

# Effects of sludge pretreatment on sludge reduction in a lab-scale anaerobic/anoxic/oxic system treating domestic wastewater

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**Abstract** Excess sludge disposal is one of the serious challenges in biological wastewater treatment. Reduction of sludge production would be an ideal way to solve sludge-associated problems rather than the post-treatment of the sludge produced. In this study, a new wastewater treatment process combining anaerobic/anoxic/oxic system with thermochemical sludge pretreatment was tested in a laboratory scale experiment. In this study, the effects of the sludge pretreatment on the excess sludge production in anaerobic/anoxic/oxic were investigated. The system was operated in two Runs (1 and 2). In Run 1, the system was operated as a reference and in Run 2, a part of the mixed liquid was pretreated thermochemically and was returned to the bioreactor. The average solubilization efficiency of pretreated sludge was found to be about 35 % during the study period of 220 days. Sludge production rate in Run 2 was less than that in Run 1 by about 52 %. Total phosphorous was removed by enhanced biological phosphorous removal with the removal efficiency of 83–87 % and

81–83 % for Run 1 and Run 2, respectively. Total nitrogen removal in Run 2 (79–82 %) was slightly higher than that in Run 1 (68–75 %). The mixed liquor suspended solids/mixed liquor volatile suspended solids ratio was identical after both runs in the range 78–83 %. The effluent water qualities were not significantly affected when operated with thermochemical pretreatment at pH 11 and 60 °C for 3 h during 7 months. From the present study it is concluded that thermochemical sludge pretreatment of anaerobic/anoxic/oxic process plays an important role in reduction of sludge production.

**Keywords** Anaerobic/anoxic/oxic reactor · Biological nutrient removal · Sludge reduction · Thermochemical pretreatment

## Introduction

Wastewater containing excessive nitrogen (N) and phosphorous (P) may lead to serious environmental problems such as eutrophication when discharged into receiving waters (Yeoman et al. 1988; Lee et al. 2003). So, it is necessary to reduce the concentration of these nutrients before discharging to prevent the algal bloom. The most commonly used process is a single-sludge suspended growth system incorporating anaerobic/anoxic/oxic (AAO) stages in sequence (Ma et al. 2005). During operation, a considerable volume of sludge should be withdrawn in order to maintain appropriate level of biomass concentration in the system (Oh et al. 2007). Treatment and disposal of excess sludge accounts for about 50–60 % of the total operational cost of wastewater treatment plants (Davis and Hall 1997; Spellman 1997). The conventional disposal methods such as landfill may cause secondary pollution

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problems and are strictly regulated in many countries (Liu and Tay 2001). Hence, excess sludge reduction has been found to be one of the major challenges in this field. Several methods to pretreat waste sludge were employed to accelerate the solubilization of sludge, such as thermal pretreatment (Pinnekamp 1989; Climent et al. 2007), chemical pretreatment (Rajan et al. 1989; Ray et al. 1990; Carballa et al. 2006; Rajesh et al. 2012); and thermochemical pretreatment (Stuckey and McCarty 1984; Tanaka et al. 1997).

Among these disintegration techniques, thermochemical hydrolysis using sodium hydroxide was found to be the most efficient in inducing cell lysis (Novelli et al. 1995). When compared with other pretreatment methods, alkaline treatment was more efficient because of a simple device, convenience of operation, and high efficiency (Navia et al. 2002; Kim et al. 2003; Neyens et al. 2004; Carballa et al. 2006). The alkaline treatment destroys floc structures and cell walls by hydroxyl anions. Due to extremely high pH, the protein loses its natural shape, saponification of lipids takes place, and hydrolysis of ribonucleic acid (RNA) takes place. Chemical degradation and ionization of the hydroxyl groups lead to extensive swelling and subsequent solubilization of gels in sludge (Neyens et al. 2004). After the destruction of extracellular polymer substances (EPS), the cell walls, being exposed to a high pH, which cannot withstand the appropriate turgor pressure, leads to disruption of cells and release of intracellular substances (Erdinciler and Vesilind 2000). Thermal pretreatment was studied using a wide range of temperatures ranging from 60 to 270 °C. Temperatures over 200 °C have been found responsible for refractory compound formation (Muller 2000). The most common treatment temperatures are between 60 and 180 °C. Treatments applied at temperatures below 100 °C are considered as low temperature thermal treatments (Gavala et al. 2003).

