## Feasibility of Recycling Residual Solid from Hydrothermal Treatment of Excess Sludge

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#### Abstract

Residual solid in excess sludge treated by hydrothermal reaction was investigated as raw material for its recycling. Treated excess sludge and residual solid were also focused on their content change during hydrothermal reaction. Two kinds of excess sludge, obtained from a local food factory and a municipal wastewater treatment process, were tested under various conditions. Following hydrothermal reaction, depending on the reaction conditions, biodegradable substrates in treated excess sludge appeared to increase. The separated residual solid was a composite composed of organic and inorganic materials. The proportion of carbon varied from 34.0 to 41.6% depending on reaction conditions. Although 1.89% of hazardous materials were detected, SiO<sub>2</sub> (Quartz) was a predominant constituent of the residual solid. X-ray diffraction (XRD) experiments revealed that the residual solid was of a partially amorphous state, suggesting that the residual solids could be easily converted to stable and non harmful substances through a stabilization process. Thus, this technology could be successfully used to control excess sludge and its reuse.

Keywords: Biodegradability improvement, Excess sludge, Hydrothermal reaction, Residual solid, Resource recovery

#### 1. Introduction

Control of excess sludge discharged from conventional biological treatment processes is an urgent environmental problem. <sup>1,2)</sup> Numerous methods have been considered to address this issue. <sup>3-9)</sup> However, dealing with secondary pollutants and incinerator slag still remain a challenge in the field of sludge disposal. Sludge ocean disposal and landfill are currently prohibited in many countries and the associated regulations are becoming increasingly stricter. Potential alternatives to disposal should be developed with consideration given to sludge disposal and reactant reuse following treatment.

The hydrothermal reaction approach has been attracting attention,  $^{10)}$  due to the unique characteristics of water as a reaction medium under conditions near its critical points ( $T_{Critical}$ =374.2°C,  $P_{Critical}$ =22.1 MPa). Hydrothermal reaction processes have been used for degradation of refractory pollutants, toxic chemicals, and wastes.  $^{11-13)}$  Resource recovery from wastes under subcritical water conditions has been reported.  $^{14,15)}$  This method

has not been studied sufficiently, as a method for partial destruction of refractory pollutants, for enough to be known of its potential to improve their biodegradability, with the exception of our previous related research on treatment of chloroacetic acids<sup>16)</sup> and poly vinyl alcohol.<sup>17)</sup> Hydrothermal reaction is very useful for altering the content of excess sludge without causing much reduction of carbon.<sup>18)</sup>

The objectives of this research were to investigate the contents of residual solids in excess sludge treated by a hydrothermal reaction and to evaluate the possibility of recycling the residual solids for production of non-harmful and stable substances.

#### 2. Materials and Methods

#### 2.1. Materials

Excess sludge obtained from a local food factory was used as a raw material due to its high organic content. Generally, inorganic matter was not affected by the hydrothermal reaction. Excess sludge originating from a municipal wastewater treatment process was also used to demonstrate the general effects of the hydrothermal reaction on excess sludge. Activated sludge of an aerobic reactor in the wastewater treatment process was

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used as the seeding microorganism for water quality analysis and respirometric tests. All reagents and standard solutions were purchased from Wako Pure Chemical Industry, Ltd. (Osaka, Japan) and were used for the evaluation of products obtained from the hydrothermal reaction and respirometric tests. All chemicals were HPLC grade or a 99% purity grade.

#### 2.2. Experimental Apparatus

The hydrothermal reaction treatment was carried out using a batch reactor apparatus (TSC-006, Taiatsu Glass Corp.). The apparatus consisted of a stirrer, a pressure gauge, a reactor and a molten salt bath containing a mixture of potassium nitrate and sodium nitrate. The batch reactor was made up of hastelloy C22 (Ni, Cr, Mo alloy) and had a total inner volume of 65.9 cm<sup>3</sup>. The maximum operational conditions of the reactor were 450°C and 45 MPa. The apparatus took 7 min to reach a temperature of 400°C and heating time was dependant on the desired reaction temperature. The state of th

