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(17) Complete Treatment of Shochu Processed Wastewater by Thermophilic Oxic Process

高温・好気法を用いた焼酎廃液の完全処理

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ABSTRACT; Thermophilic Oxic Process was applied to treat the "shochu" processed wastewater which contained extremely high Biochemical Oxygen Demand(BOD) and Suspended Solids(SS). The optimum operational conditions such as BOD load, aeration rate, etc. were investigated. It was found that the BOD and Total Organic Carbon(TOC) removal efficiencies were 100% and a minute amount of excess sludge was formed when the BOD load was 6 kg・m^{-3}・d^{-1} and aeration rate was 300ℓ・m^{-3}・min^{-1}. The carbon balance showed that almost all of the input carbon was converted to carbon dioxide(CO_2). The water and thermal balances demonstrated that all of the added water was evaporated;66% and 34% of it were evaporated by the heat of biological reaction and electric heater, respectively.

KEYWORDS : Shochu wastewater, Thermophilic oxic process, Evaporation of water.

1. Introduction

"Shochu" is one of the most popular liquors in Japan. The amount of shochu produced per year is 500,000 kℓ.[1] The same quantity of wastewater which contains high BOD and SS was formed from the distillation process. Three kinds of methods had been applied in order to treat this wastewater. The first method is to utilize it as a fodder of pigs and a fertilizer. The second method is to dispose it to the ocean 50 nautical sea miles far from coasts. However, the first method is relatively expensive because this process includes drying, grinding and packing. The ocean dumping is to be restricted in 1995. The third method is methane fermentation. This process is known to be effective for the treatment of high-strength organic wastewater. Methane fermentation processes such as UASB, fluidized bed, fixed bed, etc. have been developed for the high rate treatment of soluble compounds in the wastewater. Biofilms or granules were applied in order to enhance the biamass in the reactor.[2] However, these new type reactors were not available to treat wastewaters which contain high amounts of SS.[2]

As reported in the previous paper,[3] Thermophilic Oxic Process was successfully applied to treat the red bean precessed wastewater containing high amounts of BOD and SS which were 35,000 and 4,000 mg/l, respectively. In this paper, Thermophilic Oxic Process has been used for the treatment of Shochu wastewater which contains high concentrations of BOD and SS. The objectives of this work were ① to obtain optimum operational conditions, such as BOD load, aeration rate and moisture content, and ② to determine the carbon, water and thermal balances in order to demonstrate that there was a minute amount of excess sludge and no dis-

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charge of drain water in this system.

2. Materials and Methods

2.1 Wastewater

The wastewater used in the experiment was sampled from Kyusyukako shochu plant in Kyusyu, in which the wheat was processed to produce shochu. The sampled wastewater was transported to a laboratory by express mail at 4℃, and kept in a refrigerated room of 4℃.

2.2 Absorbents

Two absorbents, cedar chips and kumazasa charcoals, were used. The size of the absorbents ranged from 2 to 7 mm. The moisture content of raw cedar chips decreased to approximately 20 % by drying them in the sun for approximately 5 days before they were put into the reactor. The structure of the cedar chips was observed by using a Scanning Electron Micro-Scope (SEM) as shown in the previous paper.[3] The kumazasa charcoals were made of kumazasa in Houkedou and charred at the temperature of 300℃. Its structure is similar to that of the cedar chips.

2.3 Apparatus and Air Heating System

The flowchart of the experiment is shown in Fig.1. The reactor was a 20 ℓ cylindrical container (effective volume was 10 ℓ) with a perforated plate at the bottom, surrounded by styrenefoam insulation(860mm by 800 mm by 800 mm). Air was supplied by an air pump through flow meter (ST-10A) and heated to 60℃. The air heater was installed in the center of the reactor and its size was ϕ120(mm)×H200(mm). The medium(10 ℓ) was put into the reactor. The temperature was determined using a thermometer which was inserted into the medium at the center of the reactor. It was monitored by the computer(NEC, PC-KD55K) and recorded by the recorder (Chino, RH720-RNN). The reactor was placed on a scale (Tanita, BEC-800)in order to continuously measure the change of weight.

