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# Effects of Liquid Fertilizer Produced from Sewage Sludge by the Hydrothermal Process on the Growth of Komatsuna

Xiao Han Sun<sup>1\*</sup>, Hiroaki Sumida<sup>2</sup> and Kunio Yoshikawa<sup>1</sup>

<sup>1</sup>Department of Environmental Science and Technology, Tokyo Institute of Technology, Yokohama 226-8502, Japan. <sup>2</sup>Laboratory of Soil Science, Department of Chemistry and Life Science, Nihon University, Fujisawa 252-8510, Japan.

#### Authors' contributions

This work was carried out in collaboration between all authors. Author XHS designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors HS and KY managed the literature searches. All authors read and approved the final manuscript.

Original Research Article

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#### **ABSTRACT**

The objective of this study is to evaluate the possibility of recycling the liquid product obtained from sewage sludge by the hydrothermal treatment as a kind of organic fertilizer and its effect on the plant growth. A small scale hydrothermal treatment experiment was performed and proved that the liquid product contains high content of nitrogen and low content of micronutrients. Therefore, the liquid product has the potential to be used as a kind of liquid fertilizer. In a seed germination test, the liquid product indicated low phytotoxicity. Moreover, in a Komatsuna cultivation experiment, the liquid product showed accelerate effect to the crop yield which is not lower than the chemical fertilizer. Through the low-temperature hydrothermal treatment, the sewage sludge was converted into liquid organic material that could be used as a delayed-release nitrogen fertilizer for the growth of Komatsuna. These results indicated the possibility of establishing a comprehensive system for recycling sewage sludge into a kind of organic fertilizer.

Keywords: Sewage sludge; hydrothermal treatment; liquid organic fertilizer.

\*Corresponding author: Email: xiaohansun1984@gmail.com;

#### 1. INTRODUCTION

In recent years, huge amount of sewage sludge production has become a serious problem in many countries. Sewage sludge is one of the principal byproducts of wastewater biologically treated by the activated sludge method or the bio-film method in wastewater treatment plants [1]. Each year, the world generates huge amounts of sewage sludge and in the coming years, it is still increasing, which causes a series of economic, social and contamination problems. Thus, the sludge pollution problem should be solved as soon as possible.

As a kind of biomass, the moisture content of sewage sludge could reach above 80% and its organic matter content is high, thus sewage sludge is very easy to rot. According to these features, at present, the methods for sludge disposal include landfill, incineration, and agricultural use and so on. However, these methods still have defects. For the method of landfill, the heavily polluted leachate will pollute the groundwater; and the landfill gas, which is mainly methane, also has a hidden peril of causing explosion and fire. In addition, the choice of suitable sludge landfill sites is also another awkward problem. On the other hand, if the sewage sludge is used for incineration, the operating cost is very expensive due to its high moisture content and the emissions of toxic air pollutants such as  $NO_x$ ,  $SO_2$  and dioxins are also problems. Therefore, there is an urgent need to find ecologically acceptable means for reutilization of sewage sludge.

Originally sewage sludge is a good agricultural fertilizer. Its nitrogen, phosphorus and potassium content are much higher than manure. It contains many kinds of molecules coming from proteins and peptides, lipids, plant macromolecules with phenolic structures or aliphatic structures, along with organic micro-pollutants and mainly microbial cells that are complex polymeric organic materials [2,3]. And as a kind of organic waste, the organic compounds inside are also capable of promoting plant growth [4]. As a soil conditioner in farmland, forests and home gardens, use of sewage sludge in crop production is ideally favorable for a sustainable society, as it can recycle a substantial content of nutrients and organic matters [5]. But unfortunately, sewage sludge also contains parasites, bacteria and so on, coupled with unpleasant odor, therefore, it is more unwelcomed as fertilizer. The accumulation of heavy metals in soils and subsequent accumulation along the food chain are the potential threat to animal and human health. Therefore, all of these issues are calling for the development of more economic and environmentally friendly approaches to the disposal of sewage sludge and simultaneously utilize its valuable components as soon as possible.

In recent years, the hydrothermal treatment process, as a new technology applicable to biomass waste, is gaining attentions in the world due to its cheap, non-toxic, non-flammable and non-explosive operation offering essential advantages. The hydrothermal treatment employing high pressure (around 2MPa) saturated steam to convert wastes into usable products is a new applicable technology to sewage sludge. It has been already applied to sewage sludge to improve their dewaterability [6]. It is proved that at certain temperature and pressure, the hydrothermal treatment will rupture the cell wall and membranes of organics in sewage sludge, and will improve the dewaterability of the sludge at the same time [7]. After the treatment, the solid product is always used as a kind of solid fuel or reused for agricultural fertilization after anaerobic digestion. Fig. 1 shows the scheme of the hydrothermal process.

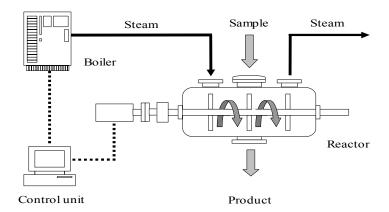


Fig. 1. Hydrothermal treatment process

Comparing with the traditional sewage sludge treatment technologies, hydrothermal treatment showed obvious merits, which were listed in Table 1.

	Thermal drying	Carbonization	Hydrothermal treatment
Merit	No solid lost	Low energy lost	low energy cost No emissions of dioxins, NOx, SOx, dust, etc
Demerit	High energy lost, emissions of dioxins, NOx SOx dust etc.	Loss of mass	Solid lost

Table 1. Comparison with others technologies

However, the liquid phase, which is rich in dissolved organic compounds, is always treated as wastewater or discarded. And on one hand, the expenditure for treating this so-called wastewater is not inexpensive cheap; on the other hand, there are actually plenty of nutrients, such as nitrogen, phosphorus and potassium and so on in the sewage sludge. During the hydrothermal processes, accompanied with the destruction of bacterial cells, a certain amount of these nutrients will also dissolve into the liquid phase. Thus treating the liquid byproduct as wastewater seems regretful. If this so-called wastewater can be recycled and utilized, it could not only solve the wastewater treatment problem of sewage treatment, but also could achieve a huge economical benefit.

