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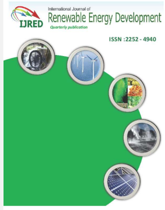

Effect of Hydrothermal Treatment Temperature on the Properties of Sewage Sludge Derived Solid Fuel

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ABSTRACT: High moisture content along with poor dewaterability are the main challenges for sewage sludge treatment and utilization. In this study, the effect of hydrothermal treatment at various temperature (120-200 °C) on the properties of sewage sludge derived solid fuel was investigated in the terms of mechanical dewatering character, drying character, calorific value and heavy metal distribution. Hydrothermal treatment (HT) followed by dewatering process significantly reduced moisture content and improved calorific value of sewage sludge with the optimum condition obtained at 140°C. No significant alteration of drying characteristic was produced by HT. Heavy metal enrichment in solid particle was found after HT that highlighted the importance of further study regarding heavy metal behavior during combustion. However, it also implied the potential application of HT on sewage sludge for heavy metal removal from wastewater.

Keywords: hydrothermal, sludge, moisture, heavy metal

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1. Introduction

In many countries, the disposal of sewage sludge -a wastewater treatment byproduct- might be technically difficult and cost intensive owing to its enormous volume and pollutant properties. For the case of China, according to the state annual statistics (Ma 2014), 69.5 billion ton wastewater as well as approx. 69.5 Mt sludge (85% moisture content) was generated in 2013. Multifarious pollutants are concentrated in sludge during the wastewater treatment including organic pollutants (PAHs, PCDD/Fs) (Stevens *et al.* 2002; Clarke *et al.* 2008), heavy metals (Babel & Mundo 2006; Pathak, Dastidar & Sreekrishnan 2009), and others (Tadeo *et al.* 2010; Lebrero *et al.* 2011; Marin *et al.* 2015). It is estimated that totally 1277.3 ton volatile phenol, 163.1 ton chromium (Cr), 112.2 ton arsenic (As), 18.4 ton cadmium (Cd) and 76.1 ton plumbum (Pb) were released into the municipal water in China in 2013 (Ma 2014) and most of them would be subsequently concentrated in sludge. Therefore, without a proper treatment and management, sludge disposal will lead to some environmental and health problems.

Sewage sludge can be utilized as derived fuel or bio-fuel (Fonts *et al.* 2012; Cano, Perez-Elvira & Fdz-Polanco 2015). However, the high contents of moisture and heavy metal are still present as the key challenges on sludge-to-energy conversion (Neyens & Baeyens 2003; Yin *et al.* 2004). For the purposes of energy recovery or low-cost disposal, dewatering and/or drying are commonly applied as preliminary processes. It is well researched that sludge is poorly dewaterable due to the high content of organic substances, which normally has less favorable dewatering properties (Colin & Gazbar 1995). Therefore, a pre-treatment process that involves the modification of sludge structure might be required for reaching low moisture content in sludge. Several methods were investigated to improve the dewaterability including thermal method (Neyens & Baeyens 2003), chemical treatment (Ruiz-

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Hernando *et al*. 2013), mechanical disintegration (Yin *et al*. 2004), freezing and others.

Recently, the application of hydrothermal or thermal hydrolysis for reducing the volume of sludge and improve its dewaterability has been attracting a great attention from researchers (Neyens & Baeyens 2003; Wang & Li 2014; Yu *et al*. 2014; Wang, Zhang & Li 2015). The possible mechanism is that hydrothermal treatment (HT) changed the floc structure by solubilizing the extracellular polymeric substances and enhanced the particle flocculation. Fisher and Swanwick (1971) showed that sludge dewaterability was greatly improved by hydrothermal treatment at the temperature above 150 °C. However, they also reported the formation of refractory COD compounds with increasing temperature. Haug, Lebrun & Tortorici (1983) showed the improvement in dewaterability of undigested and digested sludge by HT and that the temperature of 175 °C was about the limit for digestibility before digestion was inhibited. Yu *et al*. (2014) observed that the dewaterability of activated sludge was first deteriorated but then ameliorated when the temperature was raised from 100 °C to 200 °C with a threshold temperature of 130 °C under HT. For the low calorific value sludge, co-incineration of sludge with others fuels such as municipal solid waste or coal is commonly applied (Murakami *et al*. 2009; Wzorek, Koziol & Scierski 2010; Lin & Ma 2012) as the method for its disposal and energy recovery. Therefore, the reduction of pollutants emission after incineration, especially heavy metal and PCDD/Fs (Halonen *et al*. 1993; Han *et al*. 2008), is also an important issue that have to be paid attention to.

