Water softening by induced crystallization in fluidized bed

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ABSTRACT

Fluidized bed and induced crystallization technology were combined to design a new type of induced crystallization fluidized bed reactor. The added particulate matter served as crystal nucleus to induce crystallization so that the insoluble material, which was in a saturated state, could precipitate on its surface. In this study, by filling the fluidized bed with quartz sand and by adjusting water pH, precipitation of calcium carbonate was induced on the surface of quartz sand, and the removal of water hardness was achieved. With a reactor influent flow of 60 L/hr, a fixed-bed height of 0.5 m, pH value of 9.5, quartz sand nuclear diameter of 0.2–0.4 mm, and a reflux ratio of 60%, the effluent concentration of calcium hardness was reduced to 60 mg/L and 86.6% removal efficiency was achieved. The resulting effluent reached the quality standard set for circulating cooling water. Majority of the material on the surface of quartz sand was calculated to be calcium carbonate based on energy spectrum analysis and moisture content was around 15.994%. With the low moisture content, dewatering treatment is no longer required and this results to cost savings on total water treatment process.

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Introduction

Water hardness can be attributed to the presence of certain ions in water which can easily form undissolved salts (Teixeira et al., 2012). Some of the common ions include calcium ions, magnesium ions, ferrous ions, manganese ions and aluminum ions. Treatment processes often include the removal of these ions due to certain water quality requirements (Apell and Boyer, 2010).

In industries, circulating water used for cooling comprises the largest percentage in water utilization. Strict requirements exist even for circulating water as the quality of the water may also affect the overall industrial process used. An example of a situation that is brought about by uncontrolled cooling water quality is the formation of salt crusts on the surface of heat exchangers. Various dissolved scaling salts may exist in cooling water. If these salt ions are not removed,

certain physical and chemical conditions in the system may cause an increase in the concentration beyond saturation point, resulting in crystallization of various salt ions (Esfahani and Yoo, 2014). These ions adhere to the surface of heat exchangers and other equipment, forming salt crusts and requiring maintenance. Salt crusts include calcium carbonate, calcium sulfate and calcium metasilicate and so on, of which, calcium carbonate is the most common and hazardous for water systems. Nowadays, there are various methods used to prevent calcium carbonate scale formation, such as softening through heating, seeding, ion exchange, nanofiltration, and electro-deionization (Omar et al., 2010). In the field of water softening treatment, lime soda softening process is the most common one in application. This is due to the numerous advantages of the process, such as, extensive sources of lime, easy disposal of solid waste, no pollution to the natural water bodies, and a big decrease of organic matter, silicate and iron

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in effluent. However, there are disadvantages when using the traditional lime softening method due to the generation of high-moisture sludge which is also hard to sediment. Generation of such sludge is undesirable as it means additional dewatering treatments not to mention that dealing with dewatered sludge has already been an urgent problem for national governments for a long time now (Indarawis and Boyer, 2013; Chehayeb et al., 2014). Therefore, a new kind of treatment method is required to remove water hardness and at the same time, minimize the generation of sludge.

Segev et al. (2013) modeled a fluidized bed system maintaining a high alkaline environment. With this, calcium ion in solution was induced to precipitate as calcium carbonate. Then, carbon dioxide was utilized to strip calcium carbonate from the system, thereby reducing water hardness. Currently, for precipitation from solution, evidences have shown that precipitation occurs more when certain amount of tiny particles already exist or form in the solution (Da Silva et al., 2014). Nowadays, the method of fluidized induced crystallization/precipitation is being used. This method combines the technology of fluid-bed and induced crystallization precipitation technology. The process operation just utilizes fluidized bed which has a certain number of induced crystal nuclei, and then, the precipitant which could form insoluble matters with ions is added, having an effect of purifying water (Comstock and Boyer, 2014). Kim van Schagen et al. paved small pellets on fluidized bed, and used the advantage of these pellets to enhance the water softening capability by fluidized bed. Moreover, evidences showed that it was possible to keep the reactor at desired operational parameters (pellet size and bed height) under varying operational conditions in a simulation experiment. In this way, the cost of pellet softening is reduced and the situation of irregularities could be prevented. However, the potential cost limited further development of this technique because of the addition of the induced crystal nucleus (van Schagen et al., 2008; Segev et al., 2011).

