Evaluating the Fires and Oxygen Deficiency Risks Caused by Stored Agricultural Waste

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Abstract: In the Japanese agricultural industry, efforts are being made to recycle waste produced in order to use resources more efficiently. However, in some cases, when stored for long periods of time these materials generate heat through fermentation, which can eventually result in spontaneous ignition or oxygen deficiency in storage areas, resulting in the deaths of workers. In this study, we conducted a series of experiments on several types of agricultural waste (organic waste generated by agricultural production activities in Japan) frequently stored for recycling, using combinations of various thermal- and gas-analysers, in order to identify the risk factors related to spontaneous ignition and oxygen deficiency accidents. The aim of this research was to understand the circumstances leading to spontaneous ignition and oxygen deficiency accidents in storage facilities and to recommend safety measures to prevent such occurrences. Our results suggest that fermentation is likely responsible for the generation of heat and production of carbon dioxide at temperatures up to approximately 50 °C, where microbial activity diminishes. At temperatures beyond 50 °C, a transition into heat generation by the oxidation of fatty acid esters occurs. Additionally, when the barrier of heat absorption is overcome at around 100 °C due to the evaporation of water, there is a transition to thermal cracking that could lead to fire. Accidents due to oxygen deficiency may occur when a storage facility is well sealed and the amount of oxygen circulation is minimal. However, when the amount of oxygen is sufficient; the substance is stored in large deposits; and the facility is well insulated, fermentation can cause the temperature to increase. Therefore, it is desirable to periodically measure the temperature of stored materials and monitor the generated gases.

Keywords: agricultural waste; fermentation; thermal and gas analysis; spontaneous ignition; oxygen deficiency

1. Introduction

In Japan, there is a need to build a recycling-based society which reduces environmental loads, recycles limited resources, and uses them efficiently. To this end, various laws for controlling the generation of waste have been established.

In addition, various recycling methods have been developed, and products created using recycled waste are beginning to be distributed. However, accidents related to recycled materials, such as fires and explosions, have steadily increased over the years in Japan [1–4]. If a shift towards a sustainable
society is rushed and new substances are used without full safety considerations in their handling, the possibility of such accidents occurring will increase.

For example, in the agricultural industry in Japan, a project to reuse commercial agricultural waste as biomass fuel has begun. Moreover, since the Great East Japan Earthquake, an increasing focus has been placed on biomass fuels as an energy source [5,6], and the government of Japan has begun implementing policies designed to encourage their widespread application. As advanced examples of other countries, waste/wastewater can be properly managed for production of valuable products. Wastewater can be used as the nutrient source for production of biodiesel feedstock. Alternatively, the solid waste (e.g., agricultural waste) can be used directly as biofuel [7–9].

However, spontaneous ignition of agricultural waste stored for recycling can occur during storage and transport [10]. In addition, these materials have caused oxygen deficiency in storage areas due to fermentation, which could lead to injury or even death of workers [11].

Therefore, we conducted experiments on several types of agricultural waste commonly stored for recycling in order to evaluate storage risks and safety measures, using a combination of highly sensitive thermal and gas analysers. Moreover, the aim of this research was to understand the circumstances leading to spontaneous ignition and oxygen deficiency accidents in storage facilities and to recommend safety measures to prevent such occurrences.

2. Experiments

Thermal analysis was used to investigate the cause of accidents for a variety of reasons. With just a small sample (<1 g), the properties of a material can be studied in order to prevent the occurrence of fire, large quantities of poisonous gas, or smoke. The results of thermal analysis can also aid in risk assessment targeting, placing the principal focus on materials that are likely to generate heat when piled.

2.1. Samples

The agricultural waste materials used in this study included wood chips (Figure 1), wood pellets, sludge fuel, chicken dung, and soy sauce squeezing residue (Figure 2).

