

Article

Combustion Characteristics of Chlorine-Free Solid Fuel Produced from Municipal Solid Waste by Hydrothermal Processing

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Abstract: An experimental study on converting municipal solid waste (MSW) into chlorine-free solid fuel using a combination of hydrothermal processing and water-washing has been performed. After the product was extracted from the reactor, water-washing experiments were then conducted to obtain chlorine-free products with less than 3000 ppm total chlorine content. A series of combustion experiments were then performed for the products before and after the washing process to determine the chlorine content in the exhaust gas and those left in the ash after the combustion process at a certain temperature. A series of thermogravimetric analyses were also conducted to compare the combustion characteristics of the products before and after the washing process, there were increases in the fixed carbon content and the heating value of the product. Considering the possible chlorine emission, the washing process after the hydrothermal treatment should be necessary only if the furnace temperature is more than 800 °C.

Keywords: municipal solid waste; solid fuel production; hydrothermal treatment; combustion characteristics; chlorine removal; washing process

1. Introduction

Humans require a continual supply of energy, both for their present and future living needs. It should be noted that the current global energy consumption shows an increasing trend, driven strongly by the recent economic recovery. The consumption of coal as an energy supply grew by 7.6% in 2010, the fastest global growth since 2003. Coal now accounts for 29.6% of global energy consumption [1], but due to this fossil energy's non-renewable energy resource nature, it can be predicted with certainty that its supply will diminish. This particular case has stimulated the search for other alternative and at the same time an environmentally friendly energy resources.

Economic growth is creating another problem resulting from the growth effect of population and industries. Disposal of municipal solid waste (MSW) has become a big problem in many places. In developing countries, garbage is mostly disposed of in landfills by the open dumping method and this creates numerous environmental problems. The recent global trend is to prohibit or limit waste land-filling, but the citizenry does not want to increase the number of waste incineration plants in their respective countries.

Using technological improvements, waste could provide advantages and economical value due to its potential utilization as a fuel. Waste is now considered one solution to the limited energy supply problem. This social awareness provides a big opportunity for solid fuel production from MSW.

The Tokyo Institute of Technology is developing one method to produce solid fuel from MSW by employing a hydrothermal treatment system [2]. It was proven that MSW can be co-combusted with coal at certain blend ratios in previous experiments [3], and it was also shown that the devolatilization properties of coal were improved [4]. Considering its advantages, the hydrothermal treatment system can be considered as an alternative MSW treatment to produce coal-like solid fuel in order to reduce the usage of fossil fuel for combustion.

However, there are some limitations in the method, especially for MSW containing impregnated plastics which could contain chlorine originated from polyvinyl chloride (PVC) and inorganic chlorine in the form of salt. Chlorine is an element that may cause environmental and technical problems during combustion such as corrosion of the boiler tubes, the accumulation of deposits, and the reduced melting point of the ash, largely due to the low melting points of alkali chlorides and some metallic chlorides. Chlorine is released as hydrogen chloride during the heat treatment, both in an inert atmosphere and also under combustion conditions [5,6]. It is the most volatile element according to trace partition behavior during combustion [7] and it remains completely in the gas phase throughout the thermal power station and may be lost to the stack [8,9], a mean loss of 65%–75% of chlorine in thermal power stations [10]. 90%–92% of chlorine was detected to be lost, even during low-temperature (150–200 °C) ashing [11,12]. Thus, a chlorine removal process is necessary to avoid its detrimental effects in solid fuel furnaces.

Preliminary experimental results showed that the total chlorine content of raw MSW was about 1.63% or 16,300 ppm, with the majority (13,800 ppm) being organic or water-insoluble. After the hydrothermal treatment however, the organic chlorine in the sample was reduced and converted into inorganic (water soluble) chlorine [13] because of the combination of dehydrochlorination effect and the reaction with the alkaline content in MSW to produce salt [14]. The inorganic chlorine can then be easily removed by a water washing process to reduce the total chlorine content in the products [15].

To validate the necessity of the washing process after the hydrothermal treatment, an additional study is needed to determine the chlorine contents in the exhaust gas and in the ash after the combustion process in order to confirm that the process of washing the hydrothermal treatment product can reduce the exhaust gas pollution by chlorine. In this study, a comparison of chlorine behavior during combustion of hydrothermally treated MSW before and after the washing process is presented. The effectiveness of the washing process in order to suppress the chlorine emissions in the exhaust gas during combustion of the products in various combustion temperature conditions is shown.