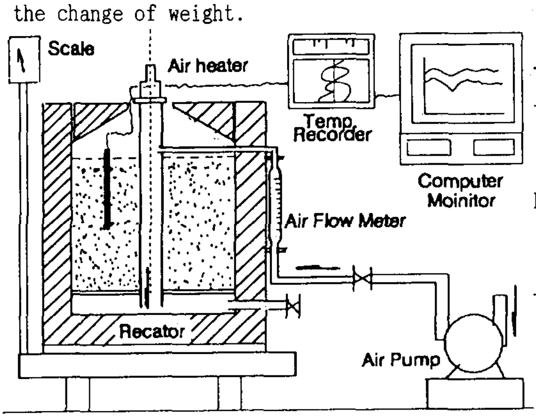

Fig.1 The Flowchart of Thermophilic Oxic Process

Table 1 Experimental Conditions

Run number	1	2	3
Medium(M)	Cedar chips	Cedar chips	Kumazasa charcoal
Ratio of Mixture*	M : W	M : W	M : W
(v/v)	10 :0.8	10 :1〜2	10 :1.2
Temperature**	〜20℃	60℃	60℃

* Ratio of mixture in one cycle except the initial stage. W : shochu wastewater. M : medium

** Temperature of the air which was introduced into the reactor.

2.4 Operation of Process

Three experimental runs were carried out by changing the medium and temperature of the air as shown in Table 1. At the initial time, 2 ℓ of raw wastewater and 10 ℓ of absorbent were completely mixed by hand outside the reactor, then transported into the reactor. Seeding was not necessary because bacteria remain in the reactor which might came from the air. Wastewater was added to the upper part of the medium in the reactor every other day. The quantities of wastewater added depended upon the BOD loads and moisture content. A lid, made of styrofoam insulation materials, was used to seal the reactor and to prevent the heat loss. Then, the air was supplied to the reactor. The lid was taken out for approximately 18 hours from the reactor 30 hours after the addition of the wastewater in order to accelerate the evaporation. Turning of the medium was conducted every 12 hours.

Aeration rates, 100, 200, 300 and 400 $\ell \cdot m^{-3} \cdot min.^{-1}$, were used. The BOD load was gradually increased by increase of the amounts of wastewater added. The same BOD load was used for one week. The temperature and moisture content of the mixture were used as indicators for the active reaction. The temperature of the air was controlled at 60°C by a electrical heater.

2.5 Carbon Balance

The carbon balance was obtained in Run 2 and Run 3 in which the cedar chips or kumazasa charcoals was used as an absorbent, respectively. The charcoal was used because it was not degraded by microorganisms. The samples were taken every 2 hours for 48 hours until the next addition of the wastewater. The evaporated water was collected by introducing off-gas into a trap vessle(1ℓ), and it was sampled from this vessle when the experiment of carbon balance was conducted. The other sampling method was same as that of the previous paper.[3)]

2.6 Analyses of Water Qualities

The following items were analysed : moisture content of mixture, COD, BOD, TOC, T-N and T-P in influent and effluent, CO_2 in the off-gas, temperature in the reactor, weight of mixture in the reactor,calorific value of wastewater, and salinity of wastewater and cedar chips. The calorific value of wastewater was measured by a calorimeter (YOSHIDA SEISAKU-SYO, UENO,TOKYO). The samples for the analysis of COD, BOD and TOC were pretreated as following ways; 50 g of sample was put into a homogenizer cup and dispersed at 10,000 rpm for 10 min. with a homogenizer(Type HM-10S ANIPPON RIKAGAKU KIKAI, CO, LTD). Then this sample was treated by ultrasonic disruptor (Type UR-200P, TOMY SEIKO,CO,LTD) at 6 of output for 5 min.. After the pretreatment, COD and BOD were determined according to JIS-K-0102. The TOC was measured by a TOC analyzer (Shimadzu, TOC-5000). The salinity of the cedar chips was measured by-S-C-T meter(YSI,Model33,U.S.A)by suspending 0.5g of dried and grinded cedar chips into 100 distillated water for 24 hours.