Wood chips (shape: chip) and wood pellets (shape: pellet) are made from logged trees for use as fuel in wood stoves and in thermal power plants [12]. Sludge fuel (shape: granule) is made from agricultural sewage sludge, and its conversion and use as fuel is expected to reduce its disposal costs [13].

Chicken dung (shape: sludge) is often used as fuel for boilers and heating equipment on chicken farms. Soy sauce squeezing residue (shape: powder) occurs as a byproduct of soy sauce production, and it is used as fuel for boilers at soy sauce factories and in farmhouses [14]. Since the composition of waste material to be used for recycling is not fixed, experiments are challenging. In this study, to keep the test conditions as uniform as possible, we used samples with consistent grain size (2 cm or less was used).

Moreover, distilled water was added to samples (water to equal 20% of the sample mass) in order to examine the effects of additional moisture on fermentation and thermal characteristics. In addition, samples were subjected to a 17-h sterilisation treatment using ethylene oxide gas (EOG), to ascertain the effects of fermentation. EOG is widely used to sterilise medical devices and precision machinery, and to kill all microorganisms [15].
2.2. Thermogravimetric Differential Thermal Analysis

A thermogravimetric differential thermal analysis (TG-DTA) system (Rigaku Thermoplus TG 8120, Japan) was used to study the overall thermal characteristics of the samples. The samples (~20 mg) were placed in an open aluminium container (0.05 mL). The thermal characteristics of the samples were examined by heating them from room temperature to 600 °C at a rate of 2 K/min, with an air circulation of 150 mL/min.

2.3. Calvet Calorimeter

A Calvet calorimeter (Setaram C80, France) was used for additional thermal testing. The C80 is a highly sensitive twin-type heat-flux calorimeter. It can reduce the effects of evaporation of water contained in the sample by using a high-pressure closed vessel (8 mL), and it can take measurements from room temperature to 100 °C, which is a temperature range that cannot be easily measured using the TG-DTA system. The temperature of approximately 1500 mg samples in sealed vessels was increased at a rate of 0.1 K/min up to 300 °C.

2.4. High-Sensitivity Isothermal Calorimeter

To examine the faint heat generation from the fermentation and oxidation of fatty acid esters in detail, we used a highly sensitive isothermal calorimeter (Thermometric TAM-III, Sweden). The TAM can measure the amount of heat generated by microbial fermentation on a nanoscale. Samples of 1000 mg were placed in sealed containers (4 mL), which were isothermally maintained at 50 °C for three days.

2.5. Gas Chromatography

To study gas emissions during storage, samples of approximately 50 g were placed in a 1 L glass bottle that was sealed airtight and placed in a thermostatically controlled oven. The resulting gas
was collected and measured by gas chromatography (Shimadzu, GC-14B, Japan) using a standard gas (CO: 0.05%, C₂H₆: 0.995%, H₂: 0.997%, CO₂: 0.996%, CH₄: 0.987%) for calibration, and a thermal conductivity detector (TCD, 200 °C, sensitivity was 50 mA, carrier gas was Ar at 20 mL/min). The column temperature ranged from 40 °C (6 min hold) to 80 °C (12 min hold) to 150 °C (10 min hold), and changed at a rate of 40 °C/min. An air cylinder (O₂: 21%, N₂: 79%) was used as the standard gas for the measurement of O₂ and N₂. For these measurements, we used a TCD (200 °C, 20 mL/min Ar carrier gas, 30 mA sensitivity) with the isothermal column temperature maintained at 30 °C.

3. Results

3.1. Thermogravimetric Differential Thermal Analysis

Figures 3–7 show the TG-DTA measurements for a scanning rate of 2 K/min. The decomposition temperature is the temperature at which the DTA curve shifts by 0.1 µV (0.01 K) from the baseline, where the DTA curve is constant.

