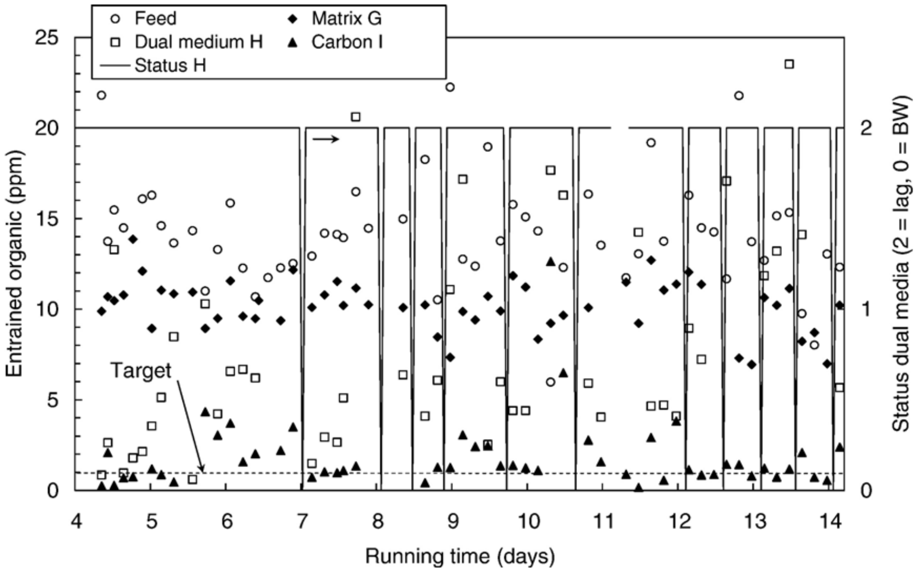

Fig. 4. Effect of the CoMatrix packing in series with activated carbon on the removal of entrained organic material from advance electrolyte at 12 m/h linear velocity.

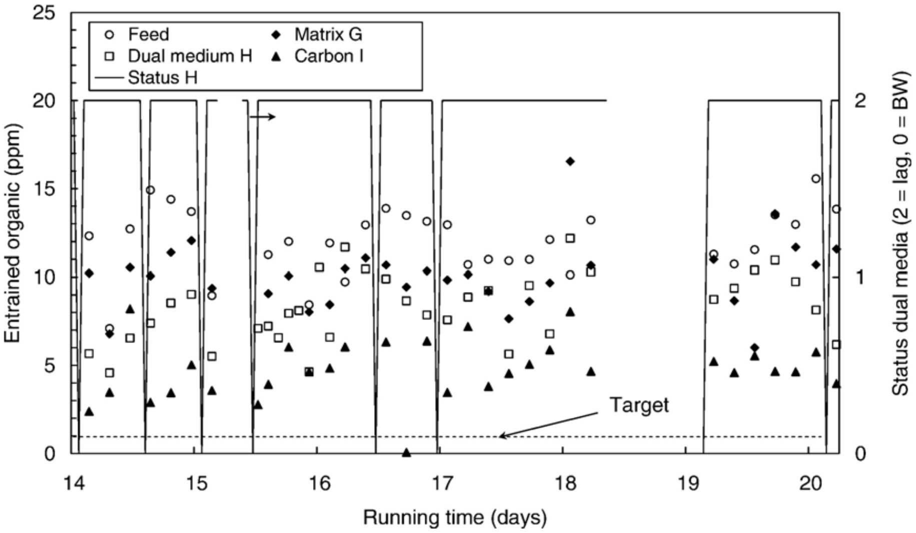

Fig. 5. Effect of the CoMatrix packing in series with activated carbon on the removal of entrained organic material from advance electrolyte at 24 m/h linear velocity.

leading dual media column, the filtrate from the secondary carbon column showed very good performance and OE values of typically 1 to 2 ppm were consistently obtained.

One of the major advantages claimed for CoMatrix systems is that they can handle flowrates of up to four times those of conventional carbon columns. It was therefore important to test the performance of the CoMatrix system under higher flowrate conditions. The performance of the column at a linear flowrate of 24 m/h is shown in Fig. 5. Under these conditions, the performance of the matrix packing remained constant (giving an output of ~10 ppm OE), but the dual media unit was again seen to offer very little contribution to organic removal. Under these conditions, the secondary carbon column was subjected to increased duty, and the performance of this unit deteriorated significantly. The combined system was not able to consistently reduce the OE in the filtrate to <1 ppm.