Despite the numerous researches regarding HT effect on the improvement of sludge dewaterability, a comprehensive study for the application of HT in the production of sewage sludge derived solid fuel is still hardly found. This study examined the effect of HT at various temperature, 120-200 °C, on the properties of sewage sludge derived solid product including its dewaterability, drying characteristic, calorific value and heavy metal distribution.

2. Materials and Methods

2.1 Sewage sludge

The sludge sample was taken from a water treatment plant in Hangzhou city, Zhejiang province, China. The capacity of this plant is 0.6 million ton per day, and the treatment system is A/A/O. The produced sludge in this plant sludge was mechanically dewatered without digestion and then directly transported to incinerator plant for burning process. The sample collection for this experiment was done for the sludge prior to the mechanical dewatering process. The collected sludge was thickened for two hours. The separated water at the top was removed and the bottom sludge, named as raw sludge, was used in our experiments.

2.2 Hydrothermal reactor and process

The hydrothermal reactor (HTR) is shown in Fig.1. It is a metal surfaced reactor with cavity volume of approx. 800 ml and complemented with a stirrer, a jacket heater connected with temperature controller, a sampling channel, a cooling channel, and the monitoring sub-systems for temperature and pressure. The cooling channel was used to cool the reaction system down to room temperature after each experiment so that the treated sludge could be maximally recovered. It was operated by flowing the cold water through the channel. The pressure monitor was used to judge the sealing condition since pressure is autogenously correlated with temperature setting under the employed saturated steam condition. The gas input and sampling parts could be used to change the atmosphere or collect the intermediate product during the process. The possible maximum operating temperature of HTR is 300°C.

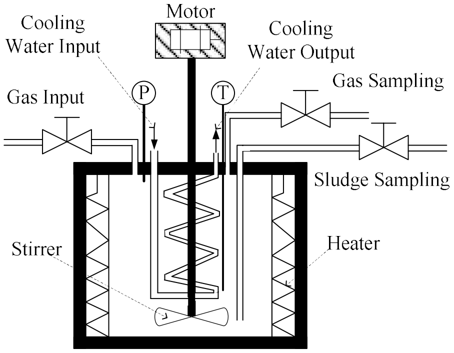

Fig. 1 Hydrothermal reactor

500 g of raw sludge was used in each experiment run. The temperature was set in the range of 120 °C – 200 °C by the increment of 20 °C. The holding time was set to 30 min. The holding time was counted after the desired temperatures setting was achieved and ended by rapidly cooling the reaction system down to room temperature. The HTR was subsequently opened to recover the treated sludge and the collected sample was stored in cool and dark container for further analysis.

2.2 Analysis

Fig.2 describes schematic diagram of the complete experiment steps and the naming system of each associated sample that will be used for following discussion (i.e. raw sludge, treated sludge, sludge cake and solid product). The treated sludge was mechanically dewatered for 10 min under various centrifugal force (0, 1.2, 4.7, 18.9, 42.5, and 57.9 kN) to separate the supernatant and sludge cake. The water content, drying character, and calorific value of sludge cake were measured. Water content was measured by the oven drying test at 105°C. Drying character was observed as a thermogravimetric profile and it was obtained using a real-time-recording thermo-balance system (Fig.3). Calorific value was measured with Calorimeter (MELL 9000). Heavy metal concentration in supernatant and cake were determined by ICP-MS (Elan DRC). The samples were digested according to HNO3-HF-HClO4 method in a PVC vessel. After digestion, the sample solution was diluted with deionized water to 50 ml.

