Effect of low temperature thermochemical pretreatment on sludge reduction potential of membrane bioreactor treating primary treated dairy wastewater
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ABSTRACT
In this present study, an aerobic submerged membrane bioreactor (MBR) was used to study the effect of thermochemical pretreatment on the efficiency of sludge reduction. For this purpose, two MBRs were fabricated. Between the two MBRs, one acted as a control reactor (CMBR) and the other acted as an experimental reactor (EMBR). The MBRs were operated with a mixed liquor suspended solids (MLSS) concentration in the range of 6,800–7,200 mg/L for a period of 160 days. In the EMBR, part of the MLSS was withdrawn at a ratio of 1% of Q and was pretreated by low temperature thermochemical treatment. The sludge pretreatment was carried out at 60°C and an alkali dosage in the range of 0.49 to 0.56 mg NaOH/mg MLSS. During the pretreatment, 42% of COD solubilization and 22% of SS reduction were observed. The pretreated sludge was returned to the reactor for further degradation where it was found to be 42% reduced. During the 160 days of reactor operation, both of the MBRs maintained a relatively constant transmembrane pressure. The sludge digestion does not have any impact on the COD removal efficiency of the reactor.

Key words | COD removal, membrane bioreactor, sludge pretreatment, sludge reduction, TMP

INTRODUCTION
Dairy industries release large quantities of wastewater, often in the order of thousands of cubic meters/day (Banu et al. 2008). Generally, the primary treatment of dairy wastewater is carried out anaerobically because of its high organic concentrations (Hawakes et al. 1995; Vidal et al. 2000; Banu et al. 2006). It is known from the literature that the primary treated high strength wastewater requires further treatment before being discharged into the environment (Banu et al. 2009). The activated sludge process is the most widely used biological wastewater treatment for industrial plants in the world (Metcalf & Eddy 2003). As a biological technology, the activated sludge system has been most widely employed throughout the world to treat secondary treated dairy wastewater. Most of the industrial treatment system uses the activated sludge system, or a modification of it, as the core part of the treatment process. A considerable volume of sludge is generated during the operation of activated sludge, part of which should be withdrawn and disposed of in order to maintain the appropriate level of biomass concentration in the aeration basin (Van et al. 2009). The treatment and disposal of excess sludge accounts for about 50–60% of the total cost of wastewater treatment (Davis & Hall 1997). Conventional disposal methods such as landfill or ocean dumping may cause secondary pollution problems and are strictly regulated in many countries (Liu & Tay 2001). Therefore, sludge reduction is very important for economical treatment.

The membrane bioreactor (MBR) has the advantages of complete solids removal from the effluent, effluent disinfection, high loading rate capability, low/zero sludge production and rapid start-up among others, but also has a
more compact size and lower energy consumption. A combination of sludge disintegration techniques and advanced wastewater treatment processes such as the MBR may also be an interesting approach. The MBR process has been known as a process with a relatively high decay rate and less sludge production because of significantly longer biomass retention in the reactor (Visvanathan et al. 2000). A number of processes including thermal energy (Li & Noike 1992), ozonation (Yasui & Shibata 1997), high pressure (Dollerer & Wilderer 1993), mechanical disintegration (Kopp et al. 1997), and ultrasound (Tiehm et al. 1997) have been investigated for decomposition and pretreatment of waste sludge. Compared with other methods, thermochemical treatment has several advantages: simple manufacturing of device, easy to operate and high efficiency (Young et al. 2007). Most of the investigations exhibit an increase of soluble chemical oxygen demand (SCOD) or a decrease of volatile suspended solid (VSS) during alkaline treatment or during a combination with thermal treatment (50–200 °C) (Neyens et al. 2003; Vlyssides & Karlis 2004). A combination of alkaline treatment and temperature is investigated in this study. The aim of this study is to investigate the effect of the thermochemical sludge pretreatment on controlling the excess sludge production in the MBR. The performances of the thermochemical pretreatment on the MBR system including membrane fouling and effluent quality were evaluated and compared with a reference system under the same conditions.

**MATERIALS AND METHODS**

**Wastewater**

The dairy wastewater treated by the hybrid upflow anaerobic sludge blanket (HUASB) reactor at an organic loading rate of 12 kg CODm⁻³/day was used as a feed for the present study. The characteristics of the wastewater are given in Table 1.