To improve the efficiency of sludge reduction, researchers used different pretreatment methods such as ozonation (Yan et al. 2009), microwave (Eskicioglu et al. 2008) and ultrasonication (Li et al. 2009; Salsabil et al. 2009). According to previous reports, combinations of different pretreatments that accelerated the sludge reduction process were as follows: Bernal-Martinez et al. (2005) reported sludge reduction using the combination of ozonation and anaerobic digestion where ozonation of anaerobically digested sludge improved the PAH (polyaromatic hydrocarbon) removal rate (61 %). An additional enhancement (up to 81 %) of the PAH removal rate was obtained by the addition of hydrogen peroxide during ozonation. Dogan and Sanin (2009) used a combined pretreatment method of alkaline solubilization (using NaOH) and a relatively new technology of microwave (MW) irradiation (160 °C) for sludge reduction. For combined pretreatments, they

achieved soluble COD to total COD ratio (SCOD/TCOD) of waste activated sludge (WAS) increased from 0.005 (control) to 0.18, 0.27, 0.34 and 0.37 for combined methods of MW and pH 10, 11, 12 and 12.5, respectively.

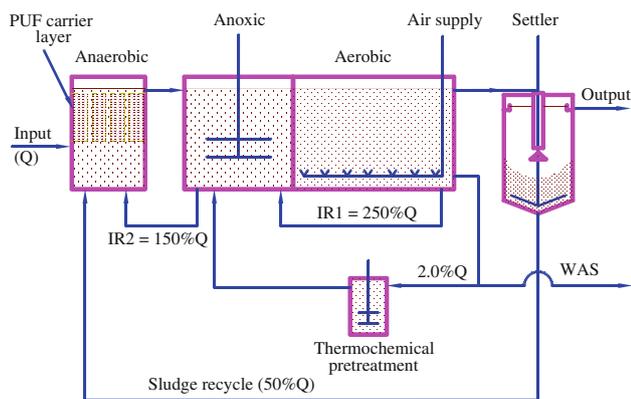
Integration of sludge pretreatment with aerobic biological treatment system is one of the interesting approaches in controlling excess sludge production. Pretreatment enhances the biodegradability of the sludge and its subsequent aerobic biodegradation demands more energy. However, integration of these two technologies reduces considerable energy savings as biodegradation occur at free of cost (Young et al. 2007) and improvement in nitrogen removal efficiency by the soluble organics in the pretreated sludge (Uan et al. 2009). Rocher et al. (2001) reported that it is possible for partial integration of sludge alkaline heat treatment loop in the conventional activated sludge processes. Sludge alkaline heat treatment allows the sludge production yield to be decreased significantly due to the biomass solubilization by the alkaline heat treatment and the cryptic growth occurring in the bioreactor (Rocher et al. 2001). Introduction of pretreated sludge into other wastewater treatment processes such as the AAO reactor may also be an interesting approach.

The objectives of this study were to investigate the effect of thermochemical sludge pretreatment on the excess sludge reduction and wastewater treatment performances in a lab-scale AAO reactor. The temperature used in the study was 60 °C for 3 h at pH 11 for thermochemical pretreatment of sludge.