Fig. 6 shows the performance of the CoMatrix-carbon system measured at a linear flowrate of 6 m/h. Under these conditions, the matrix packing again showed consistent performance, even under widely varying values of the OE in the feed stream. The dual media behaviour was erratic and highly inconsistent, with a large number of slugs of organic material breaking through on a

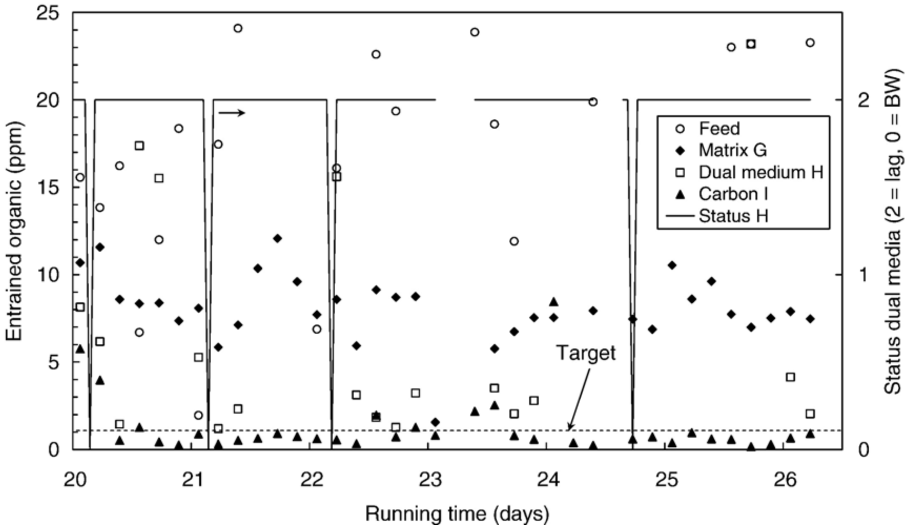

Fig. 6. Effect of the CoMatrix packing in series with activated carbon on the removal of entrained organic material from advance electrolyte at 6 m/h linear velocity.

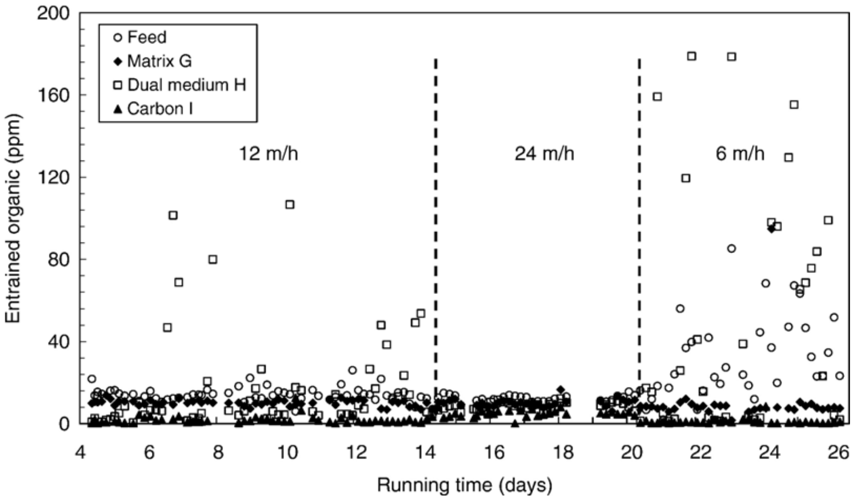

Fig. 7. Effect of operating conditions on efficiency of the CoMatrix packing followed by activated carbon on the removal of entrained organic material from advance electrolyte. (Expanded *y*-axis).

regular basis. Nevertheless, excellent performance was still achieved by the secondary carbon column, consistently giving outlet OE values of <1 ppm.

An interesting insight is gained from Fig. 7, which combines the data of Figs. 4, 5 and 6, but plotted using an expanded *y*-axis scale to allow better assessment of the outlier data. Although the dual medium filter appeared to offer the most stable performance at high solution flowrates (24 m/h), it did not show significant benefit over the matrix packing alone under these conditions. The dual media component of the CoMatrix system effectively became redundant at higher flowrates. As the feed flowrate to the column decreased, the performance of the dual media became increasingly erratic, and a large number of slugs of organic material broke through the column at regular intervals. The lower the flowrate, the worse this phenomenon became. This behaviour was not improved or influenced by the frequency of the backwash of this column. These organic breakthroughs had OE values up to two orders of magnitude greater than that permitted by EW, indicating a very undesirable situation.