Quartz sand is a kind of silicate mineral which is hard, wear-proof and has stable chemical properties. Furthermore, it is an irreplaceably important raw material of fireproofing, glass-making and building industries because of the abundant sources and low cost. Taking some quartz sand with specific proportion and particle size into wastewater and keeping it in fluidized state by adjusting the rate of inflow, quartz sand could serve as a crystal nucleus to induce precipitation of insoluble matters on the surface. This shows that it has properties of good settling ability, high solid content, and good sludge dewatering ability. The precipitate could also be used as building materials. Compared with traditional methods, the method of crystal quartz sand was easy to conduct and had a small floor coverage, which could reduce capital expenditure and running cost (Du et al., 2011). In this study, quartz sand was utilized as induced crystal nucleus, and the research combined the technology of fluid-bed and induced crystallization precipitation to explore water softening process.

1. Materials and methods

This study was aimed to solve the problem of water hardness of the secondary effluent from Guodian Longshan Sewage Treatment Station. A laboratory-scale process was established to optimize various parameters of fluidized induced crystallization/precipitation at metastable region. Factors affecting calcium carbonate removal were also studied to determine the best runtime conditions to achieve good effluent quality as required by government standards. The test device and the experimental flowchart are shown in Fig. 1.

The reactor is a 5.5 L cylindrical transparent Plexiglas with a total height of 2400 mm including the conical bottom, the cylindrical body and the water outlet. The column was connected using screws and nuts and a rubber gasket was added to prevent water leakage. Size 200 mesh filters were placed at the top and bottom of the cylindrical column to serve as the support for the quartz sand bed layer. The lower part of the column is the fluidized bed and has a height of 2 m and a diameter of 5 cm. The upper part of the column is used for the water, having a height of 20 cm and an inner diameter of 10 cm. The water inlet and dosing point inlet are located at the bottom of the reactor. Water and softening agents enter the column in a tangential direction. The water inlet pipe is 8 cm. Located 30 cm with increments of 10 cm from the bottom are 10 sampling ports. A 100 mesh screen was installed near the outlet of the sampling port in order to prevent outflow of quartz sand.

To start the process, the column was filled with washed quartz sand. After loading the sand into the reactor, water was pumped into the reactor from the bottom, bringing the quartz sand to a fluidized state. Flow meters were installed to control the influent flow. Once the fluidized state stabilized, sodium carbonate solution was added into the reactor to serve as the precipitant, with quartz sand as the induced nucleus. This resulted in the formation of calcium carbonate precipitate on the surface of quartz sand, achieving the purpose of removing calcium ions. Treated water flowed out from the upper part of the column.

In order to identify the optimum operating conditions, experiments were done with controlled variables while varying others. The experiments were performed at a room temperature of 25°C, using water solution with calcium ion concentration of 450 mg/L. The concentration used in the research was chosen by taking the average value of the concentration of calcium ions in actual water samples obtained from the plant. Dynamic experiments were conducted to determine the effects of pH conditions, fixed-bed height, particle size, flow rate, and reflux ratio on the removal efficiency.

1.1. Wastewater samples

This study focused on the optimal conditions for the removal of calcium ions from wastewater. Actual water samples from the sewage treatment plant had high calcium ion concentrations (ranging from 449 to 456 mg $CaCO_3$/L). In order to simulate the conditions of the actual wastewater, an equally high concentration of 450 mg $CaCO_3$/L was used during the experimental phases to test the effects of pH conditions, fixed-bed height, particle size, flow rate, and reflux ratio on the removal rates.

