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PHOSPHATE-ASSISTED REMOVAL OF NICKEL AND TRACE METALS FROM GOLD-PROCESSING TAILINGS WATER USING A THERMALLY MODIFIED SUGAR-REFINERY LIME WASTE–BENTONITE COMPOSITE

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Abstract:

This study evaluates the combined use of a thermally modified sugar-refinery lime waste (defecate)–bentonite composite and monoammonium phosphate (MAP) for the removal of nickel and associated trace elements from gold-processing tailings water. Defecate was calcined at 600 °C for 30 min and blended with local bentonite at 80:20 by mass. Tailings-pond water containing 66.0 mg/L Ni, 21.0 mg/L Cu, 21.0 mg/L Co, 4.80 mg/L Sr, 2.40 mg/L U, 1.30 mg/L Mn, 1.00 mg/L Se and 0.88 mg/L As was treated with 5 g/L of composite alone and with 5 g/L of composite plus 15 mL/L of 10 % MAP solution. The composite alone removed 99.87 % of Mn, 99.98 % of U and 98.86 % of As, but only 80.30 % of Ni, 66.67 % of Co and 47.62 % of Cu. Co-dosing MAP raised nickel removal to 97.53 %, reducing the residual concentration from 13.0 to 1.63 mg/L, and gave 93–96 % removal of Cr, Al and Zn, while potassium remained unchanged at 0.21 % removal. A phosphate budget constructed from the measured concentration changes shows that 91.9 % of the dosed phosphorus is consumed by calcium and only 8.1 % by the target metals, which reconciles the 15-fold difference between the laboratory dose (1.50 g/L) and the design dose adopted in the process flowsheet (0.10 g/L): the latter corresponds almost exactly to the stoichiometric requirement of the target metals alone (0.107 g/L). The laboratory dose also introduces an estimated 189 mg/L of ammonium into the treated water, which the stoichiometric dose reduces to 13.5 mg/L. Sequencing calcium removal ahead of phosphate addition is therefore proposed as the governing design principle for this reagent pair.

Keywords: sugar-refinery lime waste, thermally modified defecate, bentonite composite, monoammonium phosphate, nickel removal, gold-processing tailings water, phosphate precipitation, reagent stoichiometry.

1. Introduction

Tailings-pond water at gold-processing plants occupies an awkward position between process water and effluent. It is too contaminated to be returned to the flotation or leaching circuits and too voluminous to be treated by methods developed for dilute streams. Its composition reflects the chemistry of the process that produced it: an alkaline matrix carrying residual cyanide and thiocyanate together with base metals, alkaline-earth metals and, at plants processing uraniferous ores, actinides. In arid regions the problem is compounded because evaporation concentrates the pond rather than diluting it, and because the water lost cannot easily be replaced.

Sugar refining offers such a substitute. Beet-sugar production generates a lime cake, known as defecate, at 8–12 wt % of the beet processed, it consists mainly of calcium carbonate with 10–15 % organic matter, and only a small fraction is returned to the land. Ławińska et al. [1] developed a disc-granulation route that converts this material into a handleable, dosable form. Its capacity as a sorbent has been demonstrated in several matrices: Marti and Zeidan [2] used beet-sugar carbonation sludge to remove synthetic dyes from aqueous media, Türk and Arslanoglu [3] pyrolysed carbonation cake with vinasse to obtain both a potassium product and a wastewater adsorbent, and Baganna et al. [4] applied sugar lime sludge to landfill leachate. The mechanistic basis for using a carbonate solid on metal-bearing water was set out by Wierzba et al. [5], who showed that calcium carbonate binds heavy metals through adsorption, ion exchange and precipitation acting together rather than through any single route. Mogashane et al. [6] applied the same principle directly to iron-rich acid mine water, recovering ferric hydroxide using calcium carbonate with a gypsum scale inhibitor.