#### 2.3. Experiment

Prior to experimentation, the two kinds of excess sludge were gravitationally settled for 1 h until reaching approximately 22 000 mg suspended solids/L. The thickened sludge was cleaned and separated to obtain pure sludge by centrifugation (RS-206, Tomy Corp.) repeated twice at 5 000 rpm for 5 min. After each centrifugation, the liquid phase was replaced by dechlorinated tap water. Next, the two kinds of sludge were used as raw materials for hydrothermal reaction treatments conducted at various temperatures (200-400°C) and pressures (1.6-30 MPa). The reaction times were defined as the elapsed time from the moment that 90% of each reaction temperature was reached 16,17) until the end of treatment. The desired pressure was obtained by adjusting the initial sample amount. During each hydrothermal reaction, reaction pressures were verified from the pressure gauge of a batch reactor apparatus. In each reaction, sample was placed into a reactor. The reactor was sealed, and then the air inside was purged using pure nitrogen gas. The reactor was placed in a preheated molten salt bath for the desired reaction time. All reactions were quenched by placing the reactor in a water bath.

#### 2.4. Analytical Methods

Total organic carbon (TOC) and dissolved organic carbon (DOC) were measured with a TOC analyzer (TOC-5000A, Shimadzu Corp.). Chemical oxygen demand (COD<sub>Cr</sub>) was analyzed with a COD analyzer consisting of a COD reactor (P/N 45600-00, HACH Corp.) and a spectrophotometer (DR/3000, HACH Corp.). Biochemical oxygen demand (BOD) was determined with a BOD tester (BOD Tester 200F, Taitec Corp.). Suspended solids (SS) and volatile suspended solids (VSS) were analyzed following the methods described in a book, "Standard methods for the examination of water and wastewater". <sup>19)</sup> Before analysis of DOC, all samples were filtered to separate solid mate-

rials, using syringes and filters with pore size of 0.45 µm. The fraction of organic content was determined based on a respirometric test conducted following the methods reported by Kappeler and Gujer<sup>20)</sup> and Henze et al.<sup>21)</sup>

The residual solid was prepared for analysis using the following protocol: Excess sludge treated by hydrothermal reaction was settled for 30 min. The separated (settled) substances were dried in a drying oven at  $105^{\circ}$ C for 12 hr, then cooled in a desiccator for at least 30 min, or until the mass stabilized. The residual solid was weighed and 2-3 g aliquots were placed in 100 mL test tubes. De-ionized water (50 mL) was added to each test tube and the suspension mixed for 1 hr in an ultrasonic bath. The aqueous phase was separated by centrifugation at 300 rpm for 20 min. The separated aqueous phase was filtered using filter paper (Advantec, 150 mm×1.2 µm) and the volume brought to 100 mL using de-ionized water. The first prepared liquid phase was analysed for water soluble contents by inductivity coupled plasma (ICP) - atomic emission spectrometry (AES) (Optima 3300DV, Perkin Elmer Inc.).

The used filter papers were air-dried in a laboratory hood for later reuse. The test tube containing the insoluble phase was dried in a drying oven and cooled in a desiccator until the mass stabilized. Aqua regia (40 mL) was added and the test tube was placed into a laboratory hood for 1 day. The acid soluble liquid phase was filtered using the dried filter papers and diluted to 250 mL using de-ionized water. The second prepared liquid phase was analysed by ICP-AES for acid soluble contents.

The used filter paper was again dried in a laboratory hood, and cooled in a desiccator until the mass stabilized. The re-dried filter paper was put into an alloy pot. It was heated until the mass stabilized following carbonization by using a gas burner. Then 1 mL of sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) and 5 mL of hydrofluoric acid (HF) were added to the pot. The pot was heated again until the mass stabilized. During this procedure, the mixture of residuals dissolved in sulphuric acid and hydrofluoric acid was evaporated until only white solid remained in the pot. This reaction removed silicon dioxide (SiO<sub>2</sub>) in residual solid as it reacted with hydrofluoric acid then disappeared as silicofluoric acid gas (SiF<sub>4</sub>). The difference in weight of the alloy pot, before and after the acids were added corresponded to the amount of silicon dioxide.

For the next analysis, 1.5 g of sodium carbonate ( $Na_2CO_3$ ) was placed in the alloy pot which was then heated until the contents were mixed thoroughly. Following the procedure, the outside wall of the alloy pot was cleaned using hydrochloric acid (HCl) diluted with de-ionized water (50% w/v). Then the diluted hydrochloric acid was added to the alloy pot until the contents in the pot were solved completely. For the last step, liquid phase from the alloy pot was filtered with filter paper, diluted to 200 mL using de-ionized water, and analysed by ICP-AES. The total contents of residual solid were the sum of the results of ICP-AES at each step.

