Agricultural recycling of the by-product concentrate of livestock wastewater treatment plant processed with VSEP RO and bio-ceramic SBR

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Abstract One of the problems in a reverse osmosis process for livestock wastewater treatment is disposal of the by-product concentrate. The agricultural recycling of the concentrate is more cost saving than a further treatment. Application of the concentrate reduces the chemical fertilizer amendment. The agricultural recycling of the concentrate from the Kimhae livestock wastewater treatment plant, processed with the VSEP RO and bio-ceramic SBR, was studied. The concentrate includes non-biodegradable humic substance and residual inorganic ions (NH4+, NO3–, PO43–, K+, etc.). The contents of N, P and K were 1,650, 382 and 2,059 mg L–1, respectively. The total acidity of humic acids extracted from the concentrate was 5.17 cmol(+) g–1, composed of 2.38 cmol(+) g–1 of carboxylic group and 2.79 cmol(+) g–1 of phenolic hydroxyl group. Coliforms and E. coli were not detected in the concentrate. The yield of rice plant with the concentrate applied to it resulted in similar production to that with chemical fertilizer applied. The water extractable nitrate content of the concentrate-applied land did not exceed that of chemical fertilizer applied, at soil depths of 30 and 60 cm. The percolated amount of nitrate into the water table in arable land with the concentrate applied showed a similar level to that treated with the chemical fertilizer.

Keywords Agricultural recycling; concentrate; odor; pathogens; piggery wastewater; reverse osmosis

Introduction
The fast growth in livestock farming since early 1980s has resulted in a vast production of animal wastewater in Korea. Livestock wastes may cause serious environmental and agricultural problems such as eutrophication of surface water by phosphorus deposition, nitrate leaching to ground water, odor nuisance, crop damage by ammonia emission and deposition, and disturbance of soil fertility (Taiganides, 1992; Ten Have, 1993; El-Ahraf and Willis, 1996; Aarnink, 1997). Especially, problems associated with piggery wastewater have drawn tremendous attention over the past years, since piggery wastewater containing high concentration of organic matters and nutrients has caused a limitation in efficient treatment. The livestock industry has been restricted within a limited land area and operated with dense animal feeding in Korea. Issues on environmental pollution, pathogens, odors and sanitary problems are caused by piggery wastewater. The piggery wastes were disposed several methods such as: composting, liquefying by fermentation, and discharge by wastewater treatment plant. Korean government has been encouraging the livestock wastes to be recycled into agricultural land as a potential source of valuable nutrients and for the establishment of sustainable agriculture which will reduce inorganic fertilizer use. About 87% of annual generation was recovered to agricultural land by composting and fermentation. However over-application and improper management in the agricultural recovery of livestock wastes causes some secondary environmental and agricultural problems. Therefore, integrated nutrient management to crop plants is becoming important. Pretreatment is needed for the mitigation of environmental risk and crop injury...
before the agricultural input of piggery wastes. The agricultural recycling of the concentrate from the Kimhae livestock wastewater treatment plant, processed with VSEP (vibratory shear enhanced process) RO (reverse osmosis) and bio-ceramic mediated SBR (sequencing batch reactor), was studied. The technical reasonability of recycling piggery wastes within the agricultural production cycle was examined, and the use of non-biodegradable humic substance and residual inorganic ions (NH\textsubscript{4}\textsuperscript{+}, NO\textsubscript{3}\textsuperscript{−}, PO\textsubscript{4}\textsuperscript{3−}, K\textsuperscript{+}, etc.) as valuable plant nutrients of the concentrate were analyzed. Rice crop yield response to the application of the concentrate was investigated by field experiments, and environmental effects such as nitrate leaching and phosphate deposition were investigated. Sanitary problems concerned with coliforms and \textit{E. coli} were also investigated.

