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Accelerated Seeded Precipitation Pre-Treatment of Municipal Wastewater to Reduce Scaling

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Accelerated seeded precipitation pretreatment of municipal wastewater to reduce scaling

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Abstract

Membrane based treatment processes are very effective in removing salt from wastewater, but are hindered by calcium scale deposit formation. This study investigates the feasibility of removing calcium from treated sewage wastewater using accelerated seeded precipitation. The rate of calcium removal was measured during bench scale batch mode seeded precipitation experiments at pH 9.5 using various quantities of calcium carbonate as seed material. The results indicate that accelerated seeded precipitation may be a feasible option for the decrease of calcium in reverse osmosis concentrate streams during the desalination of treated sewage wastewater for irrigation purposes, promising decreased incidence of scaling and the option to control the sodium adsorption ration and nutritional properties of the desalted water. It was found that accelerated seeded precipitation of calcium from treated sewage wastewater was largely ineffective if carried out without pre-treatment of the wastewater. Evidence was presented that suggests that phosphate may be a major interfering substance for the seeded precipitation of calcium from this type of wastewater. A pH adjustment to 9.5 followed by a one-hour equilibration period was found to be an effective pre-treatment for the removal of interferences. Calcium carbonate seed addition at 10 g L^{-1} to wastewater that had been pre-treated in this way was found to result in calcium precipitation from supersaturated level at 60 mg L^{-1} to saturated level at 5 mg L^{-1}. Approximately 90% reduction of the calcium level occurred 5 min after seed addition. A further 10% reduction was achieved 30 min after seed addition.

Keywords: calcium, interference, phosphate, effluent, water reuse, irrigation

1. Introduction

The world's growing human population is increasingly drawing upon our planet's fresh water resources. It is estimated that many capital cities in Australia will not have sufficient water supplies to meet their growing populations (Horticulture Australia Limited, 2006). Recycling and reuse of water is therefore becoming an increasingly important water management option.

Treated sewage effluent (commonly known as wastewater, recycled water or reclaimed water) is a water resource that has been under-utilized in Australia. About 94% of Melbourne's sewage ($855 \ 10^3 \ m^3 \ d^{-1}$) is treated at two main treatment plants (Melbourne Water, 2007a). Less than 10% of this water resource is treated and utilized. The remainder is discharged to the environment (Horticulture Australia Limited, 2006).

Treated sewage plant wastewater contains considerable quantities of nutrients, making it an attractive option for its use in irrigation. It also, however, contains considerable quantities of salt. Approximately 45% of the salt in sewage that flows to one of Melbourne's sewage treatment plants comes from industry and about one-quarter comes from households. Salt in its various forms is widely used in manufacturing processes and is a by-product of many operations (Melbourne Water, 2007b).

The treated wastewater from Melbourne sewage treatment plants typically contains approximately 1050 mg L^{-1} total dissolved solids (TDS) . Investigations have shown that the most appropriate and sustainable uses of recycled water require a salinity level of less than 550 mg L^{-1} TDS (Melbourne Water, 2007b). Therefore salinity reduction is required before sewage treatment plant effluent can be used in unrestricted irrigation applications.

Technologies for desalination include membrane processes, thermal distillation and ion exchange. Membrane and thermal distillation processes are the most commonly applied technologies in large desalination plants. Membrane based desalination processes are increasingly becoming accepted as the method of choice for a wide range of applications. Reverse osmosis (RO) for brackish water desalination is the most utilised method in Australia (Barron, 2006).

One of the major impediments to the widespread use of membrane based processes is the fouling of membranes, leading to higher energy requirements and irreparable damage to the membrane. This fouling can be caused by colloidal material (Lee et al. 2004), microorganisms such as bacteria (Park et al., 2005), organic material such as humic acids (Tang et al., 2007) or inorganic material such as mineral salt scale (Sheikholeslami, 2003).

Mineral salt scaling can occur when ions such as Ca^{2+}, Ba^{2+}, CO_3^{2-}, SO_4^{2-}, F^- and PO_4^{3-} in the membrane concentrate stream are concentrated above the solubility limit of sparingly soluble salts (e.g., calcium carbonate, calcium sulfate, calcium fluoride, calcium phosphate, barium sulfate). This scaling leads to permeate flux decline, lower water recoveries and shortening of membrane life (Rahardianto et al., 2006). Although membrane elements can be cleaned using a variety of chemicals, this cleaning may require interruption of the desalination process and generates secondary effluent for disposal.

