DEWATERING OF ELECTROPLATING SLUDGE USING DIMETHYL ETHER

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The suitability of using dimethyl ether (DME) in dewatering coal for the treatment of sewage sludge has been found empirically to be highly effective. We investigated the effectiveness of a method involving the use of DME for dewatering electroplating sludge and tried to clarify the mechanism and characteristics of the dewatering process. Two zinc-containing sludge samples were examined and the effect of the volume of liquefied DME on the dewatering process was assessed. During the initial dewatering phase, the dewatering ratio is dependent only on the saturation degree of water in liquefied DME. In the later phase, it depends proportionally on the water concentration in the sludge and has a pattern that reduces the dewatering rate as dewatering is proceeded. Moreover, the dewatering pattern can be expressed using one dewatering rate constant, k .

The water contained in the sludge samples was analyzed using thermogravimetry/differential thermal analysis and the evaporation temperature range was determined. The water remaining in the samples after dewatering was found to be thermally stable and tightly bound. We further determined the amounts of total carbon and metal elements in the dewatered water and compared the concentrations to those specified in the Japan Water Pollution Quality Control Act. An important observation is that the dewatered water from the samples requires further processing before it can be discharged into the drainage system due to the presence of large amounts of Cu, Pb and Zn.

Key Words : *electroplating sludge, dewatering, dimethyl ether, normal temperature, TG-DTA*

1. INTRODUCTION

In realizing the transformation into a sound material-cycle society, waste management and energy management pose major challenges. In Japan in 2006, the total discharges of municipal solid waste and industrial waste were 52.0 and 420 million tons, respectively^{1), 2)}. The total discharge of sludge in the country was 190 million tons, which accounted for 44.3% of the industrial waste. The sludge was in the form of a slurry and had high water content; its volume reduction ratio in intermediate treatment was the highest among industrial wastes³⁾. Owing to the immense energy required for evaporating the water, a demand exists for an energy-saving dewatering technology.

The 75 million tons of sewage sludge produced an-

nually in Japan has recently been regarded as a carbon-neutral biofuel. Studies are being carried out to convert the sewage sludge into fuel by dewatering, drying and gasifying^{4), 5)}.

The electroplating sludge produced by an electroplating wastewater treatment process is inorganic. At plating plants, the sludge is dewatered down to about 70%–80% of the water content by using methods such as the filter press, and it is finally disposed of or used as soil backfill. The different types of electroplating include gold plating, silver plating, chrome and nickel plating for rolled material, and zinc plating for nuts and bolts. About 65,000 tons of electroplating sludge is produced in Japan every year⁶⁾. Since the cost of transporting and processing electroplating sludge is determined by the weight inclusive of water, the re-

duction of water in sludge results in a decrease in the cost of final disposal or resource recycling.

Various energy-saving dewatering methods are being examined in the field of thermal power generation. The objective is to improve the power generation efficiency by dewatering high-moisture coal (subbituminous coal and brown coal), which accounts for half of the world's extractable coal reserves, and to enhance the coal's combustibility. Classical techniques involving high energy consumption include dewatering with supercritical CO_2 ⁷⁾, in addition to the following methods: flash drying, steam tube drying, steam fluidized bed drying, mechanical thermal expression⁸⁾, hydrothermal drying, the Koppelman process⁹⁾, an upgraded brown coal process¹⁰⁻¹⁷⁾, the Syncoal technique¹⁸⁾, and the Encoal technique¹⁹⁾.

Recently, a dewatering process for high-moisture coal has been developed that uses liquefied dimethyl ether (DME) as the water extracting agent²⁰⁾. DME is the simplest ether and exists in the vapor state at normal temperature and pressure. Its normal boiling point is -25°C and it can be liquefied at 0.59 MPa, even at normal temperature. Because DME does not produce sulfur oxides or soot when burned and the volume of nitrogen oxides produced can be markedly reduced, its environmental impact is quite small. In addition, it causes no greenhouse effects and does not affect the ozone layer; thus, its effect on ecological systems is very small. It therefore has many uses such as fuel for diesel vehicles and power plants; it is also used as a substitute for liquid petroleum gas, especially in China²¹⁾.

The DME dewatering method is as follows. First, high-moisture coal and liquefied DME are mixed. Approximately 7–8 g of water is extracted when 100 g of liquefied DME is used²²⁾. The liquid mixture of liquefied DME and water is then separated from the dewatered coal. By decompressing the mixture at normal temperature, only the DME is evaporated. The water then can be extracted in liquid form. The evaporated DME is liquefied by compressing or cooling and can be reused. Theoretical calculations show that the energy consumed in this method is half of that required in the classical techniques²³⁾, and its prototype is in operation²⁴⁾.

