

(44) **PHYSICO-CHEMICAL CHARACTERISTICS AND STABILIZATION
OF ABOUT 23 YEARS OLD LANDFILLED MUNICIPAL SOLID WASTE**

埋立後約23年経過した都市ごみの物理化学的特性と安定度の研究

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ABSTRACT; A detailed physico-chemical characteristics of 23 years old municipal solid waste disposed of in a landfill was investigated. The results are compared with other investigations and degree of stabilization of about 20 years old landfilled waste is discussed. After about 20 years being disposed of, landfilled waste may be considerably stabilized but a minor fraction of slowly biodegradable organic matter still remains.

KEYWORDS; old landfill, sanitary landfill, municipal solid waste, elution test, metal content.

1. Introduction

It is important to estimate how many years it will take for landfilled solid waste to be stabilized, when the environmental impact, or utilization of a completed landfill site is considered. Belevi et al.¹⁾ estimated the period for leachate quality to meet with quality standard for running waters in Switzerland, and consequently concluded that it will take less than ten years for heavy metals, and several hundred years for organic pollutants, organic carbon, nitrogen

Recently, in Japan, criteria for the closure of sanitary landfill sites was established in terms of generation rate of landfill gas, temperature of refuse layer and leachate quality. The term "closure" of landfill site is defined as a state in which maintenance and management of the treatment plant and equipment is not required. But, the true goal of a landfill is stabilization of landfilled solid waste, i.e attainment of "final storage quality". It is important how to define the stabilization, how to measure, and how to estimate it. There have been a few investigations about stabilization behavior of long-term landfilled solid waste. Matsufuji et al.²⁾ reported composition analysis in landfill gas, generation rate and composition analysis and elution test of 18 years old landfilled solid waste in order to construct school buildings. Nagano et al.³⁾ reported physical composition and ignition loss of 12 years old landfilled solid waste, and yearly variation of leachate quality, generation rate and composition of landfill gas in the landfill site. Samejima et al.⁴⁾ reported physical composition, chemical composition and elution test of 8 years old landfilled solid waste. Belevi et al.⁵⁾ reported physical composition and chemical composition of 11 and 18 years old landfilled solid waste in Switzerland. Heyer et al.⁶⁾ reported physical composition and ignition loss of about 14 and 32 years old landfilled solid waste in 2 landfill sites of Germany, respectively. In order to elucidate the stabilization behavior, however, more investigations are necessary. In this work, physico-chemical characteristics of 23 years old Landfilled solid waste are presented and degree of stabilization is discussed. The understanding of the stabilization behavior will proceed by accumulating many observations and informations such ones in this paper.

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2. Outline of the investigated landfill site

2.1 Outline of investigation

A landfill site for present investigation is Y landfill site without seepage control works, which was used for final disposal of municipal solid waste between 1967 and 1986 for a period of 20 years. The completed landfill site hasn't been almost designated for any future utilization. One section of this landfill site, disposed of from January of 1974 to March of 1975 was investigated. Area of the section is 3.0 ha and calculated weight of waste disposed of in the site is 82,000t. The construction of a sludge treatment facility is planned on the section after digging. The dug waste has to be disposed of on another landfill site.

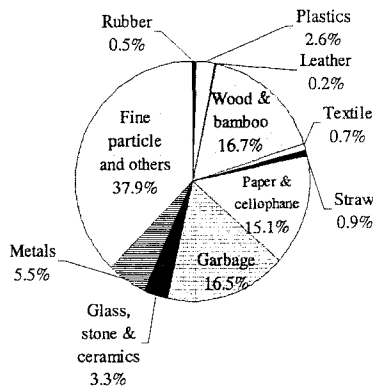


Fig.1 Estimated composition of the whole Waste disposed of in the investigated site on wet weight basis

2. 2 Estimation of landfill content

The amount and composition of landfilled solid waste was collected from the cleansing bureau and used for the estimation of the waste composition disposed of in the investigated section. The source of data were as follows:

- (1) Document of the ratio of domestic refuse and commercial waste at 1974 in the site
- (2) Document of waste composition of commercial waste by the survey for 19 of July, and 2 and 3 of August in 1971 at O landfill site.
- (3) Yearly composition of domestic refuse.