**Materials and methods**

**Wastewater treatment system**

Kimhae livestock wastewater treatment plant has been installed with the major processes of solid separation, BCS (bio-ceramic mediated SBR), and RO filtration by VSEP module for the treatment of piggery wastewater (Figure 1). The concentrate has been produced as the by-product of the VSEP RO process. About 130 m\textsuperscript{3} of piggery wastewater collected from nearby small-sized stock farmers has been treated per day. In the BCS unit, about 91% of ammonium ions are nitrified effectively during the aeration period. During anoxic mixing, NO\textsubscript{2} and NO\textsubscript{3} are denitrified and emitted to atmosphere as N\textsubscript{2} gas. Thereafter BCS effluent is treated further by VSEP RO filtration process in which non-biodegradable humic substance and residual inorganic nutrients were removed with an efficiency above 70%.

**Analysis of fertilizing ingredients**

The plant nutrients of the concentrate generated in the VSEP RO process were analyzed. TKN, T-P, K, Ca and Mg were determined as major plant nutrients, and Mn, Cu, Fe, Mo and Zn were determined as micro-nutrients. Samples of the concentrate were digested in a heating block under H\textsubscript{2}SO\textsubscript{4} using K\textsubscript{2}SO\textsubscript{4} and CuSO\textsubscript{4} as catalysts. TKN and T-P were analyzed by Kjeldahl distillation method and vanadate method, respectively. K, Ca, Mg, Mn, Cu, Fe, Mo and Zn were analyzed by atomic absorption spectrometry after wet digestion by H\textsubscript{2}SO\textsubscript{4}-HClO\textsubscript{4}. Also, in order to characterize the chemical properties of non-biodegradable humic substance included in the concentrate, the pH of the concentrate was adjusted to pH 1.5 with 6N-HCl, and precipitated particles were centrifuged and lyophilized. The cation exchange capacity of humic acid extracted from the concentrate was characterized by the determination of functional groups such as carboxyl and phenolic hydroxyl groups that have buffering capacity to absorb plant nutrients (Stevenson, 1994).

**Analysis of microbial characteristics**

In order to investigate the pathogenic risk in the agricultural application of the concentrate, Coliforms and \textit{E. coli} were tested. Coliforms and \textit{E. coli} represent pathogenic risk indirectly. These were isolated using the selective media (RAPID E.coli2; BIO-RAD). Coliforms and \textit{E. coli} were incubated at 37 °C and 44 °C for 24 h, respectively.

![Figure 1 Process flow diagram of Kimhae livestock wastewater treatment plant (1 bio-ceramic mediated SBR process, 2 vibratory shear enhanced process adopting the reverse osmosis membrane)](https://iwaponline.com/wst/article-pdf/49/5-6/405/420813/405.pdf)
Field application

Experimental site. Field experiments were conducted from 7th March to 15th November 2000 at the Korea University Experimental Farm located at Duckso, Kyungki, Korea. Duckso, located at 127°N, 36°E, has a temperate climate with hot wet summers and cold winters. The physical and chemical properties of soils at different depths are listed in Tables 1 and 2. The soil texture was silty clay loam (SiCL) at a depth of 15 cm, and silty clay (SiC) at depths of 30 cm and 60 cm. The clay content was over 40% and field moisture content (FMC) was about 40%. Available SiO₂ content, organic matter content, and cation exchange capacity (CEC) were 128 mg kg⁻¹, 1.96% and 14.3 cmol(+) kg⁻¹, respectively.

Treatments and cultivation. Rice cultivars (Oryza sativa) were cultivated with the concentrated application. The application levels were 50%, 100%, 150% and 200% of the recommended quantity of N fertilizer (110 N kg ha⁻¹) as shown in Table 3. The results were compared with the yield response of conventional agriculture using inorganic fertilizer. Experimental site was compartmented with 18 plots, each plot was 20 m² (5 m × 4 m). Each treatment plot had three replicates. Each treatment plot was arranged randomly. The rice crop was transplanted early in June and harvested in the middle of November. The yield response was estimated through the survey of 1,000 grains weight, number of spikelets, panicles, and tillers. The collected data were analyzed by Duncan’s multiple range test (Gomez and Gomez, 1984). To investigate the downward flow of nitrate, soil samples were collected periodically at depths of 15, 30 and 60 cm. Soil samples before physical and