In the TG curve, the downward direction indicates a reduction in weight, and the upward direction indicates an increase in weight. In the DTA curve, the downward direction indicates an endothermic reaction, and the upward direction indicates an exothermic reaction. The thermal decomposition results of the four samples are divided into three stages: dehydration, thermal decomposition, and char combustion.

The TG curve demonstrates weight lost by the samples as a result of dehydration between room temperature and 100 °C. The highest rate of weight loss at this stage was observed in the chicken dung.

![Figure 3. TG-DTA results for wood chips.](image-url)

![Figure 4. TG-DTA results for wood pellets.](image-url)
3.2. Calorimetry

The C80 calorimeter is more sensitive than the TG-DTA system; consequently, a lower scan rate was used. If a high-pressure, closed vessel is used, the influence of vaporization of the contained moisture can be reduced. As a result, detailed thermal-behaviour information, including the existence of low levels of heat generated at temperatures $\leq 100 \, ^\circ C$, which are difficult data to obtain by TG-DTA, can be obtained with the C80 calorimeter.
Figures 8–12 show the C80 results for a scanning rate of 0.1 K/min. The exothermic onset temperature was taken as the temperature at which the rate of heat generation increased to 0.005 mW.

The samples of wood chips, chicken dung, and soy sauce squeezing residue showed heat generation due to fermentation or oxidation immediately following the start of the measurement (around 25°C).

![Figure 8. C80 results for wood chips.](image1)

![Figure 9. C80 results for wood pellets.](image2)

![Figure 10. C80 results for sludge fuel.](image3)
3.3. High-Sensitivity Isothermal Calorimeter

The TAM-III calorimeter can measure the change in activity of microorganisms over time. Figures 13–17 show the TAM-III calorimetry curves of the various fuel samples prepared without and with the addition of distilled water (equal to 20% w/w of the sample), and Table 1 summarises the amount of heat generated.

Heat generation was considered for two periods: 0–24 h, and 24–72 h. In addition, heat generation was considered for a combination of both time periods. The TAM-III temperature was held at 50 °C for all tests, because microbial activity occurs most vigorously in this temperature range but disappears at higher temperatures [16,17].

In all samples, an increase in heat generation was observed with the addition of distilled water. The greatest increase was observed in the chicken dung, in which heat generation increased by approximately 19 times when distilled water was added.
Figure 13. TAM results for wood chips.

Figure 14. TAM results for wood pellets.

Figure 15. TAM results for sludge fuel.
Figure 16. TAM results for chicken dung.

Figure 17. TAM results for soy sauce squeezing residue.

Table 1. Heat generation at 50 °C.

| Sample                          | Heat Generation (J/g) 0–24 h | Heat generation (J/g) 24–72 h | Heat generation (J/g) 0–72 h |
|---------------------------------|------------------------------|-------------------------------|-----------------------------|
| Wood chip                       | 4.8                          | 6.3                           | 11.1                        |
| Wood chip + Distilled water 20% | 12.3                         | 1.9                           | 14.2                        |
| Wood pellet                     | 1.3                          | 1.6                           | 2.9                         |
| Wood pellet + Distilled water 20% | 8.0                         | 10.4                          | 18.3                        |
| Sludge fuel                     | 17.7                         | 11.1 *                        | 28.9 **                     |
| Sludge fuel + Distilled water 20% | 35.1                        | 8.52                          | 43.6                        |
| Chicken dung                    | 3.7                          | 5.7                           | 9.4                         |
| Chicken dung + Distilled water 20% | 80.4                        | 96.8 *                        | 177.2 **                    |
| Soy sauce squeezing residue     | 11.1                         | 4.2                           | 15.3                        |
| Soy sauce squeezing residue +    | 12.3                         | 3.8                           | 16.1                        |
| Distilled water 20%             |                              |                               |                             |

* 24–64 h; ** 0–64 h.

