Hydrochar production by hydrothermal carbonization of faecal sludge

K. Fakkaew, T. Koottatep, T. Pussayanavin and C. Polprasert

ABSTRACT

Faecal sludge (FS) management is a serious problem in developing countries which has caused environmental pollution and health risks. Hydrothermal carbonization (HTC) is an alternative technology that can be used to treat FS and convert it into a valuable solid product called hydrochar. This study evaluated the technical feasibility of hydrochar production from FS, determined the reaction kinetics of HTC of FS, and developed an empirical model which could estimate energy content of the produced hydrochar using the relevant parameters such as temperature (*T*), reaction time (*t*), moisture content (*M*), and volatile solid concentration (VS) in FS. The experiments were conducted with a 1-L high pressure reactor operated at the following conditions; *T* of 180–250 °C, *t* of 0.5–10.0 h, *M* of 70–95%wt, and VS of 40–340 g/L, which could produce at the energy content and hydrochar yield of 17–20 MJ/kg and 65%–80%, respectively. From these experimental data, an empirical model for determining energy content of the produced hydrochar was developed and validated satisfactorily with the literature data. Feasibility of applying the HTC process for FS treatment was discussed.

Key words | energy content, faecal sludge, hydrochar, hydrothermal carbonization, reaction kinetics, regression model

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INTRODUCTION

Most developing countries do not provide sewer systems with centralized treatment for the majority of urban residents. Human excreta containing faeces and urine are commonly disposed into septic tanks, cesspools or pit latrines and the accumulated sludge from these systems, so called faecal sludge (FS), is periodically removed in urban areas for further treatment and disposal. The United States Agency for International Development (USAID) (2010) reported that large amounts of FS in several developing countries such as Thailand, Vietnam and India are not properly treated and are being discharged into nearby canals, land and paddy fields. Because FS generally contains high concentrations of organic matter and enteric micro-organisms, this untreated FS could cause serious environmental and health risk problems.

Hydrothermal carbonization (HTC) is a thermochemical process which was found to be effective in converting high moisture biomass into carbonaceous solids, commonly called hydrochar, within a short period of time (1–12 h) at a relatively low temperature range (130–250 °C) and corresponding pressures (up to 20 bar) (Libra *et al.* 2011; Titirici 2013). The chemical structure and energy content of hydrochar are similar to natural coal, which make it suitable for use as a solid fuel in conventional combustion processes. The main advantages of HTC over other thermochemical conversion technologies (such as pyrolysis, gasification, and incineration) are its ability to convert wet feedstock to become carbonaceous solid products (hydrochar) at relatively high yields without preliminary dewatering and drying (Libra *et al.* 2011; Lu *et al.* 2012) and, consequently, requiring less energy. The energy required for the HTC process is expected to be substantially lower than that required for

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pyrolysis of such wet feedstock (moisture content of 75–90%) (Libra *et al.* 2011).

Some studies on HTC of human wastes (untreated faeces and FS) and sewage sludge have been previously reported. The experiments of Danso-Boateng *et al.* (2013) showed the decomposition kinetics of the HTC process to be described by the first-order reaction, and energy contents of the produced hydrochar ranged from 21.5 to 23.1 MJ/kg. The conversion of sewage sludge to an alternative solid fuel using the HTC process was studied by He *et al.* (2013) who found the fuel characteristics (e.g., energy content, H/C and O/C atomic ratio, and fuel ratio) of the produced hydrochar to be comparable with lignite. Probably due to the low volatile matter and energy content of raw FS (average 17.3 MJ/kg) (Muspratt *et al.* 2014) and its fluctuating characteristics as compared with other biomass materials, application of the HTC process for FS treatment has not been as extensively researched and reported.

The aims of this research were to evaluate the technical feasibility of applying the HTC process to produce hydrochar from raw FS which was the accumulated sludge emptied from septic tanks, cesspools or pit latrines, to determine the reaction kinetics of HTC of FS, and to develop an empirical model which could estimate the energy content of the produced hydrochar.