**MBR operation**

Schematic diagrams of the MBRs are shown in Figure 1. Among the two MBRs, one is designated as a CMBR, which acts as a control and the other is designated as an EMBR, where sludge reduction was carried out. The working volume of both the MBRs was 13 L. The wastewater was fed into the reactor at a flow rate of 36 L/d (Q) using a feed pump. A liquid level sensor, planted in an aerobic basin of the MBR, controlled the flow of influent. The HRT (hydraulic residence time) of the MBR was 8 h. The dissolved oxygen (DO) concentration in the aerobic basin was maintained at 3.5 mg/L and was monitored continuously through a DO meter. The solid–liquid separation occurs in the aerobic basin with the help of a membrane. A flat sheet type membrane with a pore size of 0.22 μm was used for solid–liquid separation and the characteristics of the membrane used are given in Table 2. A suction pump connected the membrane in which provision was made to measure the transmembrane pressure (TMP) during suction. The suction pump was operated in a sequence of timing which consists of 10 min switch on and 2 min switch off. For both MBRs, the MLSS (mixed liquor suspended solids) concentration was maintained at around 7,000 mg/L.

**Sludge pretreatment**

The present study uses the combination of thermal and alkaline sludge pretreatment. The mixed-liquid from the aerobic basin of the EMBR was withdrawn at a flow rate of 1% of the inflow (Q) and was subjected to the thermochemical pretreatment. The pretreatment experiments were carried out in a batch reactor containing samples submerged in a thermostatic water bath at 60 °C and pH 12. The reactor was covered with an aluminum foil, to avoid water evaporation. The sample in the reactor was kept in suspension by an overhead stirrer to ensure temperature homogeneity. The alkali sodium hydroxide (NaOH) was used to raise the pH of the mixed liquor. The choice of

### Table 1 | Characteristics of the influent wastewater

<table>
<thead>
<tr>
<th>Unit</th>
<th>pH</th>
<th>COD mg/L</th>
<th>sCOD mg/L</th>
<th>Alkalinity mg/L</th>
<th>TP mg/L</th>
<th>TN mg/L</th>
<th>TSS mg/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>Max</td>
<td>7.5</td>
<td>360</td>
<td>230</td>
<td>1,600</td>
<td>14</td>
<td>28</td>
<td>65</td>
</tr>
<tr>
<td>Min</td>
<td>7.2</td>
<td>250</td>
<td>180</td>
<td>1,300</td>
<td>8</td>
<td>18</td>
<td>45</td>
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<tr>
<td>Mean</td>
<td>325</td>
<td>195</td>
<td>1,450</td>
<td>11</td>
<td>22</td>
<td>52</td>
<td></td>
</tr>
</tbody>
</table>
this alkaline agent was made by reference to different studies indicating that NaOH was more efficient than other alkaline agents in solubilizing the sludge (Kim et al. 2003; Lin et al. 2007).

**Analysis methods**

Samples for analysis of soluble constituents were centrifuged at 5,000 rpm for 5 min, and then the supernatant was filtered through a membrane with a mesh size of 0.45 μm (GD/X PVDF, Whatman). Suspended solids (SS), VSS and chemical oxygen demand (COD) were determined in accordance with Standard Methods for the Examination of Water and Wastewater (APHA et al. 2005). pH was measured using a pH meter (Horiba Navi®, Model F-54).

**RESULTS**

**MBR operation**

Figure 2 presents data on the MLSS profile of both the MBRs. One of the advantages of the MBR over a conventional reactor is the fact that it can be operated with a
high MLSS concentration. The MBRs were seeded with return sludge from the activated sludge treatment plant, located at Kerala, India. The reactor was initiated with a MLSS concentration of 6,500 mg/L. The concentration starts to increase steadily with an increase in the period of the reactor operation and reached a value of 7,400 mg/L on day 13. Subsequently, the MLSS concentration was maintained in the range of 6,800–7,200 mg/L by wasting the excess activated sludge produced. In the present study, sludge reduction experiments were carried out at a fixed MLSS range of 6,800–7,200 mg/L. It is evident that sludge reduction can be increased by working the MBR at relatively high MLSS concentrations. At high MLSS concentrations, the sludge yield is lower and the designed pretreatment Q is not available. Sludge reduction at high MLSS concentrations is an optimum range for the performance of the MBR (Banu et al. 2011).