Table 2 Water Quality of Wastewater(n=5)

Items	Concentration
BOD (mg/ℓ)	70,000±450
COD (mg/ℓ)	48,000±4,240
TS (mg/ℓ)	110,000±3,580
VS (mg/ℓ)	105,000±465
SS (mg/ℓ)	103,000±578
TOC (mg/ℓ)	52,000±1,340
T-N (mg/ℓ)	21,00±670
T-P (mg/ℓ)	450±60
Salinity(mg/ℓ)	141±5
Viscosity(MPa·s)	158±14

3. Results and Discussions

3.1 Treatment of Wastewater Without Using Heater

As shown in Table 2, the shochu processed wastewater contained very high concentration of BOD, TOC and SS which were 70,000, 50,000 and

103,000 mg/ℓ ,respectively. The ratio of BOD : T-N : T-P was 100:3:0.6.

When the wastewater was put into the reactor, it was absorbed into the cedar chips. The chips has a relatively high specific surface area and water holding capacity as shown in the Table 3. When the absorbed organic matter was decomposed, the temperature of the mixture increased and water was evaporated due to the high temperature.

Table 3 Characteristics of Media

Media	Diameter of the Pores (μm)	Water Holding Capacity (WHC) (g/g)	Specific surface area ($m^2 \cdot g^{-1}$)
Cedar Chips	40～50	2.53	2.5
Kumazasa Charcoals	30～40	1.30	36

Fig. 2 shows the change of maximum and minimum temperatures and moisture contents without using heater under the condition of BOD load of 3.5kgBOD $\cdot m^{-3} \cdot d^{-1}$ and aeration rate of 100ℓ $\cdot m^{-3} \cdot min^{-1}$. During the initial stage of the experiment(2 days), the temperature increased to higher than 60℃, while moisture content remained lower than 70%. However, with the continuous addition of wastewater,the moisture content increased, and the temperature decreased. After the operation for one week, the moisture content increased from 61% to 70% and the temperature decreased to 50 ℃. Therefore, the BOD load was reduced from 3.5 to 2 kgBOD $\cdot m^{-3} \cdot d^{-1}$. However, the temperature still decreased to lower than 50℃, resulting in the formation of a malodous odor. The condition in the reactor became anaerobic when the moisture content was 72%. Fig.3 shows the change of weight of the mixture and accumulated wastewater added to the reactor and accumulated drain water. The weight of mixture increased by 18.8% because approximately 30% of the added water accumulated in the reactor. The other 40% and 30% of the added water was drained and evaporated, respectively. Since some of the added wastewater accumulated in the absorbent because of its high viscosity, anaerobic condition resulted. It was clarified from these results that the moisture content of the absorbent in the reactor should be lower than 72% to keep an aerobic condition. When the average BOD load was 2.5 kgBOD $\cdot m^{-3} \cdot d^{-1}$ at the aeration rate of 100 ℓ $\cdot m^{-3} \cdot min^{-1}$, aerobic condition was kept for 12 days. During these 12 days, the removal efficiencies of BOD and TOC were approximately 70% and 60%, respectively. It is, therefore, concluded that moisture content in the reactor must be reduced by draining or electrical heating.

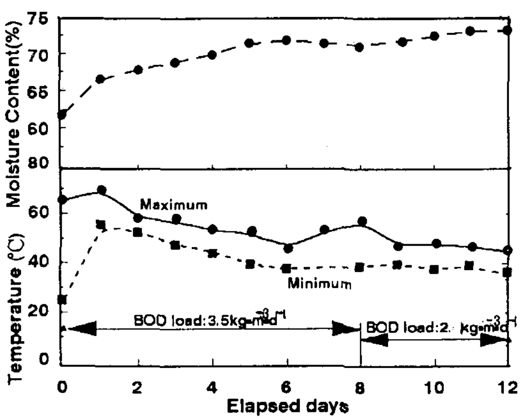

Fig. 2 Changes in Temperature and Moisture Content
The cedar chips were used as medium and the aeration rate was 100 $l \cdot m^{-3} min^{-1}$