MATERIALS AND METHODS

Faecal sludge

FS samples were collected from a municipal emptying truck which serviced residential areas in a city located near Bangkok, Thailand. Moisture contents of the collected samples, which were originally about 95%wt with the volatile solid concentration of 40 g/L, were adjusted to be 90%wt, 80%wt, and 70%wt using a water bath before feeding to the HTC reactor, while volatile solid concentrations of these samples were about 70 g/L, 150 g/L, and 340 g/L, respectively.

HTC experiments

The experiments were conducted with a 1-L high pressure reactor made of stainless steel and equipped with pressure gauge, thermocouple and gas collecting ports, as illustrated in Figure 1. An electric heater equipped with a control panel (Figure 1(a)) was used to adjust temperature and reaction time of the reactor. Each HTC experiment was performed with 350 mL of FS sample and the operating conditions were controlled at temperatures of 180, 220, and 250 °C and reaction times of 0.5, 1.0, 5.0, and 10.0 h. The pressure was monitored and recorded during HTC operation. At the end of each experiment, the reactor was rapidly cooled to the ambient temperature with water in a cooling jacket (Figure 1(b)) at the cooling rate of about 45 °C/minutes to quench the reaction. After collection of the gas samples, the carbonized FS remaining in the reactor was separated for solid (hydrochar) and liquid products using vacuum filtration (Whatman filter paper, 1.2 μm). The produced hydrochar was subsequently dried in an oven at 105 °C for at least 12 h to remove the remaining moisture. The produced hydrochar, liquid, and gas samples were analysed for their physical and chemical characteristics as described in the following section.

Analytical methods

The volatile solid concentration of FS samples was analysed according to APHA/AWWA/WEF (2005). Energy content of the produced hydrochar was determined by a bomb calorimeter (AC500, Leco, USA). The proximate analysis (moisture, volatile matter, fixed carbon, and ash content) and ultimate analysis (carbon, hydrogen, nitrogen, and sulphur) of the produced hydrochar were measured using a thermogravimetric analyser (TGA701, Leco, USA) and CHNS analyser (Truspec, Leco, USA), respectively. The total organic carbon (TOC), chemical oxygen demand (COD) concentrations, total nitrogen (TN), and total phosphorus (TP) of the liquid samples were analysed using a high temperature combustion method (TOC-V CPH, Shimadzu, Japan), closed dichromate reflux method, persulfate method, and colorimetric method (APHA/AWWA/WEF 2005), respectively. The collected gas samples were analysed for; CO_2, CH_4, O_2, and N_2 using a gas chromatograph instrument (GC 7890A, Agilent, USA) equipped with flame ionization detector; and total volatile organic carbon (VOC) using a VOC analyser (MiniRAE 2000, RAE systems, USA).

