



## Effect of initial LSI on wastewater softening: simultaneous removal of polyacrylamide residues

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### ABSTRACT

Industrial effluent was tested for softening and polyacrylamide (PAC) residue removal and correlated with the Langelier saturation index (LSI). Contrary to previously reported methods, the initial LSI ( $LSI_{in}$ ) was calculated using the pre-reaction pH, the inherent alkalinities of the original wastewater and the alkalinities added during softening, under the assumption that no physical or chemical reaction had started. During the softening optimization tests, higher  $LSI_{in}$  predicted efficient softening, which was further confirmed by lower final LSI ( $LSI_{fin}$ ). The NaOH dose adjustment was more effective with 100% softening than that achieved by adjusting initial pH. Calcite seeding was more effective at pH 7 with lower  $LSI_{in}$  in the range  $-2$  to  $2$  than that at pH 10 with  $LSI_{in}$  in the range  $1-4.7$ . During NaOH dose optimization,  $LSI_{in}$  was higher and  $LSI_{fin}$  was lower at a higher soda ash to hardness molar ratio of  $1.3:1$  than that at a lower ratio of  $1:1$ . Compact accelerated precipitation softening process removed 60% of the calcium and 41% of the PAC residues because calcium carbonate precipitates worked as a coagulation aid. Direct correlations between  $LSI_{in}$  and softening efficiency in all processes ( $R^2 = 0.95-0.98$ ) confirmed the importance of LSI and explained the effects of calcite addition.

*Keywords:* Industrial wastewater pretreatment; Softening; Polyacrylamide residues; Langelier saturation index

### 1. Introduction

Increasing urbanization, abrupt climatic changes, and improper water resource management have increased the demand for recycled wastewater. Reverse osmosis (RO) is a frequently used treatment process [1]. Membrane fouling is caused by organic and inorganic pollutants present in wastewater. RO maintenance cost is significantly reduced by water pretreatment before

RO [2–4]. Simultaneous removal of organic and inorganic contaminants by a single process is difficult.

Hardness due to the presence of calcium and magnesium causes scale deposits of calcium carbonate ( $CaCO_3$ ) and magnesium compounds ( $Mg(OH)_2$ ,  $MgCO_3$ ,  $Mg_4(CO_3)_3(OH)_2 \cdot 3H_2O$ ) [5] that are responsible for the membrane fouling. Various physicochemical and membrane processes have been tested to minimize scaling [5–7]. Chemical precipitation is a commonly used method for making calcium and magnesium carbonates that settle easily. Chemical

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precipitation is affected by the factors that control the speciation of alkalinities, and formation and precipitation of respective carbonates.

The Langelier saturation index (LSI) is an indicator of the stability of water and its degree of  $\text{CaCO}_3$  saturation [8,9]. Water with LSI near to zero is saturated with calcium carbonate and is considered of good quality. Water with negative or positive LSI values is under or over saturated with respect to calcium carbonate and is termed potentially corrosive or scaling, respectively. LSI is the difference between the measured pH of the solution and the pH at which water is saturated with  $\text{CaCO}_3$  (pH of saturation (pHs)).

In published studies, the saturation index (SI) of water before softening and after process completion was calculated on the basis of determined alkalinities and the absence of any correlation between softening and SI hindered process optimization [8,10–12]. Contrary to previously reported methods, we calculated initial LSI ( $\text{LSI}_{\text{in}}$ ), at reaction commencement, using the pre-reaction pH, under the assumption that no physical or chemical reaction had taken place. LSI was calculated using the pre-reaction pH, the inherent alkalinities of the original wastewater determined by analysis and the alkalinities added for softening.

Compact accelerated precipitation softening (CAPS) is a process tested for enhancing the softening efficiency [8,13]. In CAPS, calcite in water mixture or in cake on membrane induces the nucleation of calcium carbonate, which in turn causes crystallization of calcium carbonate. Oren et al., simplified the process using a submerged filtration unit [12]. Addition of aluminum salts removed other contaminants like silica [10].

The polyacrylamide (PAC) family of polymers is used in water treatment by coagulation/flocculation [14], and the efficiency depends upon the stirring speed and particulates present in the water. With low particulate concentration, the flocculation of PAC becomes less efficient. Industrial applications of aqueous solutions of PAC often involve stirring, pumping, injecting, and movement through porous media. Each of these processes has the potential to cause mechanical degradation of the polymer used [14–16]. PAC polymers of smaller molecular weights are difficult to flocculate and sometimes need post-treatment to remove them from the water.