Table 1 Physical properties of paddy soil

<table>
<thead>
<tr>
<th>Depth (cm)</th>
<th>Soil textural classes</th>
<th>Sand</th>
<th>Silt</th>
<th>Clay</th>
<th>FMC ¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>SiCL</td>
<td>14</td>
<td>46</td>
<td>40</td>
<td>41</td>
</tr>
<tr>
<td>30</td>
<td>SiC</td>
<td>14</td>
<td>43</td>
<td>43</td>
<td>–</td>
</tr>
<tr>
<td>60</td>
<td>SiC</td>
<td>7</td>
<td>46</td>
<td>47</td>
<td>–</td>
</tr>
</tbody>
</table>

¹ Field moisture capacity

Table 2 Chemical properties of paddy soil

<table>
<thead>
<tr>
<th>Depth (cm)</th>
<th>pH (H₂O)</th>
<th>pH (KCl)</th>
<th>Available P₅O₅</th>
<th>Available SiO₂</th>
<th>TKN¹</th>
<th>OM²</th>
<th>CEC³</th>
<th>Exchangeable cations</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Na</td>
</tr>
<tr>
<td>15</td>
<td>6.25</td>
<td>4.93</td>
<td>21.80</td>
<td>128</td>
<td>1.3</td>
<td>1.96</td>
<td>14.3</td>
<td>0.88</td>
</tr>
<tr>
<td>30</td>
<td>6.58</td>
<td>5.19</td>
<td>3.26</td>
<td>–</td>
<td>0.8</td>
<td>0.96</td>
<td>14.6</td>
<td>0.88</td>
</tr>
<tr>
<td>60</td>
<td>6.14</td>
<td>4.89</td>
<td>2.88</td>
<td>–</td>
<td>0.8</td>
<td>0.34</td>
<td>15.0</td>
<td>0.85</td>
</tr>
</tbody>
</table>

¹ total Kjeldahl nitrogen, ² organic matter content, ³ cation exchange capacity

Table 3 Nitrogen application rates for field experiments

<table>
<thead>
<tr>
<th>Treatments</th>
<th>Nitrogen application rates (kg ha⁻¹)</th>
<th>Fertilizing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control¹</td>
<td>0</td>
<td>Non-application</td>
</tr>
<tr>
<td>C 50%²</td>
<td>55</td>
<td>Concentrate of VSEP RO process</td>
</tr>
<tr>
<td>C 100%</td>
<td>110</td>
<td>Chemical fertilizer</td>
</tr>
<tr>
<td>C 150%</td>
<td>165</td>
<td>Chemical fertilizer</td>
</tr>
<tr>
<td>C 200%</td>
<td>220</td>
<td>Chemical fertilizer</td>
</tr>
<tr>
<td>NPK³</td>
<td>110</td>
<td>Chemical fertilizer</td>
</tr>
</tbody>
</table>

¹ non-application, ² application rate of the concentrate for the recommended N quantity (110 N kg ha⁻¹), ³ application of chemical fertilizer
chemical analysis were screened through a 2 mm sieve and homogenized. All samples were
dried at room temperature.

