

Application of magnetic activated sludge process for a milking parlor wastewater treatment with nitrogen and phosphorus recovery

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Abstract

Milking parlor wastewater contains high concentration suspended solid (SS), nitrogen, and/or phosphate as well as organic compounds. A new biological wastewater process by magnetic separation, magnetic activated sludge (MAS) process, was applied to milking parlor wastewater treatment process. A three step wastewater treatment process of coagulation sedimentation / ammonia stripping (C/S), magnetic activated sludge process and contact oxidation (CO) was proposed for removal of these pollutants. First step, C/S process recovered 96% TN and 96% PO₄³⁻-P as resource for fertilizer from the wastewater. 81% biochemical oxygen demand (BOD) in wastewater was removed after MAS process. As a results, all pollutant concentrations satisfied Japanese effluent standards. Most of residual BOD and SS were removed by the CO process. It was estimated that the proposed process could reduce the process space to 1/7.

Keywords: ammonia stripping, coagulation, dairy wastewater treatment, magnetic separation, nitrogen and phosphate recycle

1. INTRODUCTION

Magnetic separation hardly been applied in water environmental conservation technology because almost pollutants have not ferromagnetic property. However, new wastewater treatment processes using magnetic separation is researched in physicochemical or biological treatment process, recently. By magnetic seeding with magnetite, many kinds of pollutant, suspended solid in paper factory wastewater [1], dye [2], aquatic organisms [3], heavy metal [4], ferrous ions [5] were able to separate from water effectively. Also activated sludge can separated magnetically by magnetic seeding [6-8]. Magnetic separation made possible to operate easily activated sludge process. Magnetic separation is considered to have a large potential to bring innovative advance to water treatment technology. In order to enlarge of applications of superconductive magnet, it is expected to actively challenge to apply of magnetic separation to water treatment technology.

In Japan, due to "the act on the appropriate treatment and promotion of utilization of livestock manure", every dairy farm with more than 10 cows must have a wastewater treatment plant. Milking parlor wastewater is composed mainly of washing water of milking equipment and the floor. The wastewater contains generally high concentrations of organic compounds, nitrogen compounds, and phosphate. Moreover, the chemical

oxygen demand (COD) concentration of dairy wastewater fluctuates seasonally. It was found to be higher COD in winter and lower in rainy seasons [9]. Some wastewater of milking parlor includes high concentration SS of cow dung origin, that make the stable wastewater treatment difficult. Some no primary settling tank process has led to increases of inorganic suspended solids in the activated sludge, negatively affecting water treatment plant performance [10]. Biological process is able to remove biodegradable fraction at highly level, on the other hand, other treatment strategies should be applied for refractory and/or slowly biodegradable COD [11]. Coagulation is used generally for removal of suspended solid. Kushwaha et al. reported dairy wastewater treatment by inorganic coagulants [12]. To optimize the chemical pretreatment of dairy wastewater, the effects of dosages of coagulants and adsorbents were examined [13]. There are a few studies that were carried out by the organic coagulants such as chitosan [14] or cellulose-base flocculants [15]. Some milking parlor wastewater also includes high concentrations of ammonium ions generated from cow's urine. If the value of C/N ratio is below 4, it might be necessary to add of a suitable amount of hydrogen donor for biological nitrogen removal. In Japan, the provisional effluent standards of nitrogen compounds, 500 mg-N/L as ammonium, ammonium compounds, nitrite compounds and nitrate compounds was allowed until 2022 for livestock farmer. In future, the provisional effluent standard will be tight until the national uniform effluent standard of 100 mg-N/L after certain mitigation period. Development of a simple, low

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cost and high-performance wastewater process is considered to be important and urgent issue for many livestock farms.