Actually, in recent years, the use of the hydrothermal treatment for converting organic wastes into more valuable substances has been investigated. According to the investigation about microalgae, an additional benefit of the hydrothermal processing had been found, which has the potential to recycle liquid byproduct rich in nutrients, as well as other mineral matter and polar organics [8]. The hydrothermal treatment was also demonstrated to be able to produce high yield of amino acid from biomass waste by controlling the reaction temperature and reaction time range [9]. It is reported that fish meat and silk fibroin can be converted into organic acids and amino acids by the hydrothermal treatment [10-11]. As more detailed investigation, hydrothermal conversions of cellulose and disaccharides were also studied and had been found to be readily convertible into glucose and low-molecular-weight carboxylic acids [12-16].

In the application of the hydrothermal treatment to sewage sludge, most of the previous researches have been focused on the improvement of the dewatering performance for solid fuel production [17], or the anaerobic digestion [18]. Nevertheless, due to the complicate components in the sludge, few researches are focused on the nutrient release during the hydrothermal processes. Considering the abundant nutrient concentration in the hydrothermal treated liquid product, it is also possible to make use of it as a liquid fertilizer. In our past research, we found that during the hydrothermal process, a certain amount nutrient could move into the liquid product, and both the reaction temperature and the reaction time are all affecting the nutrient solubilization and the form. Therefore, the fertilizer effect of the liquid product from sewage sludge by the hydrothermal treatment is gaining interest.

This paper presents the results of a study aimed at developing a liquid product from sewage sludge as a kind of liquid fertilizer. For this purpose, the hydrothermal treatment was used for extracting the nutrient from sewage sludge. The presence of nitrogen, phosphorus, potassium and heavy metal contents in the liquid product were analyzed, and a germination experiment was also carried out to evaluate its phytotoxicity. Also, a small scale plant cultivation experiment using Komatsuna was carried out to investigate the liquid product's fertilizer effect. The results are expected to be favorable for utilizing the liquid product obtained from sewage sludge by the hydrothermal treatment process and creating a new component for constructing the systematic hydrothermal theory to use sewage sludge as a resource in a maximum range.

#### 2. EXPERIMENT METHODS

# 2.1 Characteristics of Sludge

In this research, the sewage sludge was obtained from a wastewater treatment facility located in Shimane province, Japan. The nutritional value along with other properties of the sludge is provided in Tables 2 and 3.

Table 2. Characteristics of sewage sludge

рН	Moisture (%)	N (%) db	C (%) db
6.28	85.94	7.2	39.95

Table 3. Nutrient and heavy metal of sewage sludge

Raw	unit	Р	K	S	Na	Mg	Ca	Fe	Al	Mn	Pb	Zn	Cu	Cd
db	mg/g	13.88	1.43	7.60	0.35	1.4	7.44	20.69	8.71	0.16	0.02	0.33	-	-

#### 2.2 Methods

#### 2.2.1 Hydrothermal treatment

In this research, a bench-scale hydrothermal reactor with 0.5L capacity was used. The schematic view of the facility is shown in Fig. 2. The reactor is a batch type (MMJ-500, Japan) which is equipped with an automated stirrer, a pressure sensor and a temperature controller. 60g of sludge (as received based) mixed with 60ml of distilled water, was introduced into the reactor without any pretreatment. After sealing the reactor, the air inside

the reactor was purged by inert gas (argon) to prevent combustion during the treatment. Initial pressure inside was set to near atmospheric. Then, the reactor was heated to the target temperature (180, 200 and 220°C) with the average heating rate of 7°C/min and the constant stirring speed of 100 rpm. The pressures under different temperatures were 1.0 MPa, 1.5MPa and 2.3 MPa respectively. After reaching the target temperature, the mixture was further kept in the reactor for 60 minutes. Once it is completed, the reactor was cooled down (<90°C) and depressurized. Then the treated mixture was taken out and was subjected to centrifugation (3000 rpm, 30 min) for separating the solid and liquid phases. The liquid phase was filtered through sterile analytical filter units (Thermo Scientific, USA), with a membrane (Millipore, Ireland) of 0.2 $\mu$ m pore size and kept at 4°C until analytical measurements.

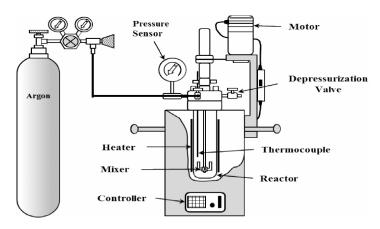


Fig. 2. Schematic view of small-scale hydrothermal treatment reactor

#### 2.2.2 Germination experiment

As the liquid product contains a lot of organic components that are useful for plants, an attractive application is to use it as a fertilizer; however, if the organic components contain toxicity, the liquid product still can not be utilized, therefore, the phytotoxicity test is necessary. In this investigation, a germination experiment was carried out on the liquid product and compared with distilled water [19]. The liquid products obtained under different hydrothermal treatment parameters were diluted to different solutions based on the TOC concentration and the EC value. The detail is shown in Table 3. During the experiment, 10 ml of diluted liquid product was pipetted into a sterile petri dish (Askul, Japan) lined with Tanepita (Fujihira, Japan). Fifty Komatsuna seeds were evenly placed in each dish (three replicates for each sample) and the seeds were incubated at 25°C in the dark at 75% of humidity. Distilled water was used as a control. The seed germination ratio and the root length in each plate were measured after passing 72 hours. The seed germination ratio (SGR) and the mean root length (MRL) were calculated with the following formula [20,21].

$$SGR(\%) = \frac{\textit{Numbers of seeds germinated in the liquid product}}{\textit{Number of all the seeds}} \times 100$$
 
$$MRL = \frac{\textit{sum of all the root length in the liquid}}{\textit{Number of all the germinated seeds}}$$