Blending a carbonate solid with a clay adds a second class of surface. Casucci et al. [7] added zeolite and bentonite to carbonation lime from the sugar industry and improved its capacity to sequester nickel, and AlTowyan et al. [8] removed manganese from industrial wastewater with local and commercial bentonites. These results motivate the composite examined here, in which thermally modified defecate supplies alkalinity and calcium while bentonite supplies aluminosilicate surface and coagulating capacity.

Alkaline precipitation nevertheless has a known ceiling. Metals bound in stable anionic complexes do not follow the hydroxide solubility curves, and in gold-plant water the relevant complexants are cyanide and thiocyanate. Kuyucak and Akcil [9] reviewed cyanide and its removal options in gold mining and metallurgical effluents, Anning et al. [10] surveyed the determination and detoxification of cyanide in gold mine tailings, and Khodadad et al. [11] reported detoxification of a gold-plant tailings water using calcium and sodium hypochlorite. The presence of such complexes is the most plausible explanation for the incomplete removal of copper and cobalt reported in Section 3, and it is the reason a second, chemically different precipitant was investigated.

The reagent conventionally used to neutralise and precipitate such waters is lime. Balladares et al. [12] followed the neutralisation and co-precipitation of heavy metals when lime is added to the acid-plant effluent of a copper smelter and quantified the removal achieved for each metal, while Miller et al. [13] separated the contributions of adsorption and co-precipitation in limestone-based treatment of acid mine drainage and showed that zinc and nickel are removed by different mixes of the two mechanisms. Li et al. [14] have recently demonstrated that limestone need not be a passive reagent at all, recovering valuable metals from acid mine drainage in an electrochemically activated limestone system. What these studies share is a dependence on a purchased calcium reagent, which is precisely the cost item that a waste-derived substitute could displace.

Phosphate is that precipitant. Ma et al. [15] established that phosphate rock immobilises lead from aqueous solution and from contaminated soil, and Mavropoulos et al. [16] examined the mechanisms by which hydroxyapatite fixes lead. The principle extends beyond lead: Oliva et al. [17] removed cadmium, copper, nickel, cobalt and mercury from water in column experiments with a biogenic apatite, and Corami et al. [18] treated lead, copper, zinc and cadmium with phosphate rock. For uranium the phosphate route is particularly well characterised, because the uranyl phosphates that form are among the least soluble uranium solids known. Simon et al. [19] removed uranium from groundwater with hydroxyapatite, Mehta

et al. [20] showed that the extent and mechanism of U(VI) immobilization by calcium and phosphate depend on the order in which the reactants meet, Fanizza et al. [21] followed uranyl phosphate precipitation at the pore scale in a model groundwater, and Zhang et al. [22] applied a hydroxyapatite-coated sand barrier to uranium-bearing water. Phosphate has been dosed into real gold-tailings water before, but for a different purpose, amended tailings water with phosphate to stimulate in-situ thiocyanate biodegradation. Its use as a chemical precipitant for metals in this matrix does not appear to have been reported.

The remaining constituents of the water are governed by their own chemistries. Arsenic is removed either as calcium arsenate, whose formation and stability were characterised by Bothe and Brown [23], or by co-precipitation with ferric iron during lime neutralisation, studied under continuous-circuit conditions by De Klerk et al. [24] and in sulfate media by Jia and Demopoulos [25]. Selenium is more difficult, as the systematic review of Stefaniak et al. [26] makes clear. Manganese, in contrast, is comparatively straightforward at high pH, Freitas et al. [27] precipitated it oxidatively from acid mine drainage and Bao et al. [28] have reviewed the available removal and recovery routes.

The literature therefore establishes that a carbonate waste can act as an alkaline reagent–sorbent, that clay improves its performance towards nickel, that cyanide complexation limits what alkaline precipitation can achieve, and that phosphate immobilises nickel and uranium efficiently. What has not been examined is the two reagents acting together on a real multi-element tailings water, nor — more importantly for practice — how the dosed phosphate is actually distributed among the species competing for it. The present study addresses both questions, using the measured composition of the water before and after each treatment to construct a closed phosphate budget.

