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Bioelectrochemical cell (BeCC) integrated with granular activated carbon (GAC) in treating spent caustic wastewater: Effects of mixed liquor suspended solid (MLSS)

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Abstract. The study present the feasibility of a bioelectrochemical cell (BeCC) integrated with Granular Activated Carbon (GAC) as the bacterial attachment medium in treating spent caustic wastewater. BeCC is a bioelectrochemical reactor that uses activated sludge for substrate degradation while also capable in energy recovery. Unlike the general MFC configuration, the BeCC reactor is cost effective as it was operated without a proton exchange membrane (PEM). Instead, a baffle is used to reduce the oxygen transfer to the other side of the reactor and the employment of the baffle has divide the reactor into hybrid of anoxic and aerobic conditions. Also, instead of using packed GAC, the BeCC was integrated with 10 g of suspended GAC in order to increase the surface area available for bacteria to attach. The study investigated the best operating MLSS for the system to treat spent caustic wastewater whereby the BeCC was tested at various MLSS of range within 2500 mg/L to 4000 mg/L and its performance in terms of Chemical Oxygen Demand (COD) and sulfide removal as well as it open circuit voltage (OCV) were evaluated throughout 30 days of operation. From the study, the highest COD removal of the system was 95.6% achieved at MLSS of 3500 mg/L whereas the highest sulfide removal was 87.1% achieved at MLSS of 3000 mg/L. The highest OCV was 413.7 mV achieved at MLSS of 3000 mg/L.

1. Introduction

In the present study, spent caustic wastewater was treated by using a BeCC reactor. BeCC is a bioelectrochemical reactor that employs microorganisms for pollutants degradation in wastewater while also capable in producing energy. This reactor has the same application with a Microbial Fuel Cell (MFC) reactor, however unlike the general configuration of an MFC reactor, the BeCC reactor in the present study was operated without a proton exchange membrane (PEM). A PEM is required in an MFC system in order to allow the transfer of protons from anode to cathode while blocking the oxygen to pass through from cathode to anode [1]. The common material used as PEM is Nafion membrane which is an expensive material that caused the conventional MFC to be cost ineffective [2]. Therefore, several researches has been conducted to find cheaper material as the substitute to the Nafion [3]. Early research has come out with a porcelain septum made from kaolin as the PEM in MFC and reported that the system could generate a maximum power density of 788 Wm⁻² [4]. A research by Behera et al. (2010) has constructed MFC with an earthen pot as the medium for proton exchange and the research has found that the system could achieved a maximum power output of 16.8 Wm⁻³ [3]. Recent study by Gao et al. (2018) has adopted Quartz sand chamber as PEM and reported that the system achieved 228 mWm⁻³ higher power density than the system with PEM [5].



However, the employment of PEM in MFC is unnecessary as studies had also found that a PEMless MFC is also feasible in treating wastewater and generating electricity. In fact, Liu and Logan. (2004) has reported that MFCs without PEM could perform better than MFCs with PEM as there was increment in the open circuit potential when PEM was removed and the condition has attributed to higher power density [6]. Another research by Lee et al. (2015) also tested MFC performance with and without PEM has reported that both power production and COD removal were higher in MFC without PEM and the condition was explained to be due to the absence of PEM has favored the growth of the electrogens [7]. Despite the PEM selective permeability to protons, researches has demonstrated that PEM could also adversely affected the MFC performance [7] as a PEM has its own internal resistance that causes the power output to be decreased [8, 9]. Therefore, in this study, a novel configuration of a BeCC reactor without a PEM and with an employment of a baffle as a separator was constructed to serve as the cost effective alternative for spent caustic wastewater treatment and energy recovery.

In order to improve both of the wastewater treatment and energy recovery efficiency of the BeCC, the BeCC was also integrated with granular activated carbon as the bacteria attachment medium. GAC has been commonly integrated in MFC, however, most of the MFC-GAC integrations were focusing on the GAC application as the adsorptive and electrode material in MFC [10, 11]. Besides that, GAC was also commonly integrated in a packed bed form but the present study employed GAC in suspended form to allow higher GAC surface area available for the biomass to attach. The presence of the attached bacteria in GAC was expected to enhance the pollutants degradation and energy recovery of the BeCC as the attached cell was reported to have higher rates of DNA synthetic than the bacteria in suspension. A research that was conducted to evaluate the comparative growth and physiology of bacteria attached to GAC particles has found that when an organism attached to GAC, the growth rate was enhanced more than 10 times due to the availability of the substrate that was adsorbed at the GAC surface [12].