X-ray diffraction (XRD) of the residual solid was tested to clarify its composition and structure using an XRD analyzer (X-ray diffractometer, Rigaku Co.). The organic contents of the residual solids were determined by an element analyzer (CHN Corder MT-6, Yanaco Co.).

#### 3. Results and Discussion

# 3.1. Relationship between Content Change and Original Composition of Excess Sludge under Various Hydrothermal Reaction Conditions

Following the hydrothermal reaction, the fraction of organic content in each of the treated excess sludge samples was determined using the respirometric test. The results of these analyses are shown in Fig. 1 and 2, for the excess sludge obtained from the food factory and the municipal wastewater treatment facility, respectively. Slowly biodegradable substrate (X<sub>S</sub>) was defined as the BOD value. <sup>18)</sup> Excess sludge obtained from the

food factory (Fig. 1) was converted primarily to biodegradable substrates ( $S_s$ ,  $X_s$ ) and non-degradable substances ( $S_t$ ,  $X_t$ ) depending on reaction conditions. Solid non-degradable substances ( $X_t$ ) appeared to decrease at temperatures higher than 350°C. The proportion of biodegradable substrates in treated excess sludge increased with increasing reaction temperatures. And this trend that was not much different with increasing reaction times, was already reported by our previous research. Due to its high organic content, excess sludge obtained from the food factory was more than 70% mineralized by thermal decomposition at 400°C. Solubilization apparently increased throughout the hydrothermal reaction. Following the initial change, solubilization did not vary much from results obtained at 250°C, regard-

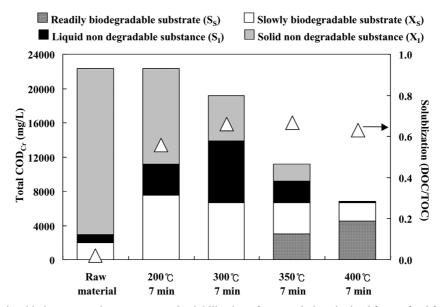


Fig. 1. Relationship between carbon contents and solubilization of excess sludge obtained from a food factory under hydrothermal reaction conditions.

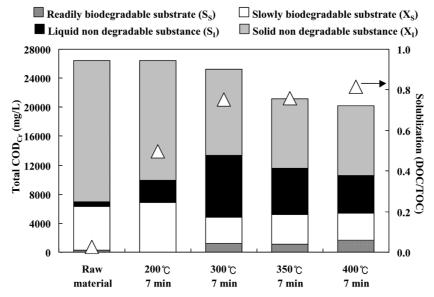


Fig. 2. Relationship between carbon contents and solubilization of excess sludge obtained from a municipal wastewater treatment process under hydrothermal reaction conditions.

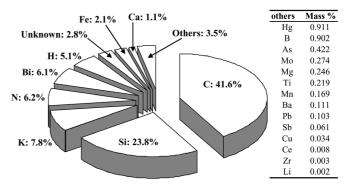
less of changes in reaction temperature.

Except for the presence of readily biodegradable substrates (S<sub>S</sub>) in the raw material, the content change of excess sludge obtained from the municipal wastewater treatment process corresponded to the results of excess sludge obtained from the food factory (Fig. 1 and 2). That is, in both cases, the proportion of resultant biodegradable substrates appeared dependent on reaction conditions. Throughout the hydrothermal reaction, solubilization did not differ between the two sample, despite the difference in carbon contents. In the case of excess sludge originating from the municipal wastewater treatment process (Fig. 2), the proportion of non-degradable substances (S<sub>I</sub>, X<sub>I</sub>) was very high and mineralization was less than 20%. This could be explained by the presence of high amounts of inorganic materials, such as sand, as is common for excess sludge originating from municipal wastewater treatment sources. These results were more representative of an actual case than those obtained using excess sludge from the food factory.