The estimated composition of waste disposed of in the investigated site is shown in Fig. 1.

3. Investigation and sampling method

3.1 Investigation outline

The investigation was performed in three successive times. An outline is given in Table 1. Furthermore, the distance between the first sampling section (Fig. 2) and the second sampling section (Fig.3) is about 50 m .

1) Pre-investigation before waste sampling

Temperature and gas composition within the waste layer at point "A" in Fig. 2 was measured for four times as shown in Table 1. Thermocouple device as well as gas sampling tubes for emitted landfill gas were placed on the point on 20 of June in 1997.

The waste layer was covered with 3cm concrete under which a steel mesh grid was placed. After taking away of these materials, an intermediate soil cover of 40cm thick was observed. Boring of 9cm diameter were performed with a hand auger. A bundle of glass tubes as well as thermocouple were placed and covered with the soil to fill up the vacant space of the boring hole. Temperature was measured and gas samples from glass tubes were taken at 0, 10, 20, 30 and 40 cm depth from the bottom of the intermediate soil.

Table1 Outline of investigation (in 1997)

| | Date | Content of investigation |
|--|------------|------------------------------------|
| Pre- investigation before waste sampling | 20 of June | Temperature and gas |
| | 24 of June | |
| | 1 of July | |
| | 10 of July | |
| First sampling | 22 of July | waste sample of No. 1 to 8, water* |
| | 23 of July | |
| Second sampling | 21 of July | waste sample of No. 9 to 12 |

* two samples of water were taken, named "stagnant water" and "seepage water"

2) First sampling

In the first phase of sampling, eight sampling points were selected as No.1 to 8, shown in Fig. 2. Sampling was carried out between 22 and 23 of July 1997. In Fig.2, the broken line shows the previous boundary of waste layer and the solid line shows the boundary after digging by a power shovel. Sampling was performed during the digging. It is concluded that the waste was disposed of in 1974, from the excavated food package, which revealed the limit date of its use. At the sampling point No.2, "stagnant water" was taken by a 50 mL injection - syringe and carried to laboratory for analysis. In addition, small hole was manually dug in the ground near the sampling point No.2 and then "seepage water" was taken by a 50 mL injection - syringe and carried to laboratory for analysis.