3.4. Gas Chromatography

The release of gases is one of the most significant risks related to the storage of agricultural waste. There have been cases in which waste in a disposal facility has fermented, filling the environment with flammable gases and causing an explosion [10]. Additionally, there have been cases in which active fermentation has occurred in a storage location, releasing a large amount of CO₂, which resulted in the death of workers due to asphyxiation (Figure 18) [11].
The results of GC analysis are shown in Table 2 (untreated samples) and Table 3 (EOG-treated samples). As demonstrated in Table 2, an increase in the amount of CO$_2$ generated was observed in all samples when distilled water was added.

![Figure 18. Oxygen deficiency accident caused by soy sauce squeezing residue. Two workers died.](image)

**Table 2.** GC results for untreated samples (25 °C).

| Sample                              | GC Results % |
|-------------------------------------|--------------|
|                                     | O$_2$  | N$_2$ | H$_2$ | CO | CH$_4$ | CO$_2$ |
| Wood chip                           | 5.3    | 77.8  | -     | -  | -      | 12.1   |
| Wood chip + Distilled water 20%     | 1.5    | 78.5  | 0.1   | -  | -      | 14.4   |
| Wood pellet                         | 19.6   | 77.0  | <0.1  | -  | -      | 0.7    |
| Wood pellet + Distilled water 20%   | 1.4    | 68.9  | <0.1  | -  | -      | 21.7   |
| Sludge fuel                         | 19.5   | 77.3  | -     | -  | -      | 0.5    |
| Sludge fuel + Distilled water 20%   | 17.2   | 76.2  | -     | -  | -      | 2.3    |
| Chicken dung                        | 9.5    | 70.1  | -     | -  | -      | 14.2   |
| Chicken dung + Distilled water 20% | 1.5    | 73.8  | 0.3   | -  | -      | 22.0   |
| Soy sauce squeezing residue         | 1.7    | 79.4  | -     | -  | -      | 16.1   |
| Soy sauce squeezing residue + Distilled water 20% | 1.7    | 79.1  | -     | -  | -      | 18.2   |

**Table 3.** GC results for EOG-treated samples (25 °C).

| Sample                              | GC Results % |
|-------------------------------------|--------------|
|                                     | O$_2$  | N$_2$ | H$_2$ | CO | CH$_4$ | CO$_2$ |
| EOG Wood chip                       | 20.3   | 76.7  | -     | -  | -      | 0.1    |
| EOG Wood chip + Distilled water 20% | 20.3   | 77.9  | -     | -  | -      | 0.9    |
| EOG Wood pellet                     | 20.5   | 77.7  | -     | -  | -      | <0.1   |
| EOG Wood pellet + Distilled water 20% | 17.6   | 76.3  | -     | -  | -      | 2.5    |
| EOG Sludge fuel                     | 19.2   | 77.2  | -     | -  | -      | 0.1    |
| EOG Sludge fuel + Distilled water 20% | 19.8   | 77.2  | -     | -  | -      | 0.5    |
| EOG Chicken dung                    | 19.0   | 77.9  | -     | -  | -      | <0.1   |
| EOG Chicken dung + Distilled water 20% | 18.3   | 78.3  | -     | -  | -      | 0.3    |
| EOG Soy sauce squeezing residue     | 18.9   | 76.7  | -     | -  | -      | 0.1    |
| EOG Soy sauce squeezing residue + Distilled water 20% | 18.9   | 77.8  | -     | -  | -      | 0.1    |

4. Discussion

4.1. Thermogravimetric Differential Thermal Analysis

Based on the results of Figures 3–7, weight loss caused by the thermal decomposition of organic components was observed in all the samples at temperatures of 180–380 °C.
Heat generation and weight loss caused by char combustion were observed at temperatures exceeding 380 °C [18–21]. The decomposition temperature ranged from approximately 170–180 °C for all samples.

To reach ignition conditions, sufficient heat has to be generated to overcome losses and sustain a temperature increase. Fire may occur upon reaching the decomposition temperature, if all other necessary conditions (thermal insulation conditions, thermal capacity, volume of air inflow, heat exchange) are fulfilled [22].