Table 3. Diluted concentration (based on TOC and EC values)

TOC(ppm)	3500	1500	500	100
EC(ms/cm)	2.5	1.0	0.3	0.06

#### 2.2.3 Plant growth experiment

Nitrogen component is the major nutrient component in the liquid product, and half of the nitrogen is organic nitrogen, so it is possible that the liquid product can be used as a kind of organic nitrogen fertilizer. Therefore, the nitrogen effect to the plant can also be seen as the most important parameter to evaluate the fertilizer value of the liquid product. Komatsuna is a kind of plant which is similar with cress, sensitive to nitrogen treatment and very easy to cultivate in the laboratory. In this experiment, Komatuna was used for evaluate the fertilizer effect of the liquid product. As different fertilizer applications will also affect the growth of Komatsuna, the experiment was set into different groups with different fertilizer quantity to investigate the optimum application.

The soil used for plant growth experiment was taken from Hiratsuka city, Japan, and was sieved through a 2mm sieve before using. Its maximum moisture, the water-holding capacity (based on 1000g dry weight of soil), pH (1:2.5,  $H_2O$ ), EC (electrical conductivity)(1:5,  $H_2O$ ) were 8.76%,396g, 6.75 and 0.08ms/cm respectively. To prevent wash-away of the growth-test materials, the growth of Komatsuna was carried out in Neubauer pots (Fujirika, Japan), and by the addition of water to maintain the moisture content of the pots, the quantity of the water was 50% to 60% of the maximum water-holding volume. In this experiment, 400g soil was prepared and different volume of the liquid product were mixed with soil and put into Neubauer pots. Every pot was arranged so that the total nitrogen, the total phosphorus, and the total potassium became 100mg respectively, which is the best fertilizer proportion and quantity for planting Komatsuna in the Neubauer pots. As the phosphorus and potassium concentrations in the liquid product were very low, to avoid the effect by different phosphorus and potassium levels, calcium superphosphate and potassium chloride were also used for supplement.

Also, to evaluate the organic nitrogen component effect, the nitrogen applications were also divided into 1 time, 2 times and 4 times groups. In the 1 time group, only 25 mg nitrogen was from the liquid product, the other 75 mg was added by ammonium sulfate. In the case of the 2 times group, 50mg nitrogen component was from the liquid product, and in the case of 4 times group, the total nitrogen component was from the liquid product. All the experiments were triplicated. Twenty seeds of Komatsuna were sown in each pot and allowed to germinate and grow under the natural sunlight in a glass house and at a controlled temperature of 25±3°C. Fifty days after sowing, the length of the stem and the width of the leaf were measured for every pot, then the tops of plants were harvested and their fresh weights were measured. The weight of the roots was ignored because it was negligibly small compared with the tops. And then the plants were dried at 60°C to have a stable weight. For comparison, experiment as a control was also carried out, where all of the fertilizers added were chemical fertilizers, containing calcium superphosphate, ammonium sulfate and potassium chloride, respectively. Finally, the nutrient quantity in the dry plant and soil were also analyzed, and the nutrient distribution was evaluated.

#### 2.2.4 Analyses

The total-C (TC) and the inorganic carbon(IC) in the liquid phase was determined by the total organic carbon analyzer (TOC-5000, SHIMADZU), and the total organic carbon (TOC) is

represented by the difference between TC and IC. The content of the organic nitrogen was measured using the Kjeldahl method, and the inorganic nitrogen in the liquid product was determined by using the Bremner's MgO method. The nitrate and nitrite were detected in negligible amount, and therefore omitted in this research. The pH and EC (electrical conductivity)values were measured using glass pH and EC electrodes(HORIBA,JAPAN). The phosphorus(P), sulfur(S) and other heavy metal were determined using the ICP emission spectroscopy (ICPE-9000,SHIMADZU), while potassium (K), calcium (Ca), sodium (Na) and magnesium (Mg) were analyzed by the atomic absorption spectrophotometer (180-50,HITACHI). The total nitrogen (Total-N) contents in the dried plant samples and soil were measured using an automatic high sensitive analyzer (Sumigraph NC-220F, SCAS, Japan). The macro and micro nutrients were analyzed using the same method with the liquid product. Analytical precision was checked against a certified standard material and most samples fell within ±10% of the certified value.

#### 3. RESULTS AND DISCUSSION

## 3.1 Effects of Hydrothermal Treatment on the Release of Nutrient

In this section, the release of nitrogen(N), phosphorus(P), and potassium(K) were investigated. N, P, and K are the three major macro nutrients for fertilizer production. Therefore, the investigation of the effectiveness of the hydrothermal treatment for the dissolutions of the three components to the liquid phase is essential. On the other hand, the organic carbon component, which is solubilized into the liquid product are also detected to evaluate the fertilizer effect. As the pH value and EC value are also very important indexes for the liquid fertilizer, they are also be analyzed. The results are presented in Tables 4 and 5.

Table 4. Basic parameters of the liquid product

Temperature	TOC(ppm)	TN(ppm)	NH <sub>4</sub> <sup>+</sup> (ppm)	TP(ppm)	TK(ppm)	рΗ	EC(ms/cm)
180°C	14000	4204	2125	282	656	6.68	6.87
200°C	13500	4483	2392	205	638	5.92	7.32
220°C	10960	4428	2290	126	460	7.29	9.42

Table 5. Micronutrient concentrations in the liquid product

Element		180°C	200°C	220°C
Na		25.1	27.7	25.65
Mg		2.06	2.13	2.59
Ca		11.49	12.00	13.47
Fe		100.5	122.5	74
Al	(mg/l)	5.45	6.00	2.49
Pb	( )	0.276	0.224	0.184
S		339	363	328
Zn		_	_	_
Mn		_	_	_

Tables 4 and 5 showed the effect of the hydrothermal treatment on the nutrient and heavy metal concentrations in the liquid product. In general, the concentration of the total organic carbon is the highest, and the nitrogen concentration also showed large values in the liquid

product. The nitrogen concentration significantly increased with the temperature increase, while when the temperature was 220°C, the total nitrogen concentration showed a decreasing trend in a relative small range, which indicated that for the same reaction time, a higher reaction temperature seems to able to transfer more nitrogen content from the solid phase to the liquid phase. But when the temperature is higher than 200°C, accompanied with the polysaccharide dissolve and solvolysis [22], a certain amount of celluloses, hemicelluloses or lignin in the solid phase also dissolved, and the volume of the liquid product increased, which also diluted the nitrogen concentration. Some previous studies also demonstrated that for the biomass conversion process in hot-compressed water, the temperature was the most critical parameter for the solubilization [23,24].