This study examines the correlation between  $\text{LSI}_{\text{in}}$  and final LSI ( $\text{LSI}_{\text{fin}}$ ) and the softening efficiency of hard wastewater, during process optimization, by varying the pH, soda ash dose, caustic soda dose, and calcite dose. The simultaneous removal of PAC and hardness from water by a single process, i.e., CAPS, is also tested.

## 2. Experimental

### 2.1. Materials and methods

Wastewater samples taken from Hong Dok Industry, Daegu, South Korea, were preserved at 4°C. The pH, total organic carbon (TOC), chemical oxygen demand (COD), and calcium values of the wastewater were 7, 3, 44, and 445 mg/L, respectively. All the chemicals used were of analytical grade and used without any purification.

### 2.2. Optimization with pH and caustic soda dose variation

The wastewater samples (100 mL) were taken in beakers and the initial pH was varied, before adding a fixed dose of soda ash, by adding solutions of HCl (1 M) and NaOH (1 M). The samples were mixed well with a magnetic stirrer at 60 rpm for 5 min and left at room temperature for 30 min before samples of supernatant were taken. The initial pH varying test was repeated with calcite seeding to further increase the softening efficiency. In the later tests, wastewater (100 mL) was taken in 250-mL beakers and pre-determined amounts of caustic soda were added. This test was performed twice with two fixed soda ash doses of 1 and 1.3 mol for each mole of hardness, i.e., 1,135 and 1,419 mg/L. The mixture was stirred for 5 min at 60 rpm and then left to settle for 30 min before supernatant samples were taken. The pH was checked before and after reaching equilibrium.

### 2.3. Calculations of LSI

$\text{LSI}_{\text{in}}$  was determined on the basis of the pre-reaction water pH, the inherent alkalinities in wastewater determined by analysis and the alkalinities added for softening. LSI was correlated with the softening efficiencies of the above processes, which helped to understand and optimize the softening process. At 25°C and total dissolved solids less than 500 mg/L, LSI can be calculated from Eqs. (1) and (2) [17]:

$$\text{LSI} = \text{pH} - \text{pHs} \quad (1)$$

$$\text{pHs} = 11.5 - \log[\text{Calcium}] - \log[\text{alkalinity}] \quad (2)$$

where 11.5 was taken from the Caldwell–Lawrence graph [17].

### 2.4. Simultaneous removal of hardness and PAC

The removal of only PAC was tested by the addition of increasing amounts of illite to 200 mL wastewater. Removal efficiencies were tested vs.

increasing time and illite dose. In another test, simultaneous softening and removal of PAC were tested by mixing 1.1 mol (1,248 mg) of soda ash for each mole of hardness with 200 mL wastewater. The mixtures were stirred for 5 min at 60 rpm and then left to settle before samples were taken at fixed time intervals.

In the continuous treatment, a small-scale CAPS process was operated in a 2-L Pyrex beaker containing 1,200 mL of total mixture and mixed with a magnetic stirrer revolving at 60 rpm. A glass fiber thimble (30 mL volume), wrapped in a glass microfiber filter from Whatman, was used as a submerged filtration unit that did not restrict the flow. The wastewater at pH 7 and soda ash solutions were kept in two separate 1-L beakers. The soda ash solution was 5 mg/mL to provide 1.1 mol (1,248 mg/L in mixed solution) for each mole of hardness. The flow rate was adjusted to 26 mL/min with a peristaltic pump giving a hydraulic retention time of 40 min. Wastewater and soda ash solutions were mixed in a ratio of 4:1.

### 2.5. Analysis

The supernatant samples were filtered through 0.45- $\mu\text{m}$  membrane. The filtrates were analyzed for pH, total hardness, calcium hardness, carbonate, bicarbonate, and hydroxide according to the standard methods for the examination of water and wastewater [18]. Total inorganic carbon (TIC) and TOC were analyzed by the TOC analyzer TOC-VCPH, Shimadzu.

## 3. Results and discussion

### 3.1. Softening at the two pH values and effect of initial LSI ( $LSI_{in}$ )

Softening is very dependent on the concentration of calcium and carbonate ions and pH. As the initial pH was increased, the softening efficiency increased (data not shown). Therefore, pH 7.0 and 10.0 were selected for further study. With increasing soda ash dose, the softening efficiency increased but the efficiency at pH 7.0 was about 10 mg  $\text{Ca}^{++}/\text{L}$  lower than that at pH 10.0, throughout the test range from 500 to 2,500 mg soda ash/L (Fig. 1). Similar tests were also performed with calcite addition to examine the role of seeding on the softening. Seeding further increased the process efficiency by further removing 10 mg  $\text{Ca}^{++}/\text{L}$  at pH 7. At pH 10 and low soda ash dose, the addition of calcite did not greatly affect the efficiency but at higher soda ash dose, i.e. above 1,500 mg/L, the softening efficiency was increased in the presence of calcite. Therefore, the maximum difference between