2. Materials and Methods

The reagent–sorbent used here is the material whose preparation, thermal behaviour and texture are reported in full in the companion paper, only the details needed to interpret the present results are repeated. Defecate was dried at 100 °C to constant mass and calcined in a SNOL muffle furnace at 600 °C for 30 min to give thermally modified defecate (TMD). The 600 °C setting is bounded on one side by the ignition of the carbon deposited on the carbonate surface and on the other by the decomposition of the carbonate itself, and was established by differential thermal analysis in that work.

Bentonite was dried, ground and dry-blended with TMD for 10 min at a TMD:bentonite mass ratio of 80:20, the ratio identified as optimal in the companion study from a series spanning 90:10 to 50:50. The composite is mesoporous, with a BET surface area of 48.91 m^2/g and a median pore width of 14.4 Å [ref. to companion paper]. This texture is worth keeping in mind when reading Section 3, because it is modest for a sorbent and the results below indicate that surface area is not what governs removal in this water.

Monoammonium phosphate solution was prepared by neutralising extraction phosphoric acid with ammonia to pH 5.5–6.0. The reagent contained 49.52 wt % P_2O_5, corresponding to approximately 80 % of the stoichiometric P_2O_5 content of pure $NH_4H_2PO_4$ (61.7 %). A 10 % solution was used throughout.

The water treated was the tailings-pond water of hydrometallurgical plant GMZ-3, an alkaline stream (pH 8.1) containing 570 mg/L Ca, 350 mg/L Mg, 710 mg/L Na, 140 mg/L K, 740 mg/L Cl, 320 mg/L HCO_3^-, 500 mg/L SCN^- and 15 mg/L NaCN, together with the trace elements listed in Section 3.

Batch experiments were carried out at room temperature on 500 mL samples. In the first series the composite alone was dosed and the suspension stirred, filtered and analysed. In the second series 5 g/L of composite was dosed together with 15 mL/L of the 10 % MAP solution, corresponding to 1.50 g/L of reagent, and stirred for 10 min before separation in a thin-layer settler. Trace elements were determined by ICP-MS, major metals by flame atomic absorption spectrometry (Agilent Technologies 55 AA), sodium by flame photometry, and anions, dry residue and pH by standard water-analysis procedures.

3. Results and Discussion

Treatment with the composite alone divides the trace elements of the tailings water into three groups (Fig. 1). Manganese, uranium and arsenic are removed almost quantitatively, strontium, nickel and

selenium are removed to between 73 and 81 %, cobalt and copper lag behind at 67 % and 48 % respectively.