The wastewater treatment capacity of the BeCC reactor was evaluated based on the COD and sulfide removal in spent caustic wastewater. Spent caustic wastewater is the hazardous industrial wastewater that has high content of harmful contaminants such as sulfide and mercaptans. Due to its noxious properties, this type of wastewater is difficult to be treated, handled and disposed [13]. Most often it is reported that conventional treatment method of spent caustic wastewater such as chemical oxidation and wet-air oxidation produce incomplete COD degradation, expensive and involved a considerable safety measures [14, 15]. Thus, in the present study, BeC as one of the highly potential method to produce effective treatment of spent caustic wastewater. Although biological method in treating spent caustic might be limited by the high alkalinity, salinity and toxicity of the spent caustic wastewater, this method is still reported to be feasible with spent caustic wastewater pretreatments, biomass acclimation and proper sludge handling [13, 16].

As mentioned, GAC was integrated as the medium for the bacteria to attach. The presence of the attached bacteria would significantly influence the MLSS concentration of the BeCC. Thus, it is also crucial to determine the best operating MLSS concentration for the BeCC integrated with GAC system. Previous researches which deal with activated sludge in treating wastewater often reported that high MLSS would improve the removal performance as the high biomass content tends to increase the biodegradable process [17-19]. However, it is also demonstrated that the increment in the MLSS beyond its ideal value could adversely affected the MFC removal and energy recovery performance [20, 21]. Previous literatures has demonstrated MLSS as an important parameter affecting the performance of a bioreactor which reflected that the BeCC integrated with GAC need to be operated at its ideal MLSS concentration for efficient removal and energy recovery performance. Therefore, the present study aims to investigate the best operating MLSS for the BeCC integrated with GAC in treating spent caustic wastewater whereby the system was operated at different MLSS concentration and its performance was evaluated in terms of COD and sulfide removal as well as its OCV.

2. Materials and Methods

2.1. Wastewater preparation

For the present study, the wastewater was obtained from a petrochemical industry located in East Coast, Malaysia. The wastewater obtained was pretreated and neutralized before being fed into the reactor reducing its COD, sulfide concentration and pH to 700 mg/L, 85 μ g/L and 7 respectively. The spent caustic wastewater pretreatment was conducted in order to create influent that has suitable characteristics for biological treatment. Table 1 shows the wastewater characteristics of the prepared wastewater.

Table 1. Characteristics of spent caustic wastewater

Parameter	Values
COD (mg/L)	700
BOD (mg/L)	70
Sulfide (mg/L)	0.085
Sulphate (mg/L)	66
Phosphate (mg/L)	3.8
Nitrate (mg/L)	14.6

2.2. Acclimatization

In the present study, the BeCC was inoculated with activated sludge that was also obtained from the petrochemical industry located in East Coast, Malaysia. The sludge was acclimatized with the prepared wastewater and was fed with glucose. The sludge was acclimatized until the biomass was able to remove 80% of nutrients and biomass MLVSS/MLSS ratio achieved higher than 60% indicating the sludge ability to survive in the new environment.

2.3. BeCC operation

Figure 1 shows the laboratory scale set up for the BeCC operation and the bioreactor was operated at ambient conditions. The reactor was made up from a polycarbonate material and has the dimensions of 26.5 cm length, 14.5 cm height and 12 cm width. The reactor design was based on a membrane-less single chamber MFC operated with both anode and cathode submerged into the reactor. As it is reported that a membrane-less MFC would possessed high oxygen diffusion into anode which could reduce the efficiency of the fuel cell [6], a baffle was inserted into the reactor as the separator to reduce the oxygen transfer to the anode side of the reactor. The employment of the baffle has allowed the reactor to operate in hybrid condition of anoxic and aerobic whereby the oxygen in the anoxic (anode side) was controlled to be below than 2 mg/L. There was a 0.2 cm gap between the baffle and the floor of the reactor to allow for protons transfer from the anodic to the cathodic side. It is reported that the employment of baffle as separator could allow fluid mixing in the anode chamber and minimize the oxygen diffusion adjacent to the cathode surface [22]. As shown in the figure, the influent and effluent of the reactor were controlled by using a peristaltic pump and an air pump was used for aeration purpose at the cathodic side. Graphite rods of 150 mm length and 3 mm diameter were used as anode and cathode electrodes. A multimeter was used to measure the OCV generated by the BeCC. A magnetic stirrer was used at the anodic side to ensure no sludge sediment at the bottom of the reactor. As for the GAC integration with the BeCC, 10 g of GAC particle size 6 \times 12 mesh was let to be suspended throughout the reactor.