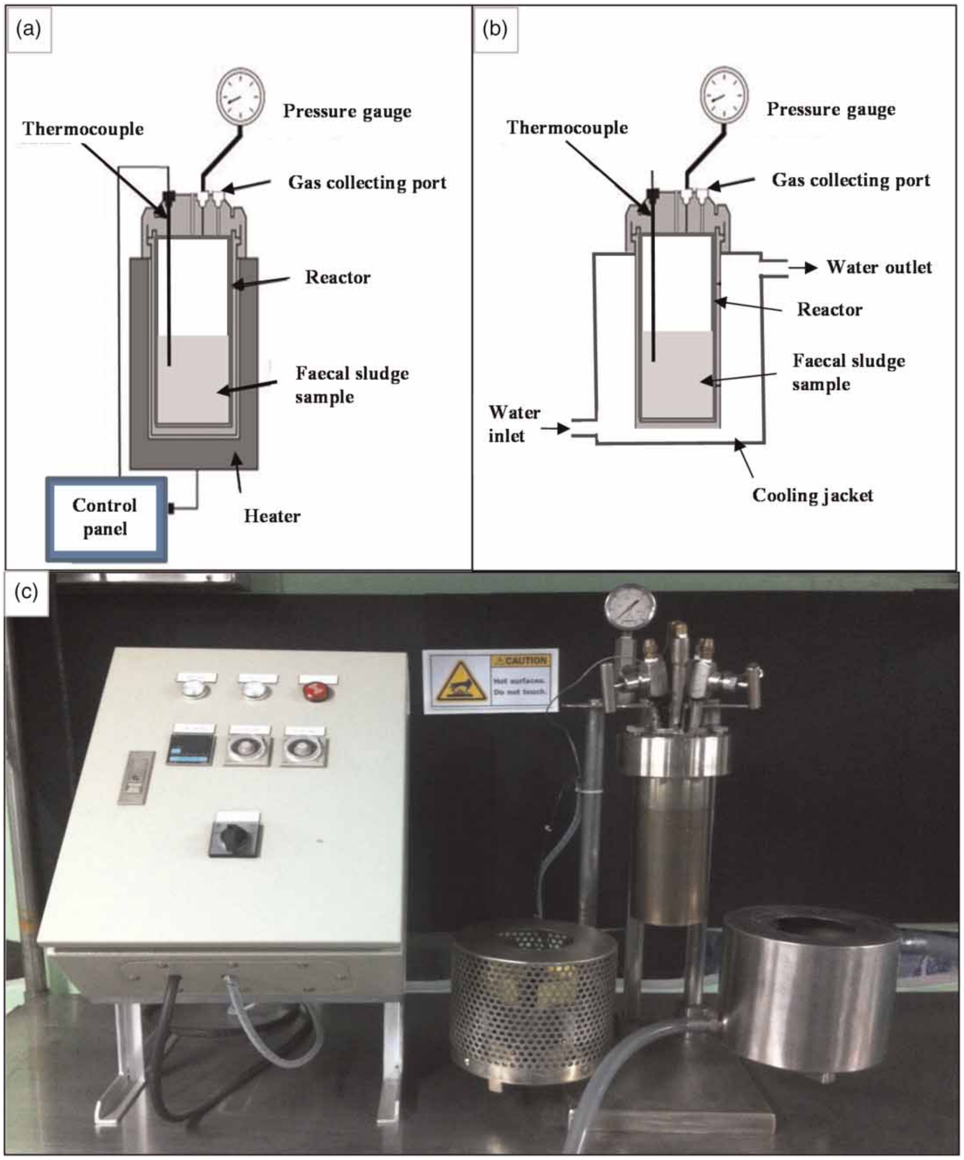


Figure 1 | Schematic of HTC reactor: (a) heating system; (b) cooling jacket; (c) photograph of HTC reactor.

RESULTS AND DISCUSSION

Effects of process parameters on energy content and hydrochar yield

Effects of moisture content on energy content of the produced hydrochar were first investigated by conducting HTC of the FS samples at the moisture contents of 70%, 80%, 90%, and 95% while temperature and reaction time were maintained at 220 °C and 5 h, respectively. Under these operating conditions, the pressure inside the HTC reactor was measured to be about 30 bar. The results from three replicates of batch experiments shown in Figure 2(a) indicated the significance of moisture content on energy content of the produced hydrochar ($p \leq 0.05$). The relatively high energy contents of 19.5 and 19.0 MJ/kg could be obtained, when the moisture contents were varied at 80% and 90%, respectively. According to Funke & Ziegler (2010), the moisture content in FS acts not only as a solvent, but also serves as a catalyst for carbonization, facilitating