2. Hydrothermal Treatment and Washing Process

Hydrothermal treatment is a technology to produce dried, uniform solid fuel or organic fertilizer by converting unutilized bio-resources such as food residues, sewage sludges, animal manures, agricultural residues, high moisture content solid wastes and MSW using low energy consumption [16]. The process began by loading the raw MSW and injecting saturated steam of about 200 °C and 2 MPa for a certain period into the stirred hydrothermal treatment reactor. After finishing the reaction and discharging the steam, a wet, uniformly-crushed material was extracted from the reactor.

An additional washing process was conducted to separate the inorganic chlorine from the product, so that a chlorine-free solid fuel product from can be obtained MSW. Figure 1 explains the hydrothermal process and the proposed additional water washing subsystem to decrease the chlorine content in the product. It can be seen from the figure that after the product is extracted from the reactor, it will be washed and filtered in the next processes step to separate the chlorine-free solid product and the liquid containing inorganic chlorine. The liquid can then be recycled for the next washing process or treated in the waste water treatment process.





Since it was necessary to identify the washing parameters which could influence the efficiency of the chlorine-free solid fuel production, a series of experiments were performed to find the optimum ratio of the water and product, the washing time, the washing speed and the washing temperature to remove the inorganic chlorine [15]. Up to 96% of inorganic chlorine could be removed only by one time washing with a water to product ratio of 32:1, a washing temperature of 40 °C, a mixing time of 5 min, and a washing speed of 400 rpm. It is obviously that inorganic chlorine removal is significantly enhanced by increasing the water to product ratio and the washing temperature, so these two parameters are the main determinants of the washing performance.

3. Experimental Method

3.1. Material Condition

Two series of experiments to obtain product samples from real MSW using the commercial-scale plant (3 m³ reactor) and alkaline-added PVCs using the laboratory-scale hydrothermal treatment were performed. The hydrothermal process in the commercial-scale plant processed 640 kg of MSW with the operating temperature of 225 °C, pressure of 2.4 MPa, and the holding period of 90 min [13].



Figure 2. Laboratory-scale hydrothermal treatment of KOH-added PVCs.

In order to confirm the behavior of chlorine during combustion, laboratory-scale hydrothermal experiments of polyvinyl chloride (PVC) and alkaline mixtures (KOH) were conducted using the autoclave shown in Figure 2.

The experiment conducted using PVC with KOH as an alkali additive was to observe the effect of presence of alkali in the plastic waste dechlorination process. From the previous research it found that the dechlorination process occurred readily and hydrochloric acid as a water-soluble chlorine species was produced during the hydrothermal treatment process. These results suggest that additional alkali is needed in the hydrothermal processing of plastic waste to reduce the corrosion due to HCl generation.

It is suggested that with an alkali additive, the hydrochloric acid produced due to dechlorination in the hydrothermal process reacted with the alkali to produce inorganic salts (in this case in the form of KCl), so in the end, the inorganic chlorine can be easily removed by the washing process.

The experiments were conducted in a 500 mL autoclave reactor, equipped with a rotor, a stirrer and a heater as shown in Figure 2. Pure water (100 g) was added to PVC (10 g, Kanto Chemical, Cat. No. 44038-02) and alkali (KOH, 10 g, Wako, purity min. = 85%) to simulate the steam input in a commercial-scale plant. Experiments with KOH-added PVCs were conducted at an operating temperature of 220 $^{\circ}$ C, a pressure of 2.2 MPa and a holding time of 30 min.

3.2. Chlorine Measurement and Material Analysis

The chlorine content analysis of product before and after the washing process for MSW and KOH-added PVC as raw material were differentiated by the chlorine type (organic or inorganic) and the form after combustion (ash or exhaust gas). The total material analysis workflow is presented in Figure 3.



Figure 3. Material analysis workflow.

In the washing process, the MSW hydrothermal treatment products were dried in a constant temperature oven at 105 °C for 24 h, and 25 g of the dried product was then mixed with 800 g of water. To accelerate the dissolution of inorganic chlorine, the dried product and water were mixed using a magnetic stirrer and a heater to maintain the washing temperature, and then filtered through a 5 μ m filter paper using a vacuum pump.

As for the hydrothermal treatment product of KOH-added PVC, 5 g of dried product were also washed under the same conditions. These washed products were used in order to compare the chlorine distribution after combustion of hydrothermally-treated products before and after the washing process.