The composition of sewage treatment plant wastewater is subject to seasonal and yearly variation. The average levels of calcium in monthly monitoring trials of

wastewater from one of Melbourne's sewage treatment plants has been reported to be approximately 35 mg L^{-1} (Southern Rural Water, 2006). This concentration would be expected to double in the concentrate stream of a membrane filtration process operated at 50% water recovery. Such an increase in calcium levels is expected to cause scaling at the membrane surface, leading to flux decline, higher power consumption and compaction of the membrane.

There are three main options to avoid or minimize the formation of scale: lowering the pH (for calcium carbonate or calcium phosphate scale), running the plant at lower water recoveries, and removing the calcium by seeded precipitation.

Treated wastewater from sewage treatment plants is often supersaturated with respect to calcium. However, supersaturation alone is not sufficient for commencement of precipitation in a solution. There must exist in the solution a number of seeds that act as centres of nucleation. Accelerated seeded precipitation involves the addition of nucleating site for precipitation of ions from this supersaturated solution. The rate of accelerated precipitation depends largely on the degree of supersaturation and the number of nucleating sites added to the solution.

The use of accelerated seed precipitation to remove the scaling ions from the first stage RO concentrate, before feed to the next stage RO, can not only decrease the incidence of calcium scaling during desalination, but also allow greater control over the composition of the recycled water. If the water is recycled for agricultural irrigation, for example, it is possible to return some or all of the removed calcium and other ions to the (low salt) recycled water, thereby improving the sodium adsorption ratio and its nutrient value.

One of the difficulties associated with the use of accelerated seed precipitation for reduction of calcium in wastewater is the potential for interference by substances such as organics and phosphates present in the wastewater. These substances can interfere with calcium carbonate precipitation mechanisms (Sawada, 1997) by a variety of mechanisms that are not fully understood (He et al., 1999), which may involve adsorption and/or co-precipitation of the interfering substance onto the calcium carbonate lattice (House and Donaldson, 1986; Sewada, 1997). Concentrations of orthophosphate as low as a few µM have been found to significantly retard the rate of calcium carbonate crystal growth in seeded solutions (Lin and Singer 2005, 2006).

For seeded precipitation of sparingly soluble salts that have a pH dependent solubility (e.g. calcium carbonate, calcium hydroxyapatite) it is often necessary to raise the pH of the wastewater. This increases the extent of precipitation due to the lower solubility of these salts at higher pH values, and increases the speed of precipitation due to the effective increase in the supersaturation of the solution.

In the current study, the feasibility of using seeded precipitation to lower the level of calcium in sewage treatment plant effluent is investigated. Treated sewage treatment plant (TSTP) wastewater was spiked with calcium to approximately 60 mg L^{-1} in order to simulate the increase in calcium concentration associated with membrane treatment. Due to the complex nature of this wastewater, the expected increase in concentration of the many other wastewater constituents was not simulated in this study.

2. Materials and Methods

Analytical reagent grade calcium carbonate (Ajax Chemicals) was used as supplied for all experiments involving calcium carbonate seed addition

One litre of TSTP was dosed with 500 µL of 100 g L^{-1} calcium (as calcium chloride) and 150 g L^{-1} carbonate (as sodium carbonate), thereby increasing the calcium concentration from the 25 to 75 mg L^{-1} (Sample No. 1) or 31 to 81 mg L^{-1} (Sample No. 2), and increasing the carbonate levels from 84 to 159 mg L^{-1} (Sample No. 1) or from 90 to 165 mg L^{-1} (Sample No. 2).

The pH of the calcium-spiked solutions was increased to 9.5 by dropwise addition of 1 M NaOH in a stirred 1 L beaker. The precipitate formed during the pH adjustment period was not removed prior to calcium carbonate seed addition. The precipitation lowered the soluble calcium concentration in the solutions. The starting concentration of soluble calcium at pH 9.5 was found to be approximately 60 mg L^{-1} (Sample No. 1) or 65 mg L^{-1} (Sample No. 2).

Ten mL samples of the solution were taken before and at various times after addition of the calcium carbonate seed material and immediately filtered through 0.2 µm membrane filter unit (Schleicher and Schuell). Experiments involving the use of wastewater that had been previously treated by calcium carbonate seed addition or pH equilibration alone were filtered through a glass fiber filter (Advatec) prior to use.