The solubilization of phosphorus and potassium showed increase trend by increasing the reaction temperature, but not so obvious, similar with those of nitrogen and carbon. In addition, it is obvious that the concentration of potassium far exceeds the concentration of phosphorus. From the high phosphorus initial content together with the presence of high Fe initial content, it can be inferred that phosphorus was added as flocculants to the sludge, is mainly in the form of insoluble FePO<sub>4</sub> precipitate. As for potassium, because its concentration in the sludge is not high, and it is very easy to dissolve in water, so when high temperature and pressure were applied, it is released simultaneously with the break up of the complex sludge flocculation network and the breakdown of the organic matter. The concentrations of the micronutrients and heavy metals showed very low levels entirely. Therefore, even though the liquid product contains many kinds of elements, including heavy metals, their concentrations are too low to cause harmful effect to the plant. Also, it is well studied that the main composition of sewage sludge is complex organic compounds, and Barlindhaug and Odegaard [25] reported that carbohydrates were easier to be degraded but more difficult to be solubilized than proteins, which indicates that not all organic compounds react in the same way. This also explains why the liquid product showed different pH values under different treatment temperature. The concentrations of N, P and K were around 4000 ppm, 200 ppm and 600 ppm, respectively, the nitrogen concentration was similar as the commercial fertilizers, while the concentrations of P and K were much lower. However, the concentrations of heavy metals were lower than commercial fertilizer, and after dilution, it can be inferred that the concentration will become even lower, which showed more safety to the plant. Particularly, As, Cd, Cu and Ni were not detected, and the concentration of Pb component was also very low. The ratio of NH4<sup>+</sup> also showed that only about half of the nitrogen content is small molecule nitrogen which can be absorbed easily by the plant. However, the huge amount of organic nitrogen also showed a new possibility of the usage of the liquid product as organic fertilizer.

The pH and EC values were measured as soon as possible after each experiment. The results are shown in Tables 4 and 5. For the case of pH value, an obvious difference was observed under different reaction temperatures. In the case of 180°C, the pH value was almost neutral; when it turned to 200°C, the pH value decreased to lower than 6, and then when the reaction temperature was increased to 220°C, the pH value increased to higher than 7. The pH decrease indicates the destruction or transformation of organic matters to organic acids in the liquid product. Under a comparatively low temperature (≤200°C), it is obvious that chemical reactions happened in the dissolved organic matters. With increasing the reaction temperature, more organic acid is produced, and this reaction is exhibited by the decrease of the pH value. Then pH increase could be due to the organic acid decomposition or acidic compounds volatilization. The EC value also increased with the reaction temperature increase, which can be attributed to some dissolved macromolecular organic

compounds decomposed into small and inorganic molecules, and also, as we had discussed before, metals, released into the liquid phase, also caused the EC value increase.

# 3.2 Germination Experiment of the Liquid Product

Fig. 2 shows the germination ratio and Fig. 3 shows the root length as functions of the TOC concentration and the EC value. Generally speaking, both of the germination ratio and the root length tended to increase by increasing dilution. The germination ratio in the distilled water was 98%, all most all of the seeds germinated. In the case of liquid product germination experiments, it showed that the lower the EC value was, the higher the germination ratio was. When the EC value was 2.5 ms/cm, all of the seeds could not germinate. When it was set at 1.0 ms/cm, under all the temperatures, about half of the seeds geminated. In the 180°C and 200°C groups, the germination ratios were 61% and 59%, and in the 220°C group it was 48%, which showed that even if the EC values were the same, the germination ratio were still not the same. As the EC value increased with the increase of the treatment temperature, it is obvious that the treatment temperature also affected the components inside the liquid product, which also caused the different germination ratios. A relatively higher treatment temperature will cause more complex chemical reactions and more ion dissolutions, and we did not measure these dissolved ions because the composition of the sludge itself is too complex to measure. When the EC value was reduced to 0.3ms/cm by dilution, all of the germination ratios increased to about 90%. When the EC value of the liquid product was diluted to lower than 0.1ms/cm, there is no obvious difference between all of the three kinds of liquid product. And we can also see that the liquid produced under the temperature of 220°C showed more harmful effect than 180°C and 200°C. On the other hand, the TOC value also decreased with the dilution of the liquid product, so at the same time, we can also see that a too high TOC concentration is also harmful to the seeds germinating.

The root length also indicated the effect of the liquid product to the plant. Similar with the germination ratio, the root length also increased with the dilution time. Particularly, when the TOC concentration was 500 ppm, nearly all of the average root lengths are the same. When it is diluted to lower than 750 ppm, the three kinds of liquid products showed almost the same effect on the root length. The average root length of the distilled water group was 28 mm and from the figure we can see that a higher EC value or TOC concentration were harmful to the root length, but when the TOC concentration was reduced to lower than 100 ppm by dilution, the liquid product showed promotional effect on the root length compared with the distilled water, which means that the micronutrient may accelerate the growth of the root. Especially, the group with the reaction temperature of 200°C and the TOC concentration of 100 ppm showed obvious promotional effect than the others.