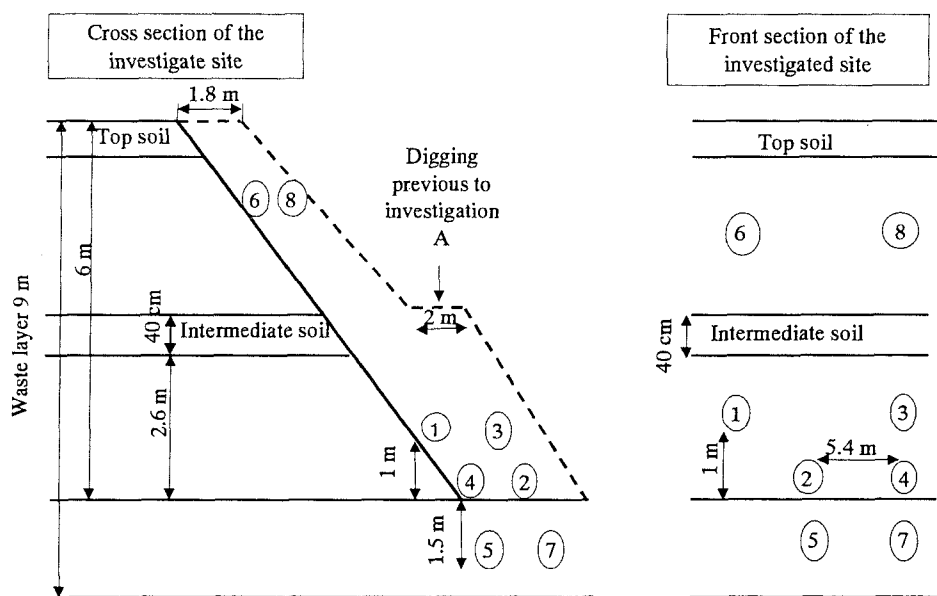


Fig.2 First sampling points

3) Second sampling

The second phase of sampling were performed in 11 of November 1997. The sampling area is shown in Fig. 3. An area of $17.7\text{m} \times 17.8\text{m}$ of waste layer was excavated to a depth of 4.5 m before several days. The perimeter of the digging area was reinforced with sheet pile. The surface of the area were covered by thin soil layer. Four waste samples, No. 9 to 12 were taken by the following procedure. (1) the area of about $3\text{m} \times 3\text{m}$ was excavated to a depth of about 2.5 m further from the surface, (2) waste samples were taken four times from the digging machine. In fact, contamination of waste sample with cover soil was not avoidable. However, waste sample No.11 is expected to be a high quality and representative sample of this section. At the first and second sampling, it has little

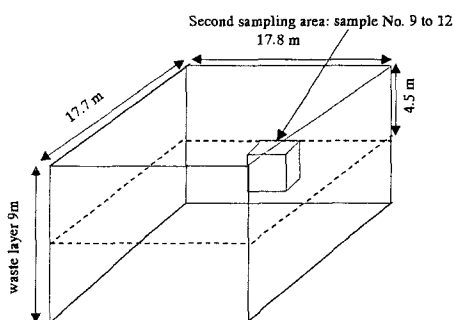


Fig.3 Second Sampling points

unpleasant odor.

3.2 Sampling and pretreatment

Waste, which was dug manually at sampling points No. 1 to 4 was carried and put on a vinyl sheet. In situ, bulky materials such as wood, block of concrete and stone, which were bigger than 5 to 10 cm were taken away. The remaining fraction was well mixed and divided into four parts. Two parts were selected, mixed again and divided in four parts. Each part was placed in a vinyl bag and carried to laboratory. This sample is called "waste sample".

Waste samples of No.5 to 8 were taken by a digging machine and pretreated by the same procedure for No.1 to 4 samples. At the sampling point No.5, however another waste sample was taken for a composition analysis on wet basis, and bulky materials was not excluded.

Waste samples No.1 to 8 were dried and sieved in laboratory. A composition analysis was performed for the fraction more than 2 mm. Fraction less than 2 mm were named "residue sample". In addition, every of residue samples No.1 to 8 was mixed together according to the calculated ratio by the assumption that every waste sampling was performed at the same amount of sampling. This mixed sample was named "composite residue".

In the second sampling, a total sampling without exclusion of bulky materials was planned. Accordingly, sampling by machine was performed four times and then waste sample, about 50 kg was spread on a vinyl sheet and carried to laboratory as it is. The weight of waste sample was measured and exclusion of bulky materials, which are bigger than 5 to 10 cm, was performed in laboratory.

4. Analysis of samples

4.1 Analytical items

For every waste sample of No.1 to 12, a physical composition was determined on dry basis. On the other hand, another composition of sample No.5 was determined on wet basis of 42kg weight. In addition, moisture content and ignition loss were also determined.

Before drying, wet waste sample was subject to a elution test in laboratory as soon as possible. This elution test was performed for two hours by reason of reduction of working hours and a filtration was performed by a filter paper with pores of $0.45\mu\text{m}$ by reason of obtaining soluble constituents. This is different from the official and routine procedure which requires six hours and glass filter with pores of $1\mu\text{m}$. The filtrated solution was used for the determination of pH, IC (inorganic carbon), TOC (total organic carbon), E260 (absorbance at wavelength of 260 nm), E220, Cl^- , SO_4^{2-} , $\text{NH}_4^+\text{-N}$, $\text{NO}_2^-\text{-N}$, $\text{NO}_3^-\text{-N}$, T-N, COD(Mn), and BOD. In addition, stagnant water and seepage water was subject to the same analysis after filtration.