For experiments involving phosphate addition, 250 mL of the pre-treated wastewater was spiked with 460 µL of 5 g L^{-1} phosphorus solution (as NaH_2PO_4) with constant stirring.

Calcium analysis was performed with a Varian Atomic Absorption Spectrophotometer using a nitrous oxide- acetylene flame. Phosphorus analyses were conducted using the Vanadomolybdophosphoric Acid Colorimetric Method (Method 4500-P C, APHA, 1998). Solutions used in the investigation of seeded precipitation without background interference were prepared by spiking deionised water to 55 mg L^{-1} calcium (as calcium chloride) and 82.5 mg L^{-1} carbonate (as sodium carbonate). The particle size of the calcium carbonate seed material was determined using a Coulter 130LS particle size analyser.

3. Results and Discussion

3.1 Characterization of TSTP wastewater samples

The chemical compositions of the two wastewater samples used in this study are shown in Table 1. The water samples have a mineral content that is typical of municipal effluent sourced from low TDS waters, and would be classed as only slightly brackish TDS < 1000 mg L^{-1}. However, the water's alkalinity provides buffering capacity and creates the potential for alkaline scale formation as the recovery increases in the RO.

Table 1: Chemical composition of TSTP wastewater samples used in this study, all concentrations are in parts per million (mg L^{-1}).

Parameter	Sample No. 1*	Sample No. 2*
Alkalinity, as $CaCO_3$	140	150
Fluoride	2.2	2.1
Chloride	350	370
Sulphur, as sulphate	81	88
Calcium	25	31
Magnesium	21	23
Sodium	240	250
Potassium	23	25
Iron	< 0.02	< 0.02
Manganese	0.004	0.001
Aluminium	0.01	0.01
Barium	< 0.002	0.003
Chromium	0.004	0.004
Copper	0.017	0.014
Nickel	0.013	0.013
Lead	0.001	0.002
Zinc	0.004	0.017
Mercury, as Hg	< 0.0001	< 0.0001
Nitrate, as N	8.3	9.7
Nitrite, as N	0.007	0.005
Ammonia, as N	< 0.1	< 0.1
Phosphate, as P	6.1	9.0
Silica, total as SiO_2	1.1	8.7

* Samples were taken directly from the discharge point of a sewage treatment plant on two different days approximately one week apart

3.2 Comparison of seeded precipitation in wastewater and calcium solutions

The potential for interference by substances such as organics and phosphates present in the wastewater has already been stated. The early parts of this research involved an assessment of accelerated seed precipitation treatment of the TSTP wastewater without prior treatment to remove interfering substances.

The calcium levels at various times after addition of various doses of calcium carbonate (Mean particle size = 32 ± 14 μm, < 1 μm = 0%, < 10 μm = 3%, < 100 μm = 100%) to wastewater that had been spiked to approximately 60 mg L^{-1} soluble calcium (75 mg L^{-1} before pH adjustment to 9.5) are shown in Fig. 1(a). The calcium level was increased by spiking to simulate the elevated calcium levels in the concentrate or reject stream of the RO membrane desalination process. Filtration of the raw wastewater prior to its use was not found to influence the calcium precipitation behaviour (results not shown), indicating that the suspended solids do not influence the calcium precipitation behaviour.

Direct application of accelerated seed precipitation of calcium from TSTP wastewater was found to be ineffective. A calcium reduction of only approximately 20% was achieved at the relatively high dose of 10 g L^{-1} calcium carbonate seed. Furthermore, the final calcium concentration achieved decreases with increasing calcium carbonate seed dose – a feature that is not consistent with an uninhibited seeded precipitation mechanism. In the absence of interferences the rate of calcium

removal but not the extent of calcium removal is expected to increase by increasing the number nucleation sites for precipitation.