As noted in Figs 4, 5 and 6, the matrix packing consistently provided an output of ~10 ppm OE, irrespective of the OE of the incoming feed stream (up to a maximum value of 85 ppm OE measured during this testwork). No organic breakthrough was observed for the matrix packing. In fact, the lowest output values from the matrix packing were recorded corresponding to the highest OE input values (see Fig. 8). Towards the end of the campaign, the average feed organic contents increased, while the matrix OE output was reduced from ~10 ppm to ~8 ppm. The matrix packing therefore performed better for higher incoming organic contents.

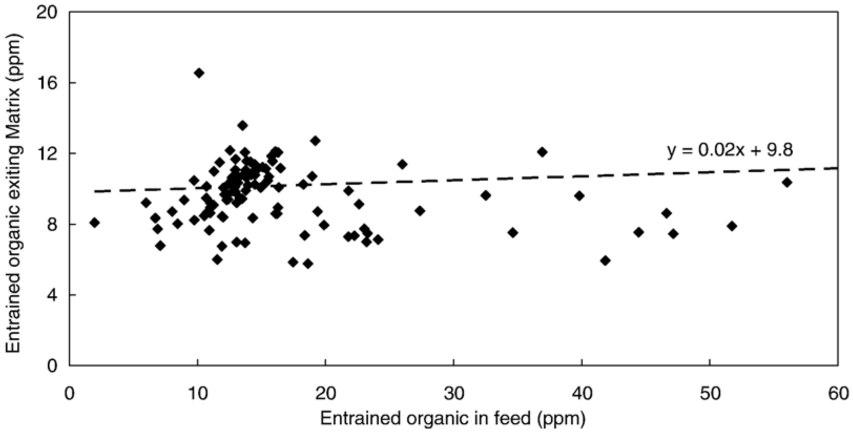

Fig. 8. Relationship between the OE content of the incoming feed solution and the output from the matrix packing. (This indicates that, within the range tested, the matrix packing consistently provided an output of ~10 ppm OE, irrespective of the feed concentration.)

Furthermore, the matrix packing acted as an excellent 'surge protector', isolating the subsequent columns from spikes in the organic levels of the feed.

The carbon column in the secondary position provided good performance throughout the campaign. The efficiency of organic removal by the carbon column was dependent on the flowrate. At the lower flowrate (6 m/h), far more consistent performance was observed for the carbon column, and the OE could be consistently maintained at <1 ppm. At 12 m/h, the OE typically ranged from 1 to 2 ppm.

It is significant to note that the secondary carbon column (Column I) was not backwashed at all during the entire pilot-plant campaign (25 days). No adverse effect on the performance was observed, confirming the effectiveness of the matrix packing in protecting the subsequent columns. By limiting the incoming OE values to a carbon column, considerable advantage could be obtained in terms of extending the lifetime of the carbon.

3.3. Loading performance of activated carbon alone in primary mode

Fig. 9 shows the organic-removal efficiency of new carbon placed in the primary position (Column J). At the start of the campaign, the flowrate was set to the design value of 12 m/h. The target output for primary loading only was 5 ppm OE.

During the first week of operation, the performance of this column was very erratic, and organic removal was poor. While very good OE values were occasionally obtained, there was high variability in the output and the levels achieved were unacceptable for EW operation. At this time, adequate performance was being measured on the CoMatrix system under similar conditions (Fig. 4). It was concluded that Column J had either been contaminated by a foreign object during commissioning, or that there was channelling or some other irregularity in the carbon packing. A decision was taken to repack the column with new carbon again. No evidence of anything that could have been considered detrimental to the column performance was found. As seen in Fig. 9, the new packing had no impact whatsoever on the performance, and OE remained unsatisfactory.

The pilot-plant backwash cycles had initially been carried out using a 2 min air scour of the bed prior to flushing the column with the backwash liquor. This is common practice in backwashing of many types of packed beds. Concerns existed, however, that this could generate fine organic aerosols or mists in the columns and that these may potentially be sources of ignition for a solvent fire. Furthermore, it was believed that the air scour may have been disrupting the bed, and thereby causing the poor performance of this column. On omitting the air scour step from the backwash procedure on Day 15, the performance did improve but the OE levels remained unacceptably high.