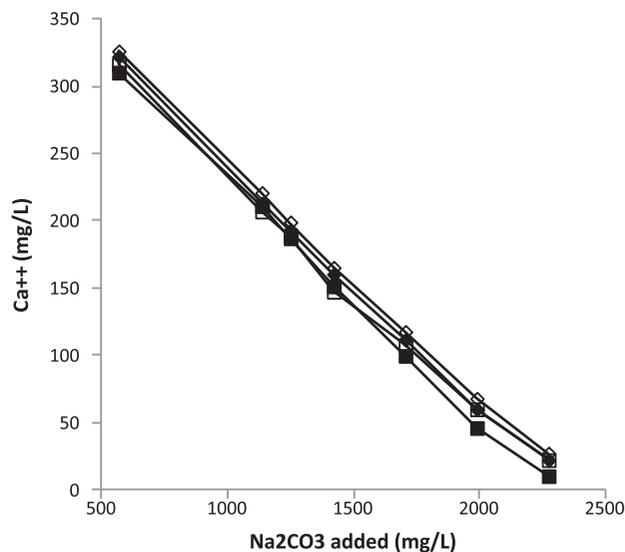


Fig. 1. Variation of softening efficiency with increasing soda ash dose and effect of calcite addition. ◇-pH 7.0, □-pH 10.0; empty marks without calcite and filled marks with calcite.

the test at pH 7.0 without seeding and that at pH 10.0 with seeding was 40 mg/L at the maximum soda ash dose. Due to the small difference between softening at pH 7 and 10, control of initial pH was not very significant.

### 3.2. Varying NaOH dose with fixed soda ash

We next monitored the equilibrium pH after increasing the NaOH dose and tested the softening efficiency. The selected soda ash doses were 1.0 (1,135 mg/L) and 1.3 mol (1,419 mg/L) for each mole of hardness. The NaOH dose ranged from 0 to 2000 mg/L. The resulting softening efficiency was higher at 1,419 mg/L dose than that at 1,135 mg/L dose, with the calcium removal increased by 50 mg/L in the low NaOH dose range (Fig. 2). With increasing NaOH dose, the difference decreased to 33 mg/L at 1,000 mg/L NaOH dose, showing that the low soda ash concentration was efficient at higher NaOH dose. This also demonstrated the importance of increasing equilibrium pH that can change bicarbonate to carbonates at higher pH.

The 1,419 mg/L soda ash dose completely removed the calcium at 1,000 mg/L NaOH dose, whereas in the previous test with the initial pH 10, the 2300 mg/L soda ash dose gave a comparable softening efficiency. At this soda ash dose, the equilibrium pH increased from 7.8 to 12 with increasing NaOH dose. This indicated that the addition of NaOH was more efficient than controlling the initial pH.

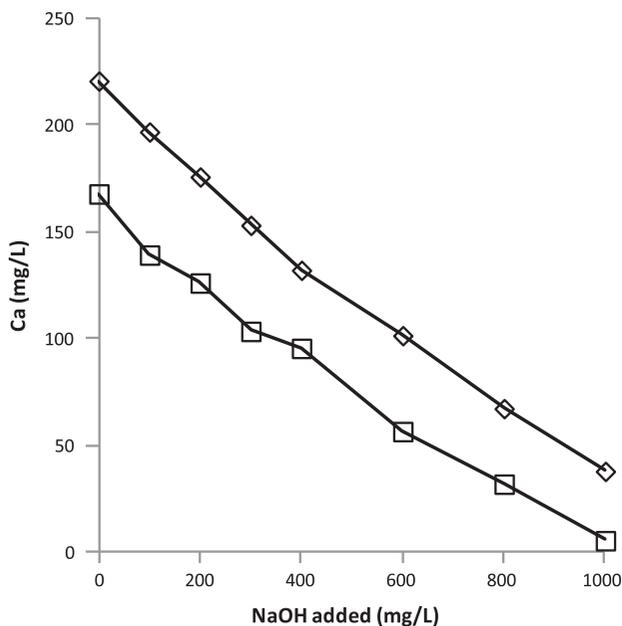


Fig. 2. Variation of softening efficiency with increasing NaOH dose at fixed soda ash dose. ◇-1,135 and □-1,419 mg/L.