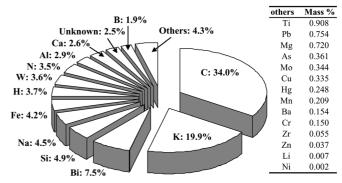
The above results indicate that the content change of excess sludge by hydrothermal reaction was dependent on its original composition. In particular, readily biodegradable substrates (S<sub>S</sub>) and slowly biodegradable substrates (X<sub>S</sub>) were produced from non-degradable substances (S<sub>I</sub>, X<sub>I</sub>) despite the difference in original excess sludge composition. The newly formed biodegradable substrates could be used as carbon sources in a bioreactor as shown by our previous research. <sup>18)</sup> To increase biodegradable substrates is important for reactant reuse. However, mineralization and solubilization of excess sludge from the municipal wastewater treatment process were not much different at temperatures higher than 250°C. For these reasons, hydrothermal reaction of excess sludge should be adjusted between the temperatures of 250°C (4 MPa) and 350°C (17 MPa).

#### 3.2. Contents of Residual Solid in Treated Excess Sludge

In order to determine the contents of residual solid, excess sludge from the municipal wastewater treatment process was treated at 250°C and 350°C for 7 min. Then the residual solid was separated and evaluated. The contents of residual solid are represented in Fig. 3. The contents were a composite of organic and inorganic materials. The proportion of carbon, mainly organic materials, was decreased from 41.6% to 34.0% with increasing reaction temperatures. The contents of composite material were very different compared to incineration slag which consists of inorganic materials.<sup>23)</sup> Under saturated vapor pressure, ion product of water increases with increasing reaction temperature from its normal value ( $K_W=10^{-14.0}$ ) at room temperature and pressure and then reaches its maximum value (K<sub>W</sub>=10<sup>-11.0</sup>) around 250°C. 17) Hydrothermal reaction around the temperature condition of 250°C is characterized by hydrolysis induced from the high ion product of water as shown by our previous research. <sup>17)</sup> That is the reason why the degree of mineralization at the temperature condition of 200°C was low as shown in Fig. 1 and 2. Due to insufficient thermal decomposition, the content of silicon (Si) in residual solid was higher than that of the temperature condition of 350°C. Although the ion product of water dramati-



(a) Contents of residual solid in excess sludge treated at  $250^{\circ}$ C and 4 MPa for 7 min



(b) Contents of residual solid in excess sludge treated at 350°C and 17 MPa for 7 min

	Carbon	B:	Boron	Hg:	Mercury	Cu:	Copper	Al:	Aluminium
H:	Hydrogen	Bi:	Bismuth	As:	Arsenic	Zr:	Zirconium	W:	Tungsten
N :	Nitrogen	Ti:	Titanium	Mo:	Molybdenum	Li:	Lithium	Fe:	Iron
Si:	Silicon	Ba:	Barium	Mg:	Magnesium	Cr:	Chromium	Pb:	Lead
K :	Potassium	Na:	Sodium	Mn:	Manganese	Zn:	Zinc	Ni:	Nickel
Ca:	Calcium								

Fig. 3. Variation in the specific contents of residual solid depending on hydrothermal reaction conditions.

cally decreases approaching to its critical point (K<sub>w</sub>=10<sup>-15.8</sup> at 374°C), a fast reaction rate and thermal decomposition induced from high temperature conditions play important roles with an increasing reaction temperature. 17) For these reasons, the carbon fraction was decreased by thermal decomposition to a greater extent with increasing reaction temperature. Besides, the content of each element in residual solid material were varied throughout hydrothermal reaction treatment at each reaction condition. However, the kinds of elements in residual solid material were relatively similar upon treatment under the two conditions. Residual solid material resistant to thermal decomposition and hydrolysis under severe conditions remained throughout the hydrothermal reaction. The presence of this residual solid material indicates that the substance could not react any further under high temperature conditions without the addition of an oxidant. In particular, silicon (Si) was one of the predominant materials in the residual solid found at both temperatures tested. It could be explained by the original composition of sludge and the characteristics of sludge at high temperature reaction.<sup>24-26)</sup> Unfortunately, 1.89% of hazardous materials were also detected at the temperature condition of 350°C (Table 1). Since sludge commonly contains heavy metals<sup>27)</sup> which are stable at hydrothermal reaction conditions, heavy metals remained in residual solid mate-

Table 1. Hazardous materials in residual solid following hydrothermal reaction at 350°C, 17 MPa in 7 min