## Materials and methods

### Experimental setup

The experimental system contained AAO basins with working volumes of 1.5, 3.75, and 4.75 l, respectively (Fig. 1). The hydraulic retention time (HRT) of AAO basin was 1.6, 4.2 and 5.3 h, respectively. In the anaerobic basin, 200 polyurethane foam (PUF) cubes (1 cm × 1 cm × 1 cm) were used as carrier material. These floating carrier materials were kept inside the anaerobic basin by using a screen having ten holes (diameter of 1 cm) to facilitate the transfer of mixed liquid to the anoxic basin. In order to facilitate nitrogen removal, the reactor was provided with the internal recycle (IR) between the aerobic and anoxic basins (IR1 = 250 %  $Q$ ; with  $Q = 21.6 \text{ l d}^{-1}$ ). The second internal recycle (IR2) between the anaerobic and anoxic basins was maintained at 150 %  $Q$ . The low speed mixers were placed in the anaerobic and anoxic basins to keep the mixed liquor suspended solids (MLSS) in suspension. The dissolved oxygen (DO) of aerobic basin was



**Fig. 1** Schematic diagram of AAO coupled with sludge pretreatment system

maintained in the range  $2.0\text{--}4.0\text{ mg l}^{-1}$ . The systems were operated for about 225 days of which 105 days were for system stability (Run 1). At Run 1, the solids retention time (SRT) of the system was maintained in the range  $6.8\text{--}8.7$  days. Sludge pretreatment was introduced into the anoxic basin (Run 2) on the 106th day. Introduction of pretreatment (Run 2) resulted in the increase of SRT to 13.5 days as solid concentrations in sludge wastage decrease considerably.

Synthetic domestic wastewater was used for the study. It was basically composed of a mixed carbon source, macronutrients (N and P), an alkalinity control ( $\text{NaHCO}_3$ ) and a microelement solution (Rajesh et al. 2008). The composition contained ( $\text{l}^{-1}$ ) macronutrient with 420 mg glucose, 200 mg  $\text{NH}_4\text{Cl}$ , 220 mg  $\text{NaHCO}_3$ , 22–34 mg  $\text{KH}_2\text{PO}_4$ , microelement solution (0.19 mg  $\text{MnCl}_2\cdot 4\text{H}_2\text{O}$ , 0.0018 mg  $\text{ZnCl}_2\cdot 2\text{H}_2\text{O}$ , 0.022 mg  $\text{CuCl}_2\cdot 2\text{H}_2\text{O}$ , 5.6 mg  $\text{MgSO}_4\cdot 7\text{H}_2\text{O}$ , 0.88 mg  $\text{FeCl}_3\cdot 6\text{H}_2\text{O}$ , 1.3 mg  $\text{CaCl}_2\cdot 2\text{H}_2\text{O}$ ). The synthetic wastewater was prepared three times a week with concentrations of COD of  $420 \pm 5\text{ mg l}^{-1}$ , TN of  $40 \pm 1\text{ mg l}^{-1}$  and TP of  $5.0 \pm 0.1\text{ mg l}^{-1}$  with pH 6–7.

The sludge from aerobic basin was withdrawn at a flow rate of  $2\% Q$ . The pH of the sludge was 6.8. The withdrawn sludge was taken in 5 l batch reactor and its pH was adjusted to 11 using 1 N sodium hydroxide. The choice of this alkaline agent was made from different studies, which indicated that sodium hydroxide was more efficient than other alkaline agents in solubilising the sludge (Kim et al. 2003 and Lin et al. 2007).

After pH adjustment, the batch reactor was submersed in a thermostatic bath at  $60\text{ }^\circ\text{C}$  for 3 h. During thermochemical pretreatment, the reactor was covered with an aluminum foil, to avoid water evaporation. The sludge in the reactor was kept in suspension by a slow speed stirrer (Digital Overhead IKA RW 20), to ensure temperature homogeneity.

## Analytical methods

Samples for analysis of soluble constituents were filtered through a  $0.45\text{ }\mu\text{m}$  membrane filter (GD/X PVDF, Whatman). MLSS, mixed liquor volatile suspended solids (MLVSS), sludge volume index (SVI), chemical oxygen demand (COD), total phosphorous (TP), total nitrogen (TN) and nitrate ( $\text{NO}_3^-$ ) were then determined on the filtrate obtained in accordance with standard methods (APHA 2005). The ammonia concentrations were measured using an ion-selective electrode (Thermo Orion, Model 95-12, Japan). pH and DO of the samples were measured with Horiba pH/DO meter (Model D-55E, Japan).