The low germination ratio indicated an adverse effect on the root growth of Komatsuna possibly due to the high concentrations of TOC and high EC values [26-27] which are known to impact the plant [28]. According to this germination experiment criterion, the liquid product required a more than 10 times dilution to reach stabilization of the organic matter to decrease the harmful effect on the plant. At 30 times dilution, the germination ratio was found to be over 80%. On the other hand, the 200°C group phenomenon indicated that proper amount of micronutrients and organic acid will promote the root growth. So, if being used as liquid fertilizer, the liquid dilution times and the components concentration modulation are also very important.

It has been reported that protein, carbohydrate and lipid are the main constituents of the sewage sludge [29]. Sewage sludge was reported to be used as the protein source, because its main constituents are protein and carbohydrate [30]. In this research, the nitrogen concentration of the raw sample is 7.2%, thus, protein was the largest constituent of the sewage sludge. During the hydrothermal process, the concentrations of the protein and carbohydrate in the liquid phase fluctuated with the increase of the reaction temperature. The reason was that soluble protein and carbohydrate were the result of a net balance between competing rates of release and degradation. With the increase of the reaction temperature, the degradation rate exceeded the release, and the concentration was observed to decrease [30]. The technology of using sewage sludge as resource for extracting the bacteria proteins, and then using chemical methods for generating the AACTE fertilizer was also investigated [31]. During anaerobic digestion or thermal processes, propionic acid, formic acid, acetic acid, and other kinds of volatile acids can also be produced [32]. Therefore, in this research, as the reaction happened, the protein and carbohydrate components also dissolved into the liquid product. With increasing the reaction temperature and the holding time, the hydrolysis reaction happened, which caused the change of the composition and the pH value. During the reaction, with the hydrolysis of the protein, more amino acid was produced, which lead the pH value of the liquid product becomes lower. But it is obvious that a higher temperature also caused a further degradation of the amino acid, which resulted in the pH value increasing and the higher ammonium concentration. It is well known that amino acid can promote the plant growth. In this experiment, it is obvious that this amino acid could also promote the root growth. It has been reported that humic acids increase the number of roots thereby stimulating nutrient uptake and plant development [33]. However, what should be attended is, too high TOC concentration, also means the chemical components are also concentrated in the liquid product, if applied to the seed directly, will hurt the seed because of fertilizer burn and the damage of the excessive ions. Therefore, the dilution of the liquid product is necessary.

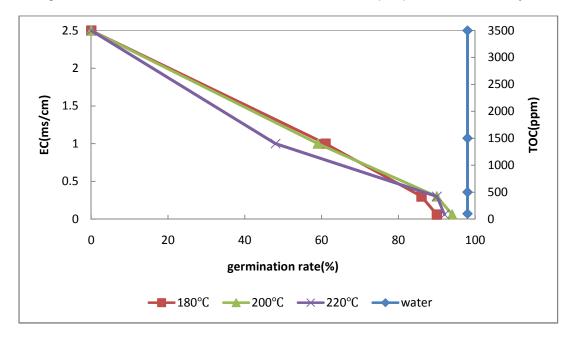


Fig. 2. Effects of TOC and EC on the germination ratio

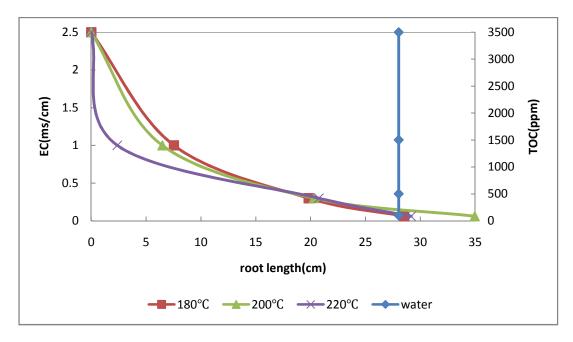


Fig. 3. Effects of TOC and EC on the root length

# 3.3 Fertilizing Value of the Liquid Product

Since the growth of plants and their quality are mainly a function of the quantity of fertilizer, to improve the utilization of the fertilizer nutrients is also very important. Organic matter affects crop growth and the yield directly by supplying nutrients [34], and also can apply a delayed nutrient release. Combined use of organic and inorganic sources of nutrients is essential to augment the efficiency of nutrients [35]. In this section, the yield change of the plant with the different fertilizer application was discussed and the uptakes of the three primary nutrients, N, P and K were also analyzed and compared with chemical fertilizers.

The plant growth condition is described in Tables 6 and 7. The stem length in all the tests were between 12 - 14 cm/plant, there was no significantly difference when the plants were fed with the liquid product, while comparing the 4 times groups under different treatment temperature, we can see that even though the total fertilizer applied were the liquid product, it still showed very good fertilizer effect, almost the same as the chemical fertilizer. In the case of the leaf width, all were centralized around 4cm/plant, and there was still no obvious difference between the chemical fertilizer group and the liquid product group. Also, the plant did not show any pathological features. That is because after the hydrothermal treatment, almost all of the parasites, bacteria were killed by the high temperature and pressure, which made the liquid product cleaner than the raw sludge.

Table 6. The length of the stem

Stem length (cm/plant)	Control	180°C	200°C	220°C
1 time		12.45±1.40	13.35±1.58	13.15±2.96
2 times	12.75±0.86	12.84±1.46	12.92±1.65	12.56±1.19
4 times		12.95±0.85	12.90±0.61	12.23±1.23

Table 7. The width of the leaf

Leaf width (cm/plant)	control	180°C	200°C	220°C
1 time		3.68±0.30	3.88±0.69	3.80±0.87
2 times	3.66±0.43	3.61±0.17	3.70±0.28	3.65±0.38
4 times		3.76±0.19	3.72±0.18	3.76±0.32

Fig. 4 showed the fresh weight of the plant after the harvest. The control group was added with the chemical fertilizer, the others were added with both the liquid product and the chemical fertilizer. We can see clearly that for every pot, the fresh weight of the plant showed difference even though it is not so obvious. As the control group, the average plant weight reached about 33.2g/pot. For the 180°C groups, different liquid product ratio also showed different fertilizer effect. When it was used as 1 time, which means only a quarter of the added fertilizer was the liquid product from the hydrothermal treatment, the fresh weight of the plant was lower than the control group. When it turned to the 180°C groups, 2 times group, the fresh weight showed almost the same value with the control group. And if we added the liquid product as 4 times, which means no nitrogen component was from the chemical fertilizer, it also showed a lower yield than the control group. In the case of 200°C groups, we can also see the similar trend of the yield, the 2 times group still showed superiority than the other times group. The 220°C groups also showed the similar trend with the other groups, even though at this temperature, 4 times group showed the lowest yield. In the case of different treatment temperatures, the same liquid product application time also showed similar yield, the difference was not so obvious, and 200°C, 2 time group showed the best yield, and compared with the control group, it showed almost the same yield.