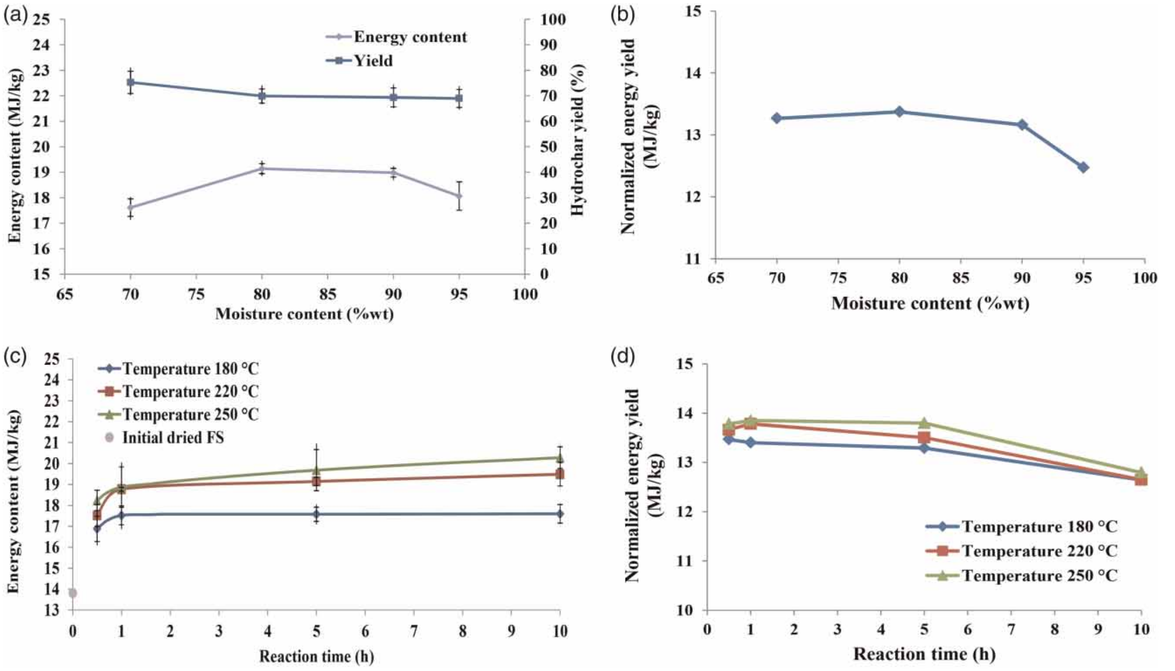


Figure 2 | Effects of process parameters: (a) energy contents and hydrochar yields at various moisture contents; (b) normalized energy yields at various moisture contents; (c) energy contents at various temperatures and reaction times operating at moisture content of 80%; (d) normalized energy yields at various temperatures and reaction times operating at moisture content of 80%.

hydrolysis, ionic condensation, and bond cleavage at elevated temperatures. However, the energy content of the produced hydrochar was decreased to 17.6 MJ/kg when the moisture content was reduced to 70%, probably because of insufficient water as a catalyst for various reactions in the HTC process. Because water acts predominantly as a solvent, giving low concentration of the hydrolyzed products which generally results in a decrease in the reaction rate, at the 95% moisture content, the energy content of the produced hydrochar was also decreased to 18.0 MJ/kg. The range of energy content of the produced hydrochar was comparable to the lignite and sub-bituminous (15.0 MJ/kg and 18.2 MJ/kg, respectively) (United States Environmental Protection Agency (U.S.EPA) 2008), which can be used as a solid fuel in conventional combustion.

According to He *et al.* (2013), hydrochar yield is the percentage of dry weight hydrochar per dried FS feedstock. Figure 2(a) shows that HTC fed with higher moisture contents of FS could generate lower hydrochar yields because the high amount of water (acting as a solvent) increased solubility of the FS and intermediate products, resulting in less hydrochar formation. However, the hydrochar yields of 65–80% were found comparable with those from the other studies (Mumme *et al.* 2011; Li *et al.* 2013), the remaining portions were in mostly liquid by-product and trace amount of gases.

To determine the optimum conditions, it is useful to compare energy content of the produced hydrochar and the normalized energy yield which is defined as the energy of the produced hydrochar per mass of dry initial feedstock (Li *et al.* 2013), as shown in Equation (1).

$$\textit{Normalized energy yield} = \frac{\textit{energy content of hydrochar} \times \textit{mass of hydrochar}}{\textit{mass of dry initial feedstock}} \tag{1}$$

Based on the data of Figure 2(b), the moisture content of 80% was found to be optimum in producing the highest normalized energy yield of 13.4 MJ/kg. The optimum temperature and reaction time of the HTC process are discussed in the following section.