Removal of water hardness is greatly affected by the pH of the water system and the saturation pH. Because crystallization
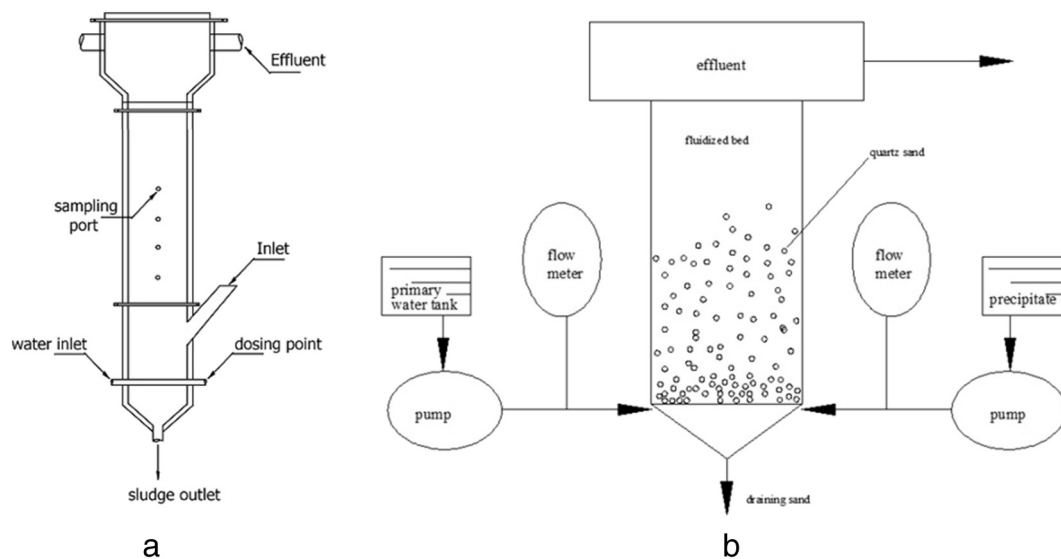
Fig. 1 – (a) Fluidized bed reactor, (b) experimental flowchart.

is highly dependent on the saturation index, which in turn, is greatly affected by the pH, the best pH conditions were determined first. By varying the pH conditions and keeping the other parameters constant, the effects of pH on calcium ion removal was determined. After which, the effects of other parameters were studied.

1.2. Analytical methods

At the end of the experiments, quartz sand samples were obtained for analysis. First, the quartz sand carrier was removed and placed inside an oven to be dried at 105°C for 3 hr. After drying, the weight was obtained and moisture content was calculated by subtracting the dry weight from the total weight before drying. Samples were then analyzed using Scanning Electron Microscope and Energy Dispersive Spectrometer SEM-EDS (Model: SSX-550) with a magnification of ×450. The energy spectrum image of the sediment was also obtained and was determined using elemental analysis.

At the end of the experiment, after drying the samples, the quartz sand samples were placed in a beaker. Deionized water and 0.1 mol/L hydrochloric acid were added and the solution was poured into a 1-L volumetric flask. The beaker was washed repeatedly with deionized water to ensure that no residue remained in the beaker. After filling the volumetric flask, an electromagnetic mixer was used to mix the solution in the flask. After which, the concentration of the calcium ion in the digestive juice was obtained. Using Eq. (1), the amount of calcium carbonate on the surface of the carrier was calculated.

$$W_{CaCO_3} = C_{Ca^{2+}} \times 1L \times \frac{W_T}{W} \qquad (1)$$

where, W_{CaCO_3} (mg) is the weight of the calcium carbonate on the carrier, C_{Ca2+} (mg/L) is the calcium ion concentration in

digestive juice, W_T (g) is the total carrier weight, and W(g) is the weight of carrier for digestive reaction.

2. Results and discussion

2.1. Effect of pH value

Depending on the pH of the system, calcium ions can exist in different forms: $CaCO_3$ at higher pH (usually close to 10.3) and H_2CO_3 at pH lower than 6.3. Calculation of the saturation index could gauge whether calcium carbonate can be precipitated or dissolved. If the saturation pH of $CaCO_3$ is less than the actual pH, the water is considered scale forming and calcium carbonate is deposited. The saturation index increases when actual pH is increased. This may explain why past researches using crystallization in fluidized bed reactors were conducted at
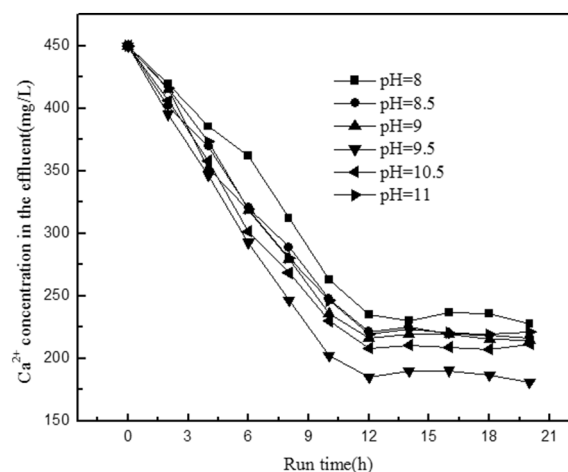