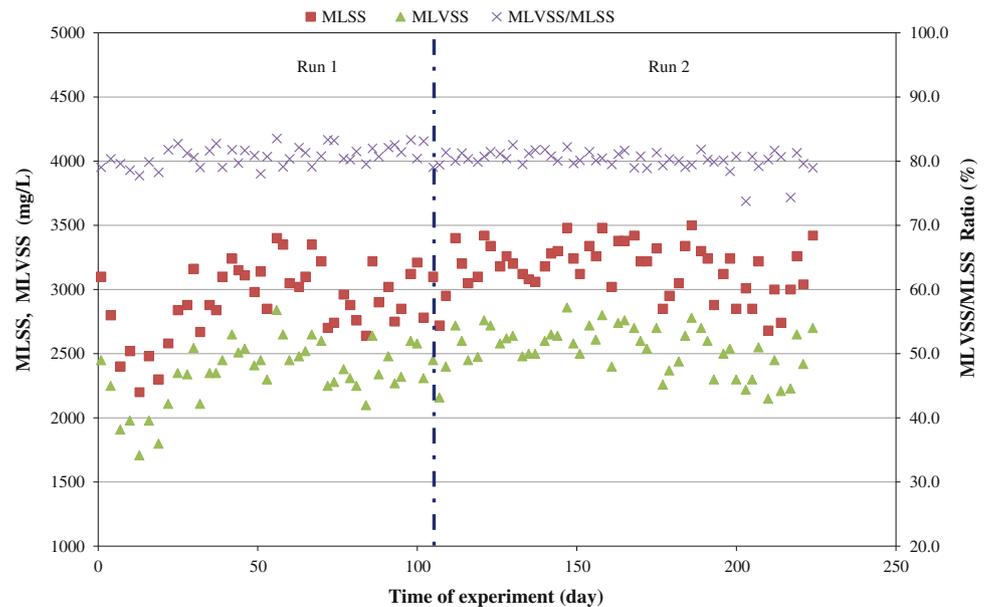
## Results and discussion

### Sludge disintegration and sludge reduction

The reactor was seeded with activated sludge taken from a pilot plant AAO reactor in Kehwang, South Korea. The MLSS concentrations in the system were maintained in the range of  $2,500\text{--}3,500\text{ mg l}^{-1}$  throughout the study. This was achieved by withdrawing  $0.6\text{--}0.7\text{ l d}^{-1}$  of excess sludge during Run 1, whereas during Run 2, it was reduced to  $0.2\text{--}0.3\text{ l d}^{-1}$ , since sludge was reduced by thermochemical pretreatment (Fig. 2). The concentration of organics in mixed liquor was estimated in the form of MLVSS, and its corresponding concentration during the study period was in the range  $2,100\text{--}2,800\text{ mg l}^{-1}$ . The sludge produced in the system during the stable operation period of Run 1 was expressed in terms of the observed yields ( $Y_{\text{obs}}$ ). The  $Y_{\text{obs}}$  was calculated by taking cumulative average of biomass produced divided by cumulative average of substrate consumed during stable operational period. The  $Y_{\text{obs}}$  for Run 1 was found to be  $0.27\text{ g MLSS/g COD}$ . In the present study the ( $Y_{\text{obs}}$ ) value was observed to be within the range  $0.24\text{--}0.4$ , which is the typical ( $Y_{\text{obs}}$ ) range for activated sludge treatment plants (Bolzonella et al. 2005). During Run 2, a part of the mixed liquor was withdrawn at the rate of  $2.0\% Q$  (about  $0.44\text{ l d}^{-1}$ ) from the aerobic basin of AAO system and was pretreated by simultaneous thermochemical pretreatment at  $60\text{ }^\circ\text{C}$  and pH 11. It is understandable that, an increase in pretreatment  $Q$  of over  $2\%$  increases the percentage of sludge reduction. However, it is reported that an increase in the pretreatment of  $Q$  of over  $2\%$  is not an economically viable option (Yan et al. 2009). Consequently, a pretreatment of  $Q$  at  $2\%$  was maintained in the present study. Sodium hydroxide (NaOH) was used to raise pH of the mixed liquor. The pH range of 11 was chosen mainly to maintain the pH level inside the aerobic basin near neutral. For example while working on sludge reduction using thermochemical treatment at pH 12,



