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Effect of freeze/thaw conditions, polyelectrolyte addition, and sludge loading on sludge electro-dewatering process

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ABSTRACT

Laboratory scale pressure-driven electro-dewatering reactor was set up to study the effect of polyelectrolyte addition and freeze/thaw conditions on solid content in the final sludge cake at different sludge loading rates. An increase in freezing temperature and extended natural freezing periods resulted in a significant increase in sludge dewatering ability. However, dry solid (DS) content in the final sludge cake after electro-dewatering was similar (39.3–41.5%) regardless the experimental strategies. The reduction in sludge loading rate from 20 to 3 kg DS/m² resulted in the increase in DS content of the final sludge cake (35.8–48.7%) using both, the polyelectrolyte addition and freezing condition sludge samples. During electro-dewatering using sludge amended with polymers, the DS content in the final sludge cake by the anode and the cathode was similar or a bit higher at the cathode either at high sludge loading or using high amount of polymers. It was found that polymer addition had a negative effect on the electro-osmotic flow.

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1. Introduction

The most important disadvantage of aerobic municipal wastewater treatment is the generation of large amounts of waste sludge. Original biological sludge from wastewater treatment plant such as activated sludge and anaerobically digested sludge are well known to have a poor dewaterability. It is known that freeze/thaw conditioning is a highly effective sludge dewatering technique. The main principle of this technique is that during freezing, ice crystals grow incorporating water molecules [1,2]. Because the structure of ice crystals is highly organized and symmetrical, it cannot accommodate any additional atoms or molecules. Each ice crystal continues to grow as long as there are water molecules available. All other impurities and solid particles are forced to the boundaries of the ice crystal where they become compressed or dehydrated. This technique changes sludge floc into a compacted form, reduces sludge bound water content and makes sludge more apt for settling and filtration [3]. In general, sludge freezing at slow freezing rates shows better dewaterability than fast freezing. Nonetheless, it has been reported that even fast freezing significantly improves sludge dewaterability [4]. Ormeci and Vesilind [2] who studied freeze/thaw conditioning effect on alum and activated sludge, concluded that freeze/thaw conditioning effectively dewatered alum and activated sludge, however, alum sludge was likely to freeze/thaw better than activated sludge due to its low dissolved ion and organic matter content. High concentrations of dissolved ions and organic material present in activated sludge promote particle entrapment during freezing and decrease the effectiveness of freeze/thaw conditioning. Alum sludge ice crystals predominantly grow in columns, whereas activated sludge ice crystals grow in dendrite [5]. The addition of dissolved solids (NaCl) to alum sludge changes ice crystals growth from columnar to dendrite. Because the dendrite ice crystals are formed at the ice/water interface, sludge particles are trapped in the ice front, resulting in a decrease in sludge dewaterability. The freeze/thaw conditioning does not only increase the sludge dewaterability but also reduces pathogenic microorganisms in sludge [6]. It is also considered as a low-cost sludge treatment technique at moderate to cold climates [7].

Electrically assisted treatments have gained popularity in environmental engineering in recent years [8–13]. Electro-dewatering method, when low level electric field is applied to the sludge cake to induce the migration of water, has been reported to significantly reduce water content in the final sludge cake [14,15]. However, the effectiveness of electro-dewatering process strongly depends on electric field strength and the contact time [16–18]. Moreover, an increase in voltage application subsequently results in reduced water content in the final sludge cake as reported by numerous researchers [14,19–21]. During electro-dewatering process, sludge type and alkalinity plays an important role in water removal rate and the final dry solid (DS) content of the sludge cake [22]. The effect of different types of polyelectrolyte and doses on electro-

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Table 1

Main	characteristics	of sludge from	n Mikkeli Waste	water Treatment I	Plant

dge after anaerobic digestion
4-34.0
60-7500
6-7.38
52-10487
7–2.87
3–1.70
1–53.4
4.0 to -15.9
7.7–345.8

dewatering was investigated by Saveyn et al. [23]. However, there was lack of studies in influence of freeze/thaw conditioning on electro-dewatering of sludge.