Fig. 2 – Removal of calcium ion at different pH.

environments with high pH, usually above 9.0 but lower than 11. In this part of the study, the pH values were changed while the following conditions remained constant: the rate of inflow was 60 L/hr, the size of induced crystal nucleus was 0.2–0.4 mm, and the fixed-bed height was 0.5 m. Fig. 2 shows the influence of calcium ion removal at various pH levels. It displayed that the effluent calcium ion concentration decreased with time and stability was achieved after a certain performance period. It can also be seen in the graph that calcium ion concentration in effluent became lower when pH level was higher. This is because carbonate ion would became more stable and the amount also increased when the system had a higher pH level. On the contrary, when the pH level decreases, carbonate ion would decrease before finally disappearing. Further study found that the removal rate of calcium ion decreased when calcium ion concentration in the effluent was stable and the pH level was higher than 9.5. The reason was that the pH level increases with increasing concentrations of sodium hydroxide. This causes a sharp increase in the partial degree of supersaturation which induces crystallization of calcium carbonate by spontaneous nucleation and results to a decrease in the heterogeneous nucleation efficiency (Li et al., 2005; Burggraeve et al., 2013).

Furthermore, when the pH value of water sample was around 10, Mg^{2+} would also react with carbonate ion to produce magnesium hydroxide precipitation, as shown in Fig. 3. Since the solubility product constant of magnesium hydroxide is much lower than the solubility product constant of calcium carbonate, this signifies that it is easier for magnesium hydroxide to form precipitates. The formation of this kind of precipitate means the consumption of hydroxide ion, thus reducing the amount of carbonate ion. This, in turn, has an effect on the bonding between calcium and carbonate ion, and eventually, on the removal rate of calcium ion (Zhani, 2013).

2.2. Effect of the height of fixed-bed

Fig. 4 shows the part of the study focused on the influence of the height of the fixed-bed. The constant parameters were as follow: size of induced crystal nucleus was 0.2–0.4 mm, the pH value was around 9.5, and the inflow was 60 L/hr. As time passed, calcium ion concentration in effluent declined, and
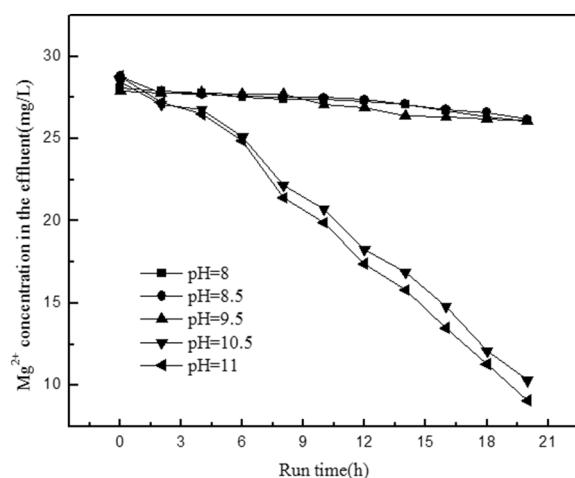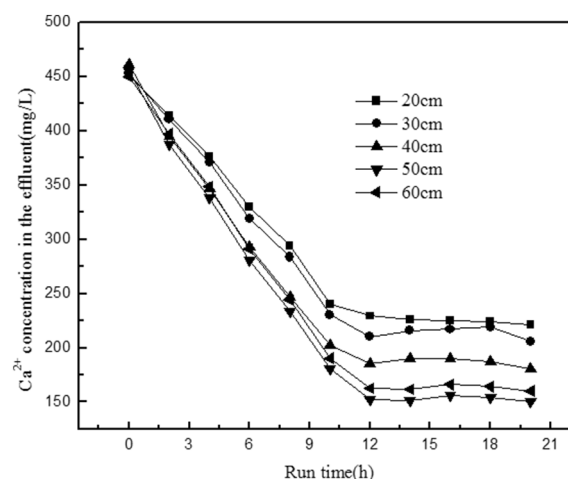