The calcium reduction in TSTP wastewater (Fig. 1(a)) can be compared to that achieved for calcium chloride solutions (i.e., in the absence of interfering substances) in Fig. 1(b). In contrast to the results obtained for the TSTP wastewater (Fig. 1(a)), accelerated seed precipitation of calcium from deionised water that had been spiked with calcium chloride solution (i.e., without background interference) was found to be very effective (Fig. 1(b)). The calcium carbonate doses tested (1, 5 or 10 g L^{-1}) were found to decrease the calcium levels to approximately 5 mg L^{-1} within the first 30 min. At 10 g L^{-1} calcium carbonate seed dose this level was achieved within the first 5 min.
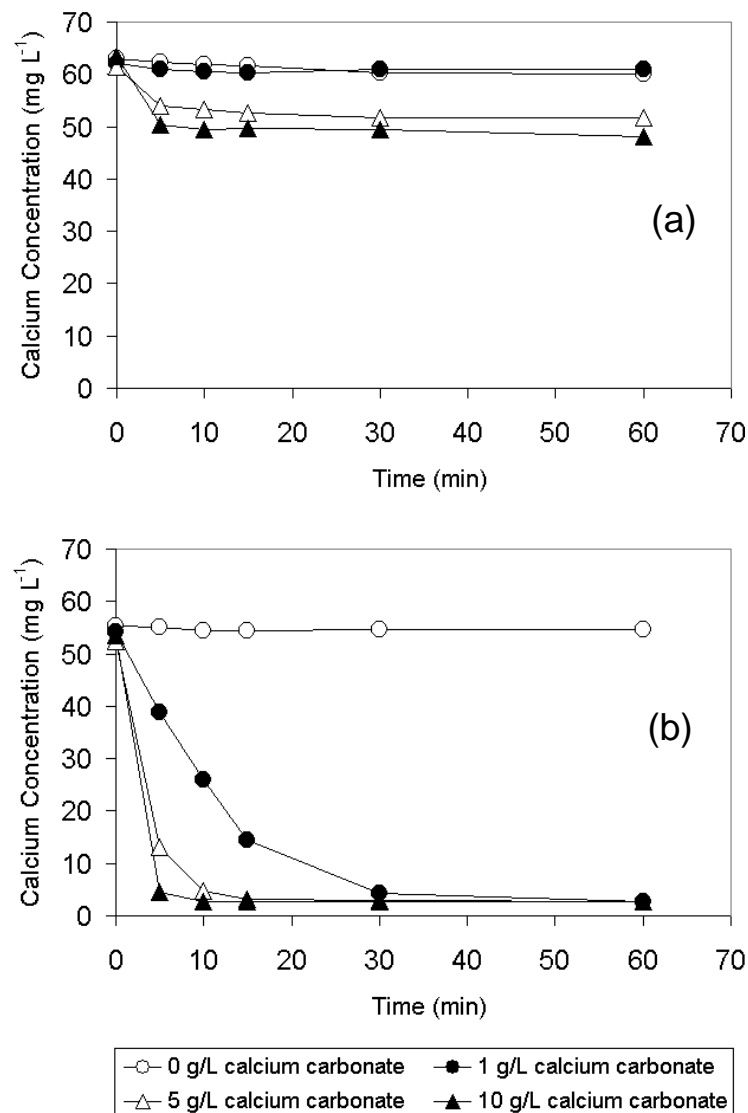

Figure 1: Effect of calcium carbonate seed dose on calcium removal from: (a) TSTP wastewater Sample No. 1 at pH 9.5, calcium concentration before pH adjustment = 75 mg L^{-1} and (b) calcium solution at pH 9.5.

3.3 Removal of interferences

The mechanism of inhibition of crystal growth by substances such as phosphate is not fully understood (He et al., 1999), but may involve adsorption and/or co-precipitation of the interfering substance as part of the calcite lattice (House, 1987; Sewada, 1997). Blockage of active crystal-growth sites by adsorbed phosphate has been widely accepted to be responsible for the reduced rate of calcite precipitation (Lin and Singer, 2006). This mechanism of interference involves the removal of the interfering substance from solution. Seed material addition to the water may, therefore, serve as a pre-treatment to remove the interfering substance. To test this, the treated TSTP wastewater used to generate the data in Fig.1 was dosed a second time with various quantities of seed material and the calcium concentration was measured. The results are shown in Table 2.

Table 2: Concentration of calcium (mg L^{-1}) 60 min after repeated dosing with calcium carbonate at pH 9.5, TSTP Sample No. 1. Contact time for first dose was 60 min, the starting calcium concentrations are those at t = 60 min in Fig. 1(a).