Residue samples were subject to the analysis of physical composition, ignition loss, metal content, CHN (carbon, hydrogen, and nitrogen) content, calorific value and oxygen consumption.

Gas samples were carried to laboratory under airtight condition. The composition of O_2 , N_2 , CO_2 , and CH_4 were determined by gas chromatography of TCD (HITACHI).

4.2 Analytical method

1) Physical composition on wet basis

It was performed for the waste sample No.5. It was classified by hand into 11 different groups such as wood, paper, textiles, garbage, plastics, urethane foam, rubber, glass and ceramics, metals, stone and others (not easily discriminated). The classification of wet sample was more difficult than dry sample

2) Physical composition on dry basis

Each waste sample of about 0.5 kg-wet for sample No.1 to 8 and about 8 kg-wet for sample No. 9 to 12 was placed in a flat plate and put in the dryer at temperature of 40°C . The temperature value was chosen in order not to melt the plastics fraction. Total duration was 2 to 3 days. Moisture content was then determined.

Dry waste sample of about 0.3 kg for sample No.1 to 8 and about 5 kg for sample No. 9 to 12 was classified into 13 groups such as wood, paper, textiles, plastics, rubber, glass, ceramics, stone, metals, shell, gypsum board & plaster and residual fraction smaller than 2 mm (previously named “residue sample”). Residue samples were separated by sieve of 2mm mesh. It is noted that before composition analysis, bulky materials bigger than 5 to 10 cm was excluded except the waste sample No.11.

In the case of waste sample No.12, the composition analysis for bulky materials was performed.

3) Ignition loss

Ignition loss was determined for waste sample and residue sample. Two samples of about 10 g were placed on oven at 600°C for two hours.

4) CHN and calorific value

Composite residue and residue sample No.11 were used. After grinding of samples, CHN is determined on the dry basis. Higher calorific value was obtained on dry basis.

5) Composition of residue sample

By the following method presented by Sekito *et al.* ⁷, the composition of the composite residue and residue sample No.11 of about 5 g was determined three times, respectively. First process is a separation by heavy liquid of specific density 1.35. Over part is a mixture of wood and plastics, and under part is a mixture of paper, metals and stone/glass. The weight ratio of wood and plastics is calculated by the procedure of perchloric acid (HClO₄) decomposition. The weight ratio of paper is calculated by the procedure of ignition in a oven of 600°C. The fraction ratio of metals and stone/glass is calculated by the further procedure of aqua regia decomposition.

6) Metal content of residual sample No.1 to 8

First, about 10g of sample was decomposed on a hot plate by aqua regia 80 mL, and then another 5mL of Nitric acid and 60 mL of HClO₄ were also added for a complete decomposition. For the solution from acid decomposition of samples, chromium (Cr), cadmium (Cd), iron (Fe), aluminum (Al), copper (Cu), manganese (Mn), molybdenum (Mo), nickel(Ni), and lead(Pb) were determined by flameless atomic absorption spectrometric method. Calcium (Ca), magnesium (Mg), and zinc (Zn) were determined by atomic absorption spectrometric method, and sodium (Na) and potassium (K) were determined by flame emission photometric method.

7) Oxygen consumption

Ten grams of composite residue, whose moisture content was adjusted to 30% was placed in an air-tight glass bin at 37 °C and the volume of carbonic acid gas produced by biological decomposition of organic matter, was measured sometimes during 750 hours.