The effects of temperature and reaction time on energy content of the produced hydrochar are shown in Figure 2(c). The highest energy content of 20.3 MJ/kg could be achieved at the temperature of 250 °C and reaction time of 10 h, while the lowest energy content of hydrochar was found at the temperature and reaction time of 180 °C and 0.5 h, respectively. These energy contents were comparable with the results of Escala *et al.* (2013) who found hydrochar produced from sewage sludge with energy contents of 18.2–19.1 MJ/kg.

In general, increasing the temperature in the HTC reactor would result in more dehydration and decarboxylation of the FS samples, resulting in increased energy content of the produced hydrochar. From these experimental results, increasing temperatures from 180 to 220 °C and from 220 to 250 °C resulted in about 10% and 4% increases in the energy contents of the produced hydrochar, respectively. The effects of temperature on the energy content of the produced hydrochar were previously reported in the literatures for the other substrates such as sewage sludge, coconut fibre, eucalyptus leaves, cellulose, and empty fruit bunches (Danso-Boateng *et al.* 2013; Liu *et al.* 2013; Lu *et al.* 2013; Parshetti *et al.* 2013). According to Titirici (2013), the typical operating temperatures of HTC are in the range 130–250 °C. At higher temperatures up to 350 °C, hydrothermal liquefaction could take place (Kruse *et al.* 2013) resulting in generation of more liquid and gas by-products and consequently, less hydrochar yield (<45%) (Liu *et al.* 2013). Therefore, the HTC process should not be operated at temperatures more than 250 °C because it would result in less hydrochar yields and high operation costs.

With respect to reaction time, it can be seen from Figure 2(c) that increasing reaction times led to increased energy contents, especially when operating the HTC at 250 °C, which had the statistical significant p value ≤ 0.05. Energy contents of 13.8 MJ/kg in the initial dried FS was increased to 18.2 MJ/kg, 18.8 MJ/kg, 19.7 MJ/kg, and 20.3 MJ/kg in the hydrochar at HTC reaction times of 0.5 h, 1.0 h, 5.0 h, and 10.0 h, respectively.

The normalized energy yields of the produced hydrochar, shown in Figure 2(d), were found to decrease with increasing reaction time, but the temperature of 250 °C still produced higher normalized energy yield than those at 220 and 180 °C. It can be deduced from the results of Figure 2(d) that the reaction time of 5 h was optimum in producing the highest normalized energy yield of 13.8 MJ/kg. As reported earlier that operating the HTC temperatures more than 250 °C could result in less hydrochar yields and higher energy consumption, it can be assumed that the temperature of 250 °C was optimum for the HTC in producing the highest normalized energy yield.

Mass balance and carbon distribution

Mass balance and carbon distribution of the FS were carried out at the optimum HTC condition, as illustrated in Figure 3. The initial dried FS consisting of carbon of 38.45%wt and the other elements (e.g. hydrogen, nitrogen, oxygen, and sulphur) of 61.55%wt was hydrothermally carbonized into the hydrochar, liquid and gas by-products of 69.86%wt, 20.72% wt and 9.42%wt, respectively. The carbon distribution indicated a significant proportion of carbon in the initial dried FS retained within the hydrochar of 28.67%wt. The rest of the carbon was shifted into either the liquid or gas by-products. TOC concentration of the liquid samples was used to calculate the carbon content in the liquid by-product which was 6.94%wt. From the mass balance analysis, a small fraction of carbon in the initial dried FS was transferred into gas by-products of 2.84%wt, which was predominantly CO_2.