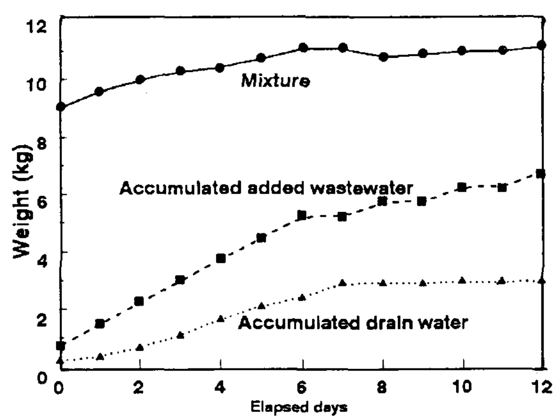

Fig. 3 Changes in Weight of Mixture and Water
The cedar chips were used as medium at the BOD load of 3.5 to 2.0 $kg \cdot m^{-3} d^{-1}$ and aeration rate of 100 $l \cdot m^{-3} min^{-1}$

3.2 Treatment of the Wastewater with Using heater

It was impossible to discharge water from the reactor because the viscosity of shochu wastewater was high. Then, it is necessary to evaporate the water by using an electrical heater.

Fig.4 shows the changes of temperature and moisture content for more than three months when a heater was used(Run 2). If the BOD load ranged from 3～6kgBOD・m^{-3}・d^{-1}, the maximum temperature was above 50℃ for approximately 30 hours in one batch cycle (2 days), and the minimum temperature was higher than 40℃. After each addition of wastewater, the moisture content increased from approximately 65% to 70%. Then the organic matter was completely decomposed and the added water was evaporated so that the moisture content decreased to approximately 65% as shown in the Fig.4. No drain water was discharged. Therefore, the one hundred percent of removal efficiencies of BOD, TOC and SS was obtained as shown in Table 4. From these results, in the condition of high temperature, the organic wastes, especially SS, could be effectively decomposed.

The low temperature from the 60th to the 70th day was due to occurrence of the anaerobic condition when the BOD load was more than 7 kgBOD・m^{-3}・d^{-1} and the moisture content was higher than 72%. The anaerobic condition was recovered again by only aeration using heater for one week.Then the temperature increased again after the addition of wastewater when the suitable BOD load was applied. From these results, it was concluded that the shochu wastewater can not be treated if heating or addition of energy source is not carried out as mentioned in the following section. In case of using kumazasa charcoals, almost same result was obtained.

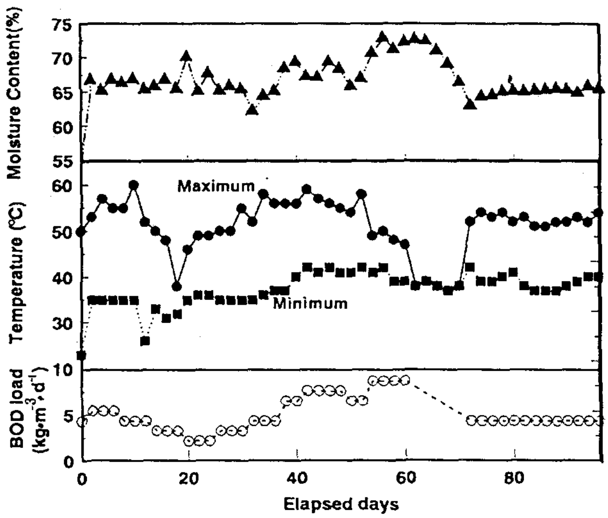

Fig. 4 Changes in Temperature and Moisture Content
The cedar chips were used as medium and the air was heateed to 60℃. Aeration rates ranged from 200 to 400 l・m^{-3}・min^{-1}..

Table 4 Removal Efficiencies of BOD, TOC and SS

Condition	Removal Efficiencies(%)		
	BOD	TOC	SS
Cedar chips with heater	100	100	100
Kumazasa charcoal with heater	100	100	100

3.3 BOD Loads and Aeration Rates

Fig.5 shows the effect of BOD volumetric loads, hydraulic loads and aeration rates on the temperature of the mixture and moisture content when the heater was used(Run 2). High temperatures, which were higher than 55℃, were obtained when BOD loads were 4 and 6 kgBOD・m^{-3}・d^{-1} at an aeration rate of 200 and 300 ℓ・m^{-3}・$min.^{-1}$, respectively. Also, the moisture contents of the mixture were kept stable at approximately 62% and 65%, respectively. From the increase of temperature, these BOD loads and aeration rates were optimum conditions for the treatment of the wastewater. When the BOD load and hydraulic load were larger than 7 $kgBOD \cdot m^{-3} \cdot d^{-1}$ and $100kg \cdot m^{-3} \cdot d^{-1}$, respectively, the temperature did not increase to higher than 55°C while moisture content increased, resulting in the anaerobic condition. This means that under the high hydraulic loads, the oxygen transfer rate was low, even though the aeration rate increased. Under these conditions, the added wastes could not be effectively decomposed and large amounts of heated air were necessary in order to evaporate all added water.