Specification	As	Cr	Cu	Hg	Pb	Zn
Total amount in	3,610	1,500	3,350	2,480	7,540	370
Residual solid	(0.361%)	(0.150%)	(0.335%)	(0.248%)	(0.754%)	(0.037%)

(unit: mg/kg, dry-weight)

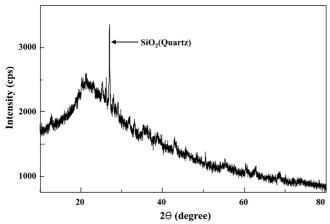


Fig. 4. Unique peak of SiO<sub>2</sub> (Quartz) obtained by X-ray diffraction.

rial followed by hydrothermal reaction. However, the detected water soluble contents of the hazardous materials were only 0.41 mg/L (As), 0.00 mg/L (Cr), 0.02 mg/L (Cu), 0.60 mg/L (Hg), 0.07 mg/L (Pb) and 1.48 mg/L (Zn), respectively. These results did not vary much at the temperatures tested. These data suggest that the residual solids could not be directly reused without any further stabilization process, due to the water soluble and inherent hazardous contents.

The results of the XRD analysis are shown in Fig. 4. There was only one detected peak which was an exact match for the structure of  $SiO_2$  (Quartz). These results were in direct agreement with the results shown in Fig. 3. Noise-like broadened peaks were also detected suggesting that the residual solid was composed of a partially amorphous state. For these two reasons,

we proposed that residual solid in excess sludge treated by hydrothermal reaction might be easily converted to stable substances such as construction materials through additional glassification or sintering. <sup>23,28,29)</sup>

### 3.3. Residual Solid Recovery and Hydrothermal Reaction Coupled with Biological Treatment Process

Fig. 5 represents the proposed flow of excess sludge recycling following hydrothermal reaction coupled with a conventional biological treatment process. Hydrothermal reaction of excess sludge could reduce the volume of solids, and improve biodegradability and solubilization. After hydrothermal reaction treatment, most of treated excess sludge could be used as carbon sources in a bioreactor, except only residual solid, mainly solid non degradable substances. The proportion of residual solid could be varied from 20% to 40% depending on the original composition of excess sludge and hydrothermal reaction conditions, as described in Fig. 1 and 2. The remainder of the residual solid might be converted to useful resources through a stabilization process as mentioned in the previous section. In the separated aqueous phase, there was a lot of phosphate, presumably because of the fracture of polyphosphate-accumulating organisms (PAO) in excess sludge during hydrothermal reaction. Phosphate in liquid phase could be easily separated using the method reported by Shimamura et al.<sup>30)</sup> Various additional methods for phosphorus recovery have recently been reported. 31,32) Hydrothermal reaction coupled with a conventional biological treatment process might secure control of excess sludge, phosphorus

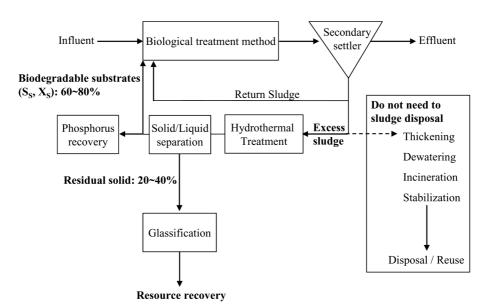


Fig. 5. Proposed recycling flow of excess sludge treated by hydrothermal reaction coupled with conventional biological treatment methods.

recovery, improvement of biological treatment processes and residual solid reuse.

#### 4. Conclusions

The following conclusions were drawn based on the results of this research.

- 1. Hydrothermal reaction of excess sludge represented similar tendency regardless of the source of excess sludge. The amount of each carbon content in treated excess sludge varied depending on its original composition.
- 2. Mineralization and solubilization of excess sludge did not vary much at temperatures higher than 250°C (4 MPa), except to increase biodegradable substrates.
- Residual solid treated by hydrothermal reaction was a composite material composed of organic, inorganic and hazardous materials.
- Since residual solid was composed of partially amorphous state and contained lots of SiO<sub>2</sub> (Quartz), a subsequent stabilization process such as glassification could be easily conducted.

Further investigation is also required to evaluate energy and material flow in a scaled-up plant setting, and to apply this technology to real excess sludge flow.

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