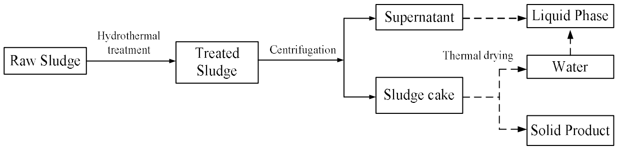

Fig.2 The schematic diagram of overall experiment steps

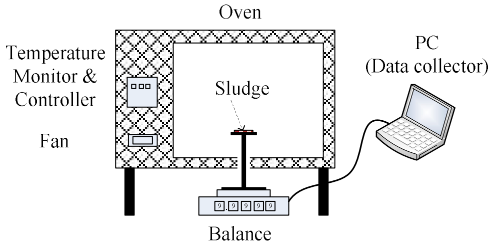

Fig.3 The scheme of thermogravimetric analysis system

3. Result and Discussion

3.1 Effect of hydrothermal treatment on the dewatering performance of treated sludge

Fig.4 shows the effect of HT temperature on the moisture content of sludge cake underwent various centrifugal forces. The samples hydrothermally treated at 120-180 °C consistently produced sludge cake with lower moisture content than that produced from the untreated sludge (HT temperature of 0) at the comparable dewatering force in the range of 18.9 – 57.9 kN. This implies that HT improved the dewaterability of sewage sludge. The dewaterability improvement might be caused by the solubilization of biopolymers (e.g. protein, polysaccharides, and humic substance) under HT condition. Solubilization of biopolymer resulted in the reductions of particle surface charge as well as the interparticle repulsive force (Wang & Li 2014; Wang, Zhang & Li 2015). Therefore, the particle flocculation was enhanced and the interstitial water was pushed out to be the free water (Wang & Li 2014; Yu *et al.* 2014; Wang, Zhang & Li 2015).

The biopolymer solubilization also can be signified from the high moisture content of HT sludge compared to raw sludge under the absence of the mechanical dewatering process (0 kN curve). The increase of sludge moisture content after HT was caused by the occurrence of solid-to-liquid transformation, e.g. polymer solubilization, in sludge particle since no additional water was put to the reactor. Temperature play significant role to the significance of this occurrence.

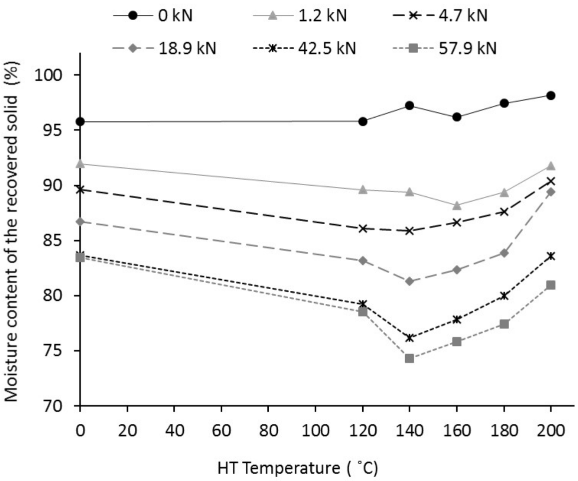

Fig.4 Effect of HT temperature and dewatering force on the moisture content of sludge cake

As also shown in Fig. 4, the increase of HT temperature up to 140 ˚C positively affected the moisture reduction of sludge cake, while the further temperature increases to 160-200 ˚C negatively affected it. These indicated that the major transformation in solid substance under HT at 120-140 ˚C is biopolymer solubilization followed by dehydration (Wang & Li 2014). Meanwhile at higher temperatures, the occurrence of biopolymer degradation that produced hydroxide (OH-) might become significant and resulted in the increase of particle surface charge. Therefore, the significant extent particle deflocculation might also took place and resulted in the reduction of dewaterability (Wang & Li 2014).

The release and breakdown of organic biopolymer also likely caused the continuous decrease of moisture content upon the application of dewatering force up to 57.9 kN. These are observable in the samples hydrothermally treated at 140-200 ˚C. In contrast, the untreated sample and the sample treated at 120 ˚C does not show any significant further decreases of moisture content upon the increase of dewatering force from 42.5 kN to 57.9 kN. These show that HT with the temperature of 140 ˚C and above enhanced the extent of dewaterability of sewage sludge.