In this study, a laboratory scale pressure-driven electrodewatering reactor was set up to investigate the effect of polyelectrolyte and freeze/thaw conditioning on water content in the final sludge cake at different sludge loading rates.

2. Materials and methods

2.1. Sludge samples

Laboratory scale experiments were conducted with anaerobically digested sludge taken from the effluent of anaerobic digestion unit of Mikkeli Wastewater Treatment Plant (SE Finland). The anaerobic digester was continuously operated and treated mixing primary and secondary sludge with 17–20 days retention time at 35–38 °C. The main characteristics of sludge are shown in Table 1. Because the DS content of this sludge was low (3.1%), the samples were pre-treated by settling to increase the sludge DS to 5%. The sludge was stored at 4 °C prior treatments. Before sludge conditioning, the sample was kept out of the cold room to reach the room temperature.

2.2. Sludge conditioning

A deep freezing room with a digital controller was used to freeze the sludge samples. The samples were frozen at -5, -10, -15, -20, and -25 (± 1)°C. The preliminary experiments (at -5°C) showed that sludge was completely frozen within 36 h, therefore the freezing time of 36 h was chosen for all the experiments. During natural freezing treatments, 51 of the digested sludge samples were kept in 81 plastic buckets placed outdoors at the beginning of January until the end of March, 2008. The average temperatures in January, February, and March are -9.3, -8.9, and -4.3°C, respectively. However, the temperature can be fluctuated from +5 to -30°C. Long term thawing results in altered sludge characteristics due to the anaerobic reactions in the sludge [2]. Thus, all the freezing sludge samples were thawed for 10 h at a room temperature.

Granular polyelectrolyte from Praestol (Praestol 855BS, Germany) was used for sludge conditioning. The polymer had a medium cationic charge, effective at the pH range from 1 to 10. Polymer solutions 4 g/l (0.4%) were prepared at least 24 h before the use [23]. A conventional Jar Test was used to mix the anaerobically digested sludge and polymer. 500 ml of sludge samples were placed in 1000 ml beakers, and then different doses of polymer solutions equivalent to around 5, 10, 15, and 20 kg/ton of DS (kg polymer per ton of DS) were added. Sludge and polymer mixture was then intensively mixed at 300 rpm for 1 min, following by 10 min of slow mixing at 50 rpm. After that sludge samples were fed into the reactor for dewatering.



Fig. 1. Schematic representation of the laboratory scale pressure-driven electrodewatering reactor.

2.3. Electro-dewatering tests

The electro-dewatering experiments were conducted in a pressure-driven reactor [24] (Fig. 1). The reactor was made of plastic (9.8 cm in diameter and 30 cm in length). The stainless steel mesh (0.5 mm thickness) and titanium plate electrode (2 mm thickness) were used as the cathode and the anode, respectively [22]. The electrodes were connected to the DC (Direct Current) power supply (GW Instek, Taiwan) to provide a constant (20 V) voltage. The cloth filter (Z104256 PP, Sigma–Aldrich), with permeability factor of 7.62 (m³/(m² min)), was placed by the electrodes. The laboratory scale experiments were performed at a room temperature ($22 \pm 1 \,^{\circ}$ C).

After the sludge samples were introduced into the reactor, sludge was subjected to a constant pressure of 5.0 bars and 20 V throughout the experiments. Removed water was collected from both, the anodic and the cathodic sides. It was assumed that the electro-dewatering process was finished, when the water removal rate at the cathode decreased below $2 l/(m^2 h)$ (liters removed per square meter filter per hour). Sludge was removed from the reactor and sampled at the anode, the cathode and the sludge cake.

Sludge loading rate was 15 kg DS/m² (kg DS per m² filter) for the experiments using natural and controlled freeze/thaw conditions and different polyelectrolyte dosages. To investigate the effect of sludge loading rate on the final sludge cake, experiments were conducted using about 3, 5, 10, 15, and 20 kg DS/m² sludge loading rates with sludge frozen at -20 °C and 15 kg/ton DS of added polymer. Blank experiments applying pressure only without electricity were performed to ensure the comparability of data. Moreover, to avoid discrepancies and ensure reliability of data, all the experiments were performed in duplicates.