The plant growth experiment was carried out in the Neubauer pot, which can prevent washaway of the nutrient, but it can not prevent the nutrient loss by volatilization. Therefore, the nutrient uptakes were also analyzed. The nutrient uptakes were shown in Figs. 5 to 7. Fig. 5 showed the nitrogen uptakes. The control group showed that only about 60% of the nitrogen can be uptake by the plant, the other 20% still remain in the soil, and about 20% was lost. As the nitrogen added into the control group was ammonium sulfate, which was a chemical fertilizer that can be taken up easily by the plant, a certain amount of ammonia, after the application, will also volatilized into the environment.

In different test groups, the nitrogen quantities remain in the soil showed different value. For the 180°C groups, in the case of 1 time group, the remained nitrogen quantity is higher than the control group, which showed that a certain amount of organic nitrogen remained in the soil. On the other hand, if we only used chemical fertilizer, only about 60% nitrogen component can be taken up by the plant, which also proved that not all of the nitrogen applied can be used. Usually, plant will take up the inorganic nitrogen first, then when the organic nitrogen was decomposed into low molecular nitrogen that can be used by the plant. With the increase of the liquid product application, the lost nitrogen did not change obviously, and also, the taken-up nitrogen quantity increased a little means that this kind of liquid product can be mineralized effectively during the plant growth. This phenomenon proved that the combined organic and inorganic nutrients can promote the efficiency of nutrients take-up. In the case of 200°C groups, 2 times application showed obvious uptakes. And in the 220°C groups, it also showed the similar trends.

The growth of the plant is not only influenced by the sunlight, water, air, fertilizer and so on, but also, by the characteristics of the fertilizer. The nutrient take up is greatly affected by environmental factors and other growth conditions [36]. And the interactions between ions in

the fertilizer also play an essential role. For some kind of plant, the uptake of total N and K depended on the N form supplied, and the uptake of P, K, Ca and Mg were affected by the pH value. And in different part of the plant it is also different [37]. The fertilizer we used for the test were obtained from the hydrothermal process, and the pH, EC value and composition were also different, which affected the plant growth. For the 180°C groups, the liquid fertilizer we used showed a relatively neutral pH, and the EC value was also lower than the other groups, therefore, the organic component inside the liquid product mainly comes from the dissolution of the big organic molecule. It seems that the organic component can be decomposed and taken up by the plant. For the 200°C and 220°C groups, still 2 times group showed good up-takes, which means 2 times mixing ratio is the best choice for Komatsuna growth. For all of the 4 times groups, it showed obvious inhibiting effect compared with other times, which may due to the high EC values. If we apply 4 times quantity to the plant, it means that we should add more liquid volume, and the water we used for watering every day was decided, which indicated that the more quantity we used, the higher the EC value would become. Too high EC value, which also means the high soluble salt concentration, is not only harmful to the plant, but also affecting the activity of the microorganism inside the soil. Plant growth and soil microorganisms are largely affected by the salt concentration, especially in arid and semi-arid regions, causing serious damages to agriculture [38]. On the other hand, some kinds of microorganism are also very sensitive to the pH value. In this test, the liquid products used in the 200°C groups and 220°C groups were also different. Not only the EC value, but also the pH value and even the components were different, this difference decided the different decomposition rate, which also decided the different mineralization rate of the organic matter. From the test result it seems that all of the tests for phosphorus and potassium uptakes showed no obvious differences. This is not a bad phenomenon because at least we can infer that the phosphorus and potassium from sewage sludge did not give harmful effect on the plant growth.