The flowrate to Column J was then halved on Day 18 to 6 m/h. Under these conditions, there was an immediate improvement in performance and OE values ranged from 1 to 5 ppm despite the fact that the feed OE values were now considerably higher. This identified a relationship between the efficiency of organic removal

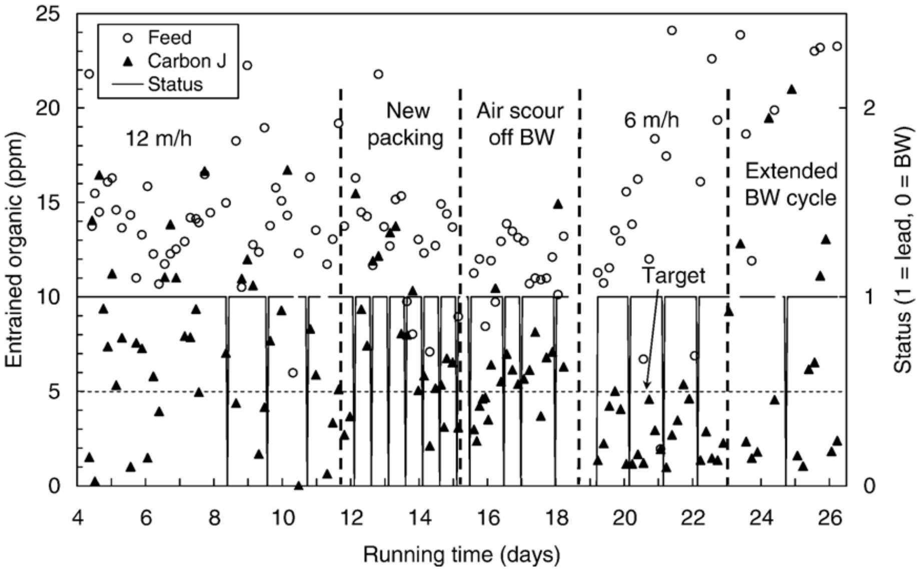

Fig. 9. Removal of entrained organic material by a single column carbon bed.

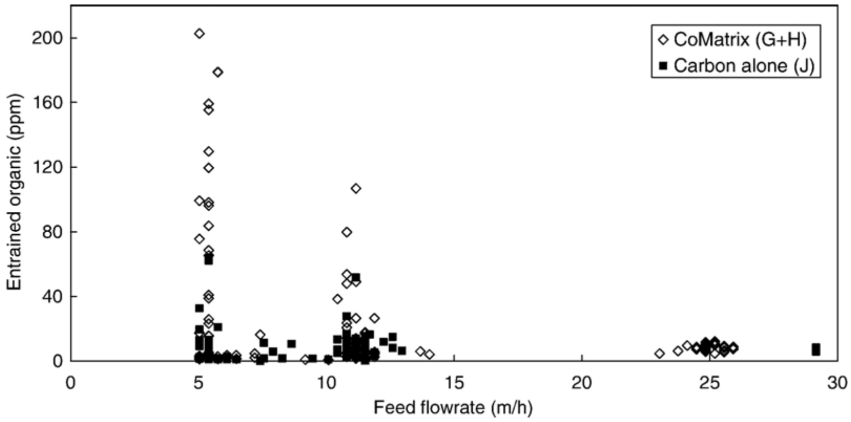

Fig. 10. Comparison of the organic-removal efficiency as a function of feed flowrate for the CoMatrix and carbon systems in primary operation.

and electrolyte flowrate through the column. When the backwash frequency was reduced (Days 22 to 25), the performance again deteriorated, confirming the importance of optimising this operating variable.

3.4. Comparison of primary loading performance of CoMatrix and carbon

Based on the data obtained during the initial periods of the pilot-plant campaign (Fig. 4), it was evident that the design flowrate selected for the testwork (12 m/h) was too high to achieve the secondary column target OE <1 ppm. Only at a linear flowrate of 6 m/h was this target consistently met (Fig. 6).