Activated sludge process is widely used for the organic wastewater treatment. The major problem of this process is sludge-liquid separation. In principle, activated sludge is difficult to be separated from water by sedimentation because the apparent density of activated sludge particle is very close to water density. To maintain the good condition of sludge settle ability is considered to be major problem on farm wastewater management. For reduction of the load of this management, a simple and stable process is expected as a dairy farm wastewater treatment process.

Magnetic activated sludge (MAS) process has been studied as new biological wastewater treatment process. In MAS process, microbes separate magnetically from water quickly. A sedimentation process which can cause to management trouble is not needed in MAS process. The magnetic separation is faster than the gravitational settling of conventional activated sludge processes [6]. The MAS process can reduce excess sludge production by operating at high concentration of microbes condition. Moreover, MAS process could remove organic matter and nitrogen simultaneously by application of intermittent aeration [7]. Ying et al. reported simultaneous removal process of organic and nitrogen compounds by a single tank MAS process with optimized intermittent aeration for milking parlor wastewater treatment [8]. However, the MAS process is not able to remove phosphate so that another process was considered to be required for phosphate removal.

Generally, milking parlor wastewater includes ammonium compounds and phosphate so that these compounds should be recycled as on-site fertilizers in the farm. It was considered that a coagulation and ammonia stripping (C/S) process before biological treatment is suitable process for recovery of the nitrogen and phosphorus compounds. The objective of this paper is proposal of an advanced MAS process applying magnetic separation including coagulation, ammonia stripping and contact oxidation process for simple and stable wastewater

process as well as recovery process of nitrogen and phosphorus on-site fertilizers. The new process proposed was evaluated by a bench scale experiment by using the milking parlor wastewater of Utsunomiya university farm.

2. MATERIAL AND METHODS

The pollutants including in university farm wastewater were shown in Table 1. These pollutant concentrations fluctuated widely at each measurement. The value of COD_{Cr} and COD_{Mn} means chemical oxygen demand by using potassium dichromate and potassium permanganate as oxidants, respectively. Average BOD/N (biochemical oxygen demand by using potassium dichromate / nitrogen) or COD_{Cr}/N (chemical oxygen demand by using potassium dichromate / nitrogen) ratio in this wastewater was 0.86 or 3.0, respectively. The proposed process, which was shown in Fig. 1, was composed of three steps, (i) coagulation sedimentation and ammonia stripping (C/S), (ii) magnetic activated sludge (MAS) process, and (iii) contact oxidation (CO).

The role of 1st step, the C/S process is to remove of ammonium, phosphate, suspended solids (SS) in the wastewater. In the C/S process, 18 L wastewater was filled into the C/S tank, and $Ca(OH)_2$ was added to be 5 g/L. The pH of the wastewater became more than 11, as a result, it

TABLE 1
COMPONENT OF DAIRY WASTEWATER IN UNIVERSITY FARM.

Parameters	Concentration (mg/L)		
	Maximum	Minimum	Average
COD_{Cr}	2600	300	1273
COD_{Mn}	899	129	436
BOD	424	257	368
NH_4^+-N	867	108	423
$NO_2^- -N$	23.3	0.90	6.93
$NO_3^- -N$	0.12	0	0.05
$PO_4^{3-} -P$	35.9	4.21	13.8
SS	327	51.5	204