2.4. Analysis

Multimeter (Fluke 110, The Netherlands) was used to monitor current fluctuation. The amount of removed water was measured by electrical balances. Temperature of sludge was measured by electronic thermometer (Fluke53 II, USA). Characteristics of sludge, such as pH, alkalinity, DS (dry solid) VS (volatile solid), and CST (capillary suction time) were determined using Standard Methods



Fig. 2. The amount of removed water at the anode and the cathode during electro-dewatering process using different conditioned sludge samples: (a) freezing at $-5 \circ C$, (b) freezing at $-25 \circ C$, (c) original sludge without treatment, and (d) 15 kg/ton DS of added polymers.

[25]. Zeta potential was determined by Malvern zeta sizer (Nano-Series Instrument, England). To measure zeta potential, the sludge sample was centrifuged at 4000 rpm for 10 min, after that liquid in the upper layer was used to dilute the original sludge sample [21,22]. The average temperature data from January to March, 2008 was taken from automatic weather station at Mikkeli Airport (61°41′14.0″N, 27°12′35.7″E).

3. Results and discussion

3.1. Effect of different freezing temperatures on electro-dewatering efficiency

After freeze/thaw conditioning, the sludge floc structure was altered and became more compacted as also observed by Vesilind et al. [3]. Increased floc size is directly proportional to the improved filterability and settleability of the sludge [26]. In comparison to the CST of the original sludge, freeze/thaw conditioning significantly reduced the CST of the treated sludge samples. During electro-dewatering process using freeze/thaw conditions at different temperatures, electro-dewatering was over after 150 min of the experiment (Fig. 2a and b). On the contrary, electro-dewatering using the original sludge samples lasted approximately 600 min (Fig. 2c) and the sludge cake was not formed at all. Therefore, sludge conditioning was necessary to improve the solid–liquid separation by pressure filtration, resulting in smooth electro-dewatering was achieved.

The freezing rate was found to be an important parameter in the performance efficiency of the freeze/thaw process. The slow freezing rate results in the significant improvement of the sludge dewaterability [4,27,28]. When the freezing rate is high, sludge particles are entrapped in the developed ice layer, which impairs the sludge dewaterability [29]. Moreover, when the freezing rate is low, flocs are rejected instead of being trapped, thus they tend to migrate in front of the growing ice crystals, which relates to a remarkable increase in the sludge dewaterability rates. The decrease in freezing temperature was mainly associated with an increase in freezing rates, therefore the CST of freezing sludge samples at -5, -10, -15, -20, and -25 °C were 94.2, 111.7, 141.1, 226.2, and 319.2 s, respectively. However, in electro-dewatering experiments using sludge samples frozen at different temperatures, water content in the final sludge cake was found to be the same regardless the freezing temperature (Fig. 3a). This could be due to the fact that electro-dewatering process was discussed being less sensitive to sludge characteristic by Yoshida [30].

Fig. 2 shows the amount of removed water as recorded at the anode and the cathode during electro-dewatering treatment of sludge samples with different conditioning methods at the same sludge loading rate. When the low level direct electric current was applied, electro-osmotic flow transported water from the anode to the cathode [31,32]. Therefore, the amount of water removed at the cathode was significantly higher than at the anode. As above discussion, a decrease in freezing temperature increased dewaterability of sludge, which can be attributed to an increase in removed water at initial state of experiments. Indeed, during experiments using sludge samples freezing at $-5 \circ C$ (Fig. 2a), 90% of total removed water was already taken out during initial 3 min in comparison to 68% during initial stages of experiment using sludge samples freezing at -25 °C (Fig. 2b). However, the total amount of removed water in both experiments was the same at the end of the treatment. This might be related to similar water content in the final sludge cake regardless the freezing temperature in above discussion (Fig. 3a).



Fig. 3. DS content of the final sludge cake, sludge by the anode and the cathode due to electro-dewatering using freeze/thaw sludge at different temperatures (a and b) and -20 °C freeze/thaw sludge at different sludge loading rate (c and d).