**Fig. 2** Variation of MLSS, MLVSS and VSS/SS ratio in the system



Yeom et al. (2005) experienced difficulty in maintaining the pH of an aerobic basin, where the pretreated sludge was recycled for subsequent biodegradation. It is very important to maintain the pH of the reactor near neutral range, which otherwise will affect the performance of the system. Hence it was decided to fix the pH of thermochemical pretreatment to 11. Time was fixed at 3 h and it is based on the experimental work by Rajesh et al. (2012) showing that increase in time over 3 h did not significantly increase the efficiency of sludge pretreatment. Various temperatures, ranging from 60 to 270 °C have been studied in literature for the thermochemical pretreatment of waste activated sludge. The usage of high temperature often resulted in the production of recalcitrant soluble organics compound and hinders its subsequent biodegradability (Wilson and Novak 2009). In addition to the above, cost-benefit analysis of thermochemical pretreatment by Uma et al. (2012) showed that the temperature 60 °C was considered to be an optimum.

During the thermochemical pretreatment, the alkali reacted with the cell walls in several ways, including the saponification of lipids in the cell walls, which led to solubilization of membrane. Disruption of sludge cells led to leakage of intracellular material out of the cell (Neyens et al. 2003). The soluble COD (SCOD) after pretreatment is presented in Fig. 3. SCOD released in the supernatant was found to be in the range 1,100–1,450 mg l<sup>-1</sup>. Müller (2001) reported that the solubilization efficiency can be used as an index for the efficiency of sludge disintegration. In the present study, sludge solubilization efficiency ( $\alpha$ ) was calculated as the ratio of the soluble COD (SCOD) increase by thermochemical pretreatment to the total COD (TCOD) of the sludge before pretreatment as follows:

$$\alpha = \frac{\text{SCOD} - \text{SCOD}_0}{\text{TCOD} - \text{SCOD}_0}$$

where SCOD<sub>0</sub> is the soluble COD of untreated sludge sample.