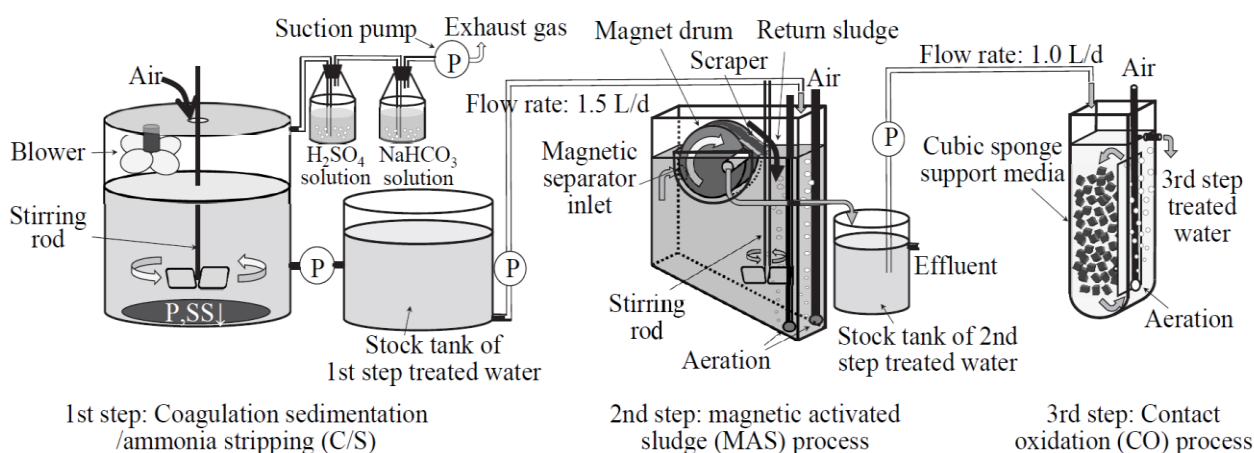


Fig. 1. Experimental apparatus for proposed process that were consisted of three steps. 1st step, coagulation sedimentation and ammonia stripping (C/S) process was a sequential batch reactor (18 L). 2nd step, magnetic activated sludge (MAS) process was a continuous reactor (1.5 L reactor with 0.1 L magnetic separator). 3rd step, contact oxidation (CO) process was a continuous reactor (1.0 L).

is expected that dissolved ammonium ion in the wastewater changes to free ammonia and is stripped from the wastewater as ammonia gas. And also, it is expected that dissolved phosphate ion in the wastewater is precipitated and removed from wastewater as calcium phosphate. The suitable stripping time was examined between 4 h and 72 h under the liquid agitation, aeration into the liquid and/or gas blowing to liquid surface in the headspace. The gas of the headspace was exhausted by a suction pump through two gas absorption bottles. One was filled with 0.5 L of H_2SO_4 (1:1) solution for trap of ammonia gas. And another was filled 0.5 L of NaHCO_3 solution for trap of H_2SO_4 mist.

After ammonia stripping process, the wastewater was settled down during 1 hour for sedimentation of suspended solids and calcium phosphate. The supernatant was sent to the stock tank and neutralized by H_2SO_4 , and then, that was introduced to MAS process (2nd step) at constant flow rate of 1.5 L/d by a peristaltic pump.

The role of 2nd step, the MAS process is removed dissolved organic compound in the wastewater. The MAS process consisted of an aeration tank (capacity: 1.5 L) with a magnet drum separator (capacity: about 0.1 L, surface magnetic flux density: 0.08 T, stripe magnetized pattern of 6 mm pitch, drum rotation rate: 10 rpm). At the start of the experiment, activated sludge suspension obtained from the milk parlor wastewater treatment plant of university farm was fed into the aeration tank. To enable magnetic separation of the activated sludge, magnetite obtained commercially was added to the activated sludge. Initial concentration of the mixed liquor suspended solids (MLSS) and mixed liquor volatile suspended solids (MLVSS) in the MAS reactor were 3400 mg/L and 1300 mg/L, respectively. Initial magnetite concentration was 2100 mg/L. Intermittent aeration of 120 min cycle (60 min-aeration / 60 min-non aeration) were adopted to remove nitrogen compound biologically.

From stock tank of 1st step treated tank, the treated water of the C/S process was fed into the MAS process as influent by constant flow rate of 1.5 L/d (hydraulic retention time (HRT): 1 d). The influent was treated biologically in aeration tank and flow into the magnetic separator as sludge suspension. The magnetic separator separated MAS from the sludge suspension, and all separated MAS was scraped off from magnet drum surface and was returned into the reactor. On the other hand, the separated water was sent to the stock tank of the 2nd step treated water. The MAS process was operated without excess sludge withdrawal during all experimental period of 325 d. From the stock tank of 2nd step treated water, The treated water was fed to the CO process (3rd step) with constant flow rate, 1.0 L/d by another peristaltic pump.