Fig. 4 – Removal of Ca^{2+} at different heights of fixed-bed.

this tendency was consistent at various heights of fixed-bed. The lowest calcium ion concentration in effluent was found when the height of fixed-bed was 0.5 m. Since the surface area of the carriers is proportional to the fixed-bed height, there was an increase in the contact time and area between water sample and sand. This made it feasible for calcium carbonate to adhere to the carriers, enhancing the removal rate (Ping et al., 2013). However, the study found that there was too much pressure when the height of fixed-bed was at 0.6 m or higher, which made the degree of fluidization become smaller. Thus, the optimal height of fixed-bed was 0.5 m.

2.3. Effect of the induced crystal nucleus sizes

Fig. 5 shows the effect of different sizes of crystal nuclei. In this part of the experiment, the rate of inflow was maintained at 60 L/h and the compacted height of fixed-bed was 0.5 m. For the size of sand spheres, the surface area was 0.02–0.05 m^2/g of beads. The size of the induced quartz crystal nuclei

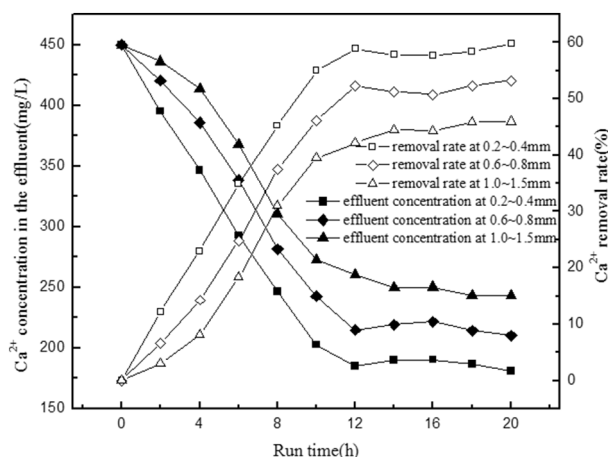**Fig. 3 – Removal of Mg^{2+} at different pH values.**

Fig. 5 – Removal of at different particle sizes of the crystal nucleus.

were varied and the following sizes were used: 0.2–0.4 mm, 0.6–0.8 mm, 1–1.5 mm.

Based on the results of the experiments, it can be seen that at longer run times, there is a decrease in the effluent calcium concentration before reaching stability. It is seen in the graph that the effluent calcium ion concentration was lowest when the size of the induced quartz crystal nucleus was 0.2–0.4 mm. Because the surface area is bigger when the size of induced quartz crystal nucleus is smaller, the chances of contact between supersaturated calcium carbonate and induced quartz crystal nucleus increases. As such, a smaller crystal nucleus is preferred for crystallization of calcium carbonate from water sample and also, for higher treatment efficiency (Li et al., 2012). However, it must also be taken into account that the size of the particle must be small enough for larger contact area but big enough to not be carried away with the effluent. Otherwise, this would bring down the removal efficiency despite having a bigger surface area (Wang et al., 2013). In this study, the size of 0.2–0.4 mm was optimal.

2.4. Effect of the flow rate

In Fig. 6, the influence of inflow rate was studied by changing inflow rates while maintaining the same values for other parameters. With ongoing run time, calcium ion concentration decreased, a common behavior at various inflow rates. The effluent containing the lowest calcium ion concentration (180.8 mg/L) and highest removal rate was obtained at an inflow rate of 60 L/hr. Theoretically, the removal rate is better at a lower inflow rate because there is an increased contact time between water sample and induced crystal nucleus (Zamankhan, 2012). However, based on the study and as shown in Fig. 6, the largest removal rate was not at the 40 L/hr inflow rate. Since the fluidized crystal nucleus could not match the minimum fluidization velocity required, it could not achieve the state of fluidization, only having contact with the sand (Aldaco et al., 2007a). In this state, contact efficiency decreased and removal rate was affected.