It was also clear that the claim of a CoMatrix system to be able to process higher flowrates than the corresponding carbon column was not validated for this system: at the higher flowrates (12 and 24 m/h), the dual media component of the CoMatrix was redundant and offered negligible benefit over the matrix packing alone, while at low flowrates (6 m/h) the dual media was characterised by frequent organic breakthroughs and stable flow could not be maintained. Fig. 10 compares the output OE values of Columns G and H in series (CoMatrix in primary mode) with those of Column J (new carbon in primary mode). The adverse effect of low flowrates on both packing materials is obvious. In general, however, the primary performance of the carbon packing appeared superior to that of the CoMatrix.

A secondary claim of the CoMatrix system is that it offers an effective filter for solids, as well as for organics. To validate this assertion, samples were taken twice daily from the feed solution and the filtrates from Columns H and J, and analysed for total suspended solids' (TSS) content. In all cases, the TSS output from the primary carbon column (average 245 mg/l) was lower than that from the CoMatrix system (average 414 mg/l). It was therefore concluded that the CoMatrix offers no particular advantage over carbon in terms of solids' filtration.

3.5. Backwash performance of the dual media and primary carbon columns

During the initial period of the piloting campaign when the performance observed was not as originally expected, a backwash was carried out 24-hourly for both Column H (dual media) (Fig. 4) and Column J (carbon in primary mode) (Fig. 9). This was to ensure that the column performance was not inhibited by saturation of the packing material with organic, as well as to remove residual fines from the packing material that may have been introduced during the packing process. Because of the inadequate organic-removal efficiency measured for both systems at this time, this was doubled. However, the 12-hourly backwash frequency proved too high, with little material being removed from the column. When this was again reduced to 24 h, no detrimental effect on the performance was observed. Once an appropriate loading condition was employed (6 m/h flowrate), the backwash frequency was further reduced (up to 60 h), with adequate backwashing still achieved at the design backwash flowrate (24 m/h). Under these flowrate conditions, the peak in the OE profiles occurred after about 2 to 3 min: the backwash was complete within 10 to 15 min and the residual OE content in the filtrate on completion of the backwash was negligible.

Optimisation of the backwash cycle could reduce the backwash time considerably, thereby limiting the time that the circuit is required to run with a reduced number of columns in forward mode. The overall effect should be to reduce the organic spikes that are often detected following backwash of a column, increase loading availability of the columns, and ultimately lower the average OE reporting to the AE.

4. Conclusions

A pilot-plant trial was carried out to validate claims with respect to the efficiency of removal of entrained organic from a zinc electrolyte using a CoMatrix filtration system compared to activated carbon. The test configuration comprised a matrix packing in series with an anthracite/garnet dual media packing, followed by a conventional carbon column.

The following conclusions were drawn under the range of conditions tested:

1) The matrix packing alone provided very consistent organic removal to a concentration of 10 ppm OE, irrespective of the feed OE concentration or flowrate.
2) The dual media component of the CoMatrix filter demonstrated limited utility: at 24 m/h linear flowrate of electrolyte, no organic removal was measured; at 12 m/h only ~2 ppm OE was removed, while at 6 m/h this column was characterised by frequent organic breakthroughs.
3) While the carbon column in primary mode showed better stability and organic removal than the CoMatrix system under all conditions, only at flowrates ~50% of design (6 m/h) could the primary target performance of <5 ppm OE be consistently achieved.
4) Carbon demonstrated greater ability to filter suspended solids from solution, giving a lower solids' concentration in the filtrate than did the dual media packing.
5) The lifetime of the carbon column in secondary mode was excellent: during four weeks of operation, this column was never backwashed and yet was able to readily recover from poor performance when required to process double the design flowrate. The secondary carbon column consistently produced <1 ppm OE.
6) Using the design backwash flowrate (24 m/h), both the primary carbon column and dual media filter were completely backwashed within 10 min to residual OE levels <1 ppm.
7) Use of an air scour during the backwash disrupted the packed beds and caused deterioration in subsequent performance.

Based on the observations of this trial, it was recommended that the most appropriate organic filtration system would comprise a matrix packing, followed by carbon columns in both the primary and secondary positions. The dual media column demonstrated no benefit for this system. The matrix packing showed robust ability to prevent periodic organic surges from reaching the packed beds, while the carbon proved more reliable than the dual media in primary mode under fluctuating incoming OE concentrations. In secondary mode, carbon proved capable of consistently producing output values of <1 ppm in the electrolyte. Opportunities exist for optimisation of the backwash programme such that the frequency and volume of backwash liquor can be reduced without adverse effect on the loading efficiency.

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