The CO process was carried out by 1 L (available volume) packed bed reactor, which was composed of 0.8 L (bulk volume) bed of sponges (1 cm^3 cubic size) for immobilizing of microorganisms and a 0.2 L air lift partition for circulation of wastewater (circulation time: about 1 min). The CO process was operated under HRT 1 d. The concentration of dissolved oxygen was more than 3 mg/L during the experiment. The immobilized bed was washed by stir once several months, and all suspended sludge was collected and returned to the MAS reactor.

There was no excess sludge emission from MAS and CO process except sampling of 50 ml sludge for measuring MLSS and MLVSS. All experiment was carried out at the liquid temperature, $25 \pm 1^\circ\text{C}$. The COD_{Cr} , COD_{Mn} , BOD, MLSS, MLVSS, $\text{PO}_4^{3-}\text{-P}$, $\text{NH}_4^+\text{-N}$, $\text{NO}_2^-\text{-N}$, and $\text{NO}_3^-\text{-N}$ were measured by the JIS methods.

3. RESULTS AND DISCUSSION

3.1. The component of farm wastewater

From Table 1, the BOD/N ratio of the farm wastewater was found to be very small for biological nitrogen removal. Zhi et al. reported that only 50% removal efficiency of TN was obtained under the BOD/N ratio of 4 [16]. The stoichiometric BOD/N ratio for the denitrification process is 2.86 (as $\text{COD}/\text{NO}_3^-\text{-N}$), however, in report by Kumar et.al., the TN removal efficiency increased with increasing in BOD/N ratio, finally, the efficiency reached to 98% under BOD/N ratio of 8.1 that is far larger than stoichiometric BOD/N ratio [17]. If BOD/N ratio of 8.1 is necessary for sufficient denitrification, it is estimated that additional 2.6 g/L BOD as hydrogen donor is required to be added into farm wastewater for achievement of biological denitrification. This means the organic loading of the wastewater increases by 7 times. Not only the cost of organic compound of additional hydrogen donor but also the 7 times larger wastewater treatment plant would be required for achievement of nitrogen removal from the wastewater. A biological nitrogen removal does not seem to be reasonable solution for the wastewater treatment of such low BOD/N ratio. It was considered that the nitrogen should be recovered from the wastewater and utilized as fertilizer in the farm.

3.2. The C/S process

An ammonia stripping is well known process for ammonia recovery method from solution. In this paper, $\text{Ca}(\text{OH})_2$ was used not only as an alkali compound but also as insolubilizing counter cation of phosphate anion and coagulants of suspended solids. Guštin et al. reported that the ammonia removal efficiency rapidly rose to 92.8% at pH values of 10.5 and 11 [18]. By adding $\text{Ca}(\text{OH})_2$ at 5 g/L, the mixture was able to be adjusted to pH 11 or more. The ammonia stripping profiles in the C/S process were shown in Fig. 2. Two types of stripping acceleration method, blowing and blowing and agitation, were examined to save the process time. The blowing and agitation were effective to save the process time on ammonia stripping process. It was found the process time was able to save ammonia stripping time to 24 h from 72 h. As another acceleration method, aeration was also effective to save process time for ammonia stripping. However, it was considered that the aeration consumed more electric power than blowing and agitation method because a large volume of air had to be blown in the mixture to reach same ammonium concentration at 24h. The concentration of $\text{PO}_4^{3-}\text{-P}$ in the farm wastewater is included 4 to 36 mg/L that was higher than Japanese effluent standard of $\text{PO}_4^{3-}\text{-P}$, 16 mg/L (8 mg/L in daily average). Phosphate ions was expected to

precipitate to the concentration of less than Japanese standard under excess calcium ions and alkaline conditions. Precipitation of calcium phosphate is be able to use as fertilizer.