2.5. Effect of reflux ratio

As it is easier for supersaturated ions to precipitate out on the surface of crystal nuclei, it seemed significant to utilize
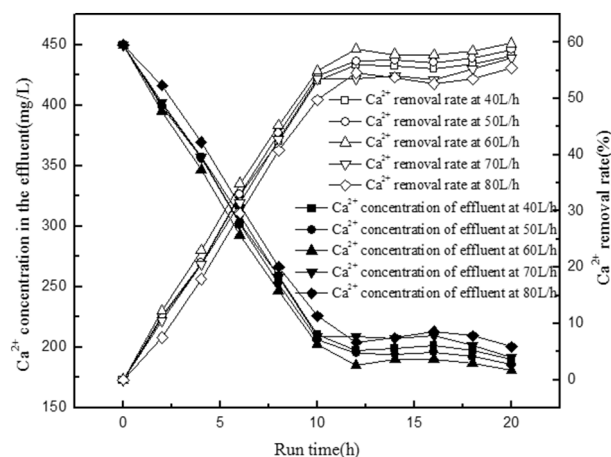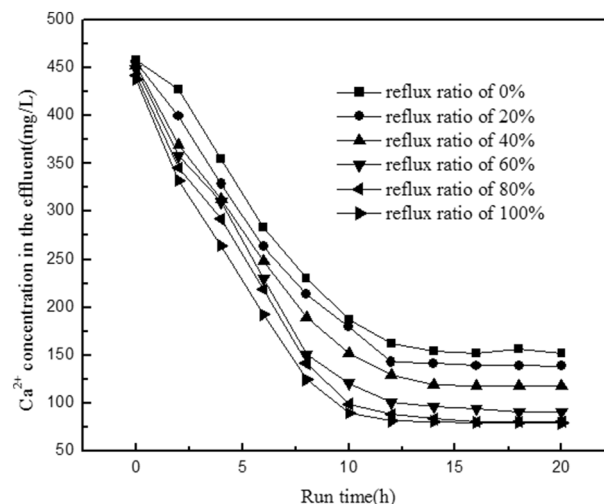

Fig. 7 – Removal of Ca^{2+} at different reflux ratios.

treated crystal nucleus to deal with water sample. With ongoing run time, the induced crystal nucleus would expand in fluidized bed, filling up the space and consequently, influencing the treatment process.

In this study, the size of induced crystal nucleus was 0.2–0.4 mm, the pH value was around 9.5, and the inflow was 60 L/h. The influence of different reflux ratios on the flow of effluent was studied. From Fig. 7, with ongoing run time, calcium ion concentration in effluent declined at various reflux ratios. When the reflux ratio increased, the treated crystal nucleus induced precipitation of supersaturated ions while lowering the pH of the system (Günther et al., 2013). The curve of reflux ratio of 0% indicated an increase in the removal rate of calcium ion from a slower rate at initial stage. While it is hard for supersaturated ions to dissolve out on the smooth surface of the induced crystal nucleus, longer run
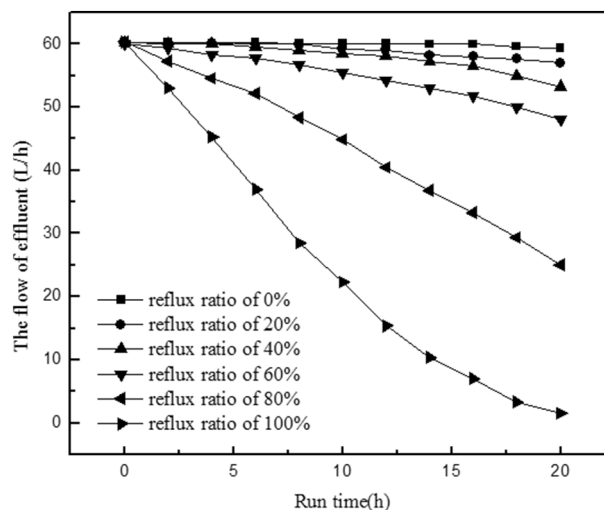

Fig. 6 – Removal of Ca^{2+} at different flow rates.