Characteristics of HTC by-products

The liquid samples obtained from the optimum HTC condition were collected and analysed for their characteristics. Liquid by-products still contained high concentrations of organic matter as indicated by COD of 33–36 g/L and

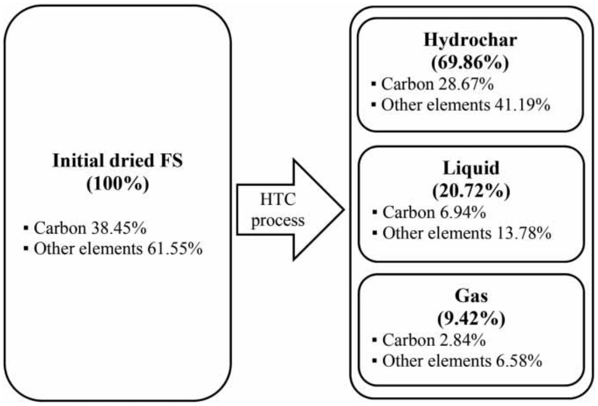


Figure 3 | Mass balance and carbon distribution for HTC of FS.

TOC of 12–15 g/L, and relatively high nutrients of TN of 3.6–3.7 g/L and TP of 0.005 g/L. These values were comparable to those reported in the literature (Danso-Boateng *et al.* 2013; Escala *et al.* 2013; Funke *et al.* 2013; Oliveira *et al.* 2013; Stemann *et al.* 2013) which showed COD, TOC, TN, and TP concentrations to be 31–53 g/L, 5–26 g/L, 2.3–4.7 g/L, and 0.01–0.16 g/L, respectively. It is apparent that these liquid by-products need to be further treated, such as by anaerobic digestion (Oliveira *et al.* 2013; Poerschmann *et al.* 2014), to minimize environmental pollution and produce useful biogas. Relatively high TN and TP contents in the liquid by-products could be safely applied on farmlands as liquid fertilizer.

Analysis of HTC gas samples showed CO_2 to be the main component (61.92%), similar to the result of Berge *et al.* (2011) and Funke *et al.* (2013), while there were trace amounts of CH_4, O_2, N_2, and total VOC at 0.70%, 1.71%, 21.50%, and 3.15%, respectively. To minimize the odour and greenhouse gas emissions, the produced gases can be further removed possibly by a wet scrubber or condenser system (Polprasert 2007).

Reaction kinetics

To understand the reaction kinetics of HTC of FS, the first-order reaction and Arrhenius equation were proposed. A typical HTC reaction is as follows:

$$\text{FS} \quad \text{Hydrochar} + \text{By-product (liquid and gas)} \tag{2}$$

The reaction rate depends on the conversion of substrate and reaction time which can be expressed as the first-order differential rate equations:

$$r = -\frac{dC_t}{dt} = kC_t \tag{3}$$

$$\ln\frac{C_i}{C_t} = kt \tag{4}$$

where r is the reaction rate; C_t is the mass of volatile matters in the produced hydrochar at reaction time (t); C_i is the mass of volatile matters in the initial FS (dry weight); k is the reaction rate constant of HTC; and t is the reaction time. The reaction rate constants (k) of various temperatures obtained from these experiments are shown in Figure 4(a). The high correlation coefficient (R^2) values indicated that the HTC of FS were a good fit to the first-order reaction.

The reaction rate constants, depending on the temperatures, can be expressed as the Arrhenius equation:

$$k = Ae^{-\frac{E_a}{RT}} \tag{5}$$

where, A is the pre-exponential factor; E_a is the activation energy; R is the gas constant; and T is the temperature (K). From the intercept and slope of the linear equation in Figure 4(b), A and E_a values were estimated to be $1005\ \text{h}^{-1}$ and 39 kJ/mol, respectively, within the temperature range of 180–250 °C. A previous study by Reza *et al.* (2013) reported the E_a values of HTC for hemicelluloses and cellulose degradations to be 30 kJ/mol and 73 kJ/mol, respectively. The E_a of HTC of FS was comparable to hemicelluloses which could be slow biodegradable carbohydrates contained in the faeces (Lentner *et al.* 1981) and decomposed under hydrothermal condition at temperature of 150–250 °C (Liu & Balasubramanian 2012).