From the point view of sludge handling process, the significant effect of HT application on solid fuel production from sewage sludge can be observed from the alteration of "liquid to solid ratio". Fig.5 shows the effect of HT condition on the Liquid to Solid ratio (L/S) of sludge cake with the treated sludge underwent 42.5 kN centrifugal force taken as the examples. Liquid to solid ratio (L/S) is an important factor for judging the viability of solid production from the watery source e.g. sewage sludge since it is determined the amount of liquid need to be handled to obtain a certain amount of dry solid. The application of HT at the temperature of 120-180 ˚C resulted in the dewatered sludge cake with lower L/S ratio than the cake produce from untreated sludge. The optimum condition for obtaining solid product with lowest L/S is HT at 140 ˚C. At this condition, the extent of biopolymer solubilization and breakdown might be optimized in sense of high enough to favor dewatering process and low enough to minimize the particle deflocculation phenomena.

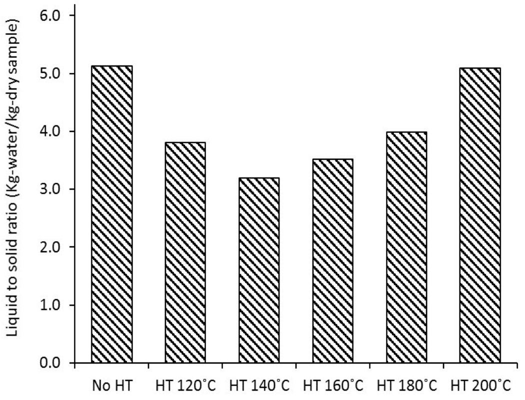

Fig.5 Effect of HT temperature on ratio of liquid to solid of sludge cake (dewatered at 42.5 kN)

The obtained L/S in this experiment is somewhere between the results of the previous researches. Long-Fei *et al.* (2014) obtained the cake with L/S as high as 4.9 - 9 after centrifugation. However, Raynaud *et al.* (2012) showed that mechanical dewatering technologies, such as centrifuges, belt pressure and filter presses generally attaining the lowest value of L/S at around 1.9 (corresponded to 65% moisture content). Wang, Zhang & Li (2015) showed that L/S value can be below 1 with the application of high temperature pressing subsequent to HT. This difference might be caused by the different feedstock properties as well as the applied process e.g. centrifugal force, dewatering temperature, additives, etc. Further process optimization from the current finding will be our future target.

3.2 Effect of hydrothermal treatment on the drying characteristic of sludge cake

Fig.6 shows the typical mass loss and mass loss rate profiles of sludge cake during drying process with the cakes dewatered at 42.5 kN following the HT conditions of: no HT and HT at 140 ˚C taken as the examples. Fig.7 shows the effect HT condition on the average and maximum mass loss rate of sludge cake underwent 42.5 kN centrifugal force. Mass loss rate is defined as the percent ratio of mass loss to the instantaneous mass of sample during some time interval. The average mass loss rate is counted during the elapsed time to reach a steady mass loss profile shown in Fig.6, corresponded to 70 min in no HT and 65 min in HT 140 ˚C. The maximum mass loss rate is defined as the highest peak occurred during the drying process, as shown in Fig.6.