First CaCO$_3$ seed dose (g L^{-1})	Second CaCO$_3$ seed dose (g L^{-1})				
	0	1	5	10	15
0	-	-	47.2	6.3	-
1	-	50.3	41.6	6.5	4.2
5	53.3	51.2	39.1	5.6	5.7
10	51.8	49.8	40.2	5.4	6.5

The data show that, regardless of the size of the first dose, a second dose of 10 g L^{-1} is required for good calcium removal. The data also show the possibility that the pre-treatment does not necessarily need the addition of seed material. Simply adjusting the pH to 9.5, waiting for 1 h and then filtering is sufficient as pre-treatment for good removal to take place using 10 g L^{-1} calcium carbonate. This indicates that interfering substances present in the wastewater are removed from solution by precipitation during the 1-h pre-treatment period. The precipitation behaviour during the process will be further discussed in section 3.5.

3.4 Pre-treatment time requirements

The kinetics of calcium removal for wastewater that had been pre-treated by equilibrating for various time periods at pH 9.5 in the absence of seed material and subsequently dosed with calcium carbonate seed at 10 g L^{-1} is shown in Fig. 2.

It can be seen that seeded precipitation after equilibration at pH 9.5 for periods of 45 min or less is not effective. The calcium reduction can be seen to be close to that observed without pre-treatment (see Fig. 1(a)). Seeded precipitation after 60 min equilibration at pH 9.5, however, was found to take place rapidly. The calcium concentration decreased to approximately 11 mg L^{-1} within 5 min, and to 6.4 mg L^{-1} within 30 min. This is in marked contrast to seeded precipitation using 10 g L^{-1} calcium carbonate without the 1 h equilibration pre-treatment period, where the minimum calcium concentration achieved was 47 mg L^{-1} (see Fig. 1(a)).
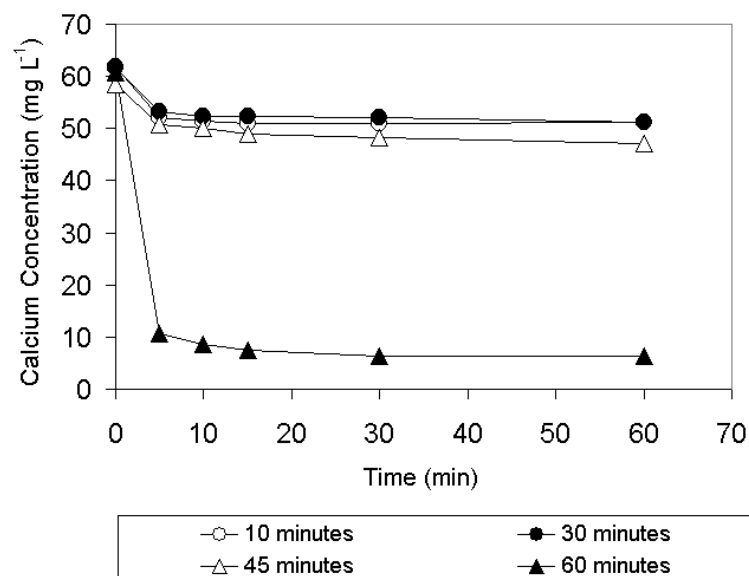
Figure 2: Seeded precipitation using 10 g L^{-1} calcium carbonate seed, TSTP Sample No. 1, pH equilibration in the absence of added seed material for various time periods prior to seed addition, contact time with added seed material = 60 min, filtration prior to seed addition.

3.5 Influence of phosphate

The identity of the interfering substance or substances in the wastewater under investigation is not known. Inspection of the wastewater composition (Table 1) reveals that phosphate levels are at levels that have been demonstrated to inhibit calcite formation in literature studies (Plant and House, 2002). It seems reasonable to suggest that the observed interferences in seeded precipitation seen for the TSTP wastewater may at least be partly due to phosphate. To investigate this, the levels of soluble phosphorus in the TSTP wastewater during the pH equilibration period were measured. The results are shown in Fig. 3.
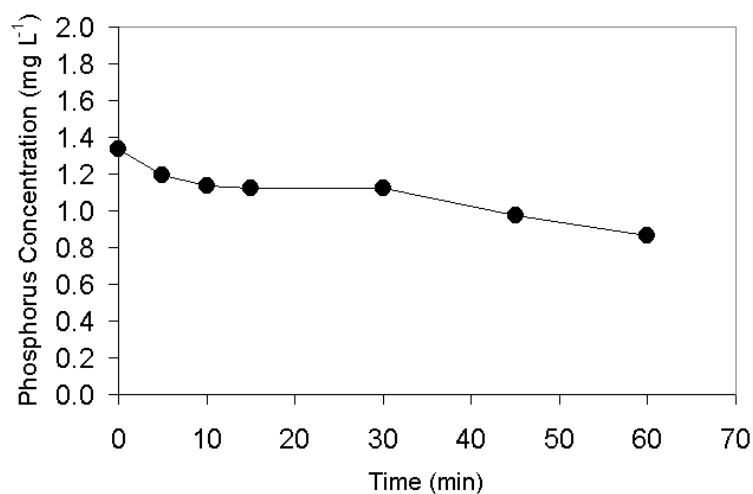

Figure3: Phosphorus levels in filtered TSTP Sample No. 1 wastewater during pre-treatment period, 0 g L^{-1} calcium carbonate. (phosphorus concentration before pH adjustment was 6.1 mg L^{-1}).