The concentrations of alkalinity, i.e., carbonate, bicarbonates, and hydroxyls, were quantified (Fig. 3). At 1,135 mg/L soda ash, increasing the NaOH dose increased the pH from 7.8 to 12, increased the carbonates from 0 to 150 mg/L, increased hydroxyl from zero to 240 mg/L, decreased the bicarbonates from 50

to 0 mg/L, and decreased the calcium from 230 to 40 mg/L. The increase in pH transformed the bicarbonates to carbonates, which facilitated the precipitation of calcium carbonate as it became insoluble.

### 3.3. Langelier saturation index

During optimization tests,  $LSI_{in}$  and  $LSI_{fin}$  of the solution were calculated. In previous studies,  $LSI_{in}$  was calculated using analytical results of alkalinity and pH during the initial time of softening [8,10–12]. In this study, however, we calculated  $LSI_{in}$  at reaction commencement, using the pre-reaction pH, the inherent alkalinity of the original wastewater determined by analysis and the alkalinity added during softening, under the assumption that no reaction had started.  $LSI_{fin}$  was calculated using the alkalinity and pH determined by analysis at equilibrium.

During softening optimization by varying the NaOH dose,  $LSI_{in}$  at 1,135 mg/L soda ash dose was less than the 1,419 mg/L dose test results (Fig. 4(a)). In both of the tests,  $LSI_{fin}$  determined at equilibrium was far less than  $LSI_{in}$  with a minimum difference of 2.  $LSI_{in}$  at 1,419 mg/L soda ash dose was higher and  $LSI_{fin}$  was lower than that at 1,135 mg/L dose. The higher  $LSI_{in}$  indicated the saturation of the mixture that forced the equilibrium toward precipitation to increase the softening efficiency. At the higher soda ash dose of 1,419 mg/L, the softening was more efficient and  $LSI_{fin}$  was less than that in the 1,135 mg/L soda ash dose test. This more efficient softening

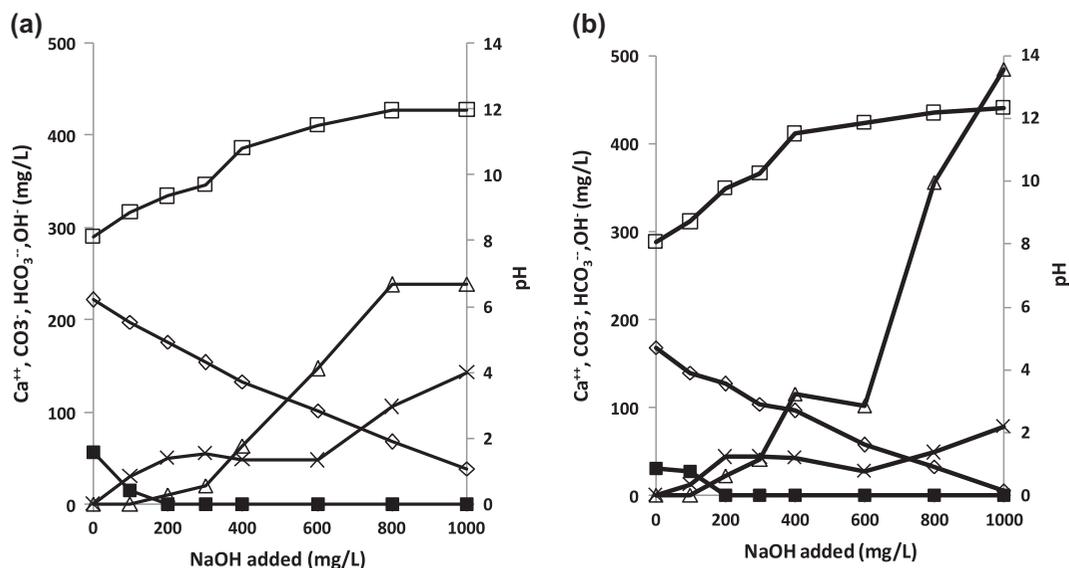


Fig. 3. Variations of concentrations of  $OH^-$ ,  $HCO_3^-$ , and  $CO_3^{2-}$  during the softening process with increasing NaOH dose at fixed soda ash dose. Soda ash dose (a) 1,135 mg/L and (b) 1,419 mg/L. ◇-Calcium, □-Equilibrium pH, Δ-Hydroxide, x-Carbonates, ■-Bicarbonates.