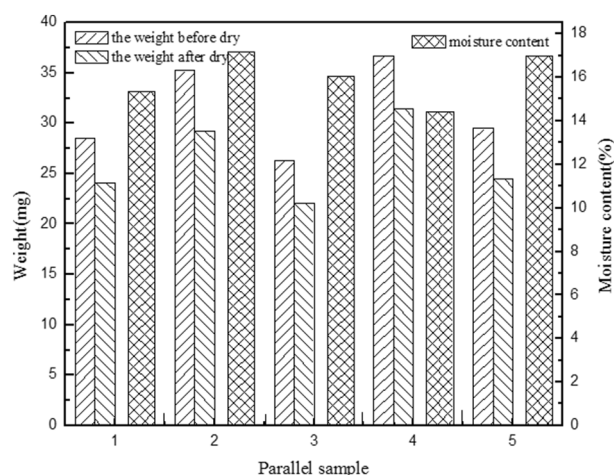**Fig. 8 – The flow of effluent at different reflux ratios.**

Fig. 9 – Moisture content of quartz sand.

time will increase the number of particles. As the surface becomes rougher, it becomes easier for the supersaturated ions to precipitate out (Hickenbottom and Cath, 2014). Thus, theoretically, the higher the reflux ratio, the better the treatment effect.

On the other hand, Fig. 8 shows the variation of effluent flow at different reflux ratios. At longer run time, the effluent flow declined at various reflux ratios, and the degree of decline became larger when the reflux ratio turned higher. A higher reflux ratio could mean more crystallization occurrence in fluidized bed when the reactor is in operation (Chmelar et al., 2013). It can be seen that the flow of effluent decreased by half when the reflux ratio was higher than 80%. In contrast, the 0%–60% reflux ratio had an acceptable range, accounting for less than or approximately 10% loss in effluent flow rate. Thus, combining with the results from other conditions, this study chose the reflux ratio of 60% as optimal level.

2.6. Identification and analysis of crystal nucleus

2.6.1. Moisture content of crystal nucleus
After a complete process of treating water sample in fluidized bed, this study attained a certain amount of induced crystal nuclei, all of which were obtained at optimum conditions. In order to ensure reliability in testing, five portions of induced crystal nucleus were weighted before and after three hours of

drying in the drying oven at 105°C, as shown in Fig. 9. The study found that the average moisture content of induced crystal nucleus was 15.99%. This value displayed a very low moisture level from crystallization fluidized bed reactor when compared with the sludge from mechanical accelerated clarifier (Plappally and Lienhard, 2012). Therefore, the use of crystallization fluidized bed reactor could reduce the volume of discharged sludge and save the cost of sludge dewatering for water softening process. Thus, it is feasible to utilize fluidized crystallization technology.

2.6.2. Surface topography of crystal shape
The SEM images of quartz sand particles before and after the fluid-bed processing are shown in Fig. 10. As shown in Fig. 10a, unused crystalline precipitates had no sediment and crystallization; in Fig. 10b, c, some obvious crystal substances were brought out on the surface of quartz sand particles, becoming rougher as the water traveled upward. A layer of calcium carbonate crystals were found on the surface (Grumiaux et al., 2012). Deposition and growth of crystal nucleus would develop after the solution obtains a state of saturation, or supersaturation. Then, once the crystal nucleus is formed, it is easier for supersaturated ions to precipitate out on rough surfaces. This shows that the reaction rate would increase with time. Calcium ion concentration would decrease slowly until it reaches stability (Aldaco et al., 2007b). Thus, this research displayed that the rate of crystallization of calcium carbonate would increase after an initial layer of calcium carbonate crystal is formed (Aldaco et al., 2007c).

Energy spectrum analysis showed the spectral image and the analysis of the elements. Fig. 11 and Table 1 display the analysis of the sediment on the surface of quartz sand. Based on the mole ratio, the sediment obtained may be calcium carbonate, magnesium hydroxide, or carbonate silicon. Because the quality proportion of magnesium ion and silicon was very low, it was concluded that the main substance was $CaCO_3$, since the mole ratio of Ca, C and O was 1:1:3 (Hermosillo et al., 2012).