4.2. Calorimetry

Based on the results of Figures 8–12, in all samples, heat generation produced by fatty acid esters was observed at temperatures ranging from 80 °C to 100 °C [4,21–23]; at temperatures higher than 100 °C, heat generation was caused by thermal decomposition [18,19].

In general, these results demonstrate that the process of heat generation that can lead to the spontaneous ignition of these materials begins with a small amount of heat generated from fermentation or oxidation. Next, the fatty acid esters contained within the materials begin to oxidise as the temperature gradually rises; even if the microorganisms causing fermentation die, the temperature of the material continues to rise and ultimately results in spontaneous ignition.

For example, one method of forecasting fires in such agricultural waste piles (Figure 19) is to monitor the internal temperature of the pile [24,25]. On the basis of these C80 results, it is recommended that the following safety measures be implemented: first, if the internal temperature of a pile is between 30 °C and 50 °C, heat dissipation measures should be taken.

At these temperatures fermentation begins, but only a small amount of heat is generated. The use of heat dissipation measures at this stage reduces the risk of a further increase in temperature that could lead to spontaneous ignition.

Second, if the internal temperature of a pile is between 50 °C and 80 °C, the surface of the agricultural waste pile should be covered entirely. At these temperatures, microbial fermentation occurs in combination with the oxidation of fatty acid esters [26,27]: self-heating of the pile is already in progress, and immediate treatment is required.

At this stage, breaking down the pile would increase the oxygen supply to the area where material is self-heating, rapidly increasing the temperature and the risk of combustion.

Therefore, the safest course of action is to prevent the inflow of oxygen by covering the surface of the agricultural waste pile entirely. These conditions should be maintained until the internal temperature of the pile decreases.

Figure 19. Agricultural waste piles (wood chips).
4.3. High-Sensitivity Isothermal Calorimeter

Based on the results of Figures 14–18, in wood chip and wood pellet samples, once distilled water was added, heat generation began immediately and increased again after 24 h. This is assumed to occur because microorganism activity increases over that period of time [28].

In all samples, once all the oxygen in the sealed sample containers was consumed by rapid fermentation, the reactions became inactive.

Sludge fuel and chicken dung are made from faecal matter and are more likely to contain a large number of microorganisms, which are assumed to become active with the addition of distilled water [29]. From this result, we conclude that waste managers must pay attention to moisture content in storage areas, especially for these materials.

Moreover, TAM added-moisture results indicate that care should be taken with regard to spontaneous ignition caused by the heat of fermentation during the rainy season in early summer (Figure 20). This is also true in early spring after the snow has melted. Therefore, it is necessary to continuously monitor the area [30].

![Figure 20. Large scale fire fighting of the wood chips.](image)

4.4. Gas Chromatography

Based on the results of Tables 2 and 3, in chicken dung and soy sauce squeezing residue, high generation of CO$_2$ was observed irrespective of the addition of distilled water. A small amount of H$_2$ generation was also observed in some samples, likely due to the high possibility of anaerobic fermentation [31–33], in addition to aerobic fermentation. CO and CH$_4$, however, were not detected.

In the EOG-treated samples, we observed a significant reduction in the amount of generated CO$_2$; no H$_2$, CO, or CH$_4$ were generated at all.

Based on thermal analysis results, it can be concluded that wood chips, chicken dung, and soy sauce squeezing residue all undergo significant fermentation, generating relatively high amounts of CO$_2$ even without the introduction of additional water. When the amount of contained moisture was increased, all materials except sludge fuel were observed to produce significant CO$_2$. In a real-world situation, this gas production could lead to oxygen deficiency accidents or spontaneous ignition.

When agricultural waste materials are stored outside, any flammable gas or CO$_2$ released diffuses into the air and their concentration decreases, so the risk from these gases is lower (Figure 21). However, when agricultural waste that is likely to ferment is transported in a tightly sealed container or stored for a long period in a warehouse, caution is required.