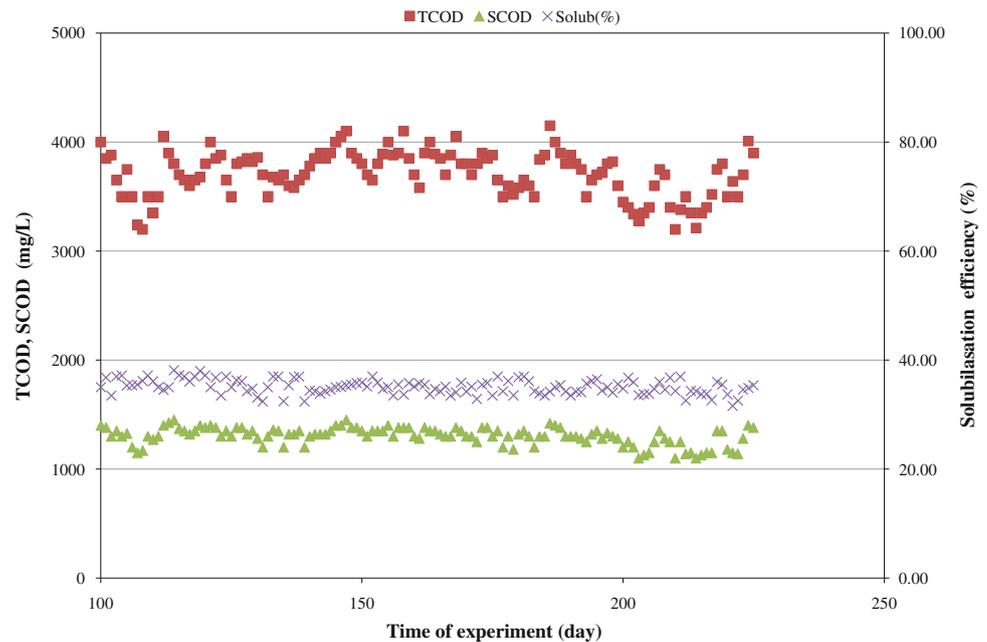
The observed values for the sludge disintegration experiments are also given in Fig. 3. The average solubilization efficiency for thermochemical treatment was found around 0.35, which could be comparable with the values obtained from the thermal-alkali by others (Penaud et al. 1999; Neyens et al. 2003). Sludge reduction in a treatment system is achieved by combining sludge pretreatment along with subsequent biodegradation. In the present study, in order to facilitate the biodegradation of the pretreated sludge, it was recycled back into the AAO system, where subsequent biodegradation takes place. As a result of pretreated sludge recycling and its degradation, the daily sludge wastage was found to decrease from 0.59–0.70 l d<sup>-1</sup> to 0.20–0.30 l d<sup>-1</sup>. At Run 2, 52 % of sludge reduction was achieved with an observed yield of 0.13 g g<sup>-1</sup> (gram of MLSS produced per gram of COD removed). The observed excess sludge reduction of 52 % in this study was comparable with the 42 % (Rajesh et al. 2011a) and also higher than the data (30 and 37 %) reported for activated treatment processes (Penaud et al. 1999; Rocher et al. 1999, 2001) and 60 % (Rajesh et al. 2011b) reported for the aerobic treatment systems integrated with sludge pretreatment.

#### Sludge characteristics

The MLVSS/MLSS ratio plays an important role in governing the treatment efficiency of an aerobic treatment