5. Result and discussion

5.1 Temperature and gas-composition of waste layer in pre-investigation

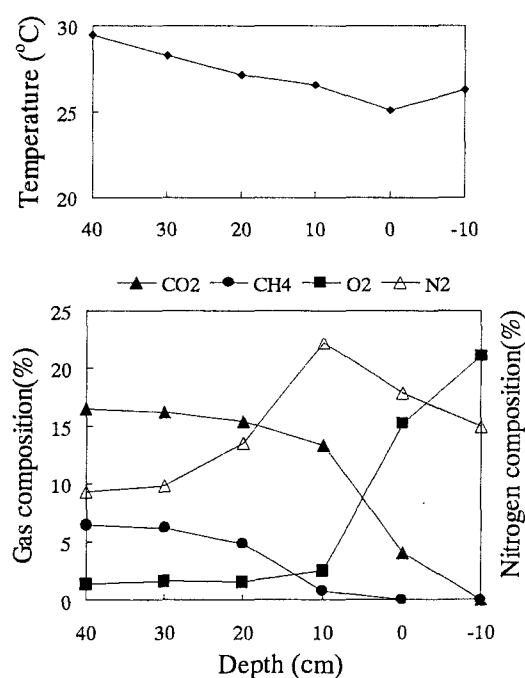


Fig.4 Variation of temperature and gas composition with depth on 10 July 1997

The last measurement (on July 10) of temperature and gas-composition in the pre-investigation is shown in Fig.4. The temperature increases with depth and it is suggested that temperature of deeper layer may be high. Regarding gas composition, methane and carbonic acid gas increases, while oxygen gas decreases rapidly with depth. Thus, enough amount of biodegradable are still remaining to keep anaerobic in the waste layer and methane generation occurs in the deeper layer. From nitrogen gas of about 75% it is concluded that nitrogen gas may be penetrating into deeper of waste layer and rate of methane generation may be weak.

5.2 Characteristics of waste sample

1) Physical composition

Physical composition of waste sample No.5, was determined on wet bases and shown in Fig.5. Combustibles constitute about 30%, incombustibles about 30%, and the others, 40% are non-distinguishables. Bulky wood remains under original configuration, but garbage is only 0.4%. Physical compositions on dry basis are shown in Fig.6. In this figure except No. 11, combustibles constitute 7 to 29%, incombustibles 20 to 44%, and under 2 mm residue about 50%. Among all waste samples, waste sample No.11 has the smallest content of under 2mm residue. The reason is because only for this sample bulky materials such as wood and plastics was not excluded.

In addition, wet physical composition of waste sample No.12 was estimated by assuming uniform moisture content, then by adding composition of excluded bulky materials.

Total composition of landfilled waste is shown in Fig.7. Consequently, there is merely a little difference between Fig.5 and 7, that is combustibles are contained 30 to 40 % and the content of plastics and wood are similar between Fig.5 and 7. Therefore, sum ratio of incombustibles and others may be attain about 70%. Then in order to elucidate the biodegradation of landfilled waste, the wet physical compositions of landfilled waste (Fig.5 and 7) were compared with the initial physical composition (estimated) of Fig.1. Garbage and paper, whose sum was estimated to be about 30 % in initial waste, can be hardly found in landfilled waste. In detail, newspaper has been degraded and crushed, but thick book such as telephone directory was inert. In addition, initial content of plastics and wood is similar to

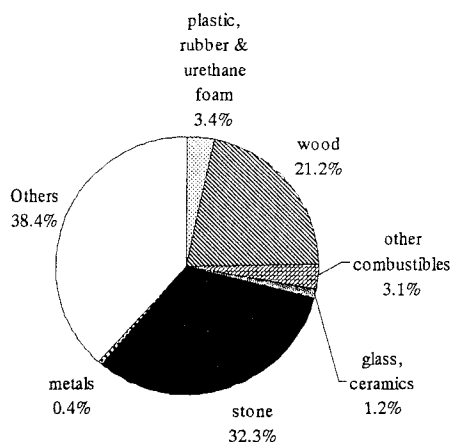


Fig.5 Composition of landfilled waste on wet weight basis including bulky materials (sample point No.5)