3.2. Effect of sludge loading rate on water content in the final sludge cake

Fig. 3c and d shows the DS content in the final sludge cake and the samples at the anode and the cathode, after electro-dewatering with different sludge loading rates using sludge frozen at -20 °C. It was observed that due to electro-dewatering process, a decrease in the sludge loading rate significantly increased the DS content in the final sludge cake. It was contradicted with blank experiments without the applied electric field, when the DS content in the sludge cake was the same regardless sludge loading rate (Fig. 3c). Results from Saveyn et al. [33] showed a clear profile of the sludge cake consisting of different sludge zones with different water contents, gradually increasing from the anode to the cathode during electrodewatering. At some point during the experiments, the contact between the anode and the sludge matrix was reduced, ceasing the electro-osmotic flow. Therefore, the DS content of the sludge cake may be limited by the dry layer of sludge at the anode. Similar dry layer with high electrical resistance formed at the anode was also observed by Ho and Chen [34]. The thickness of the final sludge cake in experiments with sludge loading rates of 3, 5, 10, 15, and 20 kg DS/m² were 3.6, 6.7, 14.6, 20.1, and 29.0 mm, respectively. The thicker sludge cake may contain more wet zones at the cathode. Thus, an increase in sludge loading rate resulted in the reduced water content in the final sludge cake. Besides, electrical gradient plays an important role in reducing water content in the final sludge cake during the electro-dewatering process [14,19,20]. Due to the sludge electro-dewatering with different sludge loading rates, the distance between the anode and the cathode increased especially when higher sludge loading rates were employed. Because of the applied constant voltage, the lower sludge loading rate resulted in

the higher electrical gradient, hence higher DS content in the sludge cake was observed (Fig. 3c).

During electro-dewatering process using freeze/thaw sludge samples, water was transported from the anode to the cathode by electro-osmotic flow. Therefore, the water content in sludge cake at the anode was found to be significantly lower than in the sludge at the cathode (Fig. 3b and d). Indeed, it was similar to the observation made earlier while investigating electro-dewatering of anaerobically digested sludge [22].

3.3. Effect of natural freezing on electro-dewatering efficiency

The average temperature from January to March, 2008 is shown in Fig. 4. According to the report from the Finnish Meteorological Institute, the period from December 2007 to February 2008 was the warmest in 100 years [35]. The average temperatures were over 0°C for many days, which allowed sludge to be subjected to multi-freeze/thaw cycles and a long curing time. The advantages of multi-freeze/thaw cycles are the significant reduction in E. coli activation [36] and increase in the permeability coefficients of soil and sludge slurries [37]. Curing time was also reported to be beneficial for the sludge dewaterability [27,28]. Due to natural freezing, the average CST of sludge after 1, 2, and 3 months was 69, 54, and 38 s, respectively, which indicated that multi-freeze/thaw cycles and also a long curing time might be facilitated an increase of the sludge dewaterability. However, when natural sludge samples frozen at various durations were used for experiments, the DS content in the final sludge cake was the same (Fig. 5). In comparison with experiments using sludge frozen at different temperatures, the water content in the final sludge cake (Fig. 3a) did not show any significant difference. Also, it was found that electro-dewatering process



Fig. 4. Average temperatures from January to March in 2006 and 2008.

using 1 month naturally freeze/thaw conditioned sludge lasted 150 min in comparison to 100 min using 2 and 3 months naturally freeze/thaw conditioned sludge under the same experimental conditions. Thus, electro-dewatering using naturally freeze/thaw conditioned sludge could significantly reduce the dewatering time.