From the experiment, the optimum conditions for the treatment of shochu processed wastewater were found that hydraulic load, BOD load and moisture content ranged from 30 ~90 $kg \cdot m^{-3} \cdot d^{-1}$, 3~6 $kgBOD \cdot m^{-3} \cdot d^{-1}$ and 61~65%, respectively.

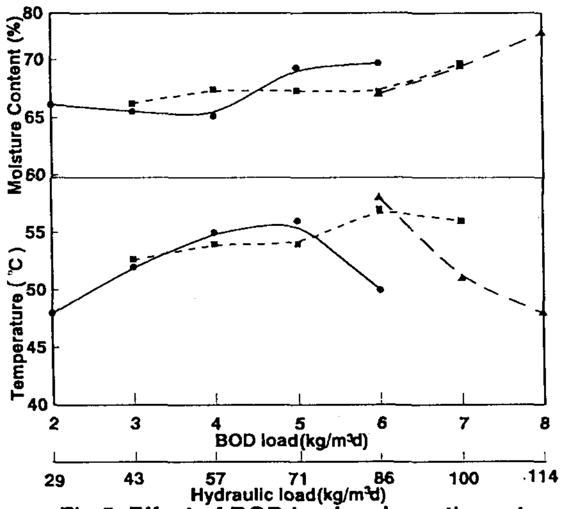

Fig.5 Effect of BOD load and aeration rate on temperature and moisture content at the aeration rate of 200(●), 300(■) and 400(▲)l·m⁻³min⁻¹.
The cedar chips were used as medium and the air was heated to 60°C.

3.4 Change of Weights of Mixture

Fig. 6 shows the change in the weights of the accumulated wastewater,mixture and drain water. The total amount of shochu wastewater treated was approximately 57 kg during a period of 100 days, and no drain was obtained. Also, the weight of the mixture was kept constant throughout during this period, though the weight of the mixture increased from the 45th to the 65th day of the experiment because of the high BOD loads. These facts indicated that all of the added wastewater was evaporated by the heat produced by decomposition of the organic matter and heater, and a minute amount of excess sludge was formed in this process. The organic wastes were completely converted to carbon dioxide and vaporized water. Therefore, the TOC, BOD and COD removal efficiencies were 100 %. No formation of drain water caused the accumulation of inorganic matter in the absorbent. The salinity in the absorbent increased from 0 to 0.013 g/g. However, no any changes happened in temperture increase and removal efficiencies of TOC,BOD and COD. The weight of the mixture also kept constant and no drain water was obtained when the kumazasa charcoals were used as medium.

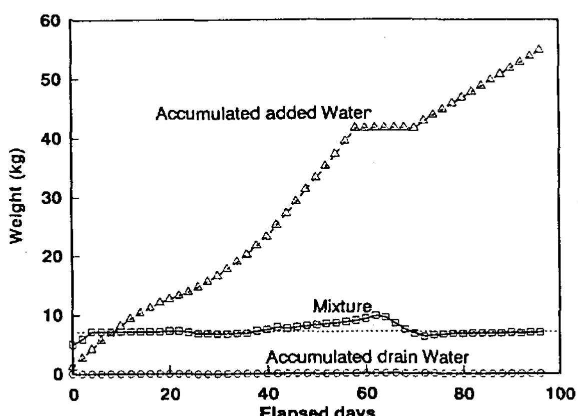

Fig. 6 Changes in Weight of Mixture and Water
The cedar chips were used as medium and the air was heated to 60°C.

3.5 Carbon Balance

There is a possibility of the decomposition of cedar chips in the experiment. Therefore, kumazasa charcoals were used in this experiment because it is not biologically decomposed. Table 5 shows the results of carbon balances following the same method as previous paper.[4] The CO_2 conversion ratios were higher than 90% when the cedar chips and kumazasa charcoals were used as media. These results indicate that a minute amount of excess sludge was formed in thermophilic oxic process and that cedar chips did not decomposed during three months.