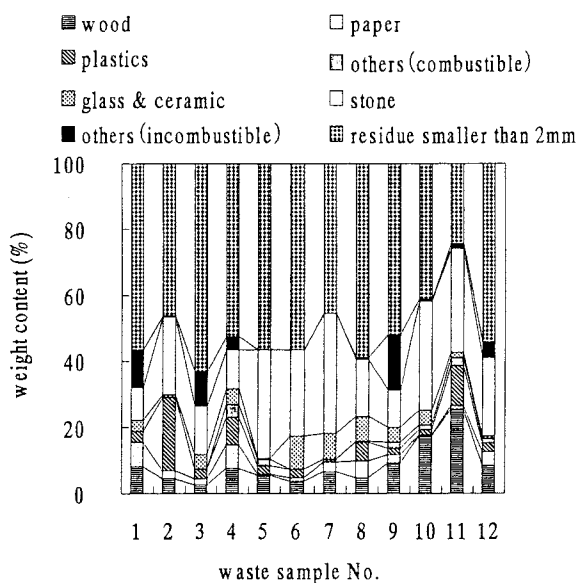


Fig.6 Composition of waste sample on dry basis, excluding bulky materials except No.11

that of landfilled waste.

The same tendency has been reported that garbage could hardly observed in 12 years old waste³⁾, 8 years old waste⁴⁾, and about 14 and 32 years old waste⁶⁾, sum ratio of incombustibles and others on wet basis are 86%³⁾, 83%⁴⁾, and 79%⁶⁾, respectively. These values exceeds this work of about 70%.

2) Moisture content and ignition loss

The results are shown in Fig.8. Waste sample No.11 has the highest ignition loss because bulky materials were not excluded. The average of ignition loss of waste sample except No.11 is 21.6%. Nagano et al.³⁾ reported that time variation of ignition loss of 0, 4, 7, and 16 years old waste were 58.3, 38.0, 24.9 and 15.5, respectively.

Ignition loss of residue samples is mostly lower than that of waste samples. The former is a little higher than that of typical soil, which is around 10 %.

5.3 Characteristics of residue sample

1) Physical composition

Composite residue sample and residue sample No.11 are given shown in Table 2. Plastics content are only 0.5% and 0.9%, respectively. On the other hand, sum of incombustibles, i.e metals and stone/glass is more than 80 %.

2) CHN composition and calorific value

Carbon/Nitrogen ratio (C/N) of the two samples, shown in Table 3, are a little higher than 10 of typical soil. As typical C/N of mixed refuse in Japan is 20 to 30, landfilled waste seems to attain 10 with biodegradation proceed. The low C/N suggest that biological degradation process was almost completed. By the measurement of Samejima et al.⁴⁾ the higher calorific value of 8 years old waste, containing 13.5% plastics on dry basis was reported to be 1,050 kcal/kg-dry, compared with original waste of 2,740 kcal/kg-dry, containing 23% plastics. As these calorific value can calculated from the content of plastics and other combustibles such as wood and paper. The difference between this and Samejima work results from plastics content and test sample, that is, sample in this work is under 2 mm residue, while that of Samejima is total waste sample.

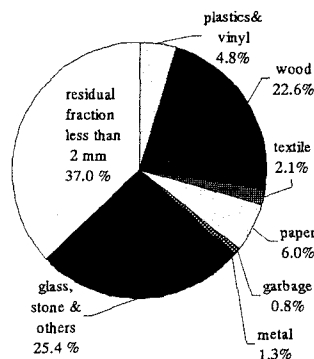


Fig.7 Estimated composition of landfilled waste on wet weight basis, including bulky material(sampling point No.12)

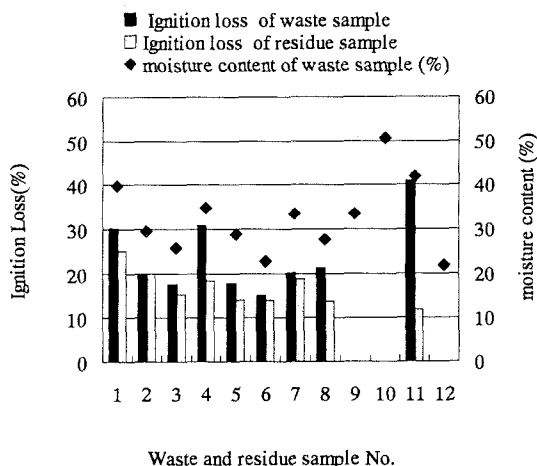


Fig.8 Ignition loss content and moisture content, excluding bulky materials except No.11

