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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.

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

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