Table 5 Carbon Balance in One Cycle (n=2)

		Charcoal		Cedar Chips	
		(g)	(%)	(g)	(%)
Input	added water(TOC)	57.2	(100)	27.5	(100)
Output	Carbon in the evaporated water	0.5	(1.0)	0.5	(1.8)
	Carbon in off-gas(g as CO_2-C)	54.9	(96.0)	24.4	(89.0)
Conversion Ratio			(97.0)		(90.8)

Note: Kumazaso charcoals: BOD load:4kg · m^{-3} · d^{-1}, Aeration rate:300 ℓ · m^{-3} · min^{-1}; TOC:52,000mg/ℓ. Hydraulic load:57 kg · m^{-3} · d^{-1}.
Cedar chips: BOD load:3kg · m^{-3} · d^{-1}, Aeration rate:300 ℓ · m^{-3} · min^{-1}; TOC:25,000mg/ℓ. Hydraulic load:57 kg · m^{-3} · d^{-1}.

3.6 Water and Thermal Balances

The moisture content did not increase and no water was discharged when the hydraulic load ranged from 43 to 86 kg · m^{-3} · d^{-1}, as shown in the Fig. 4 and 6. This means that the effect of heat from the input was not only to increase the temperature of the mixture and compensate the heat loss but also to contribute to the evaporation of all water which was added to the reactor. In order to clarify why no drain water was discharged from the reactor, the water and thermal balances in the reactor were investigated. Table 6 shows water balance. When the BOD loads were 3 and 4 kg · m^{-3} · d^{-1} at an aeration rate of 200 ℓ · m^{-3} · min^{-1}, all added water was evaporated. When the BOD loads were 5 and 6 kg · m^{-3} · d^{-1} at an aeration rate of 200 ℓ · m^{-3} · min^{-1}, 94% and 82% of the added wastewater were evaporated, respectively; 6% and 18% of the wastewater were accumulated in the medium, resulting in an increase of moisture content.

Table 6 Water Balance in One Cycle

BOD load kg · $m^{-3}d^{-1}$)	Amounts of Added Water (kg)	Evaporated Water (kg)	Accumulated Water in the Medium (kg)
3	0.85	0.85	0.00
4	1.14	1.14	0.00
5	1.42	1.34	0.08
6	1.71	1.41	0.30

Note:1.Aeration rate:200 ℓ · m^{-3} · min^{-1}.(60℃)
2.Medium:cedar chips.

The thermal balance in one cycle(two days)is described by the following equation.[4]

$$q_r + q_a = q_w + q_l + q_s \quad \cdots\cdots\cdots\cdots\cdots\cdots \quad (1)$$

input output

Where q_r represents the heat generated by the biological reaction which can be calculated by the following equation.

$$q_r = TS * Co \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots \quad (2)$$

where Co is the calorific value of the wastewater. TS is the total solid matter in the raw wastewater.

q_w represents the latent heat removed by the vaporization of the water.

$$q_w = (Qw + Sw)*Ww \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots \quad (3)$$

where Qw is the latent heat from the vaporization of water. Sw is the sensible heat of the vaporized water. Ww is the amount of vaporized water.

q_l represents the heat loss from the wall of the reactor.

$$q_l = \Sigma_i U*A(T_i - Ta_i) \quad \cdots\cdots\cdots (i = 1 \text{ to } 4) \quad (4)$$

where A is the effective surface area of the reactor(0.57 m^2). T_i is the average temperature of the mixture in every 12 hours, and Ta_i is the average atmospheric temperature in every 12 hours.

U is the overall heat transfer coefficient of the reactor. It was estimated from its relationship to the thermal conductivity of the styrenefoam insulator(λ_f), the mean thickness of insulator(L), and the logarithmic mean of surface area of the insulator surrounding the reactor (A_{lm}).

$$U = \lambda_f*A_{lm}/(LA) \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots \quad (5)$$

q_s represents sensible heat change of the mixture.

$$q_s = C_{Pr}*W_r*(dT/d\theta) \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots \quad (6)$$

C_{Pr} is the specific heat of the wastewater. Wr is the amount of added water. $(dT/d\theta)$ is the increasing rate of temperature, which is calculated from the time course of the measured temperatures. Here, it equals the temperature increase in every two hours.

q_a represents the heat from the air heater. It is difficult for it to be obtained by calculation because the heat transfers to the mixture by not only heated air but also heat exchange from the wall of the heater to mixture.