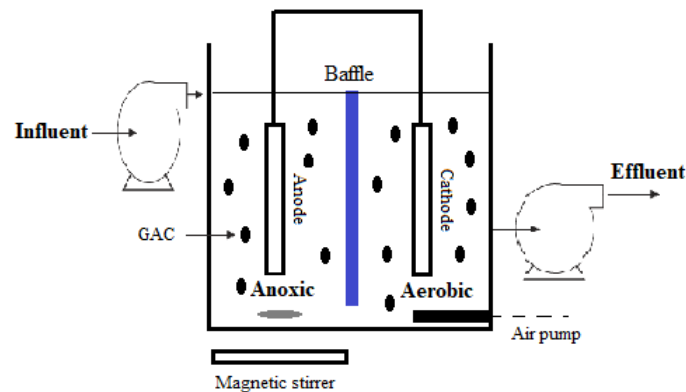


Figure 1. BeCC set up

3. Result and Discussions

3.1. The Effects of MLSS concentration on COD and Sulfide Removal Efficiency

The BeCC performance in treating spent caustic wastewater and its energy recovery was investigated at different MLSS concentration of 2500 mg/L, 3000 mg/L, 3500 mg/L and 4000 mg/L. It is crucial to determine the suitable working range of MLSS concentration for the BeCC integrated with GAC operation for efficient wastewater treatment and energy recovery. In the present study, the influent COD and sulfide concentration of the spent caustic wastewater entering the BeCC was set to be at 700 mg/L and 85 $\mu\text{g/L}$ respectively and the removal efficiencies are shown in Figure 2.

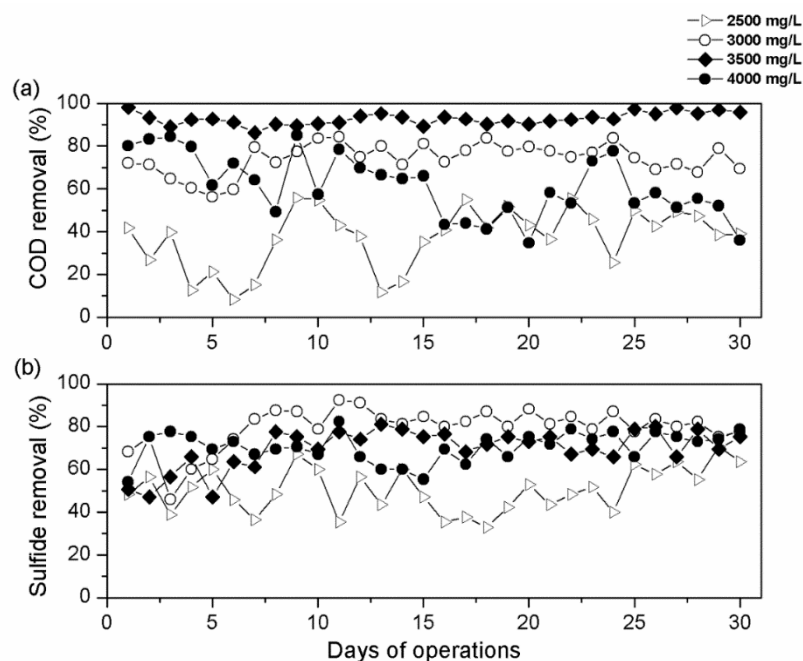


Figure 2. COD and sulfide removal efficiency at different MLSS concentration versus days of operation

Based on Figure 2, it is shown that the highest COD removal of the BeCC was 95.6% achieved at MLSS of 3500 mg/L whereas the highest sulfide removal was 87.1% achieved at MLSS of 3000 mg/L. The highest achievable COD removal efficiency for MLSS concentration of 2500 mg/L, 3000 mg/L and 4000 mg/L were 55%, 84.28% and 84.43% respectively. The overall trend of the COD removal efficiency was increasing with increasing MLSS concentration from 2500 mg/L to 3500mg/L.