Soil analysis. Soil texture was measured by pipetting method. Organic C was determined
d by dichromate oxidation (Tyurin’s method). Total N was determined by Kjeldahl dis-
tillation method after sulfuric acid digestion. Inorganic nitrogen (NH₄⁺, NO₃⁻) was
determined by a colorimetric method using an auto-analyzer (Traacs 800+, Bran+Leubbe).
pH was measured by glass electrode method (1:5), available phosphate (P₂O₅) and
available silicate (SiO₂) were measured by Lancaster method and modified Lancaster
method, respectively. The CEC was determined by distillation method after NH₄⁺ satu-
ration. Exchangeable cations were extracted with 1N-NH₄Oac (1:10), and analyzed
by atomic absorption spectrophotometry. A downward movement of nitrate was evaluated
using both distilled water extractable forms and 2-N KCl extractable forms (Bremner
and Slangen, 1981). Phosphorus deposition was evaluated using the concept of quantity
and intensity after the determination of available phosphorus (Lancaster reagent
extractable form) and labile phosphorus (0.01N-CaCl₂ extractable form) (Anderson and
Wu, 2001).

Results and discussion
Analysis of fertilizing ingredient
The concentrate of the V-SEP process is an odorless dark-brown liquid that has a high
amount of variable plant nutrients (Table 4). The color of the concentrate is caused by
dissolved humic acid. The N, P and K content was 1,650, 382 and 2,059 mg L⁻¹, respec-
tively. Since the legislated criteria for the liquefied fertilizer is restricting the nitrogen
content as over 0.1% in Korea, the concentrate has enough quality to apply for agricultural
land. The total acidity, which means the cation exchange capacity, was 5.17 cmol(+) g⁻¹,
and the charge characteristics of total acidity were composed of a carboxylic group of
2.38 cmol(+) g⁻¹ and phenolic hydroxyl group of 2.79 cmol(+) g⁻¹ (Table 5). Humic acid
improves soil fertility and soil microbial activity by providing high ion exchange capacity
and good circumstances for microbial growth (Rulkens et al., 1998). Since the cation
exchange capacity of soils was 0.05–0.18 cmol(+) g⁻¹, generally, the humic acid contained
in the concentrate may have an important role in improving chemical and physical pro-
properties in the concentrate amended soils. When we compare with the function of humic acid
extracted from peat-moss which is conventionally used in agricultural practice, total
acidity of humic acid source showed 7.27 cmol(+) g⁻¹ in peat-moss, it shows similar
chemical properties to that of the concentrate.

Table 4 Chemical composition of the concentrate

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>N</th>
<th>P</th>
<th>K</th>
<th>Ca</th>
<th>Mg</th>
<th>Na</th>
<th>Zn</th>
<th>Cu</th>
<th>Fe</th>
<th>Mo</th>
<th>Mn</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content</td>
<td>1,650</td>
<td>382</td>
<td>2,059</td>
<td>191</td>
<td>143</td>
<td>125</td>
<td>9.9</td>
<td>1.8</td>
<td>8.7</td>
<td>0.01</td>
<td>1.0</td>
</tr>
</tbody>
</table>

Table 5 Chemical composition of humic acid extracted from the concentrate

<table>
<thead>
<tr>
<th>Organic C</th>
<th>Total acidity</th>
<th>COOH group</th>
<th>Phenolic OH group</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content</td>
<td>45.5</td>
<td>5.17</td>
<td>2.38</td>
</tr>
</tbody>
</table>

¹Organic carbon content
Analysis of microbial characteristics

Figure 2 shows isolation results of coliforms and *E. coli*. The blue and violet colonies indicate coliforms and *E. coli*, respectively, and there is no colony concerned with coliforms in the concentrate. This is the reason that pathogenic microbe included in the raw livestock manure become extinct during the aerobic biological processing step.

Yield response of rice cultivars

The growth factors affecting rice yield response by the increment of the concentrate application rate was described in Table 6 and Figure 3. The 1,000 grains weight, number of spikelets per panicle and number of tillers were significantly low at 25.07, 94.4 and 15.7 ea. in the control, respectively. The increment of the concentrate application rate did not significantly affect the growth factors concerned with rice yield response.