In the first step (C/S process), COD_{Cr} , COD_{Mn} , BOD, SS, TN and $\text{PO}_4^{3-}\text{-P}$ decreased to 494, 116, 139, 88, 15 and 0.52 mg/L, respectively. Removal rate of TN, $\text{PO}_4^{3-}\text{-P}$ and COD_{Cr} by C/S process achieved 96, 96 and 61% respectively. Removal rate of each treated water was described Table 2. In addition, most of chromaticity of the wastewater was observed to be removed by C/S process (Fig. 3).

Similar method was used to anaerobically digested piggery wastewater treatment, and the removal efficiencies of the $\text{NH}_3\text{-N}$, total P and COD were over 91%, 99% and 52% respectively [19]. The C/S method was considered to be effective for simultaneous removal of ammonium and phosphate ions. All stripped ammonia gas was collected in the gas absorption tanks as ammonium sulfate. On the other hand, the removed phosphate by C/S process was collected in the portion of precipitation as calcium phosphate. The precipitation can be used to composting as raw material or additive. If this C/S process was adopted to full-scale plant of the university (4 m^3/d of wastewater loading), it would be estimated the $(\text{NH}_4)_2\text{SO}_4$ of 5.5 t/y and $\text{PO}_4^{3-}\text{-P}$ of 20 kg/y can be recovered from wastewater.

The concentrations of COD_{Mn} , BOD, SS, TN, and $\text{PO}_4^{3-}\text{-P}$ from the effluent from C/S, MAS, and CO processes were shown in Fig. 4. The dotted lines shows Japanese effluent standards (daily average values). It was demonstrated that the C/S process was able to remove to less than the Japanese effluent standards of SS, TN and $\text{PO}_4^{3-}\text{-P}$. On the other hand, the concentration of COD_{Mn} and BOD was not able to achieve the effluent standards. The MAS process as second step was necessary to achieve all effluent standards.

3.3. The MAS process

The MAS process was able to biologically decompose residual organic compounds in C/S effluent to about half values of the effluent standards (Fig. 4). The average BOD volumetric loading rate of MAS process was approximately $0.07 \text{ kg}/(\text{m}^3 \cdot \text{d})$. This value was very low as compared with $0.5\text{-}1 \text{ kg}/(\text{m}^3 \cdot \text{d})$ that is a value of standard activated sludge process. Therefore, MLVSS concentration did not increase to more than 2000 mg/L in spite of no excess sludge withdrawal. As the results, the average BOD sludge loading rate (F/M ratio, food to microorganisms ratio) became about $0.04 \text{ kg}/(\text{kg} \cdot \text{d})$. In general, it is well known that the range of F/M ratio from 0.2 to $0.4 \text{ kg}/(\text{kg} \cdot \text{d})$ and the ratio of BOD/N/P of about 100/5/1 are important for stable operation of an activated sludge process by settling separation. In spite of the low F/M ratio and low phosphate concentration conditions after the C/S process, the MAS process by magnetic separation treated successfully the wastewater for all experimental period. After the second step (MAS process), COD_{Cr} , COD_{Mn} , BOD, SS, TN and $\text{PO}_4^{3-}\text{-P}$ decreased to 172, 54, 70, 72, 13 and 0.42 mg/L, respectively.