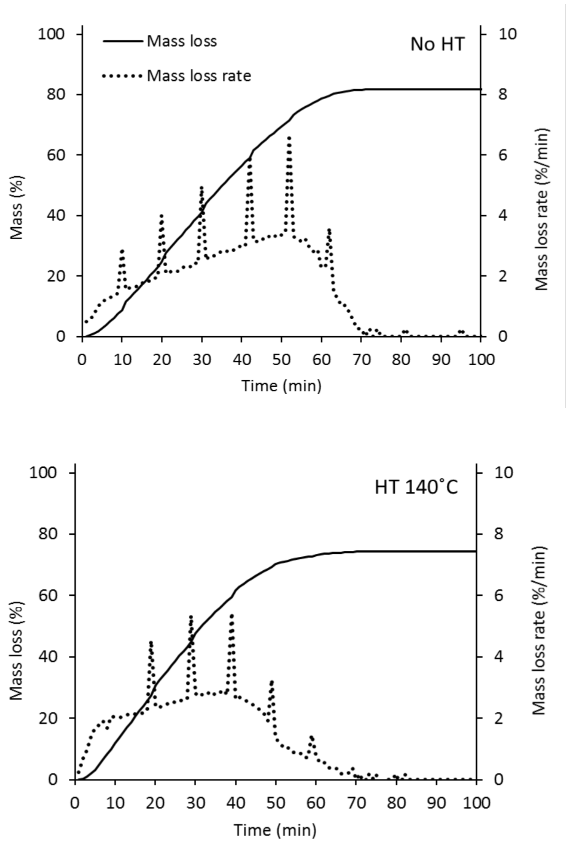

Fig.6 Drying characteristic of sludge cake (no HT and with HT at 140 ˚C, dewatered at 42.5 kN)

No clear trends are produced by the application of HT to the mass loss rate parameters. This implied that HT does not change the drying character of sewage sludge under the examined drying condition, which is likely surface diffusion controlled process. Our future works will be focused on the examination of the drying characteristic of hydrothermal treated product under the interparticle diffusion controlled process. HT is expected to breakdown the solid molecule and to enhance the interparticle water diffusion during drying process. The future examination will be performed under the high heating rate through the escalation of drying temperature to 150 ˚C.

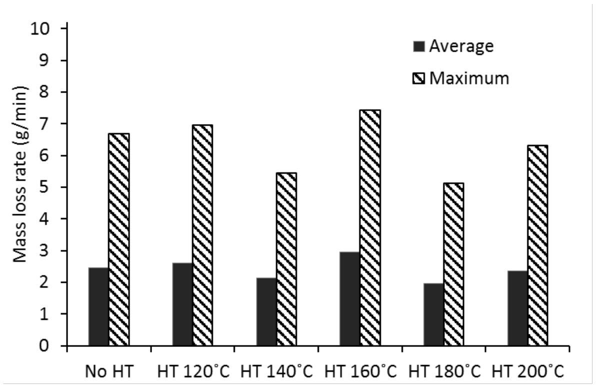

Fig.7 Effect of HT temperature on the mass loss rate of sludge cake during drying process (dewatered at 42.5 kN)

3.3 Effect of hydrothermal treatment on the calorific value of solid product and sludge cake

Evaluations of HT effect on the calorific value of the resulted products were performed in dry-base and wet-base. Dry-base HHV analysis on solid product was performed to obtain the information about some structural changes in sludge due to HT. Meanwhile, wet-base HHV of sludge cake might be the important judgment for the quality of sludge-derived solid fuel since in many cases sludge cake is managed as a final product and directly incinerated without any further drying process.

Fig.8 shows the effect of HT temperature on the dry-base higher heating value (HHV) of non-dewatered and dewatered solid product. HT on sewage sludge at the temperature of 140 °C and above produces non-dewatered solid with higher dry-base HHV than raw sludge (sludge without HT; dewatered and non-dewatered). This might be caused by the released of some volatile matter to the liquid part especially at higher treatment temperature (Wang & Li 2014; Wang, Zhang & Li 2015). The release of volatile matter is balanced by the increase fixed carbon content in the solid which subsequently increase its energy density.

On the other hand, the application of dewatering process negatively affects the dry-base HHV of the hydrothermally treated sludge. The dewatered solid products produced from the hydrothermally treated sludge are having lower dry-base HHV than the non-dewatered products. They also have lower dry-base HHV than the untreated (No HT) sludge with and without dewatering. This might be related to the release of soluble biopolymer during HT which then washed out during the dewatering process. The released substance might be relatively non-evaporable and cannot be removed by merely drying process. The biopolymer wash out is balanced by the increase of ash content of the solid product which subsequently decrease its energy density. The similar phenomenon is also observed in the previous research (Wang & Li 2014).