Modelling of HTC of FS

To develop an empirical model which could estimate the energy content of the produced hydrochar from the HTC of FS, a multiple linear regression analysis was conducted using the experimental data obtained from this study. The statistical analysis using the Minitab-17 software showing a linear regression equation was

$$E_{\text{hydrochar}} = f(26.8 + 0.020T + 0.106t - 0.131M - 0.014VS) \tag{6}$$

where $E_{\text{hydrochar}}$ is the energy content of the produced hydrochar; T is the temperature; t is the reaction time; M is the moisture content of feedstock; VS is the volatile solid concentration of feedstock; and f is the correction factor for energy content of the dry initial feedstock ($E_{\text{feedstock}}$), which was found to be $0.07E_{\text{feedstock}}$. Analysis of variance (ANOVA) showed Equation (6) to be significant at the 95% confidence level with the R^2 of 0.910.

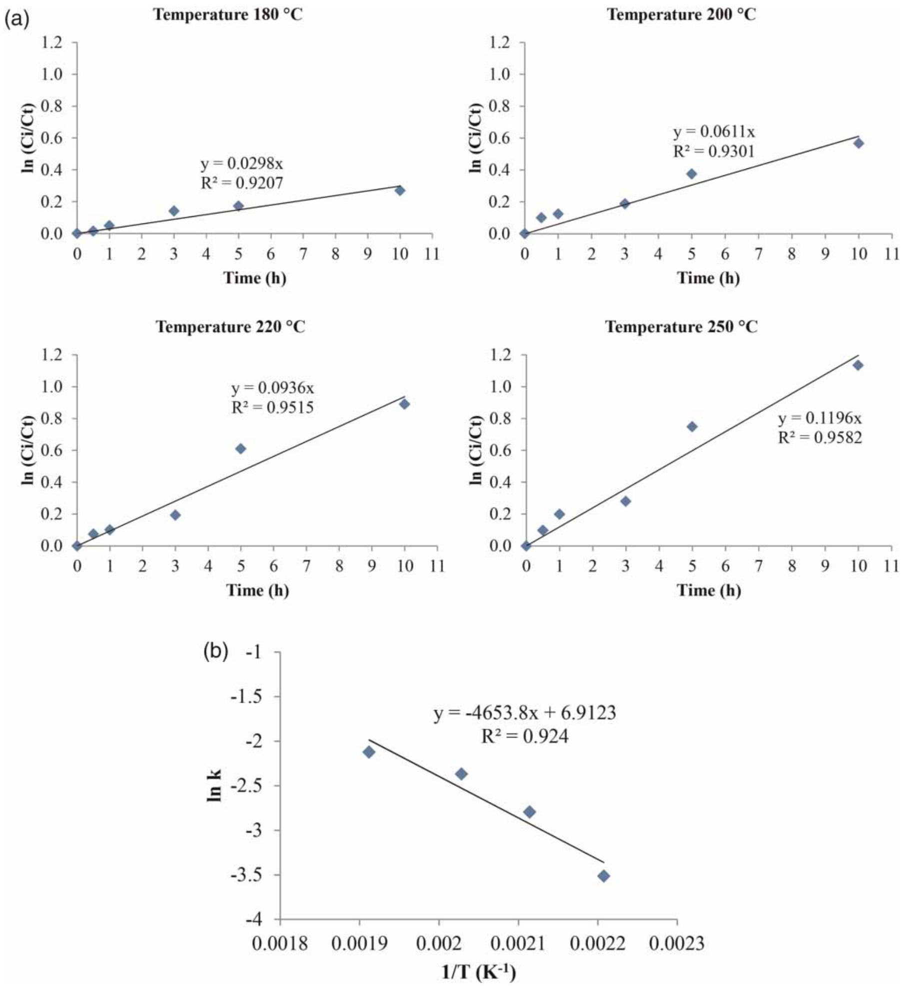
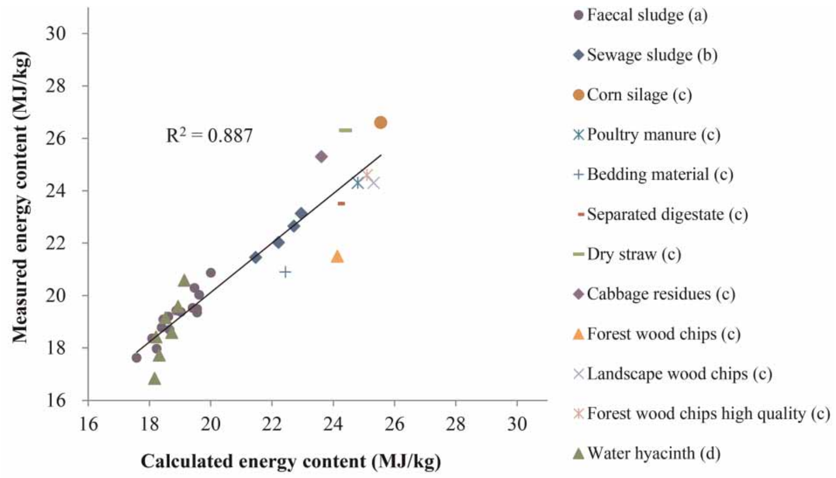