**Fig. 3** Soluble COD and solubilization efficiency during sludge thermochemical digestion



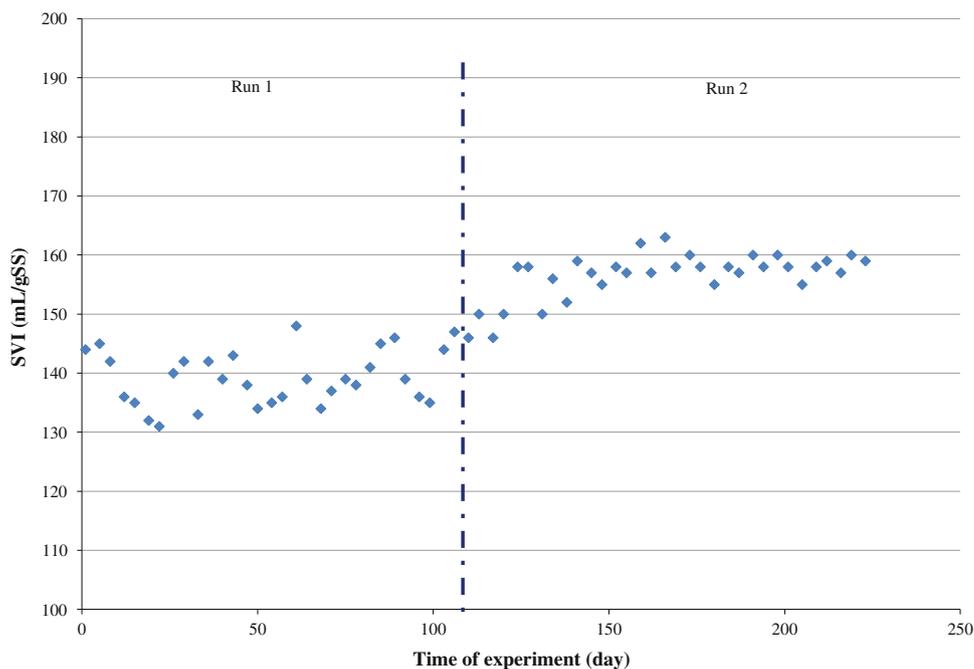
system. Any drastic decrease in this ratio affects the performance of the reactor (Rajesh et al. 2011a). One interesting observation of the present study is that the MLVSS/MLSS ratios of two Runs were almost identical. The MLVSS/MLSS ratio was in the range 78–84 % in Run 1 and 79–82 % in Run 2 (Fig. 2). Previous work on sludge reduction with chemical pretreatment in aerobic treatment system by Young et al. (2007) reported no change in volatile fraction of the mixed liquor before and after the pretreatment. The results from the present study reveal that inorganics from the disintegrated cells do not accumulate in the reactor as insoluble inorganic particulates. The inorganic particulates might be taken out of the system by wasted sludge line or discharged along with the effluent. Similar to the present study, working on sludge reduction in AAO system treating domestic wastewater, Yan et al. (2009) and Rajesh et al. (2011a) have reported that there was no change in the MLVSS/MLSS ratio. Sludge volume index (SVI) of the mixed liquor plays an important role in settling of sludge (Metcalf and Eddy 2003). Poor settling of mixed liquor in clarifier leads to the decrease in treatment efficiency of a system (Metcalf and Eddy 2003). The monovalent cation like sodium often interferes with the settling characteristics of the sludge (Higgins and Novak 1997; Katja and Mika 2007). Due to this reason it was decided to compare the SVI of mixed liquor in Run 1 and Run 2 to verify whether sodium has any profound effect on SVI. The results revealed that there was no significant difference between the sludge volume indices in two Runs throughout the experimental operation (Fig. 4). It appears that the thermochemical sludge pretreatment using sodium hydroxide does not increase the SVI of the mixed liquor

which presumably is a poor settling sludge. This may be due to the thermal effect, which on combination with alkali was found to improve the SVI of mixed liquor. From the present study, it is evident that simultaneous sludge pretreatment not only helps to improve the solubilization of sludge but also improves sludge quality.

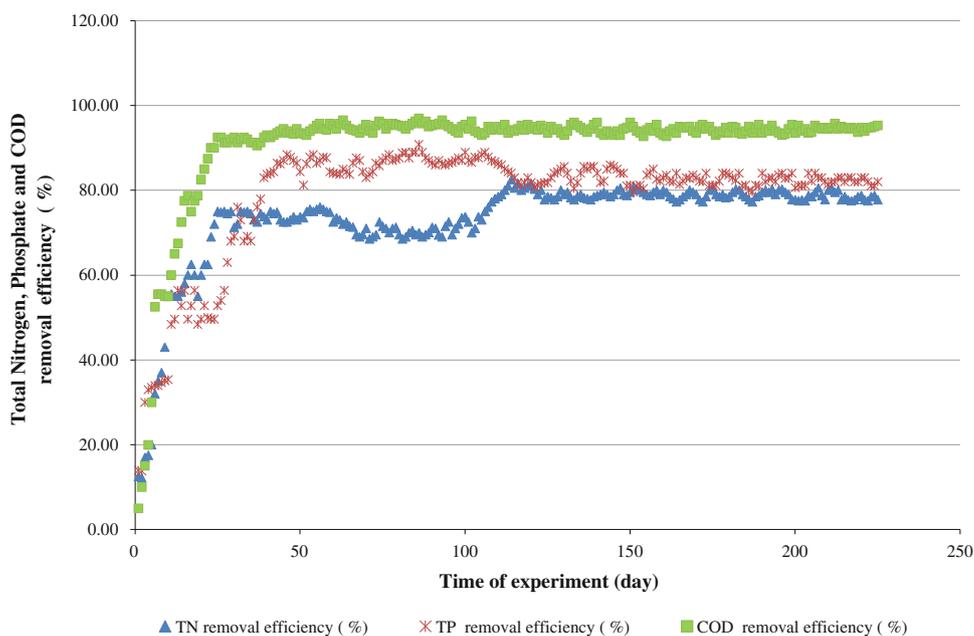
#### Effluent quality and performance of AAO