From Figure 2 it is clear that the volatile fraction of the mixed liquor solids in the CMBR and the EMBR remain unaffected by thermochemical pretreatment and are almost identical throughout the study. The MLSS/MLVSS ratio was found to be in the range of 75–80% for both the MBRs. From the results it is evident that most of the inorganic fraction of the degraded cells did not accumulate in the reactor and presumably permeated through the membrane as ionic species. Similar to our study, while working on sludge recycling in an aerobic MBR reactor treating domestic wastewater, Young et al. (2007) have reported that there is no change in the volatile fraction of the mixed liquor before and after the sludge pretreatment. In contrast to our study, Yasui & Shibata (1994) have reported a decrease in MLSS/MLVSS ratio by 10% for MBR treating pharmaceutical wastewater over a period of 9 months. This may be due to the characteristics of wastewater and not due to the sludge pretreatment process as the pharmaceutical wastewater contains a lot of inorganic substances. In that case, the extent of inorganic accumulation may not depend upon the inorganic substances released from the disintegrated cells. The observation in this study strongly indicates most of the inorganic substances from the disintegrated cells do not accumulate in the reactor as particles.

**Efficiency of thermochemical pretreatment**

Once the systems became stable, a part of the mixed liquor was withdrawn at 1% of Q from the EMBR and was treated by the thermochemical technique under the conditions 60°C, pH 12 for 24 h. It is understandable that an increase in pretreatment Q of over 1% increases the percentage of sludge reduction. However, it is reported that an increase in the pretreatment Q of over 1.5% is not an economically viable option (Uan et al. 2009). Consequently, a pretreatment of Q at 1% was maintained in the present study. The primary objective of all pretreatment techniques, regardless of the method, is the destruction of sludge flocs and the rupturing of the cell walls to release intracellular components so that they are accessible for subsequent biodegradation (Elliott & Mahmood 2007). Among the various pretreatment techniques used for controlling the excess sludge production in wastewater treatment systems, hybrid techniques such as thermochemical and alkali ozone were found to be more effective (Yeom et al. 2005). The usage of NaOH as an agent for sludge pretreatment has a serious effect on sludge management techniques (Banu et al. 2012). However, the negative influence of sodium over sludge dewaterability is reduced when NaOH is combined with other methods such as microwave and thermal (Uma et al. 2011). The conditions used in the present study for low temperature thermochemical pretreatment are based on the experimental results of Uma et al. (2011) for the pretreatment of dairy waste activated sludge. The temperature level used in the present study was a little lower than the value suggested by other researchers (Uan et al. 2009; Banu et al. 2011), and the pH level for the alkali treatment was maintained at about 12.

From Figure 3 it is clear that an alkali dosage in the range of 1.49–1.56 mg NaOH/mg MLSS was required to achieve an average COD solubilization efficiency of 42%. This dosage was equivalent to 3.2–3.5 g/L NaOH, whereas researchers working on sludge pretreatment using alkali NaOH alone required an alkali dosage of around 7 g/L to achieve 40% sludge solubilization (Kim et al. 2005). The combination of thermal and alkali sludge pretreatment could reduce this considerable alkali usage. This will help by significantly decreasing the total treatment cost. Another advantage of thermochemical combination is that the added alkali serves as a neutralizing agent to buffer the pH drop...
due to the sludge solubilization. The other sludge disintegration techniques such as acid ozone and thermal treatment produce solubilized sludge with acidic pH (Kim et al. 2003), requiring further buffering before it is reintroduced for the successive treatment. The pH of the solubilized sludge was found to be in the range of 10–10.5. It is interesting to note that this high pH of pretreated sludge, when it is reintroduced back to the reactor for subsequent biodegradation, does not increase the pH level of the EMBR over 7.5. This could be due to the dilution of the pretreated sludge by the influent of the EMBR, which resulted in the neutralization of pH.

The effect of the thermochemical pretreatment on the sludge disintegration is depicted in Figure 4. During sludge disintegration the intracellular compounds were released and are measured indirectly by estimating the amount of SCOD released. With thermo-alkali treatment, SCOD increases in the range of 3,100–3,600 mg/L were observed.

The efficiency of the sludge pretreatment was measured in terms of the COD solubilization efficiency and was calculated by the following Equation (1):

$$\alpha = \frac{(\text{SCOD}_p - \text{SCOD}_i)}{\text{TCOD}_i - \text{SCOD}_i}$$  \hspace{1cm} (1)

where, $\alpha$ = solubilization efficiency (%), $\text{SCOD}_p$ = SCOD concentration of the sludge after disintegration (mg/L), $\text{SCOD}_i$ = SCOD concentration of the sludge before disintegration (mg/L), $\text{TCOD}_i$ = TCOD concentration of the sludge before disintegration (mg/L).