3.4. Effect of polyelectrolyte conditioning on electro-dewatering efficiency

An increase in the polymer dosage resulted in a decrease of CST and the improvement of sludge dewaterability. Indeed, the CST of polymer conditioned sludge samples at around 5, 10, 15, and 20 kg/ton DS were 39.7, 12.4, 7.9, and 11.6 s, respectively, when the CST of the original sludge was 439 s. It was similar observations which were made by several authors [38-40]. In experiments using sludge samples with 5, 10, and 15 kg/ton DS of added polymers, electro-dewatering lasted for 10.0, 4.0, and 2.5 h, respectively. This shows that an increase in polymer dose considerably reduces the dewatering time. Moreover, the increase in polymer dose from 5 to 15 kg/ton DS, not only reduced the dewatering time but also significantly decreased water content in the final sludge cake in both, electro-dewatering and blank experiments (Fig. 6a). After 10 min, the amount of water removed at both sides in experiments using sludge samples with 0, 5, 10, and 15 kg/ton DS of added polymers were 2.3, 10.9, 45.8, and 57.2%, respectively. It was assumed that during the initial stage of electro-dewatering process, electroosmosis and electrophoresis did not significantly contribute to the removal of water. The filtrate flow rate at the anode and the cathode



Fig. 5. DS content in the final sludge cake, sludge by the anode and the cathode due to electro-dewatering using natural freeze/thaw conditioned sludge samples.

may be expressed by the following equation (1) [41,42]:

$$Q_p = \frac{dV_p}{dt} = \frac{\Delta p_H A}{\eta (r_c L + R_m)} \tag{1}$$

where Q_p is the pressure-driven filtrate flow rate, V_p is the volume of filtrate, Δp_H is the hydraulic filtration pressure, A is the filter surface area, η is the viscosity of the liquid medium, r_c is the filter cake resistance, L is the cake thickness, R_m is the filter medium resistance. During the process, Δp_H and A were constant, because of the same experimental conditions with applied constant pressure. Due to the short time of experiments (10 min), the filter medium resistance (R_m) was assumed constant. During the dewatering process using original or polymer conditioned (5 kg/ton DS) sludge, the filter cake resistance (r_c) is high due to the applied pressure and the sludge cake thickness (L) subsequently increases, hence, the filter flow rate (Q_p) rapidly reduces, resulting in a small amount of removed water at beginning of experiments. Therefore, in experiments using the original sludge samples, sludge cake was not formed, resulting in the high water content in the final sludge cake (Fig. 6a). On the contrary, in experiments using polymer conditioned sludge (10 and 15 kg/ton DS of added polymers), the viscosity of the liquid medium may have increased and the filter cake resistance decreased. Because of that the filtrate flow rates did not rapidly decrease or even remained constant at the beginning of the experiment due to an increase in the sludge cake thickness, which consequently resulted in the higher initial water removal (Fig. 2d). Moreover, the sludge cake was formed over the shorter period of time in comparison to the blank experiment.

An increase in polymer dosage from 15 to 20 kg/ton DS and further did not result in the reduction of sludge dewaterability, but the increase in zeta potential as also observed by Lee and Liu [38,43]. During electro-dewatering process, it was found that water content in the final sludge cake in experiments using sludge with 20 kg/ton DS of polymer loading, was higher compared to 15 kg/ton DS sludge. Also, it was found that the water content in sludge by the anode was higher than at the cathode (Fig. 6b).

CST of the sludge samples with 5 and 10 kg/ton DS of polymer addition was 39.3 and 13.0 s, respectively. This was much lower in comparison to the CST of sludge during freeze/thaw at different temperatures. However, due to electro-dewatering, the experiments using polymer added sludge at 5 and 10 kg/ton DS lasted 7 and 1.5 h more, respectively, than the experiments using freeze/thaw conditioned sludge. The DS content in the final sludge cake (Fig. 6a) was also lower than in the same experiments using freeze/thaw conditioned sludge (Fig. 3a).