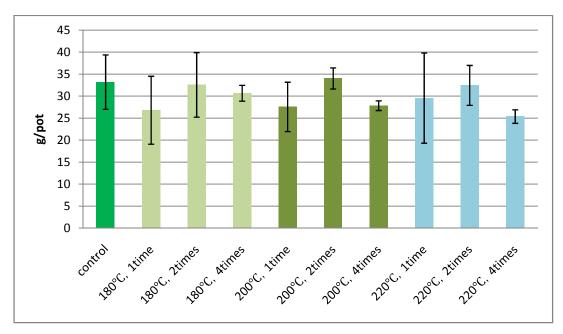


Fig. 4. Fresh weight of the plant

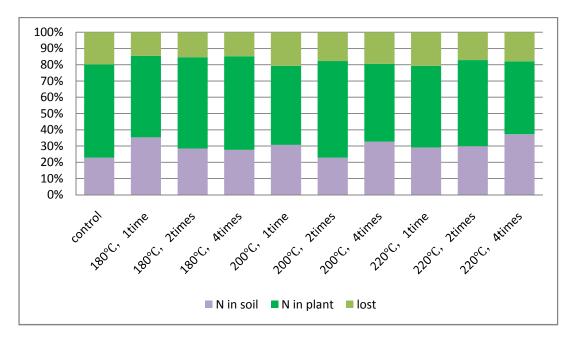


Fig. 5. Nitrogen uptakes of the plant

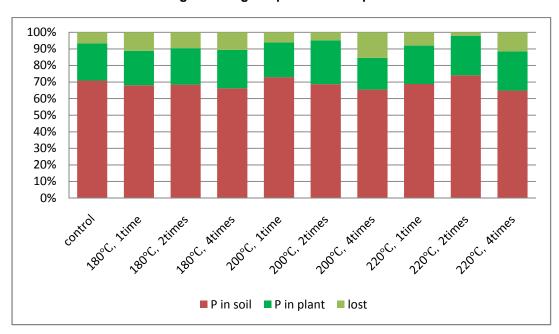


Fig. 6. Phosphorus uptakes of the plant

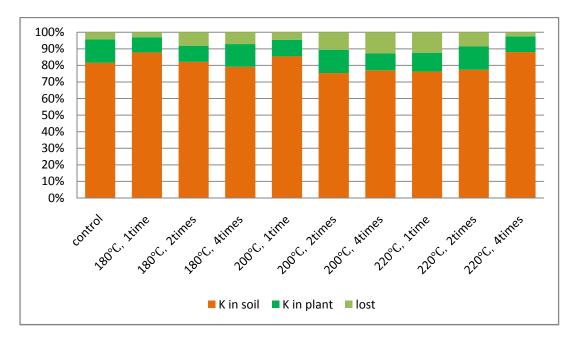


Fig. 7. Potassium uptakes of the plant

## 4. CONCLUSION

This study evaluated the possibility of recycling the liquid product of the hydrothermally treated sewage sludge as a kind of organic fertilizer and its effect on the plant growth. In this study, the application of moderate temperature hydrothermal treatment (180,200,220°C) was used as a new treatment method of nutrient recycling from sewage sludge. It is observable that the hydrothermal treatment is effective to solubilize the sewage sludge, and during the treatment process, a certain amount of macro and micro nutrients can move into the liquid product. Under different treatment temperatures, different kinds of organic and inorganic nutrients were also produced, which is considered to be used for applying nutrient to the plant. Nitrogen and potassium solubilization increased as the reaction temperature was increased, while very little of phosphorus was dissolved. By using different treatment temperature, different kinds of organic and inorganic nutrients are also produced; protein and saccharine components will be decomposed into amino acid and other organic acid, and finally into ammonia. The EC value increased according to the increase of the reaction temperature. Micro nutrients and heavy metal solubilizations are dependent on the reaction temperature, therefore, by controlling the reaction temperature, the quantity of the nutrients that dissolved into the liquid product can also be decided. After the treatment, most of the heavy metals in the sewage sludge would remain in the solid product, and the concentrations of them in the liquid are very low, therefore, after diluting, the concentration become much more lower that will not cause harmful effect to the plant. The germination experiment was conducted for testing the phytotoxicity of the liquid product, and the effects of the TOC concentration and the EC value on the seed germination were investigated. The result showed that the high TOC and EC values were harmful to the plant, but if the liquid product was diluted to the appropriate times, the germination will increase to the common level, therefore, no obvious phytotoxicity was found. The organic acid, which was produced during the hydrothermal process, can promote the growth of the plant root. Moreover, in Komatsuna cultivation experiment, the liquid product showed an enhanced crop yield which was not lower than the chemical fertilizer. The pH and EC values effect on the plant growth, therefore the adjustment of the liquid product composition also should be considered. And by comparing the nutrient distribution, the nitrogen uptake was affected by the liquid composition and concentration, 2 times application is suggested. Through the low-temperature hydrothermal treatment, the sewage sludge was converted into stable organic component that could be used as a delayed-release nitrogen fertilizer for the growth of Komatsuna. The liquid product obtained by the hydrothermal process from sewage sludge showed its wonderful effects on the plants growth and nutrients cycle. Using the liquid product to fertilizers is proposed to be appropriate way to reduce environmental damage and use resources effectively. The successful application of the hydrothermal process liquid product as liquid fertilizer here suggested a potential management of residues from biomass waste by the hydrothermal treatment in industry and provided an efficient fertilizer source for agricultural use.

#### **COMPETING INTERESTS**

Authors have declared that no competing interests exist.

#### REFERENCE

- Zhou Shao-qia, Lu Wei-dong, Zhou Xiao. Effects of heavy metals on planting watercress in kailyard soil amended by adding compost of sewage sludge. Process Safety and Environmental Protection. 2010;263–268.
- DRRSS, Disposal and Recycling Routes for Sewage Sludge. European commission. Office for Official Publications of the European Communities; 2002. Available: http://isbn:92-894-1801-X.
- 3. Tezel U, Tandukar M, Pavlostathis SG. Anaerobic biotreatment of municipal sewage sludge. In: Moo-Young M, Editor-in-Chief. Comprehensive biotech- nology, 2<sup>nd</sup> ed. In: Agathos S, editor. Environmental biotechnology and safety. Amsterdam, The Netherlands: Elsevier. 2011;6.
- 4. Day AD, Katterman FRH. Sewage sludge provides plant growth factors inarid environments. J Arid Environ. 1992;23:229–233.
- 5. Ahlberg G, Gustafsson O, Wedel P. Leaching of metals from sewage sludge during one year and their relationship to particle size. Environ Pollut. 2006;144:545–553.
- 6. Neyens E, Baeyens J. A review of thermal sludge pre-treatment processes to improve dewaterability. Journal of Hazardous Materials. 2003;98:51–67.
- 7. RuiXun, Wei Wang, Wei Qiao, Keqing Yin. Status of urban sludge treatment and hydrothermal reduction technology of enhanced dewatering. Environmental Sanitation Engineering. 2008;16(2):28-32.
- 8. Ross AB, Biller P, Kubacki ML, Li H, Lea-Langton A, et al. Hydrothermal processing of microalgae using alkali and organic acids. Fuel. 2010;89:2234–2243.
- 9. Hongbin Cheng, Xian Zhu, Chao Zhu, Jing Qian, Ning Zhu, et al. Hydrolysis technology of biomass waste to produce amino acids in sub-critical water. Bioresource Technology. 2008;99:3337–3341.
- 10. Hiroyuki Yoshida, Masaaki Terashima, Yohei Takahashi. Production of organic acids and amino acids from fish meat by sub-critical water hydrolysis. Biotechnology Progress. 1999;15:1090–1094.
- 11. Kil-yoon Kang, Byung-Soo Chun. Behavior of hydrothermal decomposition of silk fibroin to amino acids in near-critical water. Korean J Chem Eng. 2004;21:654–659.