However, further increased of the MLSS concentration to 4000 mg/L has caused the removal efficiency to decrease significantly. On the other hand, the highest achievable sulfide removal efficiency for MLSS concentration of 2500 mg/L, 3500 mg/L and 4000 mg/L were 70.58%, 81.17% and 78.82%. Unlike the COD removal trend, the overall sulfide removal efficiency of the BeCC integrated with GAC operation shows that MLSS concentration of 3000 mg/L was readily sufficient for a maximized sulfide removal efficiency in which further increased of the MLSS concentration of beyond than 3000 mg/L has caused the sulfide removal efficiency to be slightly decreased.

The result of the present study in terms of removal efficiency is in aligned with previous studies in which reported that increasing the MLSS concentration would resulted to higher removal efficiency [18, 19]. The condition was often explained in terms of higher substrates utilization at higher MLSS concentration thus higher organic removal efficiency could be achieved [18]. Besides that, Alattabi et al. (2017) also tested aerobic-anoxic sequencing batch reactor operated with synthetic wastewater at various MLSS concentration and reported that increasing MLSS concentration from 2000 mg/L to 3000 mg/L improved the removal efficiency and the optimal MLSS was obtained with 3000 mg/L to 4000 mg/L. The same scenario was agreed to have occurred in the present study, whereby higher MLSS concentration was favorable for an efficient removal efficiency of the BeCC operation. The removal trend of the present study has demonstrated that the BeCC operation integrated with GAC would behave in the same way as other bioreactor system.

From the result, it is shown that high removal efficiency was not achieved at low MLSS of 2500 mg/L. A few bioreactor literatures has reported the same trend and explained the condition in terms of high food to microorganisms (F/M) ratio which indicates excessive nutrients beyond the capabilities of the bacteria amount to fully oxidize the substrates. Due to this, the operation needed longer time to achieved constant organic removal [17]. In the present study, although there was the presence of the attached bacteria in the reactor at low MLSS, it was believed that the attached cells were also dominated by only a few types of bacterial communities which restrained for higher interactions between bacterial populations. It is reported that the electrogenic bacterial population is only capable in giving complete oxidation of simple substrates [23]. Since spent caustic wastewater spent caustic wastewater has stable contents that are not readily biodegradable [24], higher interactions among bacterial population is crucial for complete COD degradation. The result of the present study suggested that low MLSS is not favorable for higher interactions within bacterial population in the BeCC reactor.

Therefore, the BeCC integrated with GAC need to be operated at higher MLSS. As stated in previous literatures, it was agreed that higher MLSS would contribute to higher substrates uptake and degradation as well as higher bacterial communities that encourage for higher interactions among bacterial populations. In the present study, the highest COD removal was 95.6% achieved at MLSS of 3500 mg/L. Effective removal efficiency of spent caustic wastewater by the BeCC biomass at high MLSS. Besides being contributed by the low F/M which can improved the organic removal efficiency [18], the condition was also believed to be due to presence of the attached biomass in the reactor which enhanced the biomass survival despite the noxious properties of the wastewater. As reported by previous study, attached bacteria tend to be highly resistant to chlorination than the suspended one [25]. The same scenario might have occurred in the present study which has facilitate the BeCC biomass to sustain high activity and substrates degradation in spent caustic wastewater.

In terms of sulfide removal efficiency, the same trend was observed whereby the increase in MLSS concentration also increase the sulfide removal. However, unlike the COD removal trend, the result has shown that operating the BeCC at MLSS of 3000 mg/L is readily sufficient for effective sulfide removal. Slight deviation between the highest COD and sulfide removal obtained at different MLSS concentration of 3500 mg/L and 3000 mg/L respectively was agreed to be contributed by a few factors. The first factor was expected to be due to high sulfide oxidizing bacteria presence in the reactor at MLSS of 3000 mg/L. The result has reflected that sulfide- oxidizing bacteria was the dominant population at MLSS of 3000 mg/L in the spent caustic environment which encourage for higher sulfide uptake by microorganisms [26]. It is stated in that in order to create an acceptable spent caustic environment for neutrophilic sulfide-oxidizing bacteria, the pH and sodium level need to be reduced [27]. In the present study, the high sulfide-oxidizing biomass at MLSS of 3000 mg/L in the