Fig. 6c shows the DS content in the final sludge after different sludge loading rates with 15 kg/ton DS of added polymer. In comparison to the DS content of the final sludge cake in the same experiments using freeze/thaw conditioned sludge (Fig. 3c), water content in the final sludge cake was similar using the same sludge loading rates. An increase in the sludge loading rate resulted in the reduction of water content in the final sludge cake (Fig. 6c). The potential gradient at the end of experiments with sludge loading rates of about 3, 5, 10, 15, and 20 kg DS/m² was 63.4, 36.4, 18.8, 9.5, and 7.1 V/cm, respectively. An increase in the sludge loading rates significantly reduced water content in the final sludge cake and this could be related to the decrease in electrical gradient, which has already been discussed earlier. However, different sludge loading rates using polymer conditioned sludge resulted in the increase in the DS content of the final sludge cake in comparison to blank experiments and were more pronounced than those during the same experiments using freeze/thaw conditioned sludge. During electro-dewatering process, electro-osmosis removes interstitial and some of the vicinal water [44]. Therefore, the same water content was found in the final sludge cake in experiments regardless conditioning method. Conversely, freeze/thaw conditioning also



Fig. 6. DS content in the final sludge cake, sludge by the anode and the cathode due to electro-dewatering using different polymer conditioned sludge (a and b) and a sludge sample with 15 kg/ton DS polymer addition at different sludge loading rate (c and d).

reduces sludge bound water content [26,45]. Thus, water content in the final sludge cake during blank experiments using polymer conditioned sludge (Fig. 6c) was higher than in the experiments using freeze/thaw conditioned sludge (Fig. 3c). Therefore, more significant increase in the DS content in the final sludge cake was observed during experiments using polymer added sludge at different sludge loading rates.

During electro-dewatering process, electro-osmotic flow plays an important role in transporting water from the anode to the cathode, and this flow strongly depends on zeta potential. In some cases throughout the current study, an increase in zeta potential caused by the change in pH at the anode, which induced the reverse electro-osmotic flow, resulting in the higher water content at the cathode [22,46]. An increase in zeta potential with increasing polymer dosage was observed by Lee and Liu [38,43]. Indeed, zeta potential value of polymer conditioned sludge sample at 20 kg/ton was -9.6 mV in comparison to -15.2 mV of original sludge. In the current study, the DS content in sludge by the anode and the cathode was significantly different due to different sludge loading rates and using freeze/thaw conditioning (Fig. 3d). Moreover, the distinction between DS in sludge by the anode and the cathode using polymer conditioned sludge was insignificant (Fig. 6d). The DS content in sludge by the anode was lower than the cathode in experiment with sludge loading rate at 20 kg DS/m² and electrodewatering using polyelectrolyte conditioned sludge at 20 kg/ton DS. Fig. 7 shows percentage of water removed from the cathode during electrode-dewatering using freeze/thaw conditioned sludge $(-20 \,^{\circ}\text{C})$ and the sludge samples with 15 and 20 kg/ton DS of polyelectrolyte addition. In experiments using freeze/thaw sludge after 40 min of the process, all the removed water was entirely from the cathodic side. In contrast, during electro-dewatering treatment using sludge samples with the same polyelectrolyte loading, there



Fig. 7. Percentage of water removal from the cathode as a function of time during electro-dewatering process using sludge samples with 15 and 20 kg/ton DS polymer addition and -20 °C freeze/thaw conditioned sludge.

was water removed from the anodic side as well throughout the whole experiment.

4. Conclusion

In general, polyelectrolyte and freeze/thaw conditioning of sludge significantly reduced dewatering time and increased the DS content in the final sludge cake during electro-dewatering process. Smooth electro-dewatering occurred in all experiments using freeze/thaw treated sludge samples, but, the smooth electro-dewatering only happened in experiment using polymer conditioned sludge at optimum dosage.

A decrease in freezing temperature resulted in the reduced sludge dewaterability, however, natural freeze/thaw conditioning increased the sludge dewaterability. Due to electro-dewatering using freeze/thaw sludge, electro-osmosis transports water from the anode to the cathode, resulting in a much dryer sludge by the anode than the cathode.

During electro-dewatering process, an increase in sludge loading rates related to the decrease in the DS content in the final sludge cake in experiments using polymer and freeze/thaw conditioned sludge. However, the DS content in the final sludge cake by the anode and the cathode was similar or slightly higher at the cathode than at the anode in experiments using polymer conditioned sludge. This indicates that polymer conditioning had a negative effect on the electro-osmotic flow.

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