Conventionally, biological nutrient removal was carried out in the AAO reactor; the success of its design depends upon its nitrogen and phosphorous removal efficiency from the wastewater (Shammas and Wang 2010). In the AAO system, nitrogen removal was carried out by recycling the mixed liquid containing nitrate into the anoxic basin through the internal recycle line. In anoxic basin, nitrate was reduced into nitrogen gas and removed from the system. The TN removal efficiency of the system is shown in Fig. 5. During Run 1, TN removal was in the range 68–75 % and the corresponding TN concentration in the effluent was in the range 11–15 mg l<sup>-1</sup>, whereas in Run 2, TN removal efficiency was found to be 79–82 % with effluent TN of 9–12 mg l<sup>-1</sup>. This slight increase in TN removal efficiency during Run 2 may be due to the external carbon source from pretreated sludge. This additional carbon may help to improve the denitrification by heterotrophic denitrifier. The C/N ratio of the pretreated sludge solution was found to be in the range 11.5–13.5 (data not shown), which was significantly higher than the influent C/N ratio (10.5). The introduction of the pretreated sludge into the system increased the influent C/N ratio 1–3.5. In other words, the addition of the external carbon source

**Fig. 4** Variation of SVI in the system



**Fig. 5** Variation of TN, TP, COD and its removal efficiency in the system



from pretreated sludge helped the denitrification process and subsequently improved the TN removal efficiency of the system.

The effluent TP concentrations during the operation are shown in Fig. 5. The effluent TP values varied between 0.5 and 0.8 mg l<sup>-1</sup> in Run 1 and 0.7 and 0.9 mg l<sup>-1</sup> in Run 2. The present study recorded high phosphorous removal efficiency when compared to other similar systems used to treat domestic wastewater (Rajesh et al. 2008; Yu and Zhou 2010). The improvement of phosphorous removal is

likely due to the use of UCT-like recirculation which is beneficial for PAO organisms and subsequent phosphorous removal. Phosphorous removal efficiency of the system was found to be in the range 88–90 % and 80–84 % for Run 1 and Run 2, respectively. In Run 2, the solubilised phosphorous when returned into the system slightly increases phosphorous concentration in the effluent stream. Figure 5 shows the COD concentrations in the influent and the effluent of the system. The COD concentrations of the effluents were found to be in the range 12–25 mg l<sup>-1</sup> with

removal efficiency of 94 % during Run 1. In Run 2, the excess sludge was disintegrated and returned to the system as feed organics. Based on previous reports, increase in OLR was due to the return of pretreated sludge, which caused decrease in COD removal performance (Yasui and Shibata 1994; Yasui et al. 1996). However, the results from this study indicated that the presence of sludge disintegration period did not have significant impacts on the effluent COD values.

## Conclusion

Reduction of sludge production in a lab-scale AAO combined sludge pretreatment appears to be an alternative solution for sludge disposal. The present study showed that association of thermochemical sludge pretreatment along with the AAO system proved its high efficiency and reliability. A high reduction of sludge production (52 %) can be obtained when a part of the waste activated sludge (2 % of the influent flow rate) is disintegrated through thermochemical treatment at pH 11 and 60 °C for 3 h. An average solubilization efficiency of 35 % could be achieved. It appears that the solubilized fraction of the mixed liquor obtained by the thermochemical sludge pretreatment might be easily biodegraded by other microorganisms. No significant accumulation of inorganic substances was observed. It was found that introduction of pretreated sludge into the anoxic tank had no significant effect on nutrient removal.

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