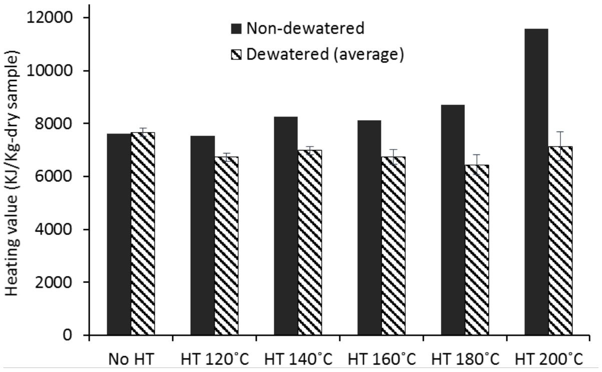

Fig.8 Effect of HT temperature on the dry-base HHV of solid product

Despite of the deterioration on the solid product dry-base HHV, the coupling process of dewatering with hydrothermal treatment might be beneficial for the overall solid fuel production from sewage sludge. This can be judged from the improvement of the wet-base HHV of sludge cake by the application of dewatering process subsequent to HT compared to the values of the raw sludge (no HT - dewatered and non-dewatered), shown in Fig. 9. The highest wet-base heating value, 1715 kJ/kg-sludge cake, is obtained under the applications of HT at 140 ˚C and dewatering process at 57.9 kN. The value is 5.3 times higher than the non-dewatered raw sludge and 1.4 times higher than the dewatered raw sludge. This is resulted from the optimization of the relatively insignificant decrease of dry-base HHV and the significant decrease of moisture content of sludge cake under that condition. As previously explained, HT application significantly improved dewaterability of treated sludge and lead to the production of low moisture content sludge cake, especially at 140 ˚C.

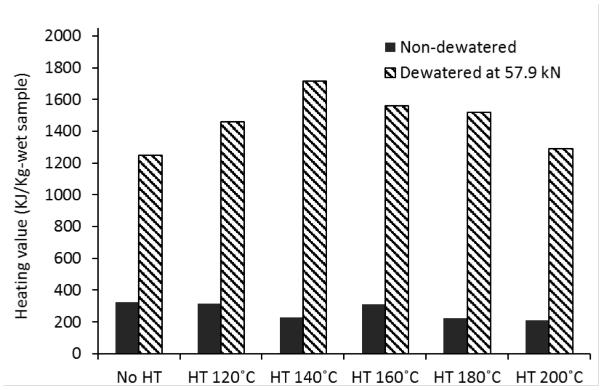

Fig.9 Effect of HT temperature on wet-base HHV of the dewatered and non-dewatered sludge

Tabel 1 Heavy metal distribution in sludge (500 g sample)

Sample	Condition	Comparison item	Pb	As	Zn	Cd	Hg
A1	Raw Sludge	Concentration(μg/g)	1.093	0.688	12.111	0.029	0.060
A2		Amount (μg)	546.41	343.98	6055.46	14.67	29.91
B1	No HT^{b}	Concentration in Supernatant (μg/g)	0.851	0.224	0.180	0.002	0.025
B2		Concentration in Sludge cake (μg/g)	2.28	2.96	70.55	0.17	0.23
B3		Amount in Liquid(μg)	407.67	107.08	86.21	0.72	11.83
B4		Amount in Solid^{a} (μg)	138.74	236.90	5969.26	13.95	18.08
B5		Solid/Liquid Amount ratio	0.34	2.21	69.24	19.39	1.53
C1	HT 160°C^{b}	Concentration in Supernatant (μg/g)	0.019	0.044	0.070	0.001	0.010
C2		Concentration in Sludge cake (μg/g)	11.58	29.39	69.19	0.30	0.54
C3		Amount in Liquid (μg)	9.18	21.08	34.06	0.61	4.97
C4		Amount in Solid^{a} (μg)	537.22	322.90	6021.41	14.06	24.94
C5		Solid/Liquid Amount ratio	58.50	15.32	176.81	23.14	5.02

^{a}: from the calculation of mass balance, ^{b}: dewatered at 57.9 kN for 30 min