The average solubilization efficiency by the thermochemical treatment was around 38–42% which could be comparable with the values obtained from the thermal-alkali pretreatment by others (Penaud et al. 1999; Neyens et al. 2005). During pretreatment, part of the SS is disintegrated and the effect of the thermochemical pretreatment on the sludge reduction (Uan et al. 2009) is plotted in Figure 4. From the figure it is evident that pretreatment reduces SS of the sludge from its initial average concentration of 7,100 mg/L to its final average concentration of 5,900 mg/L. The corresponding SS reduction during the treatment was found to be in the range of 18–22%.

**Effect of thermochemical pretreatment on sludge reduction**

The observed yields ($Y_{\text{obs}}$) for experiments without sludge pretreatment (CMBR) and with sludge pretreatment (EMBR) are presented in Figure 5. The sludge production rate was calculated based on Equation (2) and is presented in Figure 5.

$$Y_{\text{obs}} = \frac{(\text{Xo} - \text{Xe}) + \Delta X}{\text{So} - \text{Se}}$$  \hspace{1cm} (2)

where, $\text{Xo}$ = influent suspended solids (g/L), $\text{Xe}$ = effluent suspended solids (g/L), $\Delta X$ = net solids produced in MBR (g/L), $\text{So}$ = influent COD (g/L), $\text{Se}$ = effluent COD (g/L).
Both of the MBRs were operated over a period of 160 days. For the first 40 days both the MBRs were operated under the same working conditions and the corresponding period is referred to as Run 1. The average $Y_{\text{obs}}$ for Run 1 was found to be 0.24 g MLSS/g COD for both the reactors. The presently observed $Y_{\text{obs}}$ value was comparatively lower than a value of 0.4 g MLSS/g COD reported for the conventional activated sludge processes (Metcalf & Eddy 2005). From the 41st day onwards, sludge pretreatment was initiated in the EMBR, lasting for 160 days, and the corresponding period is referred to as Run 2. In Run 2, the average $Y_{\text{obs}}$ value for the EMBR and the CMBR was found to be 0.24 g MLSS/g COD and 0.38 g MLSS/g COD, respectively. On comparing the $Y_{\text{obs}}$ value of the EMBR with the CMBR, it is clear that sludge pretreatment plays an important role in the excess sludge reduction. Excess sludge removal is a regular phenomenon in the aerobic treatment system and was done to maintain the biomass balance inside the reactor (Oh et al. 2007). From Figure 5 it is evident that there was a considerable decrease in excess sludge waste of the EMBR compared with the CMBR. For example, in case of the EMBR the daily average sludge wastage was 0.84 g/day and for the CMBR it was found to be 1.4 g/day.

Mass balancing the SSs production in both the MBRs can give a clear picture of the role of sludge pretreatment in controlling excess sludge production. This mass balance equation takes into account influent SS, quantity of SS in wasted sludge, the accumulated SS in the MBR and SS lost with effluent. However, it is known that the quantity of sludge passed in the membrane permeation is zero and the accumulation of SS in the MBR could be negligible.

$$DSP_{\text{day}} = Q_{\text{WAS}} \times MLSS_{\text{WAS}} + \text{EMBR} \frac{\Delta MLSS}{\Delta t} + Q_{\text{eff}} \times MLSS_{\text{eff}}(g/L) \quad (3)$$

$$DSP_{\text{accumulated}} = \sum_{\text{day}=1}^{N} DSP_{\text{day}}(g) \quad (4)$$

The cumulative SS production for both MBRs was calculated by following Equations (3) and (4) and is presented in Figure 6. The cumulative sludge production for the EMBR and the CMBR for the stable operational period (day 40 to 160) is found to be 177 g for the CMBR and 103 g for the EMBR.