The DME dewatering method is also being studied to see whether it can be used for the treatment of sewage sludge. Sewage sludge has a water content of 80% even after mechanical dewatering and a huge amount of energy is required to evaporate the remaining water. Even sewage sludge, however, can be dewatered to a considerable extent by the DME dewatering method.^{25), 26)} However, no studies have assessed the dewatering of electroplating sludge using DME. Since sludge properties vary with the type of sludge, the dewatering characteristics of sludge and

the quality of the dewatered water using liquefied DME are expected to differ between sewage sludge and electroplating sludge. We investigated the use of the DME dewatering method for the treatment of zinc-containing electroplating sludge and assessed its effectiveness.

2. EXPERIMENTAL

(1) Equipment and methods

A schematic diagram of the equipment used in the dewatering experiment performed in this study is shown in Fig. 1.

It consisted of four main parts: a liquefied DME storage tank (100 cm^3 , TVS-1-100; Taiatsu Techno Corp., Saitama, Japan), a dewatering column (10 cm^3 , HPG-10-5; Taiatsu Techno Corp.), a liquid sampling tank (100 cm^3 , HPG-96-3; Taiatsu Techno Corp.) and a trap column (10 cm^3 , HPG-10-5; Taiatsu Techno Corp.). These parts were connected serially.

The temperature of the sludge is about 20°C , and the amount of sludge packed into the dewatering column is 1.00 g-wet. The sludge is in the form of spheres with diameters of 4.0–5.6 mm, and the packed sludge contains 12–16 spheres.

Liquefied DME was produced by cooling gaseous DME (Pure; Sumitomo Seika Chemicals Co., Ltd., Osaka, Japan) with ethanol and ice and stored in the liquefied DME storage tank. Liquefied DME was not flowed into the dewatering column until the temperature of liquefied DME in equilibrium with room temperature. Therefore it considered that temperature in dewatering column is about 20°C . And then, under predetermined conditions and nitrogen gas flow at a pressure of 0.7 MPa, liquefied DME was pushed out and transferred to the dewatering column, which contained the sludge samples.

Then, the separated liquid which mainly contained the used DME and the dewatered water was trans-

Fig.1 Schema of laboratory equipment.

ferred to the liquid sampling tank. In order to separate DME from the water, it was gasified by reducing the pressure to ambient pressure when the valve of the liquid sampling tank was opened. Gaseous DME was passed through the trap column containing approximately 7.5 g of calcium chloride (guaranteed reagent; Nacalai Tesque, Inc., Kyoto, Japan) for 1 h to trap trace atomized water.

During the extraction experiment, the pressure and the linear velocity of the liquefied DME in the dewatering column was held constant at 0.6MPa, 0.382 m/h, respectively. There were no bubbles and short-circuiting of the liquefied DME flow in dewatering column. In addition, the volumes of liquefied DME used per unit mass of the electroplating sludge (hereafter referred to as the DME: sludge ratio) were 10, 15, 20, 30, 50, 60, 70, and 80 cm³/g; the different DME volumes were obtained by varying the liquefied DME flow times.

(2) Electroplating sludge samples

We used zinc-containing electroplating sludge obtained from two plating plants in Osaka, Japan. Electroplating wastewater was treated and the raw sludge was produced by chemical polymer precipitation and dewatered with a filter press.

a) Water content and chemical composition

Water content, the amounts of organic elements, and main chemical components, Al, Cr, Cu, Fe, Mg, P, Pb, and Zn, contained in electroplating sludge A and B are shown in **Table 1**. Water content was measured according to the Japan Industrial Standard²⁷⁾. Total carbon (TC) was measured using a TOC analyzer and solid samples (TOC-V_{CSH}, SSM-5000A; Shimadzu Corp., Kyoto, Japan), and H and N were measured using a CHN coder (MT-2; Yanagimoto Corp., Kyoto, Japan). The main chemical components were measured using inductively coupled plasma atomic emission spectrometry (ICP-AES; IRIS Intrepid Duo; Thermo Electron Corp., Waltham, MA, USA).