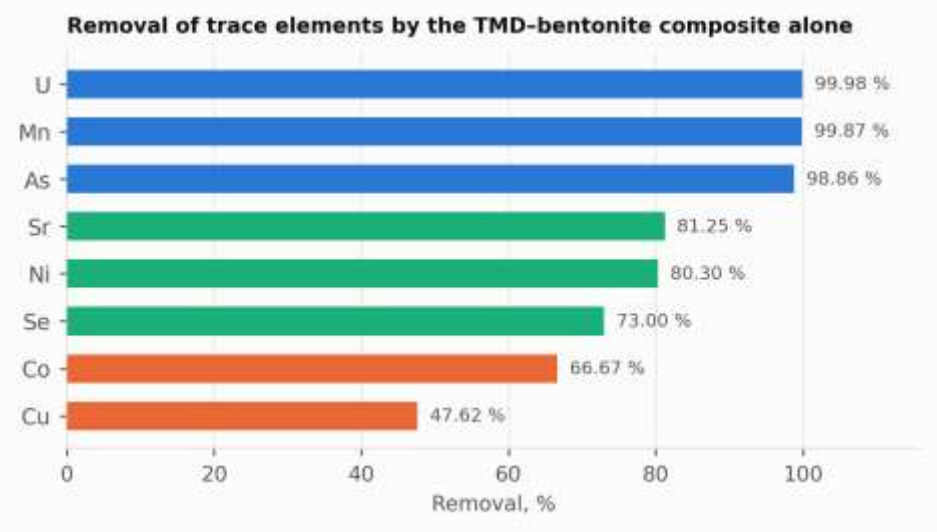

Figure 1. Removal of trace elements from the tailings water by the TMD–bentonite composite alone

The first group behaves as the literature predicts for an alkaline calcium reagent. Manganese precipitates readily once the pH is raised, in line with the routes reviewed by Bao et al. [29] and demonstrated oxidatively by Freitas et al. Arsenic is fixed either as calcium arsenate, whose stability field was mapped by Bothe and Brown, or by co-precipitation with the ferric iron already present in the water, the mechanism examined by De Klerk et al and Jia and Demopoulos. Uranium reaches 99.98 % removal, which is high even for hydroxide precipitation and suggests that carbonate and phosphate impurities in the defecate itself are already contributing, the defecate contains 0.2–0.9 % P_2O_5 on a dry basis. Selenium at 73 % is the weakest of this group, which is consistent with the difficulty documented across the technologies surveyed by Stefaniak et al.

The behaviour of copper and cobalt is the informative result. At the pH reached in these experiments, hydroxide precipitation should have removed both far more completely than 48 % and 67 %. The water, however, carries 15 mg/L NaCN and 500 mg/L SCN^-, and copper and nickel are among the metals that form the most stable cyanide complexes in gold-plant liquors. A metal held in an anionic cyanide complex does not respond to an increase in pH, which is why detoxification schemes for these waters destroy the complexant first, as in the hypochlorite treatment reported by Khodadad et al.

Adding monoammonium phosphate changes the picture for nickel in particular (Fig. 2).

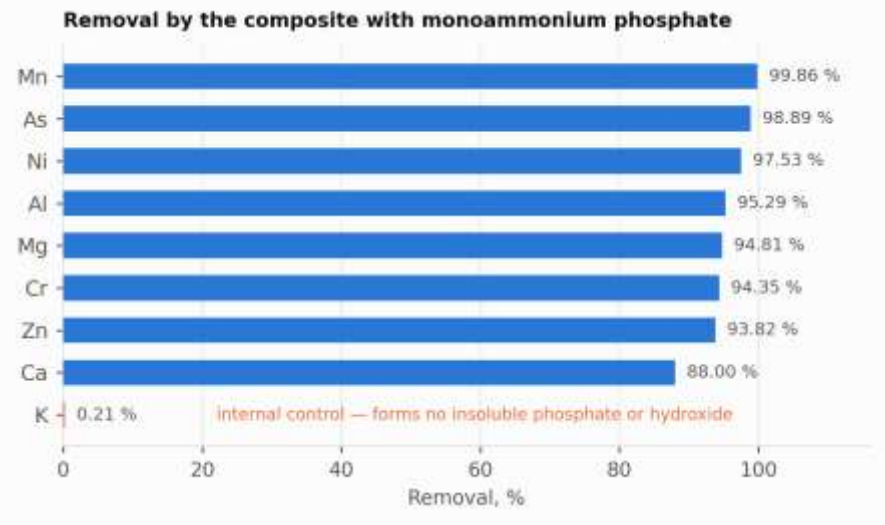

Figure 2. Removal by 5 g/L of the composite together with 15 mL/L of 10 % monoammonium phosphate. Potassium is shown in a contrasting colour as the internal control