The rice yields in control and inorganic fertilizer treatment were 5.03 and 6.32 Mg ha\(^{-1}\), and the rice yields in 50%, 100%, 150% and 200% of the concentrate application were 6.40, 5.89, 6.30 and 6.40 Mg ha\(^{-1}\), respectively. The rice yield response shows that the more

![Figure 2 Isolation test of Coliforms and *E. coli* for the inflow livestock wastewater and the concentrate of VSEP process](image1)

![Figure 3 Rice yield response curve by the concentrate application (Characters are mean separations by Duncan’s multiple range test at 5% level, and common letter are not significantly different at the 5% level.)(image2)](image2)

**Table 6** Growth factors affecting rice yield response by the concentrate application rate

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Control</th>
<th>C 50%</th>
<th>C 100%</th>
<th>C 150%</th>
<th>C 200%</th>
<th>NPK</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitrogen application rate (kg ha(^{-1}))</td>
<td>0</td>
<td>55</td>
<td>110</td>
<td>165</td>
<td>220</td>
<td>110</td>
</tr>
<tr>
<td>1,000 grains weight</td>
<td>25.07 b</td>
<td>25.36 a</td>
<td>25.17 a</td>
<td>25.35 a</td>
<td>25.45 a</td>
<td>24.38 a</td>
</tr>
<tr>
<td>No. of spikelets per panicle</td>
<td>94.4 a</td>
<td>90.8 a</td>
<td>95.5 a</td>
<td>97.3 a</td>
<td>104.0 a</td>
<td>91.1 a</td>
</tr>
<tr>
<td>No. of tillers</td>
<td>15.7 c</td>
<td>15.9 bc</td>
<td>16.0 bc</td>
<td>17.3 abc</td>
<td>18.7 a</td>
<td>17.7 ab</td>
</tr>
</tbody>
</table>

\(^1\) Characters are mean separations by Duncan’s multiple range test at 5% level, and common letter are not significantly different at the 5% level
concentrate that was applied, the higher the rice yield appeared, but rice yields caused by the increase of concentrate application rate have no statistical significance. Rice productivity when inorganic fertilizer was applied was not significantly different from that of 50% application, and the application rate of 50% of the concentrate was enough to substitute for the conventional chemical fertilizer application with respect to rice productivity. But the yield response curve, the maximum productivity appeared at the application rate of 150%.

Nitrate leaching and phosphorus deposition
The nitrate contamination of ground water by leaching of fertilizing nutrients is important in the condensed agricultural region. In this experiment, two nitrogen forms were considered by adsorption force with clay particles, one was in water extractable form and another was in 2N-KCl extractable form. The water extractable form was considered as easily movable and accelerated form by water movement, and 2N-KCl extractable form was considered as a strongly adsorbed form, that is difficult to move downward. The water extractable and the 2N-KCl extractable nitrate contents at different soil depth in arable land after the rice harvesting was shown in Figures 4 and 5.

The water extractable nitrate contents were 0.279, 7.668 and 9.108 mg kg⁻¹ in 15, 30 and 60 cm depths of chemical fertilizer applied land (Figure 4). In the concentrate applied land, the water extractable nitrate content at 15 cm depth were 0.23, 0.26, 1.20, 0.37 and 6.90 mg kg⁻¹ by increasing the concentrate application rate from 0% to 200%. The water extractable nitrate content at 15 cm soil depth was steeply increased by the increase of the concentrate application rate. The water extractable nitrate content at 60 cm depth showed the range 6.91–8.97 mg kg⁻¹ at each application rate. The water extractable nitrate content of the concentrate-applied land did not exceed the nitrate content of chemical fertilizer applied land at each soil depth of 30 and 60 cm.

The 2N-KCl extractable nitrate contents were 10.036, 9.058 and 10.289 mg kg⁻¹ in 15, 30 and 60 cm depths of chemical fertilizer-applied land (Figure 5). In comparison with water extractable nitrate contents, most of the nitrate form may be present as the strongly adsorbed form in surface soil. The 2N-KCl extractable nitrate contents were 8.38, 9.08, 9.84, 10.44 and 9.32 mg kg⁻¹ by increasing the concentrate application rate from 0% to 200%. The concentrate application rate of 50% was environmentally safe considering the nitrate leaching problem, and the nitrate leaching may be accelerated from the concentrate application rate of 150%.