Table 2 Composition of residue sample

| items | composite residue weight (%) | residue sample No.11 weight (%) |
|---------------|------------------------------|---------------------------------|
| plastics | 0.5 | 0.9 |
| paper& wood | 15.7 | 17.1 |
| metals | 27.2 | 21.5 |
| glass & stone | 56.6 | 60.5 |
| Total | 100 | 100 |

3) Metal content

On Fig.9 metal contents of residue samples are compared with typical values of the earth crust and soil⁸⁾. Residue samples contain Content of Cd, Pb, Zn definitely higher than earth crest, and when compared with soil, content of Fe, Cu, Mo, Ni, Pb, Zn are higher. Although metal content of Fe, Cu, Pb, Zn, Mo and Ni are high, leachability of these metals is low as described below in the result of elution test.

4) Oxygen consumption

Measured maximum rate of oxygen consumption for the composite residue is 0.020 mg-O₂/(h·g-dry). Matsuo et al.⁹⁾ reported 0.04 and 0.06 mg-O₂/(h·g-dry) for compost. In addition, the rate of artificial mixed refuse and bottom ash are 0.96 and 0.001 mg-O₂/(h·g-dry), respectively¹⁰⁾. Therefore, composite residue is almost stabilized, but a little of slowly degradable organic matter still remains.

Cumulative oxygen consumption per volatile organic matter of composite residue is calculated to be 21 mg-O₂/(g-VS) at final stage. Heyer et al.⁶⁾ reported that the oxygen consumption of about 14 and 32 years old waste were 82 and 21 mg-O₂/(g-VS) at 1000 hours, respectively.

5.4 Elution test of waste sample

The results of elution test summarized in Table 4 giving average and standard deviation for the groups of samples No.1 to 8 and No. 9 to 12. For reference, results of elution test by Horibe¹¹⁾ for compost made out of garbage and sewage sludge as well as bottom ash from municipal solid waste incinerator are also shown in Table 4. Mean value and standard deviation of metal concentrations for each group

are shown in Table 5. From Table 4, COD, BOD, TOC were relatively low. The value of TOC/E260, index of biodegradable matter is close to 100. When the index is lower than 100, solution contains little biodegradable matter¹²⁾. Therefore, it is concluded that this landfilled waste has been considerably stabilized. Oxides, such as sulfate and nitric ions were detected which suggests that oxidized substances penetrate into deeper layer.

Table3 CHN and calorific value of residue

| items | Composite residue | Residue of sample No.11 |
|-------------------------------|-------------------|-------------------------|
| Carbon (C) (%) | 6.82 | 6.42 |
| Hydrogen(H) (%) | 1.07 | 0.9 |
| Nitrogen (N) (%) | 0.55 | 0.37 |
| C/N | 12.4 | 17.4 |
| Calorific value (kcal/kg-dry) | 540 | 630 |

C, H, N and calorific value are on dry basis

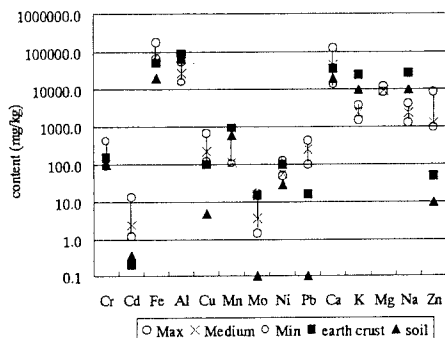


Fig.9 Metal content of residue sample (No.1 to 8), compared with the earth crust and soil