Adjusting the pH from the native pH of 7.8 to the seeded precipitation pH of 9.5 prior to the pre-treatment stage was found to decrease the soluble phosphorus concentration from the native concentration of 6.1 mg L^{-1} (see Table 1) to 1.3 mg L^{-1} (40 µM), indicating the formation of phosphate containing precipitate during pH adjustment to 9.5. Adjustment of the pH from the native pH to 9.5 prior to the pre-treatment stage was also found to decrease the soluble calcium concentration from approximately 75 to approximately 60 mg L^{-1}. This decrease in calcium concentration (0.375 mM) is approximately double the P concentration decrease (0.155mM), suggesting that approximately half of the available Ca ions go towards the formation of a calcium phosphate salt and half go toward the formation of $CaCO_3(s)$ during pH increase from 7.8 to 9.5 prior to the pre-treatment and seeded precipitation stages of the experiment.

The identity of the calcium phosphate salt formed during pH adjustment from 7.8 to 9.5 is not known. Literature calcium phosphate speciation data (Moon et al., 2007) would suggest that, at the wastewater concentrations, this solid does not contain $CaHPO_4$ but may contain other calcium phosphate species such as octacalcium phosphate ($Ca_4H(PO_4)_3$) and hydroxyapatite ($Ca_5(PO_4)_3OH$). Indeed, Moon et al. (2007) found that although a solution may be undersaturated with respect to $CaHPO_4$, the solid phosphate phases can tend to transform from $CaHPO_4$ to $Ca_4H(PO_4)_3$ to $Ca_5(PO_4)_3OH$ with time.

The phosphorus concentration during the subsequent equilibration period at pH 9.5 (in the absence of added seed material) was found to further decrease from 1.3 to 0.9 mg L^{-1} (Fig. 3). This slow decrease may attributed to further precipitation of calcium phosphate species and/or adsorption of $CaHPO_{4(aq)}$, which exists in equilibrium with its solid forms, onto the freshly precipitated $CaCO_3(s)$ at pH 9.5. Calcium hydrogen phosphate ($CaHPO_{4\ (aq)}$) is believed to be the species that adsorbs on the calcite surface and inhibits calcite precipitation (Lin and Singer, 2006).

It is important to emphasise here that all solids were filtered out of the wastewater after pre-treatment (i.e., after equilibration at pH 9.5 for 1 h). Without this step in the procedure, the precipitated calcium phosphate would be expected to act as a large source of interfering calcium phosphate species, leading to inhibition of subsequent seeded precipitation of calcium carbonate.

Comparison of Figs. 2 and 3 suggests that a threshold phosphorus concentration exists for the two-stage process used here (i.e., pre-treatment by equilibration at pH 9.5, followed by seeded precipitation using 10 g L^{-1} calcium carbonate seed). Below this threshold, the phosphorus' influence as an interfering substance for seeded precipitation using 10 g L^{-1} calcium carbonate seed is negligible. This threshold is reached between 45 and 60 min into the pH 9.5 pre-treatment equilibration period. The data in Fig. 2 and Fig. 3 suggest that, at phosphorus concentrations below this threshold, the adsorbing phosphate species responsible for the interference is insufficient to block all the available nucleating sites on the calcium carbonate seed (at 10 g L^{-1}), leaving sufficient active crystal growth sites for effective seeded precipitation. This explanation is supported by the data in Table 2 which show that a 1 h equilibration period at pH 9.5 (i.e., at 0 g L^{-1} first calcium carbonate dose) is insufficient to achieve the lower threshold concentration required for seeded precipitation with 5 g L^{-1} calcium carbonate seed dose. The lower seed dose of 5 g L^{-1} would be expected to require a longer pre-treatment time than 60 min to achieve a phosphorus concentration that is low enough to not block all the available nucleating sites on the calcium carbonate seed.