Figure 6 shows the effects of the concentrate application rate on phosphate deposition. Since the availability of phosphorus was affected by many kinds of physical and chemical characteristics of soil (Brookes et al., 1982; Hedley et al., 1982; Stewart and Tiessen, 1982), the effects of the concentrate application rate on phosphate deposition were also examined. The results showed that the phosphate deposition was not significantly affected by the different application rates of concentrate.

**Figure 4** Changes of H₂O extractable nitrate content at different soil depth by concentrate application rate (Each application rate indicates percentages for the recommended N quantity; 110 N kg ha⁻¹, and NPK means the use of conventional chemical fertilizer.)
it was difficult to interpret the state of phosphorus nutrition accurately. In Figure 6, total available phosphate means the deposited form, and labile phosphate means the form easily used by plants. Generally, the content of the easily used phosphate is very low in arable soil although phosphate is highly deposited (Sharpley et al., 1977). The deposited and labile phosphate contents were 24.75 and 0.25 mg kg\(^{-1}\) in the land with chemical fertilizer applied. The deposited phosphate content was significantly low in the control. The deposited phosphate was in the range of 22.77–27.14 mg kg\(^{-1}\) in the land with organic or inorganic fertilizer applied, and they were not significantly different. The contents of labile phosphorus were 0.23, 0.28, 0.26, 0.25 and 0.36 by the increasing of the concentrate application rate from 0% to 200%, and were lower by 100 times than the deposited phosphate contents. The sorption of phosphate by soils is affected by the content of organic matter, of amorphous Al and Fe oxides, of carbonate and clay (Moshi et al., 1974; Borggard, 1983; Brennan et al., 1994). The soil at the experimented field was silty clay loam with a high capacity to adsorb phosphate, but there is no evidence of significant deposition. But it showed a significant increase of 0.36 mg kg\(^{-1}\) of labile phosphate content at the concentrate application rate of 200%, this we infer will improve the fertilizing effect on phosphorus nutrition in the over 200% concentrate application rate but will not increase the phosphorus deposition. Therefore, the diffusion of phosphorus has occurred to a very low degree, and the loss of phosphate adsorbed clay particles has become a priority management concern (Sharpley et al., 1994).

Conclusions

One of the problems in a reverse osmosis process for livestock wastewater treatment is the disposal of the by-product concentrate. The agricultural recycling of the concentrate is
more cost saving than a further treatment. However, there are some limits to agricultural recycling such as pathogenic microbes, runoff and infiltration of underground water. The agricultural recycling of the concentrate from the Kimhae livestock wastewater treatment plant, processed with the VSEP RO and bio-ceramic SBR, was examined for the above limits. The concentrate can be used as a low concentration liquid fertilizer including non-biodegradable humic substance and residual inorganic ions (\( \text{NH}_4^+ \), \( \text{NO}_3^- \), \( \text{PO}_4^{3-} \), \( \text{K}^+ \), etc.). The contents of N, P and K were 1,650, 382 and 2,059 mg L\(^{-1} \), respectively. The total acidity of humic acids extracted from the concentrate was 5.17 cmol\((+)\) g\(^{-1}\), composed of 2.38 cmol\((+)\) g\(^{-1}\) of carboxylic group and 2.79 cmol\((+)\) g\(^{-1}\) of phenolic hydroxyl group. The concentrate can be used without pathogen problems because Coliforms and \( E. \text{coli} \) were not detected in the concentrate. The yield of rice plants with the concentrate applied resulted in similar production to that of chemical fertilizer applied. The water extractable nitrate content of the concentrate-applied land did not exceed that which chemical fertilizer applied, at soil depths of 30 and 60 cm. The amount of nitrate percolated into the water table in arable land when concentrate was applied showed a similar level to that treated with chemical fertilizer.

References