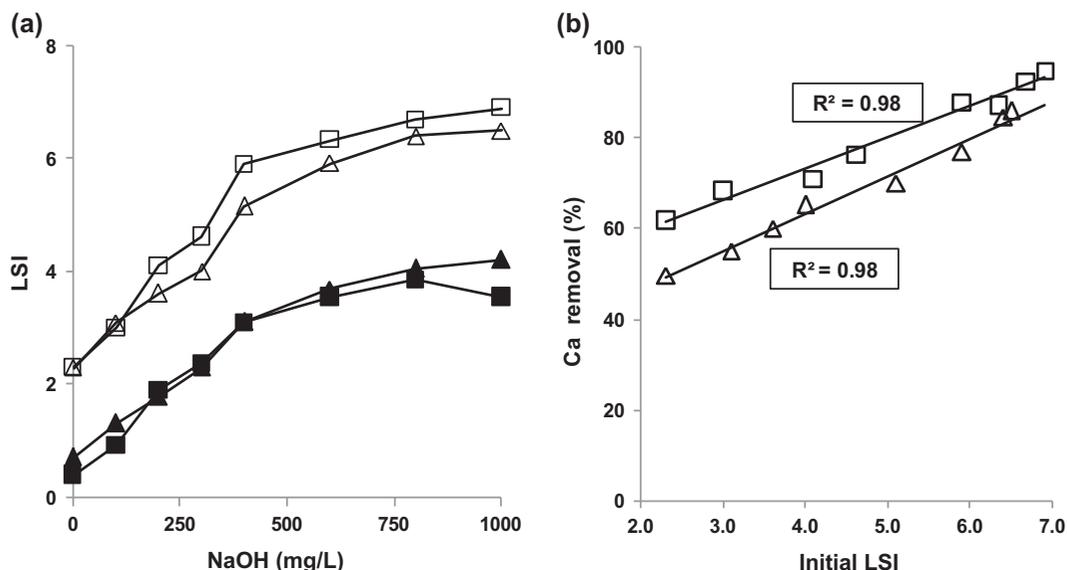


Fig. 4. (a) Variations of initial and final LSI with increasing NaOH dose and (b) correlation between initial LSI and calcium removal efficiency. Soda ash dose:  $\Delta$ -1,135 mg/L and  $\square$ -1,419 mg/L; empty marks for initial LSI and filled marks for final LSI.

reduced  $LSI_{fin}$  further, suggesting that  $LSI_{in}$  and  $LSI_{fin}$  were informative parameters to monitor during the softening process. At higher  $LSI_{in}$ , the softening efficiency was higher and  $LSI_{fin}$  was lower, and vice versa. Higher  $LSI_{fin}$  indicated that the degree of softening can be increased by further process optimization. At 1,000 mg/L NaOH dose,  $LSI_{fin}$  at 1,419 mg/L soda ash dose was slightly lower than that at 1,135 mg/L dose. The larger difference of  $LSI_{fin}$  at this NaOH dose may have been due to the lower concentration of carbonates in the water, although  $LSI_{fin}$  was higher.

A direct correlation was found between  $LSI_{in}$  and calcium removal efficiency (Fig. 4(b)) with  $R^2 = 0.98$  at both soda ash doses. This direct correlation confirmed the importance of  $LSI_{in}$  for predicting the softening efficiency. The correlation was equally effective at both soda ash doses. The only difference was the higher intercept value at the higher soda ash dose (45.3) than that at the lower soda ash dose (30.0). This showed that higher soda ash dose was also an effective parameter to be considered during softening efficiency. The slope for the two trend lines was similar (7.0 for higher dose and 8.35 for smaller dose), and hence, the trend lines were almost parallel. This revealed the importance of  $LSI_{in}$  for predicting the softening efficiency of wastewater.

During softening optimization by initial pH control, at pH 7.0,  $LSI_{in}$  was very low with a maximum value of 1.5 and  $LSI_{fin}$  was slightly less than that with a minimum value of 0.5 (Fig. 5(a) and (b)). Whereas at

pH 10,  $LSI_{in}$  was much higher, with a maximum value of 4.5 and  $LSI_{fin}$  was much less than that with a minimum value of 1. This confirmed the effect of controlling initial pH. The direct correlation between  $LSI_{in}$  and the softening efficiency, with  $R^2 = 0.95$ – $0.97$  (Fig. 6(a) and (b)), both with and without calcite, confirmed the importance of  $LSI_{in}$  and its value as an informative indicator of expected softening efficiency.