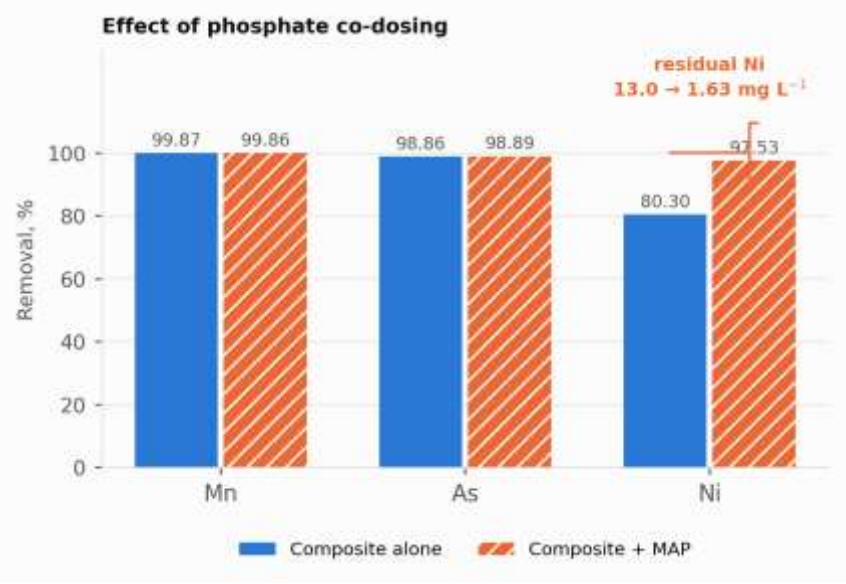

Figure 3. Effect of phosphate co-dosing on the three elements measured under both conditions

Nickel removal rises from 80.30 % to 97.53 %, and the residual concentration falls eightfold, from 13.0 to 1.63 mg/L. Chromium, aluminium and zinc are all removed to better than 93 %. The result is what the phosphate literature would lead one to expect: Oliva et al. recorded efficient nickel and cobalt uptake by apatite in column operation, and the phosphate salts of these metals are far less soluble than their hydroxides.

Potassium provides an internal control that makes the mechanism unambiguous. It is removed to the extent of 0.21 %, which is indistinguishable from analytical scatter, while every metal that forms an insoluble phosphate or hydroxide is removed to above 88 %. Removal in this system is therefore chemical precipitation, not non-specific sorption on the composite surface — an alkali metal exposed to 5 g/L of a mesoporous solid with a 48.91 m²/g surface passes through essentially untouched. This distinction matters for scale-up, because a precipitation-controlled process is dosed stoichiometrically whereas a sorption-controlled process is dosed from an isotherm.

That observation invites the obvious next question: where does the dosed phosphate actually go? A budget can be constructed directly from the measured concentration changes, assigning each removed cation to its expected phosphate phase. The dose of 15 mL/L of 10 % solution supplies 1.50 g/L of reagent, equivalent to 0.743 g/L P_2O_5 and 10.47 mmol/L of phosphorus.