The water contents of sludge A and B were 71.8% and 84.1%, respectively, and their respective ignition losses were 22.3% and 23.2%, nearly identical to the reference value²⁸⁾. The main component in the sludge was zinc; the zinc contents in sludge A and B were 24.4% and 17.6%, respectively. In particular, electroplating sludge A also contained 11.0% Cr, three times that of the average reference value²⁸⁾. In zinc plating, a chromating process is sometimes carried out to immobilize the surface after the plating process. Therefore, chrome is assumed to be present in the sludge²⁹⁾.

b) Particle size distribution

Figure 2 shows the particle size distribution of the electroplating sludge. The particle size distributions were measured with the laser diffraction particle size analyzer (SALD-2200; Shimadzu Corp.). In the case of sludge A, the average particle size was 108 μm ; the

Table 1 Initial properties of zinc containing electroplating sludge (n=3).

		Sludge A	Sludge B
Water	%	71.8	84.1
IL	% dry base	23.2	22.3
C	% dry base	2.55	4.39
H	% dry base	3.94	4.14
N	% dry base	0.39	0.18
Zn	% dry base	24.4	17.6
Al	% dry base	6.37	8.57
Fe	% dry base	8.24	4.41
P	% dry base	N.D.	N.D.
Cr	% dry base	11.0	2.0
Cu	mg/kg-dry base	3,120	2,180
Mg	mg/kg-dry base	7,870	30,400
Pb	mg/kg-dry base	98	647

IL: Ignition loss. N.D.: Not detected.

Fig.2 Sludge particle diameter distribution.

median particle size, 86 μm ; and the peak particle size, approximately 130 μm . In the case of sludge B, the average, median values were 146 μm , 127 μm , and peak values were approximately 350 μm and 130 μm , respectively.

(3) The form of the water in the sample

The form of the water contained in the sample was determined with thermogravimetry/differential thermal analysis (TG-DTA, Thermo Plus 8110; Rigaku, Osaka, Japan). Water evaporation is endothermic, and the endothermic peak in the TG-DTA curve indicated the evaporation temperature range. Furthermore, the water contained in the samples consisted of combined water and free water: the greater the amount of combined water in the samples, the greater the change in the

temperature corresponding to the TG-DTA endothermic peak, T_1 (°C). After the temperature of sludge sample was increased from about 20°C to 120°C at a rate of 5°C/min, it was maintained constant for 30 min. As a reference, the TG-DTA curve for the pure water was measured and the TG-DTA endothermic peak, T_2 (°C), calculated. We calculated the difference between T_1 and T_2 as ΔT : the greater the value of ΔT , the tighter the water in the sample is bound to the sludge.

(4) The quality of dewatered sludge and water in DME dewatering

To investigate the change in the quality of the zinc-containing electroplating sludge after dewatering by the liquefied DME, the amounts of carbon and some heavy metal components, Cr, Cu, Fe, P, Pb, and Zn, contained in electroplating sludge were measured.

The quality of dewatered water by the liquefied DME is very important information in connection with the water environment. Therefore, chemical components; Cr, Cu, Fe, Mn, P, Pb and Zn in dewatered water were also measured.

3. RESULTS AND DISCUSSION

(1) Time-dependent change of dewatering

Fig. 3 shows the relationship between the DME:sludge ratio and the ratio of the amount of water removed to the initial amount of water (hereafter referred to as the dewatering ratio). For small values of the DME:sludge ratio, the rate of increase in the dewatering ratio with the DME:sludge ratio is significant. The rate of increase gradually decreases at higher DME:sludge ratio. For a DME:sludge ratio of 80 cm³/g in the case of sludge A, the dewatering rate is 95.1%. At the same volume, sludge B is almost completely dewatered.

The water concentration is assumed to be uniform in the sludge and that the rate of water extraction from the sludge is proportional to the difference between the water concentration of the sludge and the water concentration of DME. Thus, when an approximate model is constructed by assuming to supply pure DME without any water to sludge sample at any time, the dewatering process is represented by

$$\frac{dW_t}{dt} = -kW_t \quad (1)$$

$$\ln\left(\frac{W_t}{W_0}\right) = -kt + a \quad (2)$$

where k (h⁻¹) is the dewatering rate constant, t (h) is the dewatering time, W_t (g) is the amount of water in the sample, and W_0 (g) is the initial amount of water in the sample. a is a constant.

Incidentally, flow volume of the liquefied DME for t hour were expressed by the both of DME:sludge ratio: Vt and linear velocity: u in Eq. (3).

$$A \cdot u \cdot t = Vt \cdot M \quad (3)$$

where Vt is the DME:sludge ratio in cm³/g for t hour, A is the cross-sectional area of the dewatering column (105 mm²), M is the initial amount of sample (1.00 g-wet), and u is the linear velocity (0.382 m/h).