Table 4 Organic pollutant concentration of the solution from elution test of waste, sample compared with compost of garbage, compost of sludge and ash of incineration

| items | No. 1 to 8 | No. 9 to 12 | Stagnant | Seepage | Compost | Compost | Bottom Ash |
|--|-------------|-------------|----------|---------|------------|-----------|-----------------|
| | Mean ± SD | Mean ± SD | Water | water | of garbage | of sludge | of incineration |
| pH | 7.68 ± 0.32 | 8.6 ± 0.4 | 7.4 | 7.1 | 6.7 | 8.2 | - |
| E260 | 0.26 ± 0.15 | 0.51 ± 0.31 | 0.31 | - | 5 | 10 | 0.6 |
| E220 | 0.75 ± 0.25 | 1.31 ± 0.61 | 1.49 | - | 16 | 24 | 1.7 |
| IC (mg/L) | 8.0 ± 1.1 | 11.3 ± 3.8 | 62.2 | - | - | - | - |
| TOC(mg/L) | 18 ± 7.0 | 37 ± 19.0 | 129 | 160 | 1290 | 999 | 111 |
| TOC/E260 | 109 ± 28 | 70 ± 6 | 418 | - | 281 | 101 | 202 |
| COD ₆₀ (mg/L) | 16.3 ± 5.3 | 34 ± 13.0 | 46.9 | - | 860 | 431 | 85 |
| BOD(mg/L) | - | 26.4 ± 14.1 | ND | - | 231 | 441 | 95.4 |
| Cl ⁻ (mg/L) | 150 ± 14 | 60 ± 40 | 159 | 75.9 | 290 | 412 | 440 |
| SO ₄ ²⁻ (mg/L) | 470 ± 270 | 170 ± 80 | 1040 | 1208 | - | - | - |
| NH ₄ ⁺ - N(mg/L) | 5.3 ± 3.6 | 19.1 ± 3.6 | 8.8 | 26.9 | 124 | 200 | - |
| NO ₂ ⁻ - N(mg/L) | 0.1 ± 0.05 | 0.07 ± 0.02 | ND | 1.39 | - | - | - |
| NO ₃ ⁻ - N(mg/L) | 0.3 ± 0.2 | 1.3 ± 1.2 | 0.29 | 0.76 | - | - | - |
| T-N (mg/L) | 7.0 ± 5.0 | 27.0 ± 4.0 | 14.2 | - | 290 | 430 | 12.5 |

"-": not measured

ND: not detected

SD: standard deviation

From Table 5, average concentration of all metals except lead are lower than environmental standard or a tenth of effluent criterion. However, two samples for iron & lead and one sample for nickel & molybdenum in twelve waste samples exceed environmental standard or a tenth of effluent criterion.

6. Conclusion

Detailed investigation of about 23 years old landfilled municipal solid waste was presented. These results are compared with other investigations. Consequently at 20 years after landfilling, landfilled waste has been considerably stabilized but minor fraction of slowly biodegradable organic matter still remains.

7. References

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Table 5 Soluble metal concentration of waste sample by elution test (mg/l)

| items | No. 1 to 8 | No. 9 to 12 | Reference |
|-------|--------------------|--------------------|-----------------------------|
| | Mean \pm SD | Mean \pm SD | |
| Cr | 0.003 \pm 0.001 | 0.004 \pm 0.003 | 0.2** |
| Cd | ND | 0.0005 \pm 0.001 | 0.01* |
| Fe | 0.09 \pm 0.14 | 1.03 \pm 0.85 | 1 as soluble ** |
| Al | 0.36 \pm 0.82 | 1.57 \pm 1.85 | — |
| Cu | ND | 0.03 \pm 0.04 | 0.3** |
| Mn | 0.09 \pm 0.21 | 0.087 \pm 0.07 | 1 as soluble ** |
| Mo | 0.006 \pm 0.007 | 0.055 \pm 0.068 | 0.07 as surveillance term * |
| Ni | ND | 0.3 \pm 0.6 | — |
| Pb | 0.001 \pm 0.0008 | 0.018 \pm 0.02 | 0.01* |
| Ca | 508 \pm 376 | 100 \pm 106 | — |
| K | 12.1 \pm 3.5 | 60 \pm 43 | — |
| Mg | 12.1 \pm 3.5 | 7.85 \pm 4.58 | — |
| Na | 15.5 \pm 13.5 | 58.5 \pm 37.0 | — |
| Zn | 0.016 \pm 0.008 | 0.09 \pm 0.12 | 0.5** |

Mean and SD are calculated as assumption, ND = 0 mg/L

* : Environmental Standard

** : a Tenth of effluent criterion