3.4 Effect of hydrothermal treatment on heavy metal distribution in solid and liquid

Table 1 shows the heavy metal concentration in liquid phase, solid phase and their total value. The concentrations of Zn, 12.111 μg/g, and Pb, 1.093μg/g, in raw sludge are much higher than the value of other metals, which is consistent with the typical sewage sludge sample (Pathak, Dastidar & Sreekrishnan 2009). In addition, the dry-base concentrations of Zn, 286.99 μg/g, and Pb, 25.90 μg/g, are low compared to the concentrations of Zn and Pb reported in previous review by Pathak, Dastidar & Sreekrishnan (2009): 354-3096 μg/g and 26-465 μg/g, respectively.

The application of HT prior to mechanical dewatering resulted in the significant increase of heavy metal concentration in sludge cake (sample C2 in Table 1) compared to those in the sludge cake without HT (sample B2 in Table 1). The most significant changes occurred in the concentrations of Pb and As which reached the supernatant concentrations as low as 0.019 μg/g and 0.044 μg/g from previously 0.851 and 0.224, respectively. A clearer observation can be seen in Fig.10 which shows that almost all of heavy metal persisted in sludge cake after dewatering by the application of HT, indicated by the heavy metal fractions in sludge cake that are close to 1. Those are in contrast with the heavy metal fractions in sludge cake without HT which are relatively low for Pb, As, and Hg.

These results indicate that HT significantly reduced heavy metals leaching to liquid part as well as stabilized them in solid part. As previously reported (Weng *et al.* 2014), heavy metal stabilization in sludge during drying process might occur through the transformations of the acid-soluble, the reducible, and the oxidizable species into the structurally stable forms. In addition, the biosorbant properties of sludge (Smith *et al.* 2009; Sassi *et al.* 2010) was likely enhanced by the application of HT. The similar phenomena was reported by Jin *et al.* (2010) that implement the heavy metals removal from wastewater and heavy metal stabilization in fly ash by a single hydrothermal treatment process with the addition of sodium carbonate.

The heavy metal examination results suggest the potential application of the hydrothermally treated sewage sludge as an effective heavy metal remover from wastewater. However as a solid fuel, heavy metal enrichment in solid particle also highlights the importance of further detail study regarding heavy metal behavior during combustion. This continuation study might be essential to circumvent the harmful pollutant generation during the sludge-to-energy conversion process.

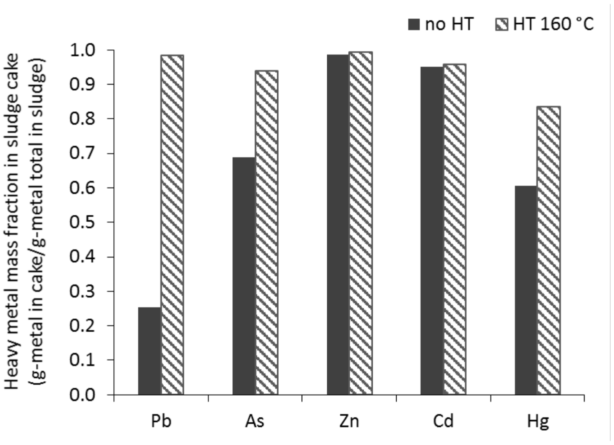

Fig.10 The mass fraction of heavy metal in sludge cake before and after hydrothermal treatment

4. Conclusion

The effect of hydrothermal treatment temperature (120-200 ˚C) on the properties of sewage sludge derived solid fuel was investigated. HT improved the dewaterability of sewage sludge with optimum temperature of 140 °C. The reduction of moisture content leads to the increase of wet-base HHV of the sludge cake up to 7.5 times higher than the non-dewatered raw sludge and 2.4 times higher than the dewatered raw sludge. No significant alteration of drying characteristic was produced by HT. Heavy metal was concentrated in solid particle after HT that highlights the importance of further study regarding heavy metal behavior during combustion. However, it also implies the potential application of HT on sewage sludge for heavy metal removal from wastewater which worth further trail and research.

5. Acknowledgments

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