The importance of calcite presence during the softening process at different initial pH values was also indicated by the correlation of  $LSI_{in}$  with the soda ash dose (Fig. 5(a) and (b)). At pH 7.0,  $LSI_{in}$  was not affected by the addition of calcite. At equilibrium, however,  $LSI_{fin}$  without calcite was higher than that with calcite, which was attributed to the increased efficiency gained by the addition of calcite softening and the completion of the process near to  $LSI < 1$ . The solution without calcite had  $LSI_{fin}$  above 1 and a higher concentration of calcium than that of the solution without calcite. This means that the higher  $LSI_{fin}$  indicated an inefficient softening process. At initial pH 10, the addition of calcite was not very significant at 500 mg/L soda ash dose. However, at soda ash dose above 1,500 mg/L, calcite addition decreased  $LSI_{fin}$ , which indicated an improvement in process efficiency. This means that the seeding of calcite is more important at pH 7.0 at any soda ash dose, but at pH 10, calcite was only marginally effective at high soda ash dose.  $LSI_{fin}$  decreased with increasing soda ash dose, which indicated the extent of reaction completion. It was concluded that at initial pH 10,  $LSI_{in}$  was high enough to

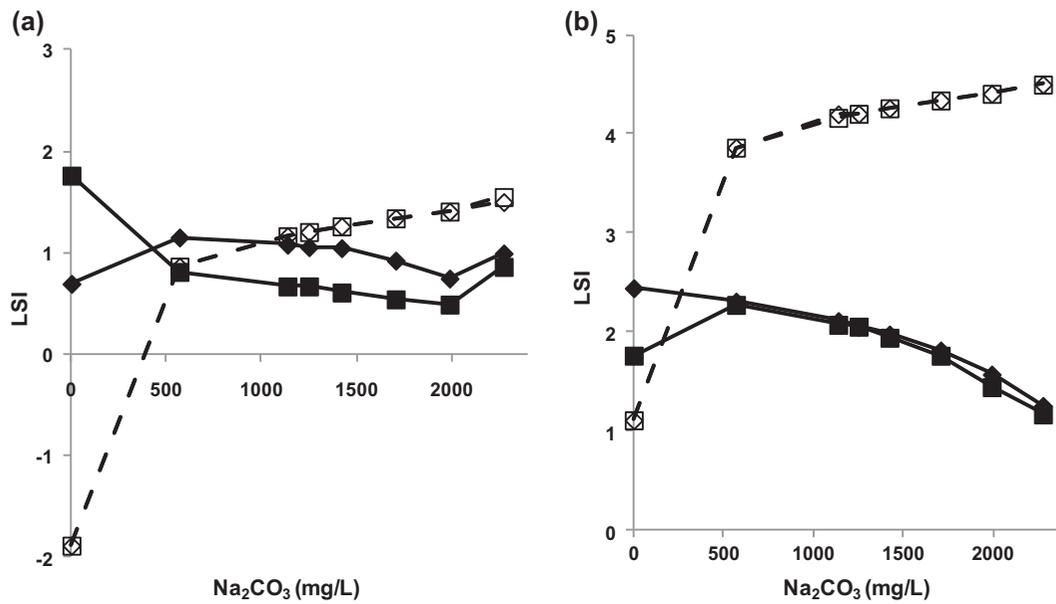


Fig. 5. Variations of initial and final LSI with increasing soda ash dose at fixed initial pH, (a) pH 7.0 and (b) pH 10.0. □-With and ◇-without calcite; empty marks for initial and filled marks for final LSI.