Organic and inorganic chlorine contents were obtained by washing of the dried solid product with distilled water, followed by vacuum filtration. In order to ensure that all inorganic chlorine was already removed from the solid product, the filtered products were washed again and filtered twice. Washing was performed by adding forty volumes of distilled water into the product with a mixing time of 30 min, a washing temperature of 60 °C and a mixing speed of 400 rpm. The mixtures were then subjected to the vacuum-filtration process using a vacuum pump and a 5 μ m filter paper in order to separate the solid and liquid parts of the product. The liquid part obtained from the filtered water was analyzed using ion-selective electrodes (Orion 4 Star and Chloride Electrodes, Orion 9617BNWP). The organic chlorine in the residual solid was then measured by using a J-Science HSU-20 temperature combustion furnace set at 1000 °C.

For the ultimate analysis of these solid products before and after the washing process, a LECO CHNS932 organic elemental analyzer was used to measure the carbon, hydrogen and nitrogen contents in the product and the total chlorine content was measured using the J-Science HSU-20. The proximate analysis was done using a DTG analyzer (Shimadzu D-50) to obtain the weight loss profiles with nitrogen and air as the inert gas. The heating values were analyzed according to the Japanese standard JISM8814. The samples were dried at 105 °C, crushed, and each sample was analyzed three times using a Shimadzu CA-4PJ bomb calorimeter with less than 0.5% variance.

3.3. Combustion Simulation Experiments

A series of combustion simulation experiments were performed for products before and after the washing process to determine the chlorine contents in the exhaust gas and in the ash after the combustion process at a certain temperature. The combustion experiments were conducted using a quartz tube cylinder reactor with an inner diameter of 5 cm and a height of 40 cm equipped with a heater as shown in Figure 4 under certain combustion process conditions set by using 500 mL/min air flow as the oxidizing gas. Hydrothermal treatment product (2 g) was inserted into the reactor for 15 min at the constant temperatures of 900 °C, 700 °C and 500 °C. The chlorine content in the exhaust gas was determined by capturing the exhaust gas during combustion using three 125 mL impinger units containing distilled water. After finishing, the ash was collected for further chlorine content analysis. Three runs were carried out for each sample in order to confirm the reproducibility. A blank run was conducted and all the measurements were corrected for the buoyancy effect due to the air flow under the same temperature conditions.



Figure 4. Combustion simulation experiments.

3.4. Combustion Characterization by Thermogravimetric Analysis

The characteristics of the hydrothermal products before and after the washing process were studied using a DTG analyzer (Shimadzu D-50) to obtain the weight loss profiles as a function of time and temperature. A sample of Indonesian coal was also studied for comparison of the combustion characteristics with the hydrothermal treatment product before and after the washing process. Use of a DTG system has been mentioned in previous research to be an effective way to analyze pyrolysis and combustion behaviors because of its ability to quickly assess different kind of materials such as coal, biomass and their blends [4,17,18].

In order to check the effect of the heat and mass transfer, some preliminary tests were conducted by changing the heating rate and the sample mass. A non-isothermal DTG with the constant heating rate of 5 °C/min from the ambient temperature to 700 °C using 10 mg dried sample on an alumina pan was found to be suitable to eliminate the heat and mass transfer effects and side reactions. The maximum temperature of 700 °C was selected because no further weight loss was detected.

An oxidizing atmosphere was established during the temperature-programmed combustion by injecting a continuous air flow at 150 mL/min. In order to confirm the reproducibility, three runs were carried out for each sample. A blank run was conducted and all the measurements were calibrated for the buoyancy effect under the same heating conditions.

The TG thermogram results were then analyzed to find the characteristic parameters. After the initial moisture removal under a constant temperature, the temperature at which the weight loss started was denoted as the volatile release temperature, and the burnout temperature was decided based on the end of the weight loss. The DTG parameters showed a good reproducibility as determined by multiple tests with the standard error within ± 3 °C.

A non-isothermal oxidative TGA with the constant heating rate of 2 °C/min from the ambient temperature to 1000 °C using 10 mg of a dried sample on an alumina pan was used to measure the weight loss profiles for several alkali-chlorine salts (KCl, NaCl and CaCl₂) in order to analyze the effect of the combustion temperature on the shift of inorganic chlorine to the exhaust gas fraction.