BeCC reactor was feasible as spent caustic wastewater was pretreated before being fed into the reactor. In addition with its attachment on the GAC particles, which also enhanced the sulfide-oxidizing bacteria survival in the spent caustic environment. Besides being contributed by the well functioned sulfide-oxidizing bacteria, the high sulfide removal efficiency at MLSS of 3000 mg/L was believed to be contributed by the sulfide's role as the electron donor. Therefore, as reported by many literatures, sulfide does not only served as the substrate for biomass uptake, but it also involved in the redox shuttle between biocatalyst and insoluble electron acceptor [26]. In other words, sulfide was also well utilized for BeCC energy recovery, thus sulfide content of the effluent can be reduced. The condition reflected that the highest sulfide removal efficiency obtained at 3000 mg/L can also be correlated with the highest voltage output that was also achieved at MLSS of 3000 mg/L.

The BeCC integrated with GAC was further tested at MLSS of 4000 mg/L and it is observed that both of the COD and sulfide removal was decreased. However, comparing the COD and sulfide removal, only a slight decrease in sulfide removal of BeCC at MLSS of 4000 mg/L and good sulfide removal efficiency could still be achieved despite the high MLSS concentration. The condition was agreed to be due to the survival of the sulfide –oxidizing bacteria that still remains as the dominant bacteria presence in the reactor at high MLSS. However, in terms of the removal efficiency, the result obtained was associated with the F/M ratio in which further increase of MLSS concentration has caused the F/M ratio to decrease. As reported in previous literatures, too low of F/M ratio would adversely affects the performance of the bioreactor as it would cause to overall population competition for substrates. Alatabi et al. (2017) has stated that decrease in F/M ratio resulted in the reduction of microorganism's activity [20].

3.2. The Effects of MLSS concentration on OCV

The energy recovery efficiency of the BeCC integrated with GAC operation at different MLSS concentration was evaluated in terms of its voltage output in open circuit configuration and the result is shown in Figure 3.

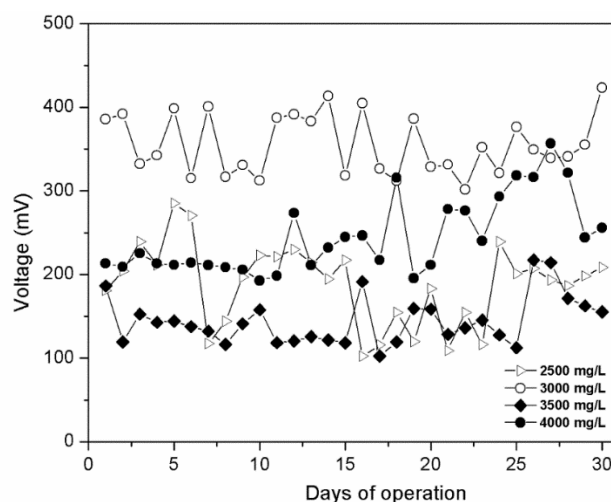


Figure 3. OCV at different MLSS concentration versus days of operation

Based on Figure 3, it is shown that the highest voltage output was 413.7 mV achieved at MLSS of 3000 mg/L whereas the lowest voltage output was 132.8 mV achieved at MLSS of 4000 mg/L. It is observed that the voltage output at MLSS of 2500 mg/L was highly fluctuated and its trend shows that the voltage output was increasing up to 15 days of operation. After day 15 of the operation, the voltage output was rapidly decreased and the voltage output was observed to be increased again from day 16 until the end of the operation duration which is day 30. Low voltage obtained at MLSS of 2500 mg/L was agreed to be due to less amount of microorganisms' presence in the reactor, thus low rates of substrates degradation occurred. It should be noted that as substrates degradation occur, electrons and protons were generated simultaneously which means at MLSS of 2500 mg/L, less electron transfer

rate occurred with respect to its low electrons generation by microorganisms. The condition was in parallel with the low COD degradation by microorganisms at MLSS of 2500 mg/L.