According to Eq.(3), t and Vt are related by the expression.

$$t = \frac{Vt \cdot M}{A \cdot u} = 2.49Vt \times 10^{-2} \quad (4)$$

The model by Eq.(2) is similar to that of the falling rate drying period, which reduces the drying rate in proportion to the water in wet materials and when the diffusion of water is predominant³⁰⁾. Eq.(2) indicates that

Fig.3 Relationships between DME:sludge ratio and dewatering ratio on (a) sludge A and (b) sludge B.

the water in the sample decreases exponentially. The value of k in Eq.(2) was calculated by fitting against the experimental result, and the fitting curve is represented in **Fig. 3** by the solid curve. One sees that k is 1.50 h^{-1} for electroplating sludge A and 2.47 h^{-1} for electroplating sludge B.

Furthermore, the dewatering ratio could be proportional to the flow volume of the liquefied DME or to the dewatering time when water is also present outside the sludge, i.e., immediately after dewatering begins. Considering that the saturation percentage of the water in the liquefied DME at 20°C is 7.20 weight % and the liquefied DME density is $0.667 \text{ (g/cm}^3\text{)}^{22}$, the behavior of dewatering ratio when liquefied DME is saturated with water is represented by the dotted line in **Fig. 3**. The experimental data at the smallest DME:sludge ratio were nearly fitted not only to the solid curve but also to the straight dotted line in the both of **Fig 3(a)** and **Fig 3(b)**. Therefore, the behaviour of the dewatering ratio could follow the straight line in the initial dewatering phase. As shown in **Fig. 2**, since the diameters of most sludge particles are a few hundred micrometers, a possibility exists that some of the water lies in the void space between the particles. In this case, the time for the water to diffuse toward liquefied DME is likely to be short and can be ignored. The dewatering rate could be constant according to the liquefied DME volume in this period, similar to the constant rate drying period as that defined in “material drying theory”³⁰⁾.

Thus, the dewatering rate could be constant in the initial dewatering phase and could later be expressed as a first-order reaction type.

(2) The form of water in dewatered sludge

In the TG-DTA, the difference between the DTA peak temperature of the dewatered sample and that of the reference curve (pure water) is calculated as ΔT . If ΔT is positive, then the DTA peak temperature of the sample is higher than that of pure water: the greater the value of ΔT , the greater the difficulty in evaporating the water by high-temperature processes, suggesting that the water is tightly bound to the sludge.

The relationship between ΔT and the DME:sludge ratio is shown in **Fig. 4**. ΔT became negative in some

cases. But we consider the DTA analysis included with an uncertainty of about 5% because this analysis is sensitive. For a DME:sludge ratio of $70 \text{ cm}^3/\text{g}$ and above, ΔT increases dramatically. This fact makes it clear that similar to water evaporation by heat, in the dewatering process, free water is extracted first and then the extraction of thermally stable water bound to the solids follows.

(3) The quality of sludge dewatered by DME

The changes in the amounts of total carbon and some heavy metals, Cr, Cu, Fe, Pb and Zn, contained in electroplating sludge A and B due to dewatering are presented in **Table 2**. The total carbon decreases as the dewatering process progresses because liquefied DME is a solvent with a strong affinity for organic compounds. In the case of other heavy metals, significant changes are not observed.

(4) Properties of removed water

The total carbon in the liquid separated from electroplating sludge A and B, and the concentrations of Cr, Cu, Fe, Mn, P, Pb, and Zn, which are included in the Discharge Standard of Pollutants in the Japan Water Quality Pollution Control Act, were measured. Cr, Mn, and P were not detected in either sludge.

Fig.4 ΔT calculated from DTA peak temperature.

Table 2(a) The change of composition of dewatered sludge A.

DME:sludge ratio	cm^3/g	0	10	15	20	30	50	60	70	80
Total C	g-C /kg-dry base	25.5	32.8	26.7	23.8	21.5	14.7	13.9	13.4	14.7
Cr	% dry base	11.0	10.2	10.8	11.2	10.7	11.1	10.5	10.5	10.6
Fe	% dry base	8.24	8.05	8.53	8.68	8.20	8.40	8.43	8.38	8.08
Cu	% dry base	0.31	0.30	0.30	0.30	0.32	0.33	0.32	0.29	0.30
Pb	% dry base	0.098	0.058	0.103	0.032	0.019	0.023	0.027	0.021	0.017
Zn	% dry base	24.4	24.3	25.6	26.5	25.2	26.3	25.4	25.3	23.9

Table 2(b) The change of composition of dewatered sludge B.