4. Results and Discussion

4.1. Proximate and Ultimate Analysis

The proximate and ultimate analysis data of the hydrothermally treated MSW before and after the washing process is presented in Table 1. It can be seen that light volatile matter was removed after the washing process. The ash content in the product was reduced from 20.1% to 10.8% after the washing process due to dissolution of inorganic chlorine compounds. Due to the loss of ash and some volatile matter, there were increases of the fixed carbon content and the heating value of the product after the washing process.

Water to product ratio	Before Washing	After Washing
Proximate analysis		
Volatile matter (%)	64.2	59.5
Fixed carbon (%)	15.7	29.7
Ash (%)	20.1	10.8
Heating value (MJ/kg)	16.1	16.8
Ultimate analysis *		
Carbon (%)	55.6	48.3
Hydrogen (%)	6.0	5.5
Nitrogen (%)	0.9	1.1
Oxygen by difference (%)	37.5	45.1

Table 1. Proximate and Ultimate Analysis of Hydrothermally Treated MSW.

* dry ash-free basis.

From the ultimate analysis, it can be seen that the carbon content decreased from 55.6% to 48.3% and the hydrogen content also decreased from 6.0% to 5.5% after the washing process. This means there some hydrocarbons were released after the washing process. This result confirmed the proximate analysis result indicating a decrease of the volatile matter after the washing process.

4.2. Chlorine Analysis

The chlorine contents of the raw MSW (Raw MSW), the product after the hydrothermal treatment before the washing process (Unwashed Product) and the product after the hydrothermal treatment followed by the washing process (Washed Product) are presented in Figure 5, where the total chlorine content in the raw MSW is taken as 100%. The unwashed product exhibited low organic chlorine content because it was converted into inorganic chlorine during the hydrothermal process. After the washing process, the inorganic chlorine content was significantly reduced from 87% to 3%. This result confirmed that the washing process effectively removes inorganic chlorine in the product as explained in the previous work [15].

Figure 6 shows the comparison of chlorine content distribution between Unwashed Product after hydrothermal treatment (in the form of inorganic chlorine and organic chlorine) and Unwashed Product after combustion (in the ash and the exhaust gas) with three different combustion temperatures (500 °C, 700 °C, 900 °C), where the total chlorine content in Raw MSW is taken as 100%. In the combustion temperature range of 500 °C to 900 °C, it can be seen that the major part of the chlorine content in Unwashed Product was transferred to the ash portion.



Figure 5. Chlorine contents of raw MSW and the hydrothermal products before and after the washing process.

Figure 6. Chlorine contents after combustion of Unwashed Product with different combustion temperatures.



Under the combustion temperature of 500 °C, the inorganic chlorine portion in Unwashed Product (87%) mainly shifted to the ash part (78%) and a small portion was shifted to the exhaust gas part together with the organic chlorine portion. The shifted inorganic chlorine to the exhaust gas increased in line with the increase of the combustion temperature, so it means the increase of the combustion temperature promotes the evaporation of inorganic chlorine into the exhaust gas.

Figure 7 shows the chlorine contents in the ash and exhaust gas after combustion of Washed Product compared with the chlorine contents in Washed Product, where the total chlorine content in Washed Product is taken as 100%. It can be seen that like the Unwashed Product, the major part of the chlorine content in Washed Product shifted to the exhaust gas portion, because most of the inorganic chlorine were already removed by the washing process. It is also shown that the chlorine shift to the exhaust gas was increased with increasing combustion temperature.





In order to compare the experimental data of real MSW with simulated chlorinated plastic, similar experiments using PVC were also performed. Figure 8 shows the chlorine contents of raw material (Raw Material), unwashed product (Unwashed Product) and washed product (Washed Product) from the KOH-added PVC, where the total chlorine content in Raw Material is taken as 100%. Confirming the previous works of PVC dehydrochlorination phenomenon in the similar hydrothermal treatment [14], about 59% of the PVC was converted into inorganic chlorine after the hydrothermal treatment, and most of it was removed after the washing process.

Figure 8. Chlorine contents of the raw material and the hydrothermal products before and after the washing process (KOH-added PVC).



Figure 9. Chlorine contents after combustion of Unwashed Product with different combustion temperatures.