3. Conclusions

(1) Optimal conditions for the removal of water hardness were found to be at an environment with pH 9.5, a reflux ratio of 60%, and an inflow rate of 60 L/hr. Optimum height of the fixed-bed was 0.5 m and the size of induced crystal nucleus was 0.2–0.4 mm. Addition of induced crystal nucleus had a positive effect on the fluidized
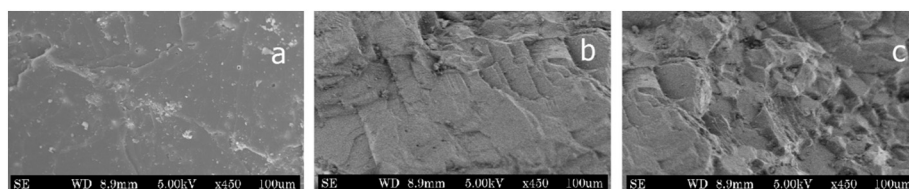

Fig. 10 – (a) SEM of unused crystalline precipitates, (b) crystalline precipitates at the center of the fluidized bed, (c) crystalline precipitates at the bottom of the fluidized bed.
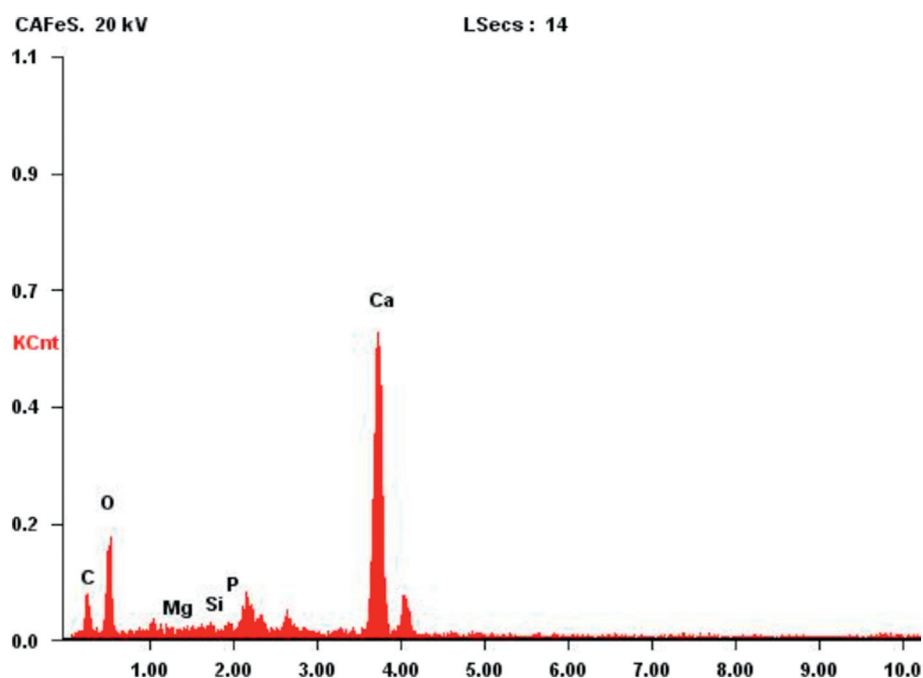
Fig. 11 – Spectrum image of quartz sand at bottom of the fluidized bed.

crystallization and is an advantage when reducing hardness in water. Concentration of calcium ion in $CaCO_3$ form in the effluent was reduced to 60 mg/L, a removal rate of 86.6% in this study. This calcium hardness index reached the standard set for circulating cooling water.

(2) In the study, the removal rate of calcium ion was low at the initial stage. As time passed, crystallization of calcium carbonate was observed on the surface of quartz sand and gradual increase in removal rate followed. This showed that water softening is possible after a period of time.

(3) Energy spectrum analysis showed that majority of the crystal substance on the surface may be $CaCO_3$, with a moisture content of 15.99%. With this low moisture content, sludge dewatering is no longer needed, thereby, reducing the cost of sludge treatment and disposal. It is recommended by the authors that for future studies, more tests should be done in determining the crystal substance.

Acknowledgments

Table 1 – Quality percentage and elemental mole.

Elemental	C	O	Mg	Si	Ca
Quality percentage (%)	12.66	49.77	00.63	00.29	36.65
Mole ratio	1.055	3.111	0.026	0.010	0.916

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