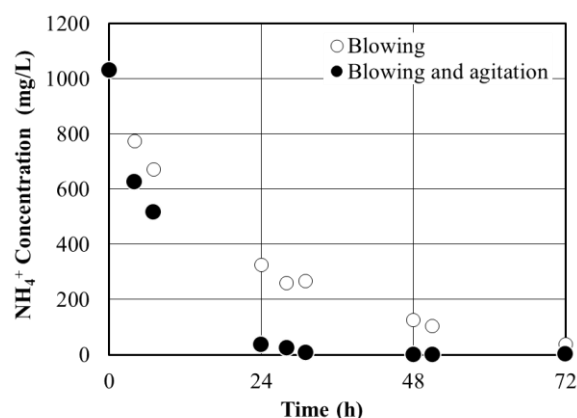


Fig. 2. Decreasing curves of ammonium ion concentration in the liquid of C/S tank under air blowing only or air blowing and liquid agitation.

TABLE 2
REMOVAL RATE OF COMPONENTS IN EACH TREATED WATER.

Parameters	Removal Rate(%)		
	C/S	C/S + MAS	C/S + MAS + CO
COD_{Cr}	61	86	94
COD_{Mn}	65	88	94
BOD	62	81	91
SS	57	70	95
TN	96	97	98
$\text{PO}_4^{3-}\text{-P}$	96	97	96



Fig. 3. Photograph of the farm wastewater, treated water after C/S, treated water after MAS and treated water after CO.

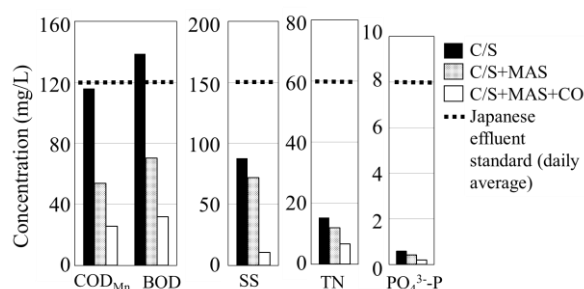


Fig. 4. Comparison of treated water in this experiment to Japanese effluent standards.

3.4. The CO process

All the effluent standard of pollutant indexes were achieved by C/S and MAS process. Therefore, CO process as the final step was not indispensable as the treatment process for the university farm wastewater. However, if an advanced purified water is required, CO process is considered to be useful to remove further the COD_{Mn}, BOD, SS and TN from MAS effluent.

3.5. Treatment performance

The relationships of BOD, COD_{Mn}, SS, TN, and PO₄³⁻-P between the farm wastewater and each treatment water after the C/S, MAS, or CO process, respectively were shown in Fig. 5. The concentration of pollutants fluctuated greatly depending on the farm works in the experimental period (325 d). For example, the range of COD_{Mn} was from 129 mg/L in minimum to 899 mg/L in maximum.

Even the C/S process alone was able to remove TN and PO₄³⁻-P until less than the Japanese effluent standards under all wastewater concentration. These removal rate was not depend on wastewater concentration. On the other hand, COD_{Mn}, BOD and SS tended to exceed the effluent standard under the condition of high wastewater concentration. Strong positive correlation (slope of regression line; 0.27, coefficient of determination; 0.93) was observed at COD_{Mn} between wastewater and C/S-treated water in the range of more than 300 mg/L COD_{Mn}. This result suggests that the C/S process alone can remove stably approximately 72% of COD_{Mn} from the dairy wastewater against large fluctuation of COD_{Mn} concentration (300 mg/L - 900 mg/L).

The C/S and MAS combined process was able to remove BOD, COD_{Mn}, SS, TN and PO₄³⁻-P until less than the effluent standards except a case of the highest COD_{Mn} wastewater condition in this experiment. In this combined process, it was also observed strong positive correlation (slope of regression line; 0.12, coefficient of determination; 0.81) at all COD_{Mn} range of the experiment.

The C/S, MAS and CO combined process was able to purified until around advanced effluent standard level of BOD, COD_{Mn}, SS, TN and PO₄³⁻-P under conditions of all wastewater concentration in this experiment. Strong positive correlation of COD_{Mn} was observed in this process, too. The slope of regression line and coefficient of determination were 0.047 and 0.97 respectively. Approximately 95% COD_{Mn} removal rate is expected to be able to be obtain stably under wide COD_{Mn} loading fluctuation (130 mg/L - 900 mg/L). On the other hand, TN removal rate was apt to decrease with decreasing of TN concentration in the wastewater.