Measuring the generated gas using a gas detection tube is recommended, in addition to temperature monitoring and regular assessment of whether fermentation is occurring or gases are being generated [34].
5. Model Case Leading to Accidents Based on Thermal and Gas Analysis Results in this Study

Based on the above results, we propose a model case of the circumstances that can trigger spontaneous ignition or oxygen deficiency (Figure 22).

From the C80 and GC results, we can conclude that fermentation is likely to be responsible for the generation of heat and production of carbon dioxide at temperatures up to approximately 50 °C, where microbial activity diminishes.

The C80 and TAM results indicate that at temperatures beyond 50 °C, a transition into heat generation by the oxidation of fatty acid esters occurs. Additionally, TG-DTA results demonstrate that when the barrier of heat absorption is overcome at around 100 °C due to the evaporation of water, there is a transition to thermal cracking that could lead to fire.

Accidents due to oxygen deficiency may occur when a storage facility is well sealed and the amount of oxygen circulating is minimal.

This allows the oxygen to be consumed by fermentation, leading to an oxygen deficiency in the storage facility. However, when the amount of oxygen is sufficient; the substance is stored in large deposits; and the facility is well insulated, fermentation can cause the temperature to increase.

This leads to the oxidation of fatty acid esters, which may result in a fire following an increase in the temperature. This also applies to storage temperatures near room temperature. Therefore, it is desirable to periodically measure the temperature of stored materials and monitor the generated gases.

6. Conclusions

Based on the experimental results, the following conclusions have been reached:
(1) The results of the TG-DTA system measurements showed that the decomposition temperature ranged from approximately 170–180 °C for all samples. To reach ignition conditions, sufficient heat has to be generated to overcome losses and sustain a temperature increase. Fire may occur upon reaching the decomposition temperature, if all other necessary conditions are fulfilled.

(2) The results of the C80 measurements showed that the process of heat generation that can lead to the spontaneous ignition of agricultural waste begins with a small amount of heat generated from fermentation or oxidation. Next, the fatty acid esters contained within the materials begin to oxidise as the temperature gradually rises; even if the microorganisms causing fermentation die, the temperature of the material continues to rise which ultimately results in spontaneous ignition.

(3) The results of the TAM measurements showed that for all samples, an increase in heat generation was observed with the addition of distilled water. This micro heat generation is the trigger of fires. Further, it is believed that moderate moisture content of an item encourages fermentation and generates heat more easily. This suggests that caution is required during the warm rainy seasons during periods of repeated rainfall. Moreover, TAM added-moisture results indicate that care should be taken with regard to spontaneous ignition caused by the heat of fermentation during the rainy season in early summer. This is also true in early spring after the snow has melted. Therefore, it is necessary to continuously monitor the area.

(4) The results of the GC measurements showed that an increase in the amount of CO2 generated was observed in all samples when distilled water was added. Furthermore, the addition of distilled water was found to result in the generation of hydrogen. Therefore, anaerobic fermentation involving combustible gases may occur alongside significant heating via aerobic fermentation. Thus, when agricultural waste that is likely to ferment is transported in a tightly sealed container or stored for a long period in a warehouse, caution is required.

(5) Accidents due to oxygen deficiency may occur when a storage facility is well sealed and the amount of oxygen that is circulated is minimal, which allows the oxygen to be consumed by fermentation and would lead to an oxygen deficiency in the storage facility. However, when the amount of oxygen is sufficient, the substance is stored in large deposits, and the facility is well insulated, fermentation likely causes the temperature to increase and leads to the oxidation of fatty acid esters, which may lead to a fire following an increase in temperature. This also applies to storage temperatures near room temperature. Therefore, it is desirable to periodically measure the temperature of stored materials and monitor the generated gases.

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