To further investigate the influence of phosphate levels on calcium removal by seeded precipitation, a sample of TSTP wastewater was equilibrated for 1 h at pH 9.5 as pre-treatment and subsequently filtered and split into two equal sub-samples for comparison. One sub-sample was left unaltered, the other was dosed with phosphate to 9 mg L^{-1} P in order to bring the phosphate level back to the original TSTP wastewater level (i.e., before pH adjustment to 9.5). Both sub-samples were adjusted to pH 9.5 and then dosed with 10 g L^{-1} calcium carbonate. The levels of calcium at various times after calcium carbonate addition are shown in Fig. 4.
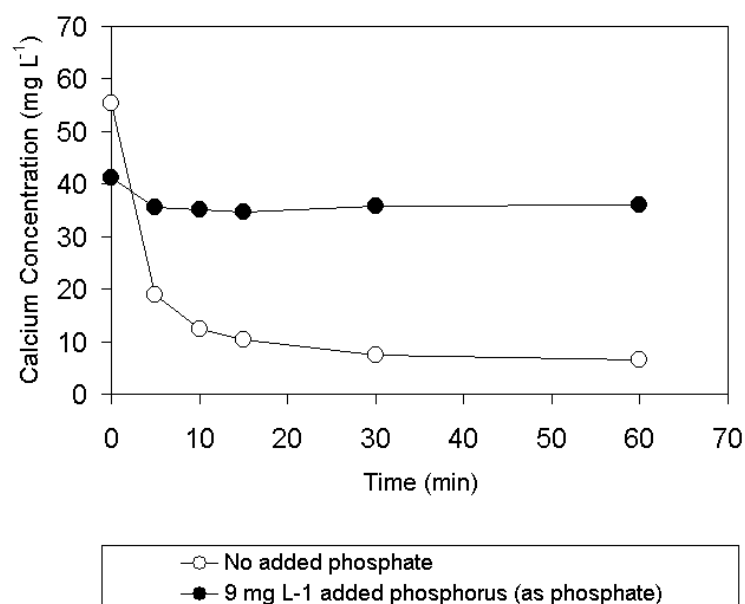

Figure4: Effect of phosphate addition, TSTP wastewater Sample No. 2, 1 h pre-equilibration at pH 9.5 in the presence of 5 g L^{-1} calcium carbonate, phosphorus concentration before pre-equilibration period: 9 mg L^{-1}, 10 g L^{-1} second dose of calcium carbonate.

The initial calcium concentration was found to be lower in the phosphate-dosed sub-samples than the control (no added phosphate) sub-sample. This can be attributed to the formation of calcium phosphate precipitate as previously discussed. The formation of sparingly soluble calcium phosphate salts has been cited as one of the major problems associated with the RO treatment of treated sewage effluents (Greenberg and Semiat, 2005; Ning and Troyer, 2007), and the cause of membrane fouling at high phosphate antiscalant concentrations during RO treatment (Al-Shammiri et al., 2000).

It can be seen that in the absence of added phosphate, the calcium concentration rapidly decreased, as was the case in previous experiments with other wastewater samples, whereas in the presence of added phosphate, the calcium concentration remained largely unaltered. This is further evidence that suggests that the poor calcium removal seen in Fig. 1(a) and in the short equilibration time data in Fig. 2 is at least partly due to the presence of phosphate.

4. Conclusions

The results presented in this study indicate that seeded precipitation is a feasible option for the decrease of calcium in treated sewage treatment plant wastewater. Adjustment of the wastewater pH to 9.5 followed by a 1-h pre-treatment/ equilibration period prior to calcium carbonate seed addition at 10 g L^{-1} was found to result in calcium precipitation to its solubility limit. Calcium carbonate seed addition at 10 g L^{-1} to wastewater that had been pre-treated in this way was found to result in calcium precipitation from supersaturated level at 60 mg L^{-1} to saturated level at 5 mg L^{-1}. Approximately 90% reduction of the calcium level occurred 5 min after seed addition. A further 10% reduction was achieved 30 min after seed addition.

Evidence was presented that suggests that phosphate may be a major interfering substance for the seeded precipitation of calcium from this type of wastewater. It was found that a 1-h pre-treatment period was effective in the removal of interferences to the seeded precipitation of calcium from treated sewage treatment plant wastewater using 10 g L^{-1} calcium carbonate seed.

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