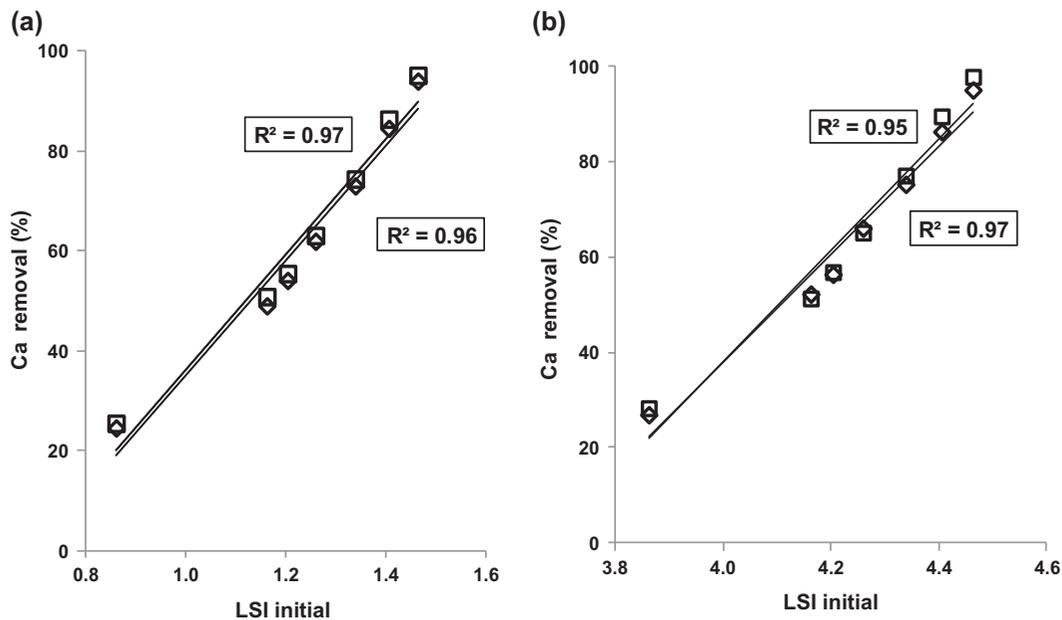


Fig. 6. Correlation between initial LSI and the calcium removal efficiency at initial pH 7.0 (a) and pH 10.0 (b). □-With and ◇-without calcite.

force the reaction toward completion without any seeding, with a minor exception at high soda ash dose. At pH 7.0, however,  $\text{LSI}_{\text{in}}$  was not high enough, so that seeding was needed to facilitate precipitation and make the process efficient.

In initial pH control, the direct correlation between  $\text{LSI}_{\text{in}}$  and the softening efficiency with  $R^2 = 0.96\text{--}0.97$  (Fig. 6(a) and (b)), both with and without calcite, showed the effect of calcite addition at pH 7 and 10. At pH 7, trend lines with and without

calcite had slopes 115.7 and 115.0 and the intercepts were  $-80.2$  and  $-79.6$ , respectively. This showed that at pH 7, throughout the tested range of  $LSI_{in}$ , the addition of calcite remained effective. At pH 10, the pattern of trend lines was different from that at pH 7. At  $LSI_{in}$  4.0, the trend lines were overlapping showing no effect of calcite addition; however, as  $LSI_{in}$  increased, the trend line with calcite showed higher efficiency due to a higher slope of 117 with the calcite test compared to 113.5 without the calcite test. This confirmed that at pH 10, calcite presence was not important at lower LSI and lower soda ash dose, but was important at higher soda ash dose and higher LSI. These graphs not only confirmed the importance of  $LSI_{in}$  for the calcium removal, but also showed the effect of calcite addition at different conditions.

This comparison showed that  $LSI_{in}$  was an informative indicator of the expected process efficiency. Once equilibrium was reached,  $LSI_{fin}$  showed the extent of process completion. For an efficient process,  $LSI_{fin}$  was less than 1 and for an inefficient process  $LSI_{fin}$  still remained above 1. This LSI study demonstrated that the presence of calcite affected the softening at lower pH, whereas calcite was needed at higher pH with higher soda ash dose only.

### 3.4. PAC removal and softening

TOC was measured as an indicator of PAC concentration because it was the only organic compound in wastewater (information provided by Hong Dok industry, Daegu, South Korea). In batch test for PAC removal from wastewater, the addition of only illite with a contact time of 60 min was sufficient to attain optimum TOC removal. Therefore, in the next test of mixing with illite, the contact time was maintained at 60 min. Increasing the illite dose increased the TOC removal but simultaneously increased the inorganic carbon as measured in TIC (supporting information). Illite is widely used as a coagulant aid and facilitates the flocculation of small residual PAC molecules that do not flocculate during the industrial coagulation/flocculation process. The increase in TIC may have been due to the colloidal-sized illite particles that remained in the water supernatant and did not settle or even dissolve.

The addition of only soda ash developed precipitates of  $CaCO_3$ , which indicated the ongoing softening process. In this wastewater treatment,

precipitated calcium carbonate also worked as a coagulant aid (Fig. 7(a)). The consequent removal of PAC led to the decreased TOC. The addition of soda ash increased TIC due to carbonates from the soda ash. These tests demonstrated the capability of soda ash to achieve considerable TOC removal during the softening process and confirmed that no illite addition was needed. The flocculation of PAC residues started once the precipitation of  $CaCO_3$  had started.

### 3.5. CAPS for softening and PAC removal

In the CAPS test, the softening was almost spontaneous and gradually increased over time until it stabilized after about 1,000 mL of wastewater treated (Fig. 7(b)). Calcium removal up to 60% was achieved in the CAPS test. In the reactor setup, the generated calcite remained in the reactor as the submerged filter only allowed the filtered water to pass whereas the precipitates remained in the reactor. This calcite accumulation in the reactor induced immediate precipitation of the incoming calcium-saturated water mixture. Therefore, as the quantity of calcite in the reactor increased over time, the softening efficiency also increased.