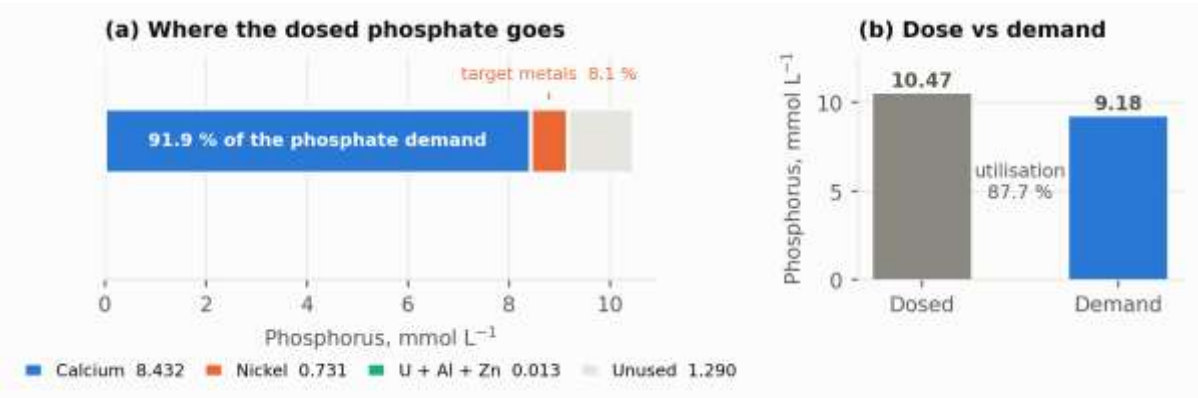

Figure 4. Phosphate budget for the combined treatment, calculated from the concentration changes of Fig. 2: (a) allocation of the dosed phosphorus among the competing sinks, (b) dose against demand

Assumed phases: $Ca_5(PO_4)_3OH$, $Ni_3(PO_4)_2$, $Ca(UO_2)_2(PO_4)_2$, $AlPO_4$, $Zn_3(PO_4)_2$. Magnesium is assigned to $Mg(OH)_2$ and consumes no phosphate, if struvite ($MgNH_4PO_4$) forms instead, the demand rises by 19.5 mmol/L, which the dose could not supply.

The budget makes the central point of this work. Calcium consumes 91.9 % of the phosphate demand and the target metals only 8.1 %. Phosphate is being spent almost entirely on an ion that the alkaline component of the composite could have removed for nothing, as carbonate. Mehta et al. showed for uranium that the order in which calcium and phosphate meet determines both the extent and the mechanism of immobilization, the same principle applies here in a purely practical form, and it points directly at a two-stage arrangement in which the composite is allowed to bring calcium down first and phosphate is added only to the partly softened water.

The stoichiometric consequence is large. If phosphate were required only for nickel, uranium, aluminium and zinc, the demand would be 0.744 mmol/L, equivalent to 52.8 mg/L P_2O_5 and 107 mg/L of the reagent — 14 times less than the 1.50 g/L used in the laboratory. This calculation also resolves an apparent inconsistency in the source material, where the flowsheet and the cost estimate adopt a MAP dose of 0.10 g/L while the laboratory experiments used 1.50 g/L. The design value is not an error: it agrees with the stoichiometric requirement of the target metals to within 6 %. What the two figures actually represent is a laboratory excess on one hand and a target-metal stoichiometry on the other, and the difference between them is the calcium term.