So it was estimated by measuring the amount of evaporated tap water rather than shochu waste water. When the 1.14 kg tap water was added to the reactor, the temperature of the mixture increased from 25 to 30℃, while the amount of evaporated water was 0.47 kg for 2 days. By using equations(2),(3),(4),(5)and(6), it was calculated that the heat from the air heater was 347 kcal.

The constants used for the calculation are summarized in Table 7.

Table 7 Constants Used for Calculation

Co = 5.284	kcal/gTS	(measured)
Qw = 575	kcal/kg vaporized water	
Sw = 10	kcal/kg vaporized water	
U = 0.344	$kcal \cdot h^{-1} \cdot ℃^{-1} \cdot m^{-2}$	
λ_f = 0.025	$kcal \cdot h^{-1} \cdot ℃^{-1} \cdot m^{-1}$	
C_{Pr} = 1.0	$kcal \cdot kg^{-1} \cdot ℃^{-1}$	

Thermal balance was calculated using water balance as shown in the Table 8. It shows that the input heat was nearly the same as the output heat, and approximately 66% of the total heat came from the biological reaction when the BOD load was 4 $kg \cdot m^{-3} \cdot d^{-1}$. This result explains why no drain water was discharged because there was sufficient energy to evaporate the added water, and the air heater was needed in order to evaporate all added water. In other words, if the calorific value of the wastewater is high, the heat from the air heater can be decreased. The calorific value is proportional to the concentration of organic carbon in wastewaters. The Ratio of Carbon to Water(C/W Ratio) can be used as an indicator of calorific value and evaporation of water. If some kinds of high organic carbon source is mixed with wastewaters, the C/W ratio increases, which accelerates the evaporation of water.

The running cost can be estimated from the above result. When the BOD load was 4 $kg \cdot m^{-3} \cdot d^{-1}$

at the aeration rate of 200 ℓ · m^{-3} · min^{-1}, it is approximately ¥6,372(1 kwH = ¥18) for one ton wastewater to be treated in this scale of reactor. In case of large scale of reactor, usually, the heat loss will be decreased from 30.8%(this reactor) to approximately 15%. So the cost will be also decreased to approximately ¥5,400/ton.

Table 8 Thermal Balance in One Cycle

BOD load ($kg \cdot m^{-3}d^{-1}$)	Output				Input				
	Temp. increase* (kcal)(q_s)	Heat loss (kcal)(q_l)	Evaporation (kcal)(q_w)	Total of Output (kcal)	Air Heater (kcal)(q_a)	Percentage (%)	Biological reaction (kcal)(q_r)	Percentage (%)	Total of Input (kcal)
3	19	282	493	794	347	41.0	490	59.0	837
4	29	310	667	1006	347	33.8	677	66.2	1024
5	36	310	784	1130	347	31.0	774	69.0	1121
6	39	254	825	1118	347	30.0	814	70.0	1161

Note:1. Aeration rate : 200 ℓ · m^{-3} · min^{-1}. 2. Medium : cedar chips.

3. No drain water was obtained in this experiment.

*. Temperature increase of the mixture.

4. Summary

The Shochu processed wastewater which contained high amounts of organic matter and SS was effectively treated by the thermophilic oxic process with a heater. From the results of experiments for 3 months, the following results were obtained.

① When BOD loads ranged from 3 to 6 kgBOD · m^{-3} · d^{-1} at an aeration rate of 200~300 ℓ · m^{-3} · min^{-1} with a heater, the average removal efficiencies of BOD, TOC and COD_{MN} were 100%, because the organic carbon was decomposed to CO_2 and wastewater was evaporated; 66% and 34 % of the added water was evaporated by the heat of biological reaction and of heater, respectively.

② The weights of the absorbent mixed with the wastewater did not change at all. This means that a minute amount of excess sludge was formed, because most of the organic wastes were converted to CO_2 and water.

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