The precipitated calcium carbonate acted as a coagulation aid by flocculating the PAC residues and depositing them on the surface of the filter to form a compact layer that decreased the pore size and increased the filtration process efficiency. In addition to the TOC analysis, COD analysis [18] results revealed that the TOC analysis used in this study was not representative of the actual organics in the wastewater because the TOC analyzer temperature was not high enough to ensure complete oxidation of all types of organics. Therefore, the COD analysis of this study was truly representative of the total organics in the wastewater. The trend of COD removal showed that over time the organics removal efficiency increased to remove almost 41% of the PAC residues from the wastewater, whereas softening was the actual process. In CAPS, the softening efficiency at a soda ash dose 1,248 mg/L was equivalent to that at pH 7 with calcite addition. This showed that CAPS gave results comparable to those of batch tests but far quicker. This confirmed that no further treatment was needed after the CAPS softening process with a submerged filter unit for removal of PAC residues, and that the calcite produced during softening could flocculate the PAC residues.

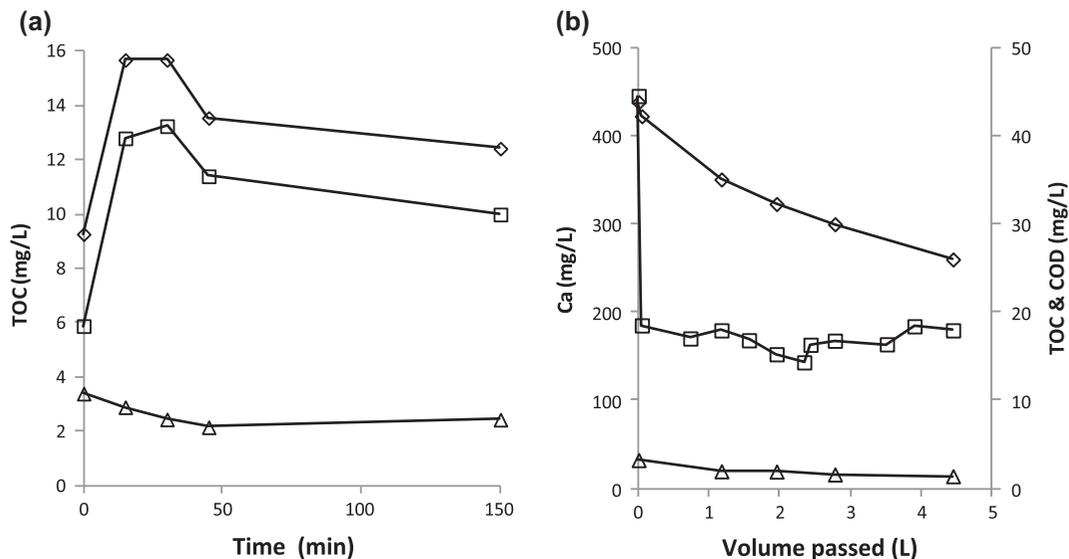


Fig. 7. (a): Variation in TC, IC, and TOC on addition of soda ash with increasing time.  $\diamond$ -TC,  $\square$ -TIC,  $\Delta$ -TOC and (b) CAPS test with variations of calcium conc., TOC and COD with increasing treated wastewater.  $\diamond$ -COD,  $\square$ -Calcium,  $\Delta$ -TOC.

#### 4. Conclusion

This study verified the importance of the calculated value of  $LSI_{in}$  for softening optimization by either initial pH or NaOH dose variation. Contrary to previously reported methods, we calculated  $LSI_{in}$  with the pre-reaction pH, the inherent alkalinities of the original wastewater and the added alkalinities, under the assumption that no physical or chemical reactions had started. The NaOH dose adjustment was more effective with 100% softening than that achieved by adjusting the initial pH. Efficient softening was predicted by higher  $LSI_{in}$  and confirmed by lower  $LSI_{fin}$ . Calcite addition increased the softening efficiency at lower  $LSI_{in}$ , like at pH 7. There was a direct correlation between  $LSI_{in}$  and softening efficiency ( $R^2 = 0.96-0.98$ ) and study of the graphical trend lines confirmed the importance of calcite addition. The CAPS process removed 60% of the calcium and 41% of the residual PAC from the tested water, aided by the calcium carbonate precipitates. In summation, the softening efficiency can be predicted by  $LSI_{in}$  and confirmed by  $LSI_{fin}$ .

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#### List of symbols

LSI	—	Langelier saturation index
pHs	—	pH of saturation

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