Figure 4 | Reaction kinetics of HTC of FS: (a) conversion of volatile matters with reaction times at temperature of 180, 200, 220, and 250 °C; (b) Arrhenius plot of reaction rate constants with temperatures.

Equation (6) was validated with 15 additional HTC experiments using FS with different HTC process conditions, and with literature data of HTC using other biomass as feedstock (Danso-Boateng *et al.* 2013; Gao *et al.* 2013; Oliveira *et al.* 2013). Figure 5 indicates that Equation (6) fits well with these data, having an R^2 value of 0.887. It should be noted that Equation (6) is applicable for predicting energy content of hydrochar produced by the HTC process operated within the conditions employed in this study. In addition, it could be useful for the HTC operators to adjust some operating parameters to achieve the desired energy content for the produced hydrochar. Further validations of Equation (6) with pilot- or full-scale HTC reactors treating FS or other biomass materials are recommended.

Application

The experimental results obtained from this study demonstrated the technical feasibility of applying the HTC process to treat and convert FS to a valuable product such as hydrochar. At present, there are no full-scale HTC reactors treating FS in Thailand or other developing countries. It is strongly recommended that some pilot-scale or demonstration experiments on HTC be conducted to test the scale-up effects and cost-benefit analysis of the systems. The application of the produced hydrochar as solid fuel, energy storage and other value-added products (Titirici & Antonietti 2010), including its marketability and social acceptance should also be studied prior to implementation of a full-scale HTC reactor treating FS.



(a) Results of additional experiments, (b) Danso-Boateng et al. (2013), (c) Oliveira et al. (2013), (d) Gao et al. (2013)

Figure 5 | Comparison of calculated and measured energy contents of produced hydrochar from FS, sewage sludge, corn silage, poultry manure, bedding material, separated digestate, dry straw, cabbage residues, forest wood chips, landscape wood chips, forest wood chips high quality, and water hyacinth.

CONCLUSIONS

Based on the results obtained from this study, the following conclusions are made:

1. The optimum conditions of the HTC of FS to produce the highest normalized energy yield were found to be: moisture content 80%, reaction time 5 h, and temperature 250 °C.
2. The energy content and hydrochar yield were 20.3 MJ/kg and 70%, respectively, comparable to lignite, sub-bituminous, and literature data.
3. The liquid by-products were found to contain high organic matter which should be further treated to avoid environmental pollution. To minimize the odour and greenhouse gas emissions, the produced gases could be removed by wet scrubber or condenser systems.
4. The HTC of FS followed the first-order reaction with the E_a value of 39 kJ/mol. A linear regression model was developed which could estimate energy content of the produced hydrochar at various operating conditions. This model was validated with some literature data satisfactorily.

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