- Jung Hoon Park, Sang Do Park. Kinetics of cellobiose decomposition under subcritical and supercritical water in continuous flow system. Korean J Chem Eng. 2002;19:960-966
- 13. Mitsuru Sasaki, Bernard Kabyemela, Roberto Malaluan, Satoshi Hirose, Naoko Takeda, et al. Cellulose hydrolysis in subcritical and supercritical water. Journal of Supercritical Fluids. 1998;13:261–268.
- Mitsuru Sasaki, Zhen Fang, Yoshiko Fukushima, Tadafumi Adschiri, Kunio Arai. Dissolution and hydrolysis of cellulose in subcritical and supercritical water. Ind Eng Chem Res. 2000;39:2883–2890.
- 15. Toshinobu Oomori, Shabnam Haghighat Khajavi, Yukitaka Kimura, Shuji Adachi, Ryuichi Matsuno. Hydrolysis of disaccharides containing glucose residue in subcritical water. Biochemical Engineering Journal. 2004;18:143–147.
- Quitain AT, Faisal M, Kang K, Daimon H, Fujie K. Low-molecular weight carboxylic acids produced from hydrothermal treatment of organic wastes. J Hazard Mater. 2002;93:209–220.
- 17. Zili Jiang, Dawei Meng, Hongyan Mu, Yoshikawa Kunio. Study on the hydrothermal drying technology of sewage sludge. Science China Technological Sciences. 2010;53:160-163.
- Christopher A. Wilson, John T. Novak. Hydrolysis of macromolecular components of primary and secondary wastewater sludge by thermal hydrolytic. Water research. 2009;43:4489-4498.
- Wong JWC, Mak KF, Chan NW, Lam A, Fang M, Zhou LX, Wu QT, Liao XD. Cocomposting of soybean residues and leaves in Hong Kong. Bioresour Technol. 2001;76:99–106.
- 20. Zucconi F, Forte M, Monaco A, Beritodi M. Biological evaluation of compost maturity. Biocycle. 1981;22:27–29.
- 21. Hoekstra NJ, Bosker T, Lantinga EA. Effects of cattle dung from farms with different feeding strategies on germination and initial root growth of cress (*Lepidium sativum* L.). Agric Ecosyst Environ. 2002;93:189–196.
- 22. Mok WSL, Antal MJ. Uncatalyzed solvolysis of whole biomass hemi-cellulose by hot-compressed water. Ind Eng Chem Res. 1992;31:1157–1161.
- 23. Neyens E, Baeyens J. A review of thermal sludge pre-treatment processes to improve dewaterability. Journal of Hazardous Materials. 2003;98:51–67.
- 24. Brooks RB. Heat treatment of sewage sludge. Water Pollut Control. 1970;69:221–231.
- 25. Barlindhaug J, Odegaard H. Thermal hydrolysis for the production of carbon source for denitrification. Water Sci Technol. 1996;34:371–378.
- 26. Wong MH. Phytotoxicity of refuse compost during the process of maturation. Environ Pollut. 1985;40:127–144.
- 27. Fang M, Wong JWC. Effects of lime amendment on availability of heavy metals and maturation in sewage sludge composting. Environ Pollut. 1999;106:83–89.
- 28. Fuentes A, Llorens M, Saez J, Aguilar M, Ortuno J, Meseguer V. Phytotoxicity and heavy metals speciation of stabilized sewage sludges. J Hazard Mater. 2004;108:161–169.
- 29. Tanaka S, Kobayashi T, Kamiyama K, Bildan MN. Effects of thermochemical pretreatment on the anaerobic digestion of waste activated sludge. Water Sci Technol. 1997;35(8):209–215.
- 30. Yinguang Chen, Su Jiang, Hongying Yuan, Qi Zhou, Guowei Gu. Hydrolysis and acidification of waste activated sludge at different pHs. Water Research. 2007;41:683–689.

- 31. Liu Y, Kong S, Li Y, Zeng H. Novel technology for sewage sludge utilization: preparation of amino acids chelated trace elements (AACTE) fertilizer. Journal of Hazardous Material. 2009;171(1–3):1159–67.
- 32. Rulkens WH. Sustainable sludge management-what are the challenges for the future, Water Science and Technology. 2004;49(10):11–9.
- 33. Alvarez R, Grigera S. Analysis of soil fertility and management effects on yields of wheat and corn in the rolling pampa of Argentina. Journal of Agronomy and Crop Science. 2005;191:321–329.
- 34. Darwish OH, Persaud N, Martens DC. Effect of long-term application of animal manure on physical properties of three soils. Plant Soil. 1995;176;289–295.
- 35. Lian S. Combined use of chemical and organic fertilizer. Technical bulletin No.11. University Pertanian, Malaysia and Food and Fertilizer Technology Centre, Taiwan. 1994;237.
- 36. Ambe S, Shinonaga T, Ozaki T, Enomoto S, Yasuda H, Uchida S. Ion competition effects on the selective absorption of radio nuclides by komatsuna (*Brassica rapa* 6ar. per 6iridis). Environmental and Experimental Botany. 1999;41:185–194.
- 37. Arunothai Jampeetong, Dennis Konnerup, Narumol Piwpuan, Hans Brixc. Interactive effects of nitrogen form and pH on growth, morphology, N uptake and mineral contents of *Coixlacryma-jobi* L. Aquatic Botany. 2013;111:144–149.
- 38. Michele Xavier Vieira Megda. Chloride ion as nitrification inhibitor and its biocidal potential in soils. Soil Biology & Biochemistry. 2014;72:84-87.

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