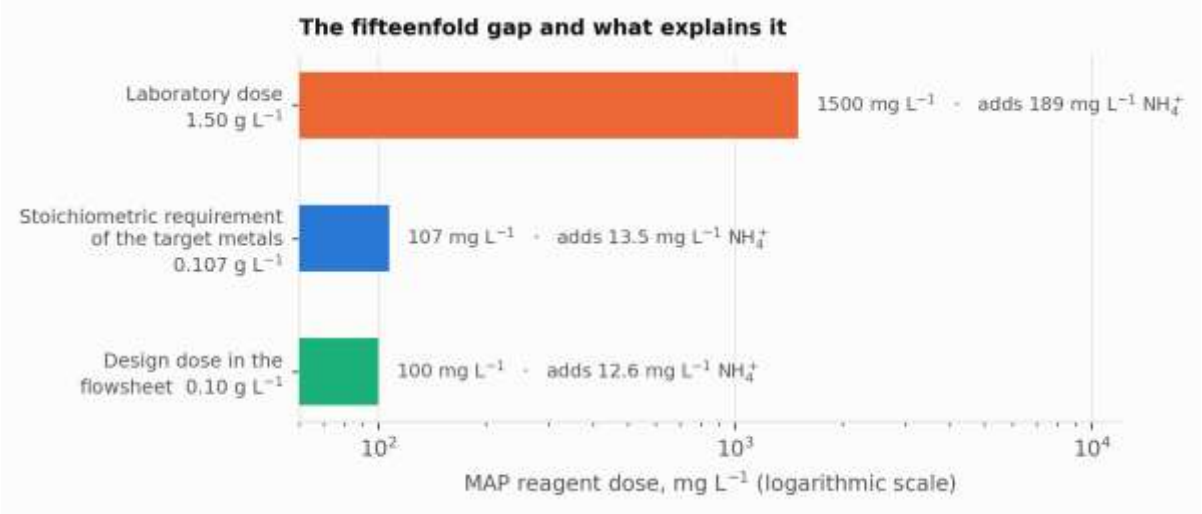

Figure 5. The laboratory dose, the stoichiometric requirement of the target metals, and the dose adopted in the flowsheet, with the ammonium each introduces

The excess is not free. At 1.50 g/L, monoammonium phosphate introduces an estimated 10.47 mmol/L of ammonium nitrogen, equivalent to 189 mg/L of NH_4^+, into water that is being treated for discharge or reuse, at 87.7 % utilisation it also leaves roughly 40 mg/L of residual phosphorus in solution. Both are regulated parameters in most jurisdictions, and both would have to be treated in turn. At the stoichiometric dose of 107 mg/L the ammonium addition falls to about 13.5 mg/L and the residual phosphate becomes negligible.

It is worth noting that ammonium is not necessarily a liability in this particular matrix. Watts et al. dosed phosphate into gold-tailings water precisely in order to stimulate the microbial community that degrades thiocyanate, and the water treated here carries 500 mg/L SCN^-. Whether the nitrogen and phosphorus left by an over-dose could be turned to that use, rather than being treated as a residual, is outside the scope of the present work but follows naturally from it.

On the reagent prices used in the source cost estimate, the difference between the two dosing regimes is direct. Treating 1000 m³ requires 0.10 t of MAP at the design dose, costing 325 000 UZS, against 1.50 t at the laboratory dose, costing 4 875 000 UZS.

4. Conclusion

A composite of thermally modified sugar-refinery lime waste and bentonite (80:20) removed 99.87 % of Mn, 99.98 % of U and 98.86 % of As from gold-processing tailings water, but only 80.30 % of Ni, 66.67 % of Co and 47.62 % of Cu. The incomplete removal of copper and cobalt is consistent with their retention in cyanide complexes in a water carrying 15 mg/L NaCN and 500 mg/L SCN^-.

Co-dosing monoammonium phosphate raised nickel removal from 80.30 % to 97.53 %, an eightfold reduction in residual concentration from 13.0 to 1.63 mg/L, and gave 93–96 % removal of Cr, Al and Zn.

Potassium was removed to only 0.21 % under the same conditions. Removal in this system is therefore governed by chemical precipitation rather than by sorption on the composite surface, and the reagent should be dosed stoichiometrically rather than from an adsorption isotherm.

A phosphate budget constructed from the measured concentration changes closes at 87.7 % utilisation and shows that calcium accounts for 91.9 % of the phosphate demand against 8.1 % for the target metals. The stoichiometric requirement of the target metals alone is 107 mg/L of reagent, which agrees to within 6 % with the 0.10 g/L adopted in the process flowsheet and explains the fifteenfold gap between that value and the 1.50 g/L used in the laboratory.

At the laboratory dose the reagent introduces an estimated 189 mg/L of ammonium and leaves about 40 mg/L of residual phosphorus in the treated water, at the stoichiometric dose these fall to 13.5 mg/L and to a negligible value. Removing calcium before phosphate is added is accordingly proposed as the governing design principle for this reagent pair, and a two-stage flowsheet embodying it is presented.

The removal efficiencies reported here are taken from the experimental record, the phosphate budget, the stoichiometric dose and the ammonium and residual-phosphorus loads are calculated from those measurements and have not been verified independently. The verification programme set out below is a condition of the conclusions in items 4 and 5.

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