The extent of SS reduction in the EMBR can be calculated by following Equation (5) and it accounts for 74 g. From the findings, it is evident that the proposed method...
of sludge disintegration is capable of controlling nearly 42% of excess sludge production.

\[
SS_{\text{red}}^{\text{EMBR}} = DSP_{\text{cumulative}}^{\text{CMBR}} - DSP_{\text{cumulative}}^{\text{EMBR}}(g) \tag{5}
\]

The 42% SS reduction achieved in EMBR depends on two important factors: sludge pretreatment and biodegradation. During thermochemical pretreatment, there is a considerable reduction in SS and it can be calculated by Equations (6) and (7). From the equations it was found that thermochemical pretreatment of sludge was responsible for 49 g of the SS reduction in the EMBR.

\[
SSR_{\text{day}} = Q_{\text{pretreated}} \times (MLSS_{\text{Int}} - MLSS_{\text{Fin}})(g) \tag{6}
\]

\[
SSR_{\text{cumulative}} = \sum_{\text{day}=1}^{1} SSR_{\text{day}}(g) \tag{7}
\]

The biodegradation of pretreated sludge happens free of cost in the EMBR when it is recycled back into the reactor. The amount of the SS biodegraded was found to be 25 g and was calculated by using Equations (8) and (9).

\[
SSD_{\text{day}} = (DSP_{\text{day}}^{\text{CMBR}} - DSP_{\text{day}}^{\text{EMBR}})
\quad - SSR_{\text{day}}^{\text{EMBR}}(g) \tag{8}
\]

\[
SSD_{\text{cumulative}} = \sum_{\text{day}=1}^{1} SSD_{\text{day}}(g) \tag{9}
\]

From the above, it is evident that SS reduction was greater for thermochemical pretreatment compared with biodegradation. Pretreatment made the sludge amenable to biodegradation and it occurs without any additional input of energy, special equipment or cost. Hence it can be concluded that the synergistic effect of these two factors plays an important role in controlling excess sludge production in MBRs and they are accountable for 66 and 34%, respectively, of the total SS reduction in the present study.

### Effect of pretreatment on effluent quality and performances of MBR

Figure 7 shows the variation in COD removal efficiency of both the MBRs during the study period. From the figure it can be seen that the COD removal efficiency of the EMBR remains unaffected before and after the introduction of sludge reduction practices. A t-test analysis showed that the differences between the EMBR and the CMBR are not statistically significant. However, it has been reported that, in wastewater treatment processes including disintegration-induced sludge degradation, the effluent water quality deteriorated slightly as a result of the release of non-degradable substances such as soluble microbial products (Yoon 2003; Yoon & Kim 2004). The COD removal increased with an increase in time during the initial phases of the reactor operation. It attains a steady state on day 11. From then onwards, the COD removal was in the range of 96–98% (calculated from the graph). During the stable operational period, the SCOD concentration in the MBRs was found to be 22 to 40 mg/L for the EMBR and 24 to 45 mg/L for the CMBR. The corresponding organic concentration in the effluent varied from 8 to 22 mg/L for EMBR and 12 to 26 mg/L for the CMBR. From this, it can be concluded that the membrane separation played an important role in providing an excellent and stable effluent quality.

The suction pump was started after the next day of seeding and was based on the SCOD of the aerobic basin. The

![Figure 7](https://iwaponline.com/wqrj/article-pdf/46/4/312/379822/312.pdf)
pump was started when the SCOD in the aerobic basin was 45 mg/L. The designed flux for the membrane was 17 L/m²/h. This was achieved by a stepwise increase of flux from 25 to 100% over a period of 8 days. Figure 8 shows the TMP variation during the operational period, indicating that the TMP increased slowly over a period of 160 days. At the end of 160 days of reactor operation, the TMP was found to be ~6 cmHg. Both MBR systems were operated without membrane cleaning for 160 days. It appears that the sludge disintegration system does not cause membrane fouling. Similar to our study, while working on sludge reduction practices in MBR, Yoon & Kim (2004) reported that the pretreatment of sludge did not cause membrane fouling.

CONCLUSION

The minimization of sludge production appears to be suitable as an alternative solution to the problem of sludge disposal. The association of low temperature thermochemical sludge pretreatment with the MBR system has proved its efficiency and reliability. A good reduction of sludge production (42%) can be obtained when a part of the biological solids were disintegrated with thermo-alkali at pH 12 and 60 °C for 24 h. Using the thermo-alkali treatment, the solubilization efficiency of 42% on average could be obtained. It appears that the solubilized fraction of the mixed liquor obtained by the thermochemical sludge disintegration might be easily biodegraded by other microorganisms. No significant accumulation of inorganic substances was observed. The recycling of pretreated sludge in the EMBR did not cause any significant increase in TMP. A COD removal efficiency of up to 97% for both MBRs was achieved. The excess sludge production in the MBR was constrained by the combined pretreatment method without any deterioration in the treated water quality and membrane performances.

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