DME:sludge ratio	cm ³ /g	0	10	15	20	30	50	60	70	80
Total C	g-C /kg-dry base	43.9	44.4	39.8	38.3	34.2	29.0	31.0	31.5	31.4
Cr	% dry base	2.0	1.9	1.8	1.8	1.8	1.7	1.9	1.8	1.7
Fe	% dry base	4.41	4.34	4.45	4.65	4.65	4.36	4.68	4.58	4.44
Cu	% dry base	0.22	0.17	0.16	0.17	0.17	0.16	0.17	0.17	0.16
Pb	% dry base	0.065	0.078	0.083	0.079	0.086	0.080	0.089	0.086	0.082
Zn	% dry base	17.6	16.7	16.5	16.9	17.8	15.7	16.9	16.1	17.5

Table 3(a) Composition of the removed water from sludge A.

DME:sludge ratio	cm ³ /g	10	15	20	30	50	60	70	80	R V
Total C	g-C/L	79.4	29.7	44.6	3.37	21.7	24.6	24.3	21.6	
Fe	mg/L	2.43	1.10	N.D.	1.21	0.30	1.04	0.17	0.17	10
Cu	mg/L	2.24	3.39	1.99	1.21	1.24	1.09	2.06	1.45	3
Pb	mg/L	0.49	0.13	0.35	0.16	0.15	0.02	ND	ND	0.1
Zn	mg/L	19.0	12.4	86.0	4.0	2.3	13.8	16.4	72.0	2

Table 3(b) Composition of the removed water from sludge B.

DME:sludge ratio	cm ³ /g	10	15	20	30	50	60	70	80	R V
Total C	g-C/L	60.3	21.3	18.8	18.1	9.57	4.66	3.44	5.26	
Fe	mg/L	0.47	0.93	1.60	1.10	N.D.	N.D.	0.07	0.07	10
Cu	mg/L	2.55	1.57	6.81	2.51	1.33	1.23	1.01	N.D.	3
Pb	mg/L	0.30	0.61	N.D.	N.D.	N.D.	0.07	0.17	0.03	0.1
Zn	mg/L	N.D.	N.D.	99.3	28.7	N.D.	5.1	2.2	N.D.	2

N.D.: Not detected.

RV: Standard reference values given in the Japan Water Quality Pollution Control Act.

RV value of Pb is item related to hazardous substances.

RV values of Fe, Cu and Zn are item related to living environment.

Cu, Fe, Pb, and Zn concentrations are listed in **Table 3** along with the standard reference values given in the Japan Water Quality Pollution Control Act. The total carbon concentration is high in the initial dewatering phase, which has a low DME:sludge ratio and a small amount of dewatered water, because DME is an organic solvent with a high affinity for organic compounds. In addition, the Fe concentrations are well below the standard value in both sludge types. The Cu concentration in electroplating sludge A is 1.01–3.39 mg/L and that in sludge B is 0–6.81 mg/L. Regardless of the DME:sludge ratio, the Cu concentrations are almost equal to the standard value. The concentrations of Pb in electroplating sludge A and B are 0–0.49 and 0–0.609 mg/L, respectively. These values are significantly greater than the standard value during the initial dewatering phase in which the DME volume is low. Zinc was not detected in some samples, but its concentration is considered to be significantly above the standard value. Since suspended solid was not detected in these samples, these metals are capable of existing in the

form of ions or microparticles in dewatered water. The obtained results show that the dewatered water cannot be flushed directly into the drainage unless processed to remove environmentally harmful elements.

4. CONCLUSIONS

This research elucidated the dewatering characteristics and dewatering mechanisms of electroplating sludge using liquefied DME, which is important in recycling of industrial waste.

Our results show that the mechanism of dewatering using liquefied DME is as follows. During the initial phase of dewatering, the water diffusion time in sludge sample can be short and ignored; it could be dependent only on the degree of saturation of water in liquefied DME. In the later phase, it is proportionally dependent on the water concentration in the sludge and shows a dewatering rate-reducing dewatering pattern. Moreover, the dewatering pattern can be expressed using one dewatering rate constant. In addition, according to TG-

DTA measurements, the final dewatered water is bound to the sludge and includes thermally stable water.

Regarding the quality of dewatered water, Cr, Mn, and P were not detected in the dewatered water. Furthermore, Fe was present in lower concentration than the standard value of the Water Pollution Control Act. However, some cases were found in which Cu, Pb, and Zn were present in concentrations above the standard values of the Water Pollution Control Act, and the dewatered water required processing to lower the concentration of these metals.

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(Received June 3, 2009)