Figure 9 shows the chlorine contents in the ash and exhaust gas after combustion of Unwashed Product compared with those of Unwashed Product. Similar to the combustion results of Unwashed Product obtained from MSW shown in Figure 6, most of the inorganic chlorine in Unwashed Product shifted to the ash part and some small amounts of inorganic chlorine transferred to the exhaust gas part at the combustion temperature of 500 °C. When the combustion temperature was increased to 700 °C, very small differences in the inorganic chlorine shift to the exhaust gas part occurred compared to that

at the combustion temperature of 500 °C, but when the combustion temperature was increased to 900 °C, most of the inorganic chlorine (up to 96%) shifted to the exhaust gas part.

To analyze the effect of the combustion temperature on the shift of inorganic chlorine to the exhaust gas part, thermogravimetric analyses were then performed for several alkali-chlorine salts. Figure 10 shows the inorganic chlorine degradation graphs using KCl, NaCl and CaCl₂. This figure shows that inorganic chlorine will evaporate in various temperatures depending on the alkaline species, for example a small amount of KCl evaporated at the temperature of 380 °C, then started to evaporate significantly at the temperature of 760 °C, so the main parameters affecting the inorganic chlorine shift to the exhaust gas part were not only the combustion temperature, but also the type of the alkali bound in the inorganic chlorine content.



Figure 10. Mass loss profiles of alkali-chlorine salts.

In view of the reduction of air pollutants in the form of HCl, if the combustion temperature is less than 800 °C then most of the inorganic chlorine will go to the ash part, and then no washing process is required. For a combustion temperature of less than 800 °C, the hydrothermal treatment product can be used for co-firing with low-rank coal or the combustion of briquetting products itself is also possible to in order to avoid the washing process, but in the case of high temperature combustion applications, for example in cement kiln applications which require up to 1400 °C furnace temperature, the washing process is required in order to remove the inorganic chlorine which will transfer to the exhaust gas during the combustion process.

4.3. Combustion Characteristics by Thermogravimetric Analysis

The combustion characteristics of the hydrothermally treated products from MSW before and after the washing process were studied using TG profiles as shown in Figure 11. The profiles confirmed the proximate analysis results that part of the light volatile matter was removed by the washing process. This leads to a rise of the volatile release temperature from 190 °C to 210 °C after the washing process.

It is also shown that the ash content in the product was reduced from 20% to 11% after the washing process. Due to the decrease of the ash content and the light volatile matter in the hydrothermal product after the washing process, it shows a closer combustion profile to Indonesia coal compared to the hydrothermal product before the washing process.

Figure 11. TG profiles of the hydrothermally treated products before and after the washing process.



Figure 12 shows the DTA profiles of the hydrothermal products before and after the washing process. The heat content changes of the hydrothermal product are indicated by a negative deflection (an endothermic effect) and positive deflection (an exothermic effect) that is shown by the differential temperature, called peaks. It can be seen that the DTA peaks after the washing process became one significant peak, and also the peak increased from 150 μ V to 250 μ V after the washing process. Due to the loss of ash and some volatile matter, the fixed carbon percentage and the heating value of the product increased after the washing process as shown in Table 1.





5. Conclusions

The combustion characteristics of hydrothermal products from MSW have been investigated by combustion simulation experiments and thermogravimetric analysis. It was shown that most of the inorganic chorine in the product will go to the ash part after the combustion process. An increased part of the inorganic chlorine was shown to shift to the exhaust gas part during combustion by the increasing the combustion temperature, but it was confirmed from the oxidation of the alkali-chloride salts in the TGA that the main parameters of the shift was not only the combustion temperature but also the type of alkali in the inorganic chlorine compounds. It was found that for NaCl, KCl and CaCl₂, combustion temperatures below 800 °C would not shift the inorganic chlorine to the exhaust gas part.

In the washed hydrothermal products, there were increase of the fixed carbon content and decrease of carbon and hydrogen contents due to the loss of volatile matter and ash in the washing process. The resulting composition leads to a rise of the volatile release temperature from 190 °C to 210 °C for the washed product and it showed a combustion profile closer to that of Indonesian coal compared to the unwashed product, increasing the confidence of the washed product usage in coal co-firing applications.

It can be concluded that the process of washing hydrothermally-treated products will improve the combustion characteristic of the products for co-combustion with coal due to low chlorine content, low ash and better mass loss profile during combustion compared to the unwashed product. The washing process, however, requires large amounts of fresh water; therefore considering the possible chlorine emissions, the washing process after the hydrothermal treatment should be necessary only if the furnace temperature is more than 800 °C.

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