The present wastewater treatment process of university farm is activate sludge process of which HRT is for 14 d. In general, HRT range of 10 d to 20 d is adopted to dairy wastewater treatment process by using activated sludge process. In the case of purification to less than Japanese effluent standard, it is enough to use C/S (HRT: 1 d) and MAS process (HRT: 1 d). The required time for the dairy wastewater treatment is expected to save to 2 d from 14 d. Therefore, it was considered that the C/S and MAS process can reduce the wastewater treatment plant space of Utsunomiya university farm to 1/7. Even in the condition

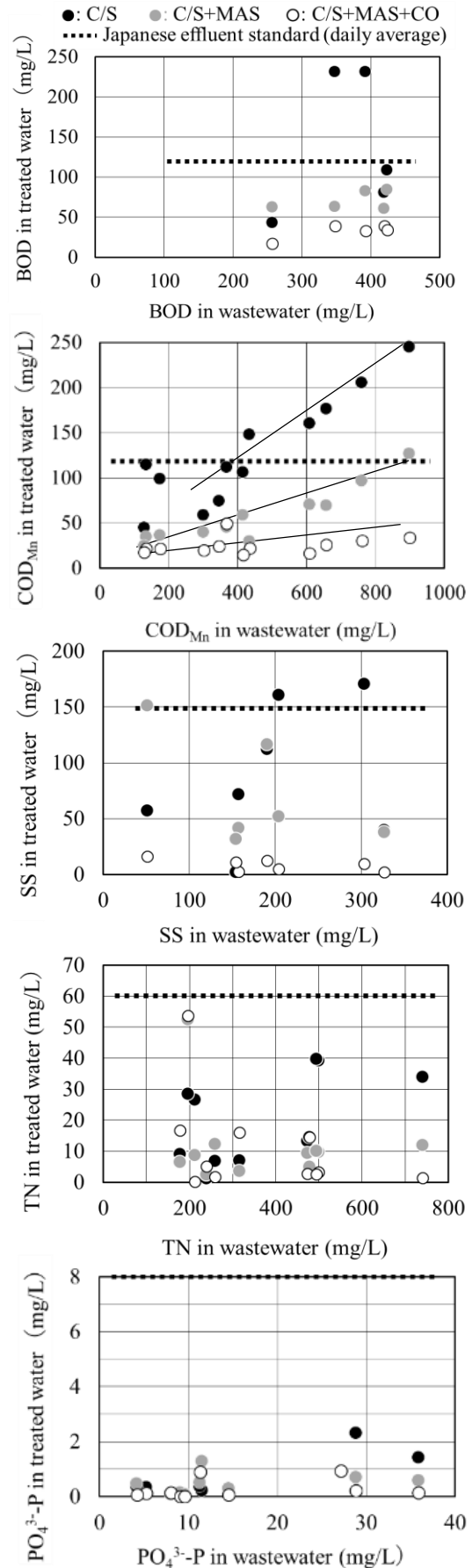


Fig. 5. Relationships of pollutant concentrations between the farm wastewater and the treated water at each step.

of advanced effluent standard, this process can be expected to save remarkably wastewater treatment plant space of many dairy farm.

4. CONCLUSION

In this paper, the three-step wastewater process of the C/S, MAS and CO processes was proposed for pollutants removal and nitrogen and phosphorous recycling in a milking parlor wastewater. The new process showed a very high performance as compared with present wastewater treatment process of the university farm. The treatment rate was shown to be 7 times faster than the present process. Ammonium and phosphate in the wastewater were able to recycle by this process. The proposed process was stable and easy to operate during 325 d experiment. The MAS and CO processes were able to operate without excess sludge withdrawal.

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