The operation was tested at higher MLSS of 3000 mg/L. At this point, the BeCC voltage was increased. The result is in aligned with previous MFC researches which obtained the same energy recovery trend with MLSS and explained the condition in terms of higher amount of biomass available to generate electrons and protons, thus higher electrons transfer rate could be obtained. As reported by Zinadini et al. (2017), increase in MLSS concentration reduced the F/M ratio which could improve the organic removal efficiency, sludge flocculation with increased in power generation [18]. High OCV obtained at MLSS of 3000 mg/L was also believed to be due to the presence of electrogenic population as the dominant bacterial population in the reactor. Previous study reported that electrogenic population has higher growth rate compared to the competing microorganisms [28]. Thus, it was also agreed that the attached bacteria on the GAC was also dominant by the electrogenic population. Not only the bacterial attachment on the GAC caused their growth to be enhanced, but the sludge flocculation and settleability could also be improved. These properties has the electrogenic population to remain in the reactor while other competing microorganisms with slower growth rate to be washed out from the system. This is as according by Penteado et al. (2016) whom stated that microorganisms with lower growth rate are washed out from the system if the removal rate is higher than the growth rate [29]. The condition explained the capability of the BeCC integrated with GAC to sustain high voltage output at MLSS of 3000 mg/L throughout 30 days of operation. Besides that, in parallel with the high sulfide removal efficiency at MLSS of 3000 mg/L, it was also believed that there was high utilization of sulfide for energy recovery of the BeCC at that point. As mentioned earlier, sulfide also served as the electron donor in a bioelectrochemical system which means the employment of sulfide as substrate could readily contribute in the energy recovery of a bioelectrochemical system. This is in accordance with a finding by Fatemi et al. (2017) which stated that sulfide can act as an electron donor as it possessed high electrochemical oxidation. Therefore, sulfide can also be considered as mediator in electron transfer to electrode and led to power generation [26].

The BeCC integrated with GAC operation was further tested with higher MLSS concentration of 3500 mg/L and 4000 mg/L and the result can be observed in Figure 3. Based on the figure, it is observed that further increase of MLSS concentration has deteriorated the energy recovery performance of the BeCC. From here, the overall trend of the AG-MFC voltage output at different MLSS concentration could be drawn whereby the increment of MLSS concentration from 2500 to 3000 mg/L increased the AG-MFC voltage. However, further increment of MLSS concentration of beyond 3000 mg/L would decreased the energy recovery performance. The overall trend is in aligned with a few MFC researches which reported that an elevated MLSS, the power generation of their respective system was reduced. [18, 21]. The condition was explained in terms of oxygen consumption by the electro-microorganisms. Oxygen served as electron acceptor in the system thus its consumption by the electro-microorganisms was unfavorable as oxygen reduction would be reduced resulting in low energy recovery performance of the bioreactor [21]. Basically, oxygen served as the electron acceptors in the bioelectrochemical system, thus higher oxygen consumption by microorganisms at high MLSS concentration is unfavorable as the amount of oxygen that is available as the electron acceptor was reduced, in turns reducing the energy recovery performance as well.

4. Conclusion

The study demonstrated the capacity of BeCC integrated with GAC i.e. 10 g of suspended GAC to produce effective treatment of spent caustic wastewater and energy recovery. For the study of the best operating MLSS concentration of the BeCC integrated with GAC, highest COD removal was 95.6% achieved at MLSS of 3500 mg/L whereas the highest sulfide removal was 87.1% achieved at MLSS of 3000 mg/L. The highest voltage output was 413.7 mV achieved at MLSS of 3000 mg/L. The overall trend shows that the performance of the BeCC integrated with GAC operation increased as MLSS was increased. Higher MLSS is required for higher substrates degradation and proton and electrons generation by microorganisms. Higher MLSS has as well encourage higher interactions between bacterial populations which is crucial for effective organic removal in spent caustic wastewater. Besides that, it can also be concluded that operating the bioreactor at MLSS of 3000 mg/L is readily

sufficient for effective sulfide removal and energy recovery, and the condition was agreed to be due to be due to well-functioned sulfide oxidizing bacteria and high sulfide utilization in electricity generation. However, further increased of MLSS to 4000 mg/L has deteriorated the BeCC integrated with GAC performance as an excessive MLSS has caused to high population competition which in